ADHESION OF ASPHALT MIXTURES

By

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DEDICATION

This thesis is dedicated specially to:

My lovely wife:

Nur Hanani Mansor

My precious little princesses:

Nur Hani Fatihah Fauzan & Nur Hasya Farihah Fauzan

My parents:

Mohd. Jakarni Mohd. Said & Bedah Ishak

My parents-in-law:

Mansor Mohd. Lazim & Zaniah Hashim

To my brothers and sisters

&

All family members and friends

To my beloved grandmother Manis Sharif who passed away in 2008 – Al-Fatihah

ABSTRACT

ADHESION OF ASPHALT MIXTURES

Adhesion is defined as the molecular force of attraction in the area of contact between unlike bodies of adhesive materials and substrates that acts to hold the bodies together. In the context of asphalt mixtures, adhesion is used to refer to the amount of energy required to break the adhesive bond between bitumen (bitumen-filler mastic) and aggregates. Thus, adhesive failure can be considered as displacement of bitumen (bitumen-filler) mastic from aggregates surface, which might indicates low magnitude of adhesive bond strength. Adhesion is considered as one of the main fundamental properties of asphalt mixtures, which can be correlated with quality, performance and serviceability. However, despite its significance, research on adhesion of asphalt mixtures is limited and yet there is no established testing technique and procedure that can be used to quantify the adhesive bond strength between bitumen (bitumen-filler mastic) and aggregates. Only in the past few years, some efforts have been conducted in developing testing techniques and procedures for measuring the adhesive bond strength of bitumen and aggregates. However, the developed testing techniques and procedures have not enjoyed universal success and acceptance, and not yet established. Hence, emphasis of this study is focused on the development of laboratory adhesion test method that can be used to directly measure the adhesive bond strength between bitumen (bitumen-filler mastic) and aggregates. Also, adhesive bond strength and failure characteristics of various combinations of asphalt mixture materials over wide ranges of testing conditions were evaluated in order to validate the reliability and efficiency of the developed laboratory adhesion test method.

This study was divided into three parts. In Part 1, a detailed review of literature on various testing techniques and procedures used to measure the adhesive bond strength in numerous areas of scientific literature and international standards was performed, in order to assess and thus to propose the most suitable and realistic approach for development of laboratory adhesion test method for asphalt mixtures. In Part 2, the proposed adhesion test method was subjected to evaluation, mainly based on trial and error experimental approach, in order to adapt and thus to develop the criteria and procedures for test setup and apparatus, specimen preparation, testing and data analysis. The established criteria and procedures were then used for detailed evaluation in Part 3, in order to quantify the test results of various combinations of asphalt mixture materials (i.e. bitumen (bitumen-filler mastic) and aggregates) over wide ranges of thicknesses of adhesive layer of bitumen, aspect ratio of specimens, testing conditions (i.e. deformation rates and test temperatures) and conditioning procedures (dry and wet conditionings). Results of the study were subjected to comparative analysis in order to determine the effect of various variables and parameters on the test results, to propose suitable testing conditions and to validate the reliability and efficiency of the laboratory adhesion test method. Upon completion of the study, a draft protocol was developed as guiding principles in conducting the laboratory adhesion test method.

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DECLARATION

I hereby declare that the thesis is based on my original work except for quotations and citations, which have been duly acknowledged. Research reported in the thesis was conducted at the University of Nottingham, Department of Civil Engineering, Nottingham Transportation Engineering Centre (NTEC), between September 2007 and January 2012. I also declare that the research has not been previously or concurrently submitted for any other degree at University of Nottingham or other institutions.

FAUZAN MOHD. JAKARNI
July 2012

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LIST OF ABBREVIATIONS/GLOSSARY OF TERMS/NOTATIONS

AASHTO	American Association of State Highway and Transportation Officials
ANOVA	Analysis of Variance
ASTM	American Society for Testing and Materials
CBT	Corrected Beam Theory
CI	Confidence Interval
COV	Coefficient of Variation
C_T	Stickiness or Tack Factor
DCB	Double Cantilever Beam
DSR	Dynamic Shear Rheometer
ECM	Experimental Compliance Method
EPFM	Elastic-Plastic-Fracture-Mechanics
G_C	Adhesive Fracture Energy
GVOC	Good-van Oss-Chaudhury
H_0	Null Hypothesis
H_1	Alternative Hypothesis
HMA	Hot Mix Asphalt
HMB	High Modulus Base
HWTD	Hamburg Wheel Tracking Device
ITS	Indirect Tensile Strength
IWP	Impact Wedge Peel
LEFM	Linear-Elastic-Fracture-Mechanics
LVDT	Linear Variable Differential Transducer
LW	Lifshitz-van der Waals
MR	Resilient Modulus
NCHRP	National Cooperative Highway Research Program
n.d.	No Publication Date
NIST	National Institute of Standards and Technology
PATTI	Pneumatic Adhesion Tensile Testing Instrument
PCC	Portland Cement Concrete
R^2	Coefficient of Determination
R^{Total}	Energy Ratio
R_a	Arithmetic Average of Roughness
SATS	Saturation Ageing Tensile Stiffness
SBT	Simple Beam Theory
SHRP	Strategic Highway Research Program
SMA	Stone Matrix Asphalt
SPS	Surface Protection System
SSD	Saturated Surface Dry
T	t-statistic
TBT	Tensile Bond Testing
TDCB	Tapered Double Cantilever Beam
TSR	Tensile Strength Ratio
USD	Universal Sorption Device
UTEP	University of Texas at El Paso
$\overline{X}$	Average
α	Level of Significance
ΔG_{SA}	Adhesive Bond Energy of Aggregates and Bitumen
ΔG_{SAW}	Reduction of Surface Free Energy
θ	Contact Angle
π_e	Equilibrium Spreading Pressure

CHAPTER 1

INTRODUCTION

1.1 General Background

Asphalt mixtures are a combination of aggregates, bituminous binder (or simply known as bitumen) and filler, mixed in a predetermined ratio in order to result in flexible pavements. Sometimes additives such as rubbers and fibres are used to improve the performance of the asphalt mixtures. Majority of roads in the Great Britain and also throughout the world are comprised of flexible pavements. Different ratios of aggregates, bitumen and filler (as well as the small proportion of air) give rise to different types of flexible pavements. Although different types of flexible pavements have different properties and serve different purposes based on the traffic level, climate, soil characteristics and other factors, all are designed and constructed to meet the demands for the following qualities; able to resist deformation, cracking and water or moisture damage, and be durable over time. However, despite the efficiency in designing and constructing to meet the demands for the aforementioned qualities, the combined effects of massive traffic growth and higher axle loads, together with environmental and ageing effects tend to lead to the rapid deterioration of the flexible pavements.

Environmental factors related to water or moisture are seen to be one of the significant factors that adversely affect the quality, performance and serviceability of the asphalt mixtures. The presence of water or moisture in the pavement structure and the detrimental effects that water or moisture has on the properties of the asphalt mixtures, commonly known as moisture damage, can contribute to variety of pavement distresses including stripping, ravelling, fatigue cracking and rutting. Moisture damage can be defined as the loss of strength and durability of the asphalt mixtures due to the presence of water or moisture in the pavement structure. Moisture damage is an extremely complicated mode of distress and can shows itself in various forms such as adhesive failure between bitumen (bitumen-filler mastic) and aggregates, cohesive failure within bitumen (bitumen-filler mastic), cohesive failure within aggregates and/or freezing of entrapped water or moisture in the pavement structure (Asphalt Institute 2007; Kim & Coree 2005). However, the most common forms of moisture damage are due to the adhesive failure (loss of adhesion) between bitumen (bitumen-filler mastic) and aggregates, which is the most prevalent form and the cohesive failure (loss of cohesion) within bitumen (bitumen-filler mastic) (Kanitpong & Bahia 2003; Kim & Coree 2005; Solaimanian et al. 2007).

Adhesion can be defined as the molecular force of attraction in the area of contact between unlike bodies (i.e. adhesive and substrates) that acts to hold the bodies together (Copeland 2007). By contrast, cohesion is the intermolecular force developed within the same body (i.e. adhesive or substrates) that forms the body. Figure 1.1 illustrates the adhesion and cohesion present between adhesive and substrates, and within adhesive respectively.

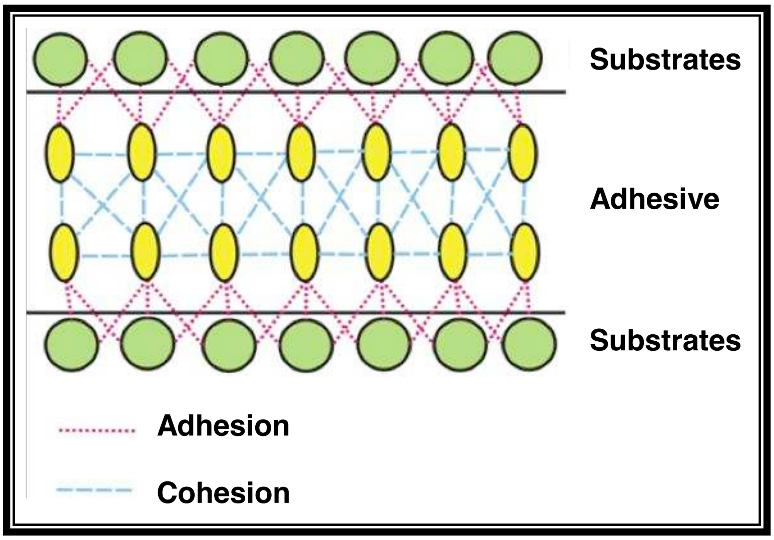


Figure 1.1 Adhesion and cohesion present between adhesive and substrates, and within adhesive (Source: Adhesive.org 2010)

Adhesive is a substance which when applied to surface of materials, capable of joining the materials and resists separation. The materials being joined are commonly known as adherends or substrates. The latter term (i.e. substrates) will be used throughout the thesis. In the thesis, adhesive and substrates are used to refer to bitumen (bitumen-filler mastic) and aggregates respectively. In the context of asphalt mixtures, adhesion may be used to refer to the amount of energy required to break the adhesive bond between bitumen (bitumen-filler mastic) and aggregates (Kanitpong & Bahia 2003).

Based on the study conducted by Fromm (1974), moisture damage is mainly characterised by the adhesive failure between bitumen (bitumen-filler mastic) and aggregates. Adhesive failure is primarily a result when bitumen (bitumen-filler mastic) coatings the aggregates is displaced by water or moisture, and a phenomenon referred to as stripping becomes visible in the asphalt mixtures. Water or moisture penetrates between the bitumen (bitumen-filler mastic) films and aggregates surface, breaks the adhesive bond and strips the bitumen (bitumen-filler mastic) from the aggregates surface due to higher affinity of some aggregates to water or moisture than to the bitumen (bitumen-filler mastic). Stripping is a complex phenomenon involving physical and chemical properties of the asphalt mixtures such as chemical composition of bitumen (bitumen-filler mastic) and aggregates, aggregates mineralogy and surface characteristics, and compositional characteristics and quantity of filler.

1.2 Problem Statement

Over the years, moisture damage has been recognised as a primary cause for pavement distresses. Based on the literature review and analysis of the past studies, moisture damage is mainly characterised by the adhesive failure between bitumen (bitumen-filler mastic) and aggregates (Fromm 1974; Kennedy et al. 1982; Majidzadeh & Brovold 1968; Tunnicliff & Root 1982). Hence, adhesion between bitumen (bitumen-filler mastic) and aggregates can be considered as one of the main fundamental properties, which can be correlated with the quality, performance and serviceability of the flexible pavements. However, despite its significance, research on the adhesion of the asphalt mixtures especially in correlation with moisture damage, is limited.

Attempts to place adhesion of the asphalt mixtures on a quantitative basis for measuring moisture damage performance have not been too successful. Currently available moisture damage performance tests such as Boiling Water test (ASTM D3625) and Modified Lottman test (AASHTO T283) only rely on the basis of comparative evaluation of mechanical properties (strength and/or modulus) of unconditioned and moisture conditioned specimens. Although this approach is helpful in terms of the comparative analysis of moisture susceptibility of various asphalt mixtures, the results cannot be used to distinguish the actual mechanisms that contribute to the moisture damage and none has been successfully correlated with the field performance data. Hence, it remains a challenge to asphalt pavement industries to develop an improved method for moisture damage performance based on the fundamental assessment of moisture damage mechanisms, especially in terms of the adhesion of the asphalt mixtures.

Adhesion and adhesive bond strength are the most important fundamental properties for surface coatings. The science and technology of adhesion and adhesive bond strength has formed a large amount of testing techniques and procedures used to measure the adhesive bond strength of coatings of composite materials such as plastic, metals and glasses. Among the most commonly used testing techniques and procedures are peel test, pull off test, double cantilever beam (DCB) test and tapered double cantilever beam (TDCB) test. However, in the pavement related areas, there are only few testing techniques and procedures known to be used for measuring the adhesive bond strength of coatings of asphalt mixtures and most of the testing techniques and procedures are used to measure the adhesive bond strength of tack coat, either in the laboratory or in the field. Tack coat is a thin bituminous layer applied between the existing pavements and the newly constructed pavements in order to promote bonding. The testing techniques and procedures used to measure the adhesive bond strength of tack coat is conducted by measuring the interaction between the thin bituminous layer and the asphalt mixtures of the existing pavement as a whole, rather than the interaction between components of the asphalt mixtures (i.e. bitumen (bitumen-filler mastic) and aggregates). Only in the past few years, have there been some efforts in developing testing techniques and procedures that can be used to directly measure the adhesive bond strength between components of the asphalt mixtures (i.e. bitumen and aggregates), such as published by Copeland (2007), Kanitpong and Bahia (2003), Kanitpong and Bahia (2004) and Kanitpong and Bahia (2005). However, the developed testing techniques and procedures have not enjoyed universal success and acceptance, and not yet established due to poor repeatability of the test results and limitations in terms of the applicability to measure the adhesive bond strength for wide ranges of asphalt mixture materials under various testing conditions (various conditioning procedures (dry and wet conditionings), deformation rates and test temperatures). Studies conducted by Copeland (2007), Kanitpong and Bahia (2003), Kanitpong and Bahia (2004) and Kanitpong and Bahia (2005) are still being carried out in order to improve the method.

Also, there is no published research in the pavement related areas that had determined the effect of different types of filler (i.e. bitumen-filler mastic) on the adhesive bond strength and failure characteristics of asphalt mixtures. Filler is a fine dust (aggregates particles less than 75 μm), used to harden the bitumen and improve the adhesion of the bitumen to the aggregates. Studies conducted by Copeland (2007), Kanitpong and Bahia (2003), Kanitpong and Bahia (2004), Kanitpong and Bahia (2005) and Marek and Herrin (1968) have only used pure bitumen (i.e. without filler) as adhesive materials for the purpose of simplification in the first step to gain basic knowledge of the adhesive bond strength and failure characteristics of asphalt mixtures.

Since adhesion between bitumen (bitumen-filler mastic) and aggregates is considered as one of the main fundamental properties of the asphalt mixtures and yet there is no established testing techniques and procedures that can be used to quantify the adhesive bond strength between bitumen (bitumen-filler mastic) and aggregates, research in this area is crucial and evidently needed. The emphasis of this study is focused on the development of laboratory adhesion test method that can be used to directly measure the adhesive bond strength between bitumen (bitumen-filler mastic) and aggregates. Previous studies on adhesion between bitumen and aggregates such as being conducted by Copeland (2007) and Kanitpong and Bahia (2005) will be referred throughout this study as guiding principle in developing criteria and procedures for the proposed adhesion test method.

1.3 Objective of Study

The main objective of this study was to develop and establish a simple, practical and reliable monotonically-loaded laboratory adhesion test method for direct measurement of the adhesive bond strength of bitumen (bitumen-filler mastic) and aggregates, and thus to quantify the adhesive bond strength and failure characteristics of various combinations of asphalt mixture materials over wide ranges of testing conditions. The specific objectives that need to be undertaken to achieve the main objective are as follows.

1. To conduct a comprehensive literature review on various testing techniques and procedures used to measure the adhesive bond strength in numerous areas of scientific literature and international standards.

2. To propose the most suitable and realistic approach among the various testing techniques and procedures for development of laboratory adhesion test method.

3. To adapt and establish the criteria and procedures for the proposed adhesion test method in order to suit the asphalt mixtures, in terms of test setup and apparatus, specimen preparation, testing and data analysis.

4. To evaluate the uniformity and repeatability of the test results in terms of thickness of adhesive layer of bitumen, total percentage area of adhesive failure, maximum tensile bond strength and tensile energy required to produce failure per unit volume, in order to validate the established criteria and procedures.

5. To further evaluate the established criteria and procedures in quantifying the test results of various combinations of asphalt mixture materials (i.e. bitumen (bitumen-filler mastic) and aggregates) over wide ranges of thicknesses of adhesive layer of bitumen, aspect ratio of specimens, testing conditions (i.e. deformation rates and test temperatures) and conditioning procedures (dry and wet conditionings).

6. To develop a draft protocol as guiding principles in conducting the laboratory adhesion test method.

1.4 Scope of Study

This study was divided into three parts based on the specific objectives and is outlined as follows.

1. Part 1: Selection and Justification of the Proposed Adhesion Test Method

   A detailed review of literature on various testing techniques and procedures used to measure the adhesive bond strength in numerous areas of scientific literature and international standards was assessed in order to propose the most suitable and realistic approach for development of laboratory adhesion test method for measuring the adhesive bond strength of bitumen (bitumen-filler mastic) and aggregates (i.e. Objectives 1 and 2). Among the testing techniques and procedures that have been taken into consideration are peel test, pull off test and double cantilever beam (DCB) test. The right selection of the approach is regarded as highly important as it will become the key success for this study. At the end of this part, a general concept for the proposed adhesion test method was developed.

2. Part 2: Development of Criteria and Procedures for the Proposed Adhesion Test Method

In this part, the general concept for the proposed adhesion test method from the previous part was subjected to evaluation, mainly based on the trial and error experimental approach, in order to adapt and thus to establish the criteria and procedures for test setup and apparatus, specimen preparation, testing and data analysis (i.e. Objective 3). In order to achieve the adhesive mode of failure, the procedures for specimen preparation were designed so that the thickness of adhesive layer of bitumen is uniform and as thin as possible. Development of the test setup and apparatus and testing were conducted in order to closely simulate the original adaptation of the proposed adhesion test method and at the same time being compatible with asphalt mixtures. At the end of this part, data analysis was conducted in order to evaluate the uniformity and repeatability of the test results in terms of thickness of adhesive layer of bitumen, total percentage area of adhesive failure, maximum tensile bond strength and tensile energy required to produce failure per unit volume (i.e. Objective 4). The final output for this part is the development and validation of the established criteria and procedures for the proposed adhesion test method in terms of test setup and apparatus, specimen preparation, testing and data analysis.

3. Part 3: Detailed Evaluation and Validation of the Proposed Adhesion Test Method

This part is a continuation from the previous part where the established criteria and procedures for test setup and apparatus, specimen preparation, testing and data analysis were subjected to further evaluation in quantifying the test results (i.e. thickness of adhesive layer of bitumen, total percentage area of adhesive failure, maximum tensile bond strength and tensile energy required to produce failure per unit volume) of various combinations of asphalt mixture materials (i.e. bitumen (bitumen-filler mastic) and aggregates) over wide ranges of thicknesses of adhesive layer of bitumen, aspect ratio of specimens, testing conditions (i.e. deformation rates and test temperatures) and conditioning procedures (dry and wet conditionings) (i.e. Objective 5). In order to consider wide ranges of asphalt mixture materials, at least two types of aggregates and/or bitumen (bitumen-filler mastic) of distinct properties that will reflect the ranges of typically used asphalt mixtures need to be utilised. Results of the study were subjected to comparative analysis in order to determine the effect of various variables and parameters on the test results, to propose suitable testing conditions and to validate the reliability and efficiency of the proposed adhesion test method. Also, at the end of this part, a draft protocol was developed as guiding principles in conducting the laboratory adhesion test method (i.e. Objective 6).

CHAPTER 2

LITERATURE REVIEW

This chapter provides background information necessary to understand the study. It defines asphalt mixtures and moisture damage in asphalt mixtures, and also discusses the two primary modes of failure for moisture damage, namely adhesive and cohesive failure. This chapter also defines adhesion and adhesive failure that occurs between bitumen (bitumen-filler mastic) and aggregates, and discusses the theory of adhesion of asphalt mixtures. A critical review on various testing techniques and procedures used to measure the adhesive bond strength in numerous areas of scientific literature and international standards is presented, in order to assess and propose a suitable and realistic approach in developing laboratory adhesion test method for measuring the adhesive bond strength of bitumen (bitumen-filler mastic) and aggregates. Also, at the end of the chapter, previous studies on tensile behaviour and failure characteristics of asphalt mixtures such as being conducted by Copeland (2007) and Marek and Herrin (1968) are reviewed, in order to provide some guidelines for development of criteria and procedures for the proposed adhesion test method.

2.1 General Background

Great Britain, which has a total land area of 227,469 km^2, was linked by 398,026 km of roads in 2008 (Department for Transport 2009; Infoplease 2008). Roads account for about 93% of the passenger-kilometres travelled and about 73% of the tonne-kilometres of goods traffic. Basically, roads can be broken down into two broad categories, namely flexible and rigid pavements. Majority of roads in the Great Britain and also throughout the world are comprised of flexible pavements.

2.1.1 Asphalt Mixtures in General

Flexible pavements are combination of predetermined ratio of asphalt mixture materials (i.e. aggregates, bitumen and filler), which must be able to resist deformation, cracking and water or moisture damage, be durable over time and yet be inexpensive and easy to construct. Sometime additives such as rubbers and fibres are used to improve the performance. Properties and interactions of the asphalt mixture materials will determine the quality, performance and serviceability of the resulting flexible pavements.

Aggregates are crushed stone, sand and fines, mixed in a predetermined proportion to provide strong structural skeleton and mechanical strength for the asphalt mixtures. Aggregates constitute about 92% to 96% by mass of the asphalt mixtures, and hence play an important part in the quality and performance of the flexible pavements (Kandhal et al. 1997). Physical properties of the aggregates are the most readily apparent properties and have the most direct effect on the aggregates performance as asphalt mixture materials. The commonly measured physical properties of the aggregates are resistance to crushing, impact, abrasion, polishing and stripping, specific gravity, water absorption, particle shape and texture, and gradation. Table 2.1 provides general guidelines for the selection of the aggregates as asphalt mixture materials based on the types of the aggregates. Also, chemical properties of the aggregates play an equally important role in determining how well the interaction between components of the asphalt mixtures (i.e. bitumen (bitumen-filler mastic) and aggregates). Physical and chemical properties of the aggregates can change over time, and hence make it difficult to accurately predict the aggregates performance.

Table 2.1 General guidelines for the selection of the aggregates (Source: Cordon 1979)

Types of Rocks	Types of Aggregates	Resistance to Crushing, Impact and Abrasion	Resistance to Stripping[1,2]	Particle Texture	Particle Shape
Igneous	Granite	Fair	Fair	Fair	Fair
	Syenite	Good	Fair	Fair	Fair
	Diorite	Good	Fair	Fair	Good
	Basalt	Good	Good	Good	Good
	Diabase	Good	Good	Good	Good
	Gabbro	Good	Good	Good	Good
Sedimentary	Limestone	Poor	Good	Good	Fair
	Sandstone	Fair	Good	Good	Good
	Chert	Good	Fair	Poor	Good
	Shale	Poor	Poor	Fair	Fair
Metamorphic	Gneiss	Fair	Fair	Good	Good
	Schist	Fair	Fair	Good	Fair
	Slate	Good	Fair	Fair	Fair
	Quartzite	Good	Fair	Good	Good
	Marble	Poor	Good	Fair	Fair
	Serpentine	Good	Fair	Fair	Fair

Notes: [1]Hydrophilic (attract water) aggregates are more likely to strip; [2]Freshly crushed aggregates with many broken ionic bonds are more likely to strip

Bitumen is a complex hydrocarbon, found as natural deposit or residue from distilling crude oil. Bitumen acts as waterproof, thermoplastic and viscoelastic binder that acts to hold together the aggregates particles in the asphalt mixtures. Although the proportion of the bitumen in the asphalt mixtures is much less than that of the aggregates (i.e. up to a minimum of 4% by mass of the asphalt mixtures), the quantity and quality of the bitumen both have a marked effect on the performance of the flexible pavements (Wignall et al. 1991). Physical properties of the bitumen can be measured using various testing techniques and procedures such as penetration test, softening point test and ductility test. In terms of the chemical properties of the bitumen, there is limited knowledge to adequately predict the performance.

Filler is a fine dust (aggregates particles less than 75 μm), used to harden the bitumen and improve the adhesion of the bitumen to the aggregates. Although bitumen constitutes the binder part of the asphalt mixtures, it is the combination of bitumen and filler known as bitumen-filler mastic that coats the aggregates and can therefore be considered as the true binder.

By weight, aggregates and filler generally account for between 92% and 96% of the asphalt mixtures, while bitumen accounts for about 4% to 8%. Different ratios of aggregates, bitumen and filler (as well as the small proportion of air) give rise to different types of asphalt mixtures of different properties and serve different purposes. However, of all types of the asphalt mixtures, there is one common problem influencing the performance and serviceability, namely as moisture damage.

2.1.2 Definition of Moisture Damage and Moisture Damage Mechanisms

Moisture damage is an extremely complicated mode of distress and represents a conditioning process due to the presence of water or moisture in the pavement structure. The interaction of water or moisture with bitumen (bitumen-filler mastic) and aggregates can result in loss of structural strength and stiffness of the asphalt mixtures. The consequences initially in the form of stripping, ravelling, surface wear, rutting and fatigue cracking, if unattended to, would lead to serious and irreparable damage, and will cause the pavements to lose serviceability earlier than expected. Although the occurrence of the pavement distresses is not necessarily initiated by the presence of water or moisture, multiple forms of pavement distress mechanisms could increase their extent and severity due to the presence of water or moisture (Miller & Bellinger 2003).

Moisture damage can show itself in various forms such as adhesive failure between bitumen (bitumen-filler mastic) and aggregates, cohesive failure within bitumen (bitumen-filler mastic), cohesive failure within aggregates and/or freezing of entrapped water or moisture in the pavement structure (Asphalt Institute 2007; Kim & Coree 2005). However, the most common forms of moisture damage are due to the adhesive failure (loss of adhesion) between bitumen (bitumen-filler mastic) and aggregates, which is the most prevalent form and the cohesive failure (loss of cohesion) within bitumen (bitumen-filler mastic) (Kanitpong & Bahia 2003; Kim & Coree 2005; Solaimanian et al. 2007).

Adhesive failure (loss of adhesion) is primarily a result when bitumen (bitumen-filler mastic) coatings the aggregates is completely displaced by water or moistures. However, based on the study conducted by Hughes and Maupin (1989), stain or discoloration of the aggregates surface left by the separation of the bitumen (bitumen-filler mastic) films is still being considered as the adhesive failure. Water or moisture penetrates between the bitumen (bitumen-filler mastic) films and aggregates surface, breaks the adhesive bond and strips the bitumen (bitumen-filler mastic) from the aggregates surface due to higher affinity of some aggregates to water or moisture than to the bitumen (bitumen-filler mastic). Pavement distress mechanisms referred to as stripping will thus becomes visible in the asphalt mixtures, as shown in Figure 2.1 (Kandhal & Rickards 2001). Kiggundu and Roberts (1988) has defined stripping as follows.

"The progressive functional deterioration of asphalt mixtures due to the loss of the adhesive bond between bitumen (bitumen-filler mastic) and aggregates surface, principally from the action of water or moisture."

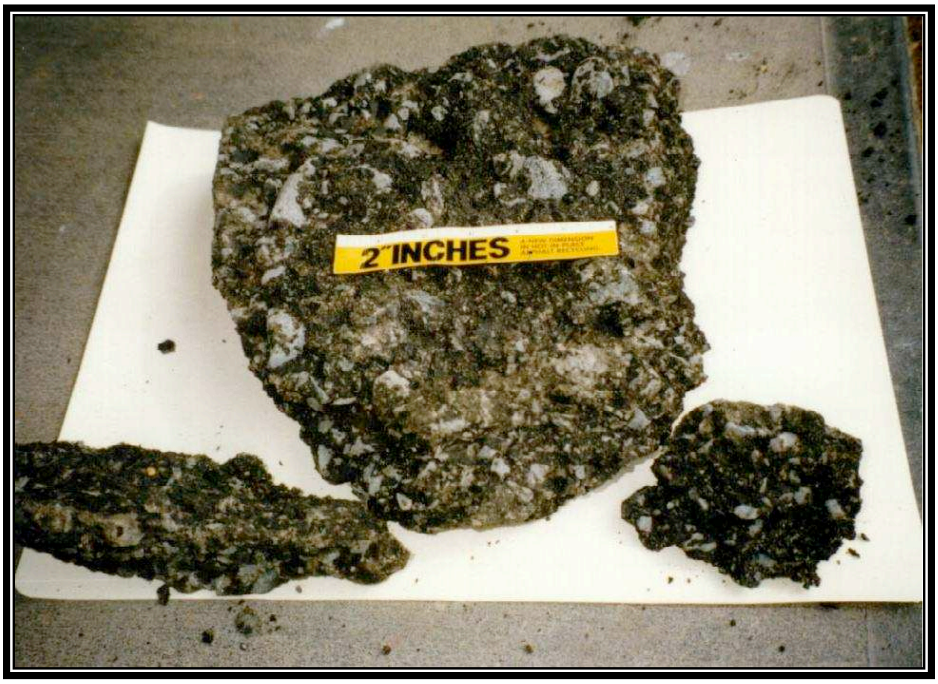


Figure 2.1 Stripping of bitumen (bitumen-filler mastic) from aggregates surface (Source: Kandhal & Rickards 2001)

According to Kandhal and Rickards (2001), there are four main factors that encourage stripping; presence of water or moisture, high air voids content, high temperature and high stress. Also, based on the studies done by Kiggundu and Roberts (1988), Taylor and Khosla (1983) and Terrel and Al-Swailmi (1994), there are several mechanisms for the bitumen (bitumen-filler mastic) films to be stripped from the aggregates surface, including detachment, displacement, spontaneous emulsification, pore pressure and hydraulic scouring.

1. Detachment is the microscopic separation of bitumen (bitumen-filler mastic) films from aggregates surface due to the presence of thin films of water or moisture without an obvious break in the bitumen (bitumen-filler mastic) films. The thin films of water or moisture probably resulted from either aggregates that were not completely dry or interstitial pore water or moisture, which vaporised and condensed on the aggregates surface (Johnson & Freeman 2002).

2. Displacement occurs when the bitumen (bitumen-filler mastic) films is removed from the aggregates surface by water or moisture. As compared to the detachment, displacement occurs due to the intrusion of water or moisture into the aggregates surface through breaks of the bitumen (bitumen-filler mastic) films. The breaks of the bitumen (bitumen-filler mastic) films may arise from incomplete coatings of the bitumen (bitumen-filler mastic) films on the aggregates surface or rupture of the bitumen (bitumen-filler mastic) films at the sharp corners or edges of the aggregates.

3. Spontaneous emulsification can be defined as inverted emulsion of water or moisture in bitumen phase. Based on the study conducted by Fromm (1974), the inverted emulsion of water or moisture in bitumen phase will cause the bituminous particles to separate from each other (cohesive failure) and ultimately leads to the adhesive failure when the emulsion boundary propagates to the coated aggregates surface.

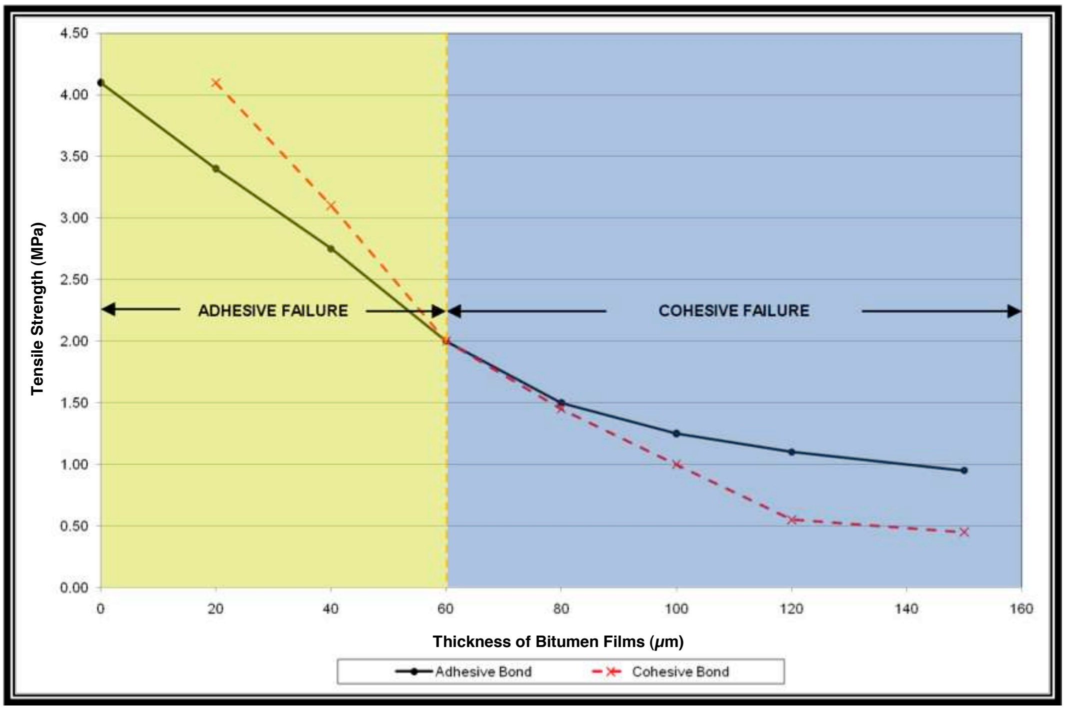
4. Water or moisture entrapped within the asphalt mixtures can lead to the pore pressure build-up due to the repeated traffic loads, and freeze and thaw cycles. Continuation of the process for the pore pressure build-up

will ultimately leads to the degradation of the adhesive bond strength of bitumen (bitumen-filler mastic) and aggregates and thus growth of the micro-cracks in the asphalt mixtures.

5. Hydraulic scouring is caused by the occurrence of capillary tension and compression around a moving traffic on saturated pavement surface. Bitumen (bitumen-filler mastic) is stripped from the aggregates surface, producing defects known as ravelling. In addition, dust is reported to mix with water or moisture and, in the presence of traffic, can enhance the abrasion of bitumen (bitumen-filler mastic) films from the aggregates.

Cohesive failure (loss of cohesion) refers to the failure within bitumen (bitumen-filler mastic) itself due to the interaction with water or moisture. Water or moisture enters the bitumen (bitumen-filler mastic) through absorption, reducing the cohesive strength via softening and causing the asphalt mixtures to lose stiffness and durability. Cohesive failure can be explained using the inverted emulsion of water or moisture in the bitumen phase. Water or moisture may behave like a solvent in the bitumen phase and result in reduced cohesive strength and increased permanent deformation.

Based on the study conducted by Lytton et al. (2005), moisture damage could occur due to either adhesive or cohesive failure, depending on the nature and thickness of bitumen films coatings the aggregates. Marek and Herrin (1968) has conducted an experimental study on the behaviour and failure characteristics of thin films of bitumen, and a part of the study has correlated the tensile bond strength and the types of failure with the thickness of bitumen films. In the study, aluminium alloy has been used as substrates due to the value of Young's Modulus of approximately 70 GPa, which is close to the typical value of aggregates and also due to the corrosion resistance properties. Using the experimental data of Marek and Herrin (1968), Lytton et al. (2005) has used the micromechanics analysis in order to reproduce the relationship between these parameters (i.e. tensile bond strength, types of failure and thickness of bitumen films) as shown in Figure 2.2. For thinner films of less than 60 μm (0.060 mm), the adhesive bond tensile strength was found to be less than the cohesive bond tensile strength; hence adhesive failure is expected to occur, and vice versa for thicker films (between 60 μm (0.060 mm) and 150 μm (0.150 mm)). Based on the micromechanics analysis, transition of the mode of failure of either adhesive or cohesive was expected to occur at thickness of bitumen of about 60 μm (0.060 mm).



Notes:
1. For thickness of bitumen films between 0 μm and 60 μm, adhesive bond tensile strength is less than the cohesive bond tensile strength; hence adhesive failure is expected to occur
2. For thickness of bitumen films between 60 μm and 150 μm, cohesive bond tensile strength is less than the adhesive bond tensile strength; hence cohesive failure is expected to occur
3. Substrates: Aluminium alloy
4. Adhesive Materials: Asphalt cement K (Penetration at 25°C is 52)
5. Conditioning Procedures: Dry conditioning at room temperature for 3 hours prior to testing
6. Testing Conditions: Deformation rate and test temperature of 0.508 mm/minute and 25°C respectively

Figure 2.2 Relationship between tensile strength and thickness of bitumen films (Source: Lytton et al. 2005)

Since the thickness of bitumen (bitumen-filler mastic) across the actual pavement structure varies considerably, generally within the ranges of 15 μm (0.015 mm) and 40 μm (0.040 mm), both adhesive and cohesive failure could occur, with one of them perhaps being dominant. However, many studies have concluded that moisture damage of the asphalt mixtures is more the adhesive mode of failure than the cohesive mode of failure (Fromm 1974; Kennedy et al. 1982; Majidzadeh & Brovold 1968; Tunnicliff & Root 1982). The emulsification of water or moisture in the bitumen (bitumen-filler mastic) will cause the bituminous particles to separate from each other (cohesive failure) and ultimately leads to the adhesive failure when the emulsification boundary propagates to the coated aggregates surface. According to Terrel and Al-Swailmi (1994), since mechanisms of the cohesive failure lead, ultimately to the adhesive failure, the final mechanisms of adhesive failure will be reported as the cause of moisture damage distress.

2.2 Adhesion of Asphalt Mixtures

In this section, definitions of the adhesion and adhesive failure in general and in the context of asphalt mixtures are presented, and theory of adhesion of asphalt mixtures is discussed.

2.2.1 Definition of Adhesion and Adhesive Failure

As has been stated before, adhesion can be defined as the molecular force of attraction in the area of contact between unlike bodies (i.e. adhesive and substrates) that acts to hold the bodies together (Copeland 2007). In the context of asphalt mixtures, adhesion may be used to refer to the amount of energy required to break the adhesive bond between bitumen (bitumen-filler mastic) and aggregates (Kanitpong & Bahia 2003). By contrast, cohesion is the intermolecular force developed within the bitumen (bitumen-filler mastic) that holds the molecules of the bitumen (bitumen-filler mastic), and is influenced by viscosity. Figure 2.3 shows the adhesion and cohesion of the bitumen (bitumen-filler mastic) and aggregates respectively.

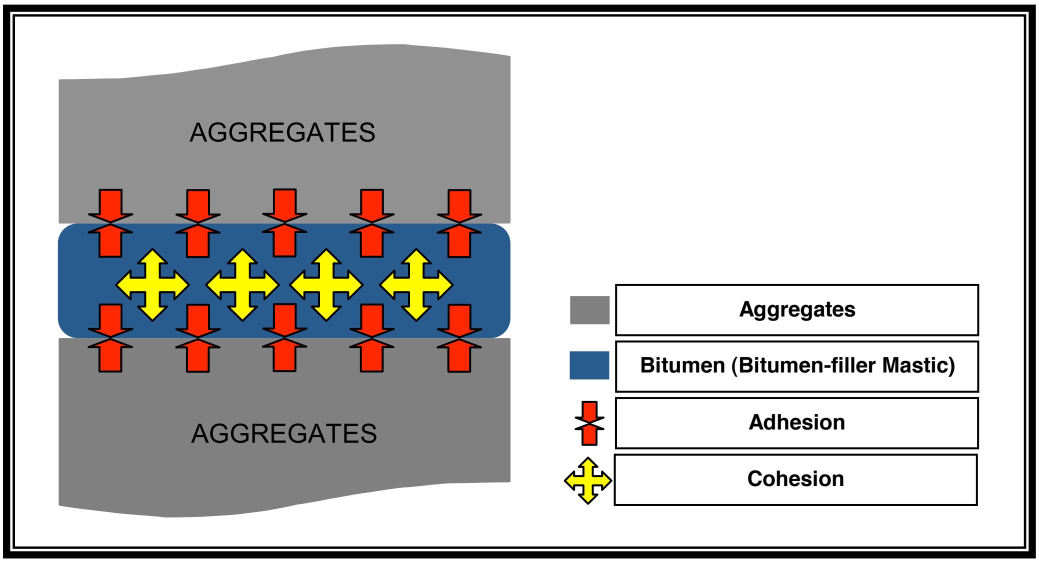


Figure 2.3 Adhesion and cohesion of bitumen (bitumen-filler mastic) and aggregates

Adhesive failure refers to the displacement of the bitumen (bitumen-filler mastic) from the aggregates surface, which indicates low magnitude of the adhesive bond strength. Adhesive bond strength between bitumen (bitumen-filler mastic) and aggregates is mainly influenced by the physical and chemical properties of the asphalt mixture materials such as chemical composition of bitumen (bitumen-filler mastic) and aggregates, mineralogy and surface characteristics, and compositional characteristics and quantity of filler. Cohesive failure refers to the failure within bitumen (bitumen-filler mastic) itself due to the low magnitude of the cohesive bond strength as compared to the adhesive bond strength. Figure 2.4 illustrates the types of failure (i.e. adhesive failure, cohesive failure and mixed cohesive and adhesive failure) that might possibly occur between bitumen (bitumen-filler mastic) and aggregates. Failure for moisture damage of asphalt mixtures is usually neither entirely adhesive nor entirely cohesive. The simplest and easiest method to identify the forms of failure is via visual observation and calculation based on the percentage area of adhesive and cohesive failure.

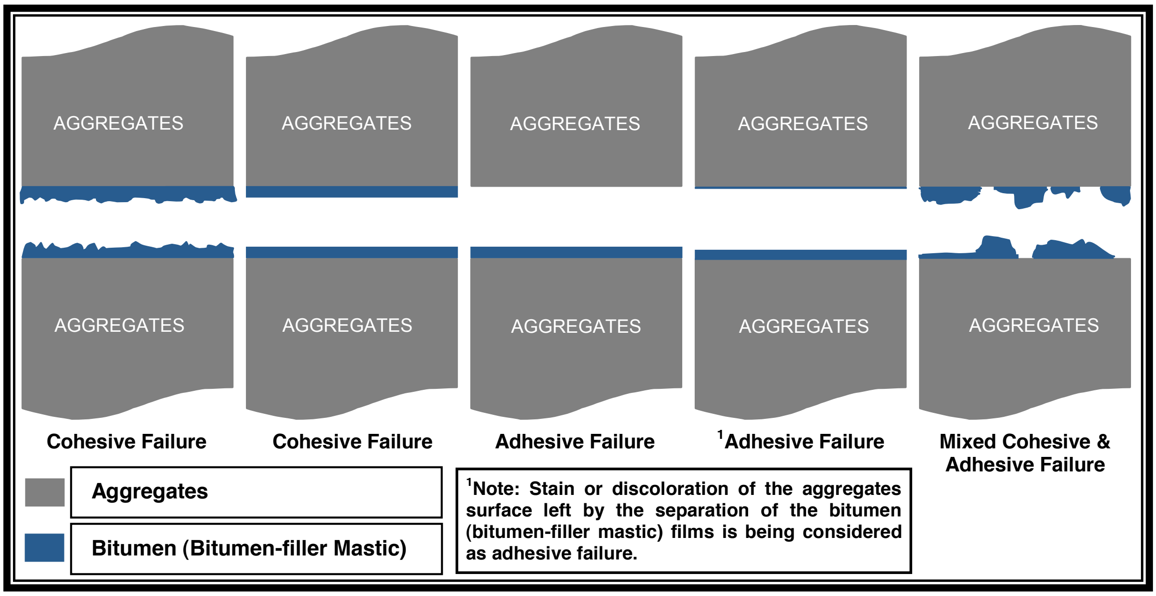


Figure 2.4 Possible types of failure that might occur in asphalt mixtures

Rand (2002) has tried to quantify the adhesion of various combinations of asphalt mixture materials (i.e. bitumen and aggregates) by performing submerged wheel tracking test. Data of the test results in terms of the percentage of adhesion remains on the asphalt mixtures after test is shown in Figure 2.5. The percentage of adhesion remains on the asphalt mixtures after test was calculated by identifying the forms of failure as either adhesive or cohesive via visual observation. Based on the study, Rand (2002) has concluded that the types of failure of either adhesive or cohesive were mainly influenced by the properties of the aggregates rather than the properties of the bitumen. Also, for hydrophobic (i.e. repulse water) aggregates such as limestone and basalt, the effect of different types of bitumen is almost negligible. Based on Rand (2002), susceptibility of the asphalt mixtures to stripping can be improved by the addition of hydrated lime as filler or application of the modified bitumen as adhesive materials, depending on the combination of the asphalt mixture materials.

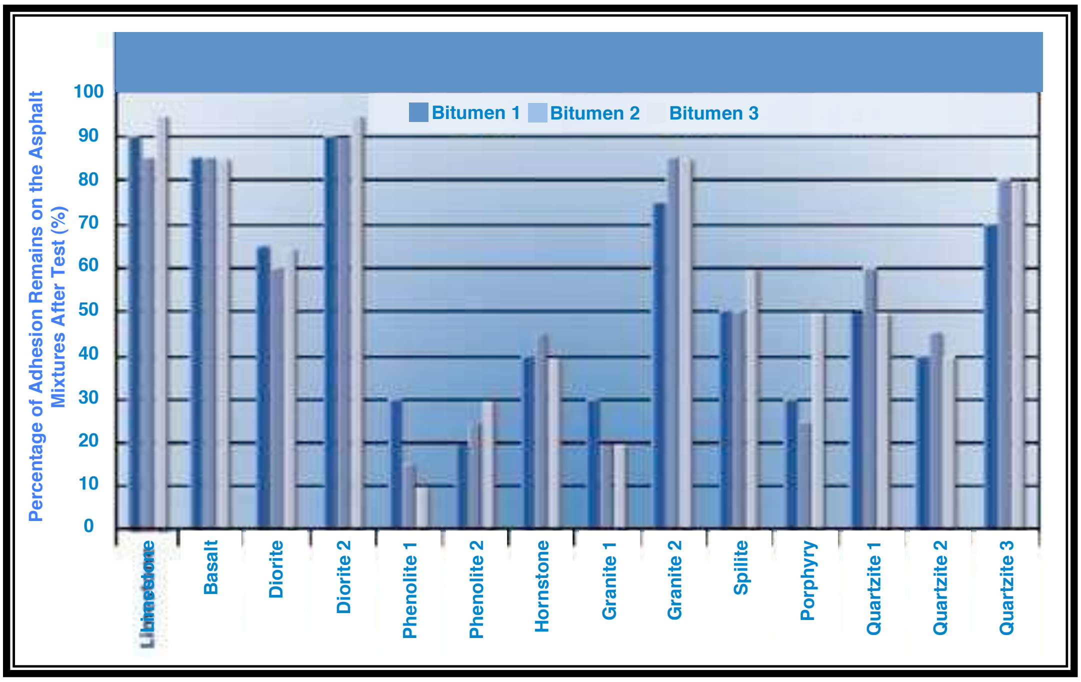


Figure 2.5 Percentage of adhesion remains on the asphalt mixtures after test for various combinations of asphalt mixture materials (Source: Rand 2002)

2.2.2 Theory of Adhesion of Asphalt Mixtures

Hicks (1991) has identified four broad theories that have been developed to explain the adhesion between bitumen and aggregates, namely as mechanical adhesion theory, chemical reaction theory, surface energy theory and molecular orientation theory. These theories each individually explain some aspects of the adhesion but do not completely capture the mechanisms.

1. Mechanical Adhesion Theory

Based on Terrel and Al-Swailmi (1994), mechanical adhesion theory has suggested that adhesion between bitumen and aggregates is affected by the physical properties of the aggregates such as particle size, surface texture, angularity, porosity or absorption and surface areas. Bitumen gets into the surface irregularities and pores of the aggregates, and hardens, causing a mechanical interlock. In general, stronger adhesive bond strength of bitumen and aggregates is created with rough, porous aggregates of large surface areas. However, according to Tarrar and Wagh (1992), aggregates having a relatively smooth surface texture are easier to coat as compared to the rough surface texture. The presence of water or moisture on the aggregates surface may decrease the mechanical interlock, thus increasing the susceptibility of the asphalt mixtures to stripping. According to Kandhal (1994), physicochemical surface properties of the aggregates are more important for moisture-induced stripping as compared to the properties of the bitumen.

2. Chemical Reaction Theory

Chemical reaction theory has been generally accepted to explain the differences in the degree of adhesion between different types of bitumen and aggregates, in the presence of water or moisture. Aggregates may be classified as either hydrophilic (attract water) or hydrophobic (repulse water) as shown in Figure 2.6. The main properties of the aggregates that determine the characteristics of either hydrophilic or hydrophobic are surface chemistry, porosity and pore size. Hydrophilic aggregates such as siliceous aggregates (e.g. granite) tend to strip easier than hydrophobic aggregates (e.g. limestone). Generally, a more acidic aggregates surface is less likely to form bonds as strongly with the bitumen and thus increase the susceptibility of the asphalt mixtures to stripping. In other words, the pH values of the aggregates surface and the bitumen affect the adhesive bond strength of the asphalt mixtures. The reason for this has been attributed to different polarities of the surface minerals in the aggregates and the bitumen.

Also, past studies have shown that the pH values of water or moisture in the pavement structure are influenced by the aggregates surface (Huang et al. 2000; Labib 1992; Scott 1978; Yoon & Tarrer 1988). Figure 2.7 shows the effect of different types of aggregates surface on pH values of water or moisture under different contacting time. In the conducted study, aggregates powders were added to the water or moisture. Based on Figure 2.7, most of the aggregates surface tends to increase the pH values of water or moisture as the contacting time is increased. The increment of the pH values of water or moisture is not restricted to the hydrophobic aggregates such as limestone, but also occurs with the hydrophilic aggregates such as granite. Hence, the classification of the aggregates as either hydrophilic or hydrophobic can only be used as rough assessment rather than absolute.

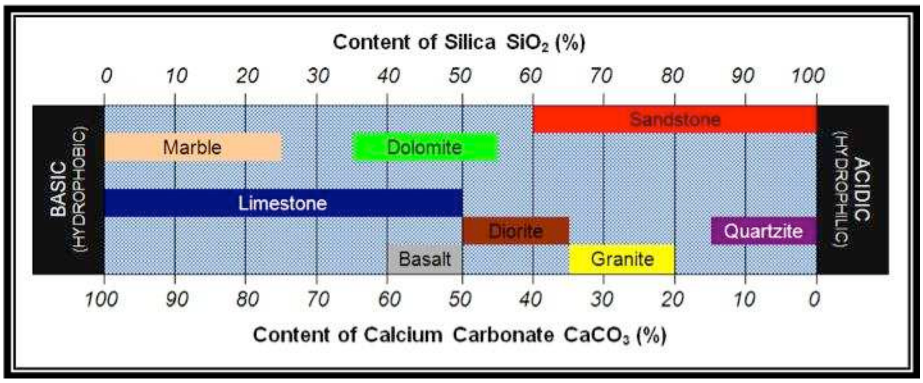


Figure 2.6 Classification of aggregates as hydrophilic and hydrophobic (Source: Huang 2004)

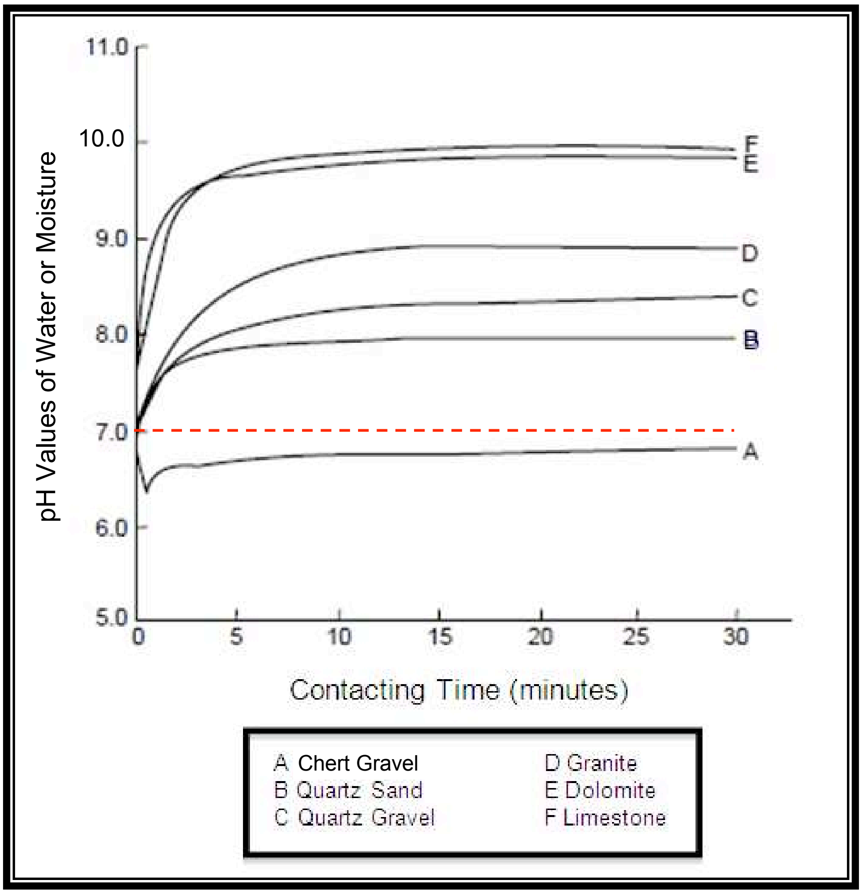


Figure 2.7 Effect of different types of aggregates surface on pH values of water or moisture under different contacting time (Source: Yoon & Tarrer 1988)

Viscosity of the bitumen (bitumen-filler mastic) may indicate the concentrations of the asphaltenes (polar molecules). Asphaltenes (polar molecules) can create greater adhesion between bitumen (bitumen-filler mastic) and aggregates due to the greater adhesion tension and molecular orientation adhesion. Therefore, asphalt mixtures of lower viscosities, which may represent lower concentrations of asphaltenes (polar molecules), are generally susceptible to moisture damage. Individual components in the bitumen such as sulfoxides, carboxylic acids, phenols and nitrogen bases can also affect moisture susceptibility (Hicks 1991).

3. Surface Energy Theory

For an effective bond, bitumen (bitumen-filler mastic) should completely coat or wet the aggregates surface. The wetting ability of the bitumen (bitumen-filler mastic) can be explained using surface energy theory. Surface energy is defined as the energy needed to create a unit area of new surface between bitumen (bitumen-filler mastic) and aggregates in vacuum condition. Rice (1959) has suggested that when bitumen (bitumen-filler mastic) and aggregates are brought in contact, adhesion tension is established. However, the adhesion tension between bitumen (bitumen-filler mastic) and aggregates is generally less than the adhesion tension between water or moisture and aggregates. Therefore, in the presence of water or moisture, bitumen (bitumen-filler mastic) will tend to be displaced from the aggregates surface. This can result in poor wetting of the aggregates surface by the bitumen (bitumen-filler mastic) films and lead to stripping. Hicks (1991) has stated as follows.

> "Water or moisture will tend to displace bitumen (bitumen-filler mastic) at the interfaces of the bitumen (bitumen-filler mastic) and aggregates where there is contact between water or moisture, bitumen (bitumen-filler mastic) and aggregates."

The adhesion tension between bitumen (bitumen-filler mastic) and aggregates varies with the types of aggregates, roughness of the aggregates surface and types of bitumen. Researchers at Texas A & M University and Western Research Institute in Wyoming have conducted research in measuring the adhesive bond strength of bitumen and aggregates based on the thermodynamic surface free energy characteristics of aggregates, bitumen and water (moisture) (Bhasin et al. 2006; Cheng et al. 2002; Masad et al. 2006).

New terms, adhesive bond energy related parameters, which consist of adhesive bond energy of aggregates and bitumen in dry condition and in the presence of water or moisture, have been introduced. A high magnitude of adhesive bond energy of aggregates and bitumen in dry condition is desirable in order for asphalt mixtures to perform as durable pavements. The adhesive bond energy of aggregates and bitumen in the presence of water or moisture is quantified based on the amount of reduction of surface free energy when bitumen debonds from the aggregates surface. A high magnitude of reduction of surface free energy of the aggregates and bitumen system would means higher propensity for water or moisture to debonds the bitumen from the aggregates surface and vice versa. Therefore, aggregates and bitumen system with high magnitude of adhesive bond energy in the dry condition and low magnitude of reduction of surface free energy in the presence of water or moisture should has reduced potential to debonds and therefore, will possess a greater resistance to moisture damage (Bhasin et al. 2006). Figure 2.8 summarises the procedures employed.

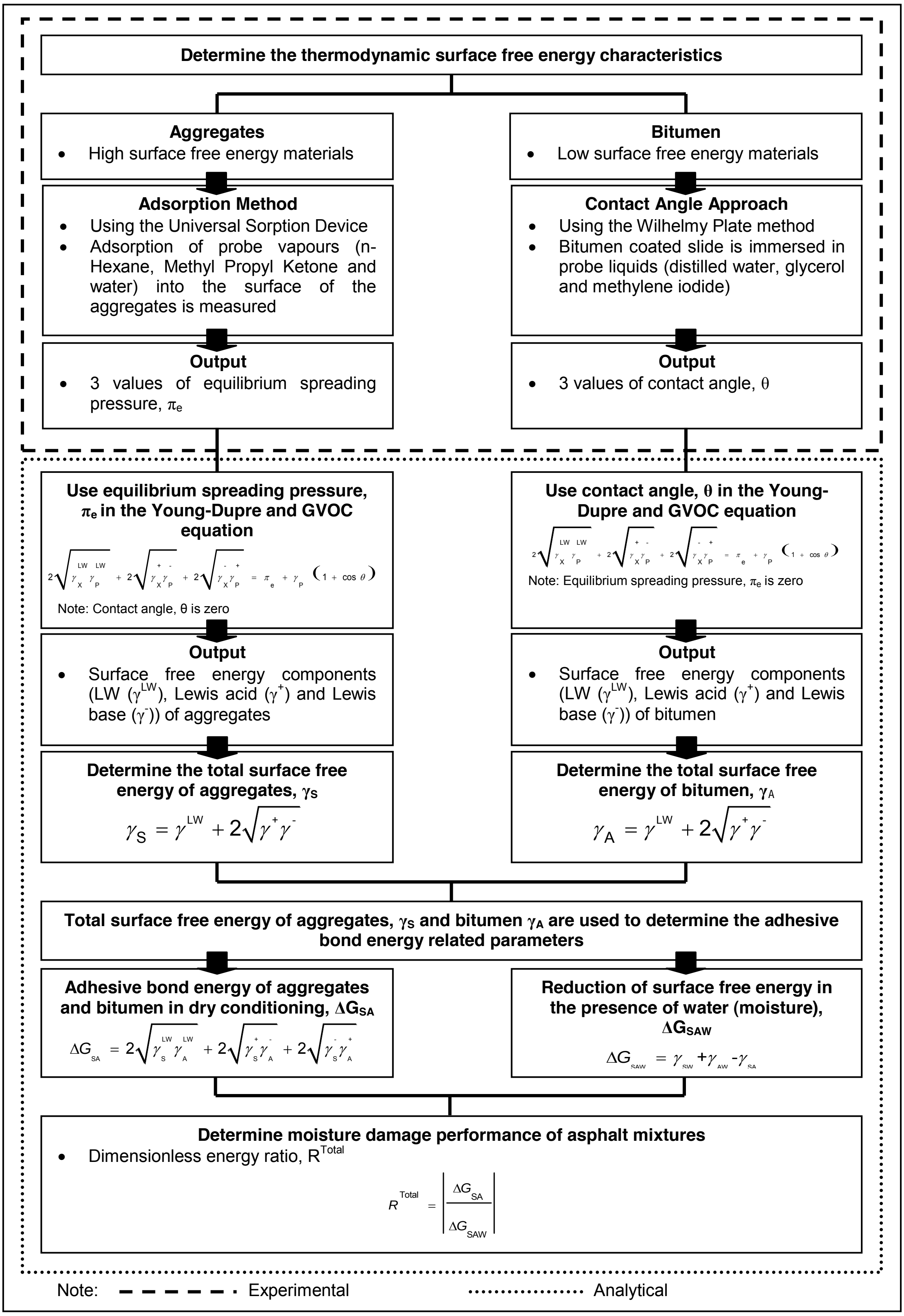


Figure 2.8 Procedures for measuring adhesive bond strength based on thermodynamic surface free energy (Source: Bhasin et al. 2006)

4. Molecular Orientation Theory

Molecular orientation theory affirms that when bitumen (bitumen-filler mastic) is in contact with aggregates surface, molecules of bitumen tend to orient themselves in order to satisfy the energy demand of the aggregates surface. Bitumen consists of a combination of non-polar (Lifshitz-van der Waals) and polar (Lewis acid and Lewis base) molecules. Hicks (1991) has stated as follows.

> "Molecules of water are dipolar. Molecules of bitumen are generally non-polar, although some components are polar. Consequently, molecules of water, being more polar, may more readily satisfy the energy demand of the aggregates surface."

Depending on the surface compositions of the aggregates, molecules of water or moisture may preferentially satisfy the energy demand of the aggregates surface, thus result in degradation of the adhesive bond between bitumen (bitumen-filler mastic) and aggregates, and increase the susceptibility of the asphalt mixtures to stripping.

2.2.3 Summary of Adhesion of Asphalt Mixtures

Adhesion and adhesive failure between bitumen (bitumen-filler mastic) and aggregates is a complex phenomenon involving numerous areas of study such as physical and chemical properties and interactions of the asphalt mixture materials, pavement mix design and construction methods, and diversified environmental and ageing conditions. Research on the adhesion of the asphalt mixtures especially in correlation with moisture damage, is limited and there is no established testing techniques and procedures that can be used to quantify the adhesive bond strength between bitumen (bitumen-filler mastic) and aggregates. Only in the past few years, there have been some efforts in developing testing techniques and procedures that can be used to directly measure the adhesive bond strength between bitumen and aggregates, such as published by Copeland (2007), Kanitpong and Bahia (2003), Kanitpong and Bahia (2004) and Kanitpong and Bahia (2005). However, the developed testing techniques and procedures have not enjoyed universal success and acceptance, and not yet established due to poor repeatability of the test results and limitations in terms of the applicability to measure the adhesive bond strength for wide ranges of asphalt mixture materials under various testing conditions (various conditioning procedures (dry and wet conditionings), deformation rates and test temperatures). Since adhesion between bitumen (bitumen-filler mastic) and aggregates is considered as one of the main fundamental properties of the asphalt mixtures and there are no established testing techniques and procedures that can be used to quantify the adhesive bond strength between bitumen (bitumen-filler mastic) and aggregates, research in this area is crucial and evidently needed.

2.3 A Review of Adhesion Test Methods

Research on the adhesion and adhesive failure has been well established for composite materials such as plastic, metals and glasses, and a large amount of testing techniques and procedures used to measure the adhesive bond strength have been developed. In this section, a detailed review of literature on various testing techniques and procedures used to measure the adhesive bond strength which can be found in numerous areas of scientific literature and international standards is presented, in order to propose the most suitable and realistic approach for development of laboratory adhesion test method for asphalt mixtures. In following sub-section, the reviewed testing techniques and procedures are divided into two; those performed on composite materials other than asphalt mixtures and those performed on asphalt mixtures.

2.3.1 Adhesion Test Methods Performed on Composite Materials Other Than Asphalt Mixtures

Adhesion is the most important property for surface coatings. The science and technology of adhesion has formed a large amount of testing techniques and procedures used to measure the adhesive bond strength of coatings of composite materials. Among the most commonly used testing techniques and procedures are peel test, pull off test, double cantilever beam (DCB) test, tapered double cantilever beam (TDCB) test, impact wedge peel (IWP) test and scratching of thin films test. These testing techniques and procedures have been successfully used in measuring the adhesive bond strength of coatings of composite materials such as plastic, metals and glasses. Adhesion and Adhesives Research Group from Imperial College London was found to provide a large number of references on these testing techniques and procedures.

2.3.1.1 Peel Test

Peel test is relatively easy, inexpensive and well developed adhesion test method, and is widely used in various engineering applications, especially in the aerospace and automotive industries in measuring the adhesive bond strength of bonded joints and laminates of various composite materials. The results from the peel test which is conducted based on the Elastic-Plastic-

Fracture-Mechanics (EPFM) approach will usually give the experienced users a good first impression of the initial capability of new adhesive materials (Kinloch 1987). One of the examples of the application of the peel test is in the packaging and electronics industries where the peel test is used in assessing the failure of flexible laminates between polymeric films and also between polymeric films and thin metallic films (Kinloch 1997). Over the years, there are various standards that have been developed for peel test, and numerous modifications for the developed and established peel test have been reported (Moore 2008). The various peel tests differ in the way that the load is applied; however remain the same in the basic principles. Figure 2.9 shows the various peel tests available in various engineering applications and Figure 2.10 shows typical peel test that are commonly used. For the typical peel test that are commonly used (i.e. Fixed Arm and T Peel), a thin flexible adhesively bonded peel arm is pulled at a specified angle and rate from the rigid substrate as in the Fixed Arm or from another thin flexible adhesively bonded peel arm as in the T Peel.

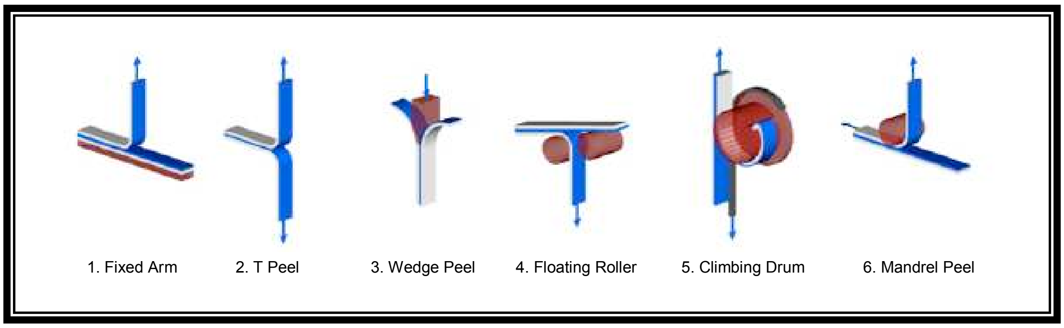


Figure 2.9 Various peel test methods (Source: Moore 2008)

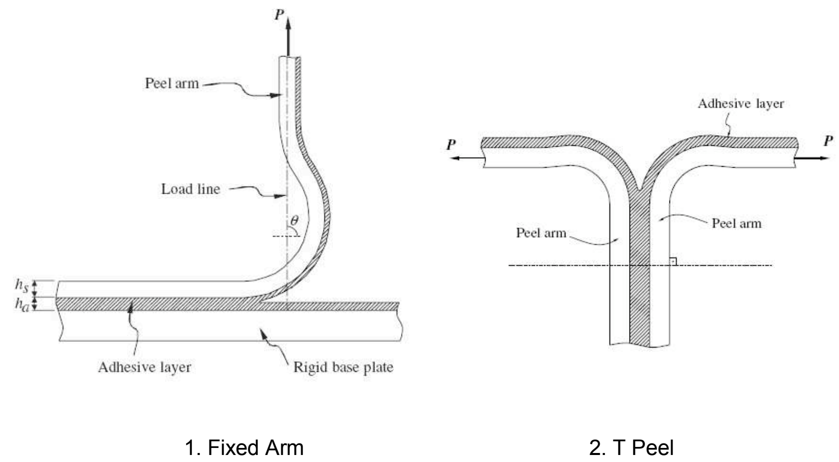


Figure 2.10 Typical peel test commonly used in various engineering applications (Source: Hadavinia et al. 2006)

Generally, for the Fixed Arm, in order to pull the peel arm from the rigid substrates, energy in the form of external work is required. Kinloch (1987) shows the relationship between energy in the form of external work and adhesive fracture energy, G_C in the following equation.

Equation 2.1

$$G_c = \frac{1}{B}\left(\frac{dU_{ext}}{da} - \frac{dU_S}{da} - \frac{dU_{dt}}{da} - \frac{dU_{db}}{da}\right)$$

where:

G_C	=	*Adhesive fracture energy*
B	=	*Width of the peel arm*
a	=	*Crack length*
U_{ext}	=	*Energy in the form of external work*
U_s	=	*Strain energy stored in the peel arm*
U_{dt}	=	*Energy dissipated during tensile deformation of the peel arm*
U_{db}	=	*Energy dissipated during bending of the peel arm*

In an ideal case, Kinloch (1987) has assumed that there is no tensile deformation of the peel arm and the bending of the peel arm is elastic. Hence, the adhesive fracture energy, G_C can be correlated with the applied peel load and peel angle as in the Equation 2.2.

Equation 2.2

$$G_c = \frac{P}{B}(1 - \cos\theta)$$

where:

G_C	=	*Adhesive fracture energy*
P	=	*Applied peel load*
B	=	*Width of the peel arm*
θ	=	*Peel angle*

However, if there is elastic-plastic deformation occurred in the peel arm, it is necessary to quantify the tensile characteristics of the peel arm (i.e. tensile strain and tensile stress), and the adhesive fracture energy, G_C can be calculated as follows.

Equation 2.3

$$G_c = \frac{P}{B}(1 + \varepsilon - \cos\theta) - \left(h_s \int_0^{\varepsilon} \sigma d\varepsilon\right) + \left(\frac{1}{B}\frac{dU_{db}}{da}\right)$$

where:

G_C	=	*Adhesive fracture energy*
P	=	*Applied peel load*
B	=	*Width of the peel arm*
ε	=	*Tensile strain*
θ	=	*Peel angle*
h_S	=	*Thickness of peel arm*
σ	=	*Tensile stress*
a	=	*Crack length*
U_{db}	=	*Energy dissipated during bending of the peel arm*

For the T Peel, adhesive fracture energy, G_C can be measured using the following equations (Lamut et al. 2008).

Equation 2.4

$$G_c = G_{c(Peel\ Arm\ 1)} + G_{c(Peel\ Arm\ 2)}$$

Equation 2.5

$$G_{c(Peel\ Arm\ 1)} = \frac{P}{B}(1 + \cos\theta) - G_{P(Peel\ Arm\ 1)}$$

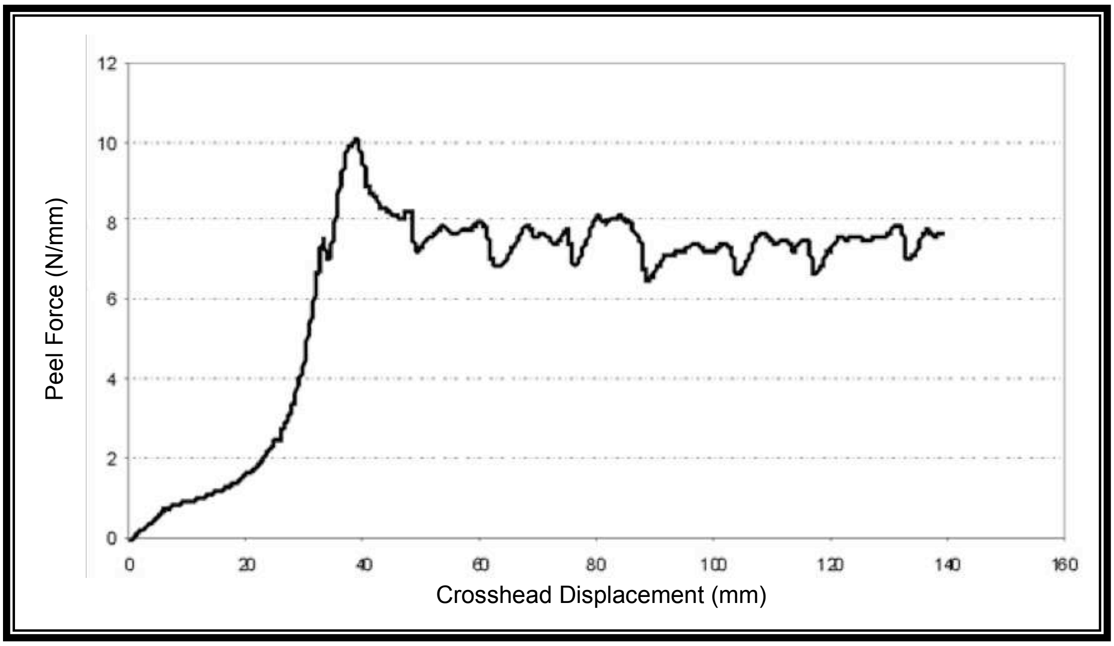
Equation 2.6

$$G_{c(Peel\ Arm\ 2)} = \frac{P}{B}(1 - \cos\theta) - G_{P(Peel\ Arm\ 2)}$$

where:

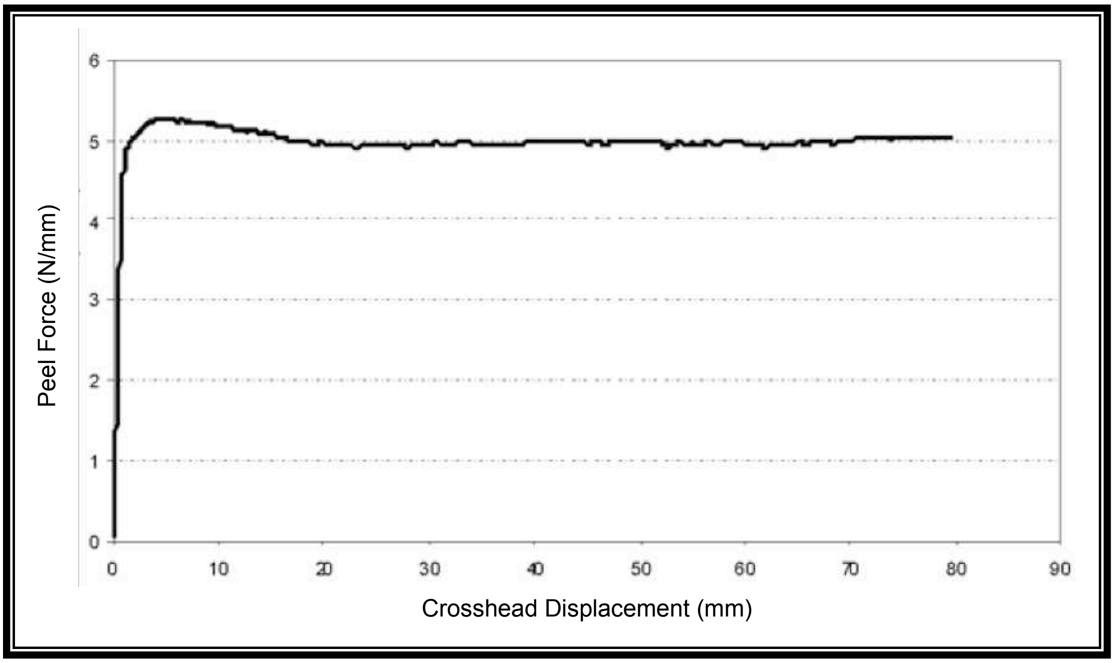
G_C	=	*Adhesive fracture energy*
G_P	=	*Plastic energy due to bending of the peel arm*
P	=	*Applied peel load*
B	=	*Width of the peel arm*
θ	=	*Peel angle*

Generally, the peel test will reach steady state conditions only after a considerable amount of displacement has been reached, and the steady state applied load is often many times larger than the required load for propagation of initial crack. Figure 2.11 shows the example of typical peel force versus crosshead displacement for T Peel test, which had used aluminium alloy as substrates and Bondmaster ESP110 epoxy as adhesive materials. Based on Figure 2.11, peel force was found to fluctuate significantly during the first 40 mm of crosshead displacement before reaches steady state conditions. However, based on Lamut et al. (2008), data of the test results of the same substrates and adhesive materials subjected to Fixed Arm test differs considerably from the data of the test results of T Peel test, as shown in Figure 2.12. Propagation values of peel force, P and calculated adhesive fracture energy, G_C for T Peel test and Fixed Arm test were 7.43 N/mm and 1370 J/m^2, and 5.00 N/mm and 922 J/m^2, respectively.



Notes: 1. Load is expressed as peel force in N per millimetre width
2. Substrates: Aluminium alloy
3. Adhesive Materials: Bondmaster ESP110 epoxy
4. Propagation Value of Peel Force, P is 7.43 N/mm
5. Propagation Value of Adhesive Fracture Energy, G_C is 1370 J/m^2

Figure 2.11 Typical peel force versus crosshead displacement for T Peel test (Source: Lamut et al. 2008)



Notes: 1. Load is expressed as peel force in N per millimetre width
2. Substrates: Aluminium alloy
3. Adhesive Materials: Bondmaster ESP110 epoxy
4. Propagation Value of Peel Force, P is 5.00 N/mm
5. Propagation Value of Adhesive Fracture Energy, G_C is 922 J/m^2

Figure 2.12 Typical peel force versus crosshead displacement for Fixed Arm test (Source: Lamut et al. 2008)

Results of the peel test usually reflect the stress and strain conditions of the bonded joints or laminates failing under conditions of extensive yielding and involve a large degree of plastic deformation of the peel arms (Cui et al. 2003). Based on Kinloch (1987), due to the effect of the applied peel load that is tend to place a very high stress and strain concentrations on the boundary line of the crack front, crack will occur easily unless the bonded joints or laminates are wide or the applied peel load is low. According to Kinloch (1997), the peel test does not measure the fundamental aspect of adhesion (i.e. intrinsic adhesion) between adhesive layer and substrates, even when the failure occurs along the interfaces of the bonded joints or laminates. Nor does the peel test directly assess the adhesive strength or toughness of the adhesive materials. The reason for this is due to the complex deformation behaviour of the peel test even though the testing techniques and procedures of the peel test can be considered as one of the simplest.

Several studies have shown that the measured peel load per unit width of the peel test does not only depends upon the degree of intrinsic adhesion and the type of adhesive materials, but also upon various factors such as peel angle, thickness and mechanical properties of the peel arms. Figure 2.13 shows the effect of the peel angle on the adhesive fracture energy, G_C for the Fixed Arm based on the studies conducted by three different laboratories. If the peel test is going to be used in pavement related areas for measuring the adhesive bond strength between bitumen (bitumen-filler mastic) and aggregates, due to the viscoelastic properties of the bitumen (bitumen-filler mastic), the measured peel load per unit width of the peel test will also depend upon the deformation rates and test temperatures (Kinloch 1997). Fracture-mechanics method using energy balance approach has been used as replacement for the Elastic-

Plastic-Fracture-Mechanics (EPFM) approach in analysing the failure of the bonded joints and laminates of composite materials.

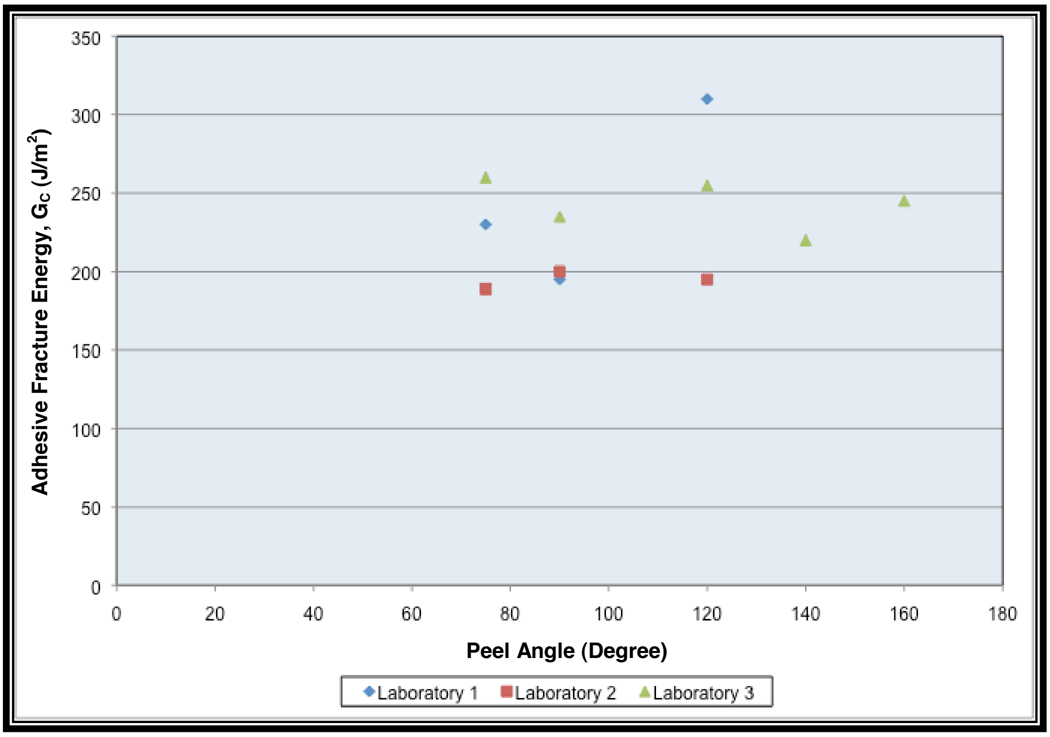


Figure 2.13 Effect of peel angle on adhesive fracture energy, G_C for the Fixed Arm test (Source: Moore & Williams 2001)

2.3.1.2 Pull Off Test

Pull off test is widely used for measuring the mechanical tensile strength of paint films, varnishes, concretes and other coatings. ASTM D4541 Standard Test Method for Pull-Off Strength of Coatings Using Portable Adhesion Testers and BS EN ISO 4624:2003 Paints and Varnishes-Pull-Off Test for Adhesion define the testing techniques and procedures for carrying out the pull off test for paint films, varnishes and other coatings. BS EN 1542:1999 Products and Systems for the Protection and Repair of Concrete Structures-Test Methods-Measurement of Bond Strength by Pull-Off specifies a method for measuring the tensile strength of grouts, mortars, concretes and surface protection system (SPS) used for the protection and repair of concretes using the pull off test.

Generally, pull off test is conducted by measuring the minimum tensile stress necessary to detach or fracture the coatings of adhesive materials in a direction perpendicular to the substrate(s). However, based on DFD® Instruments (n.d.), the word perpendicular does not have a proper meaning when testing on a curved surface of the substrate(s) and is usually used to refer to the evenly distributed tensile stress. There are various testing techniques and procedures used to conduct the pull off test. The most commonly used pull off test is conducted by inserting or casting thin films of uniform thickness of adhesive materials between two plates of rigid substrates (infinite rigid plane and rigid disc), as shown in Figure 2.14. The bonded assemblies are then subjected to increasing tensile stress until failure or fracture occurs. The failure mechanisms could occur due to either adhesive failure along the interfaces or cohesive failure through the layer of the adhesive materials, or combination of both.

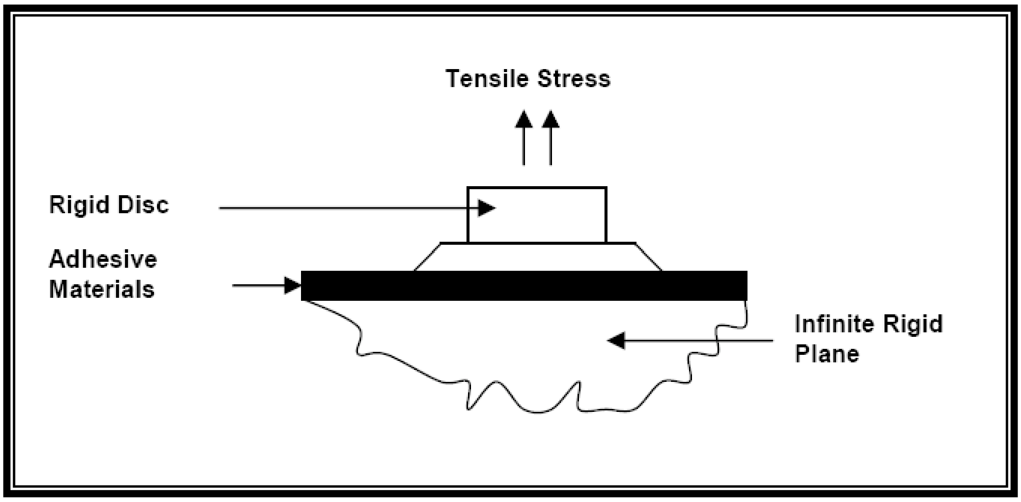


Figure 2.14 Pull off test specimen

Based on the pull off test developed by DFD® Instruments (n.d.) which is based on the ASTM D4541 Standard Test Method for Pull-Off Strength of Coatings Using Portable Adhesion Testers, as shown in Figure 2.15, the applied tensile stress must be steadily increased within the specified rate intervals and also must be applied in the perpendicular direction to the adhesive materials so that the applied tensile stress will be evenly distributed throughout the coated surfaces. Otherwise, the area where the applied tensile stress is concentrated will fracture before the maximum tensile stress has been reached elsewhere (DFD® Instruments n.d.).

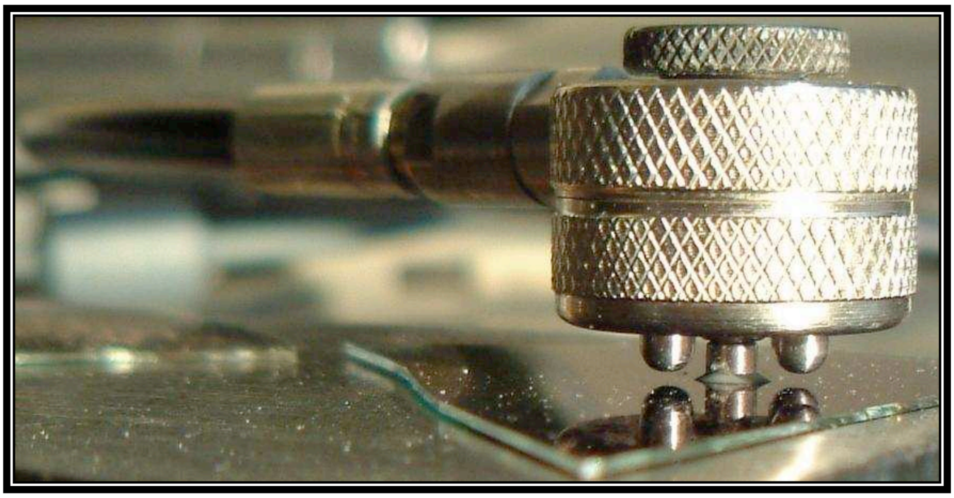


Figure 2.15 Pull off test developed by DFD® Instruments (Source: DFD® Instruments n.d.)

According to the BS EN ISO 4624:2003 Paints and Varnishes-Pull-Off Test for Adhesion, the minimum tensile stress required to detach or fracture the coatings of the adhesive materials can be calculated as in Equation 2.7 and the types of the failure which is determined via visual observation can be tabulated as in Table 2.2.

Equation 2.7

$$\sigma = \frac{F}{A}$$

where:

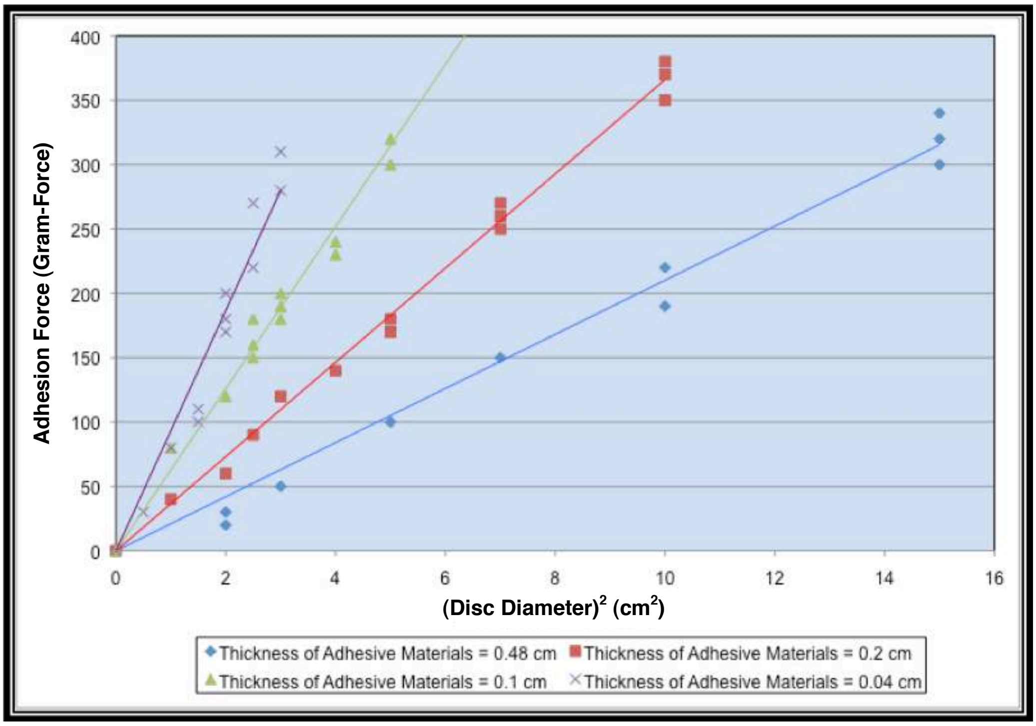
σ	=	*Minimum tensile stress (MPa)*
F	=	*Minimum tensile load (N)*
A	=	*Area of contact (mm^2)*

Table 2.2 Template for calculating the types of failure via visual observation

Abbreviation for Types of Failure	Descriptions of Types of Failure	Percentage Area of Failure (%)[1]
A	Cohesive failure within adhesive	A%
A/S_1	Adhesive failure between adhesive and substrates 1	B%
A/S_2	Adhesive failure between adhesive and substrates 2	C%
Total	Sum of All Types of Failure	A + B + C = 100%

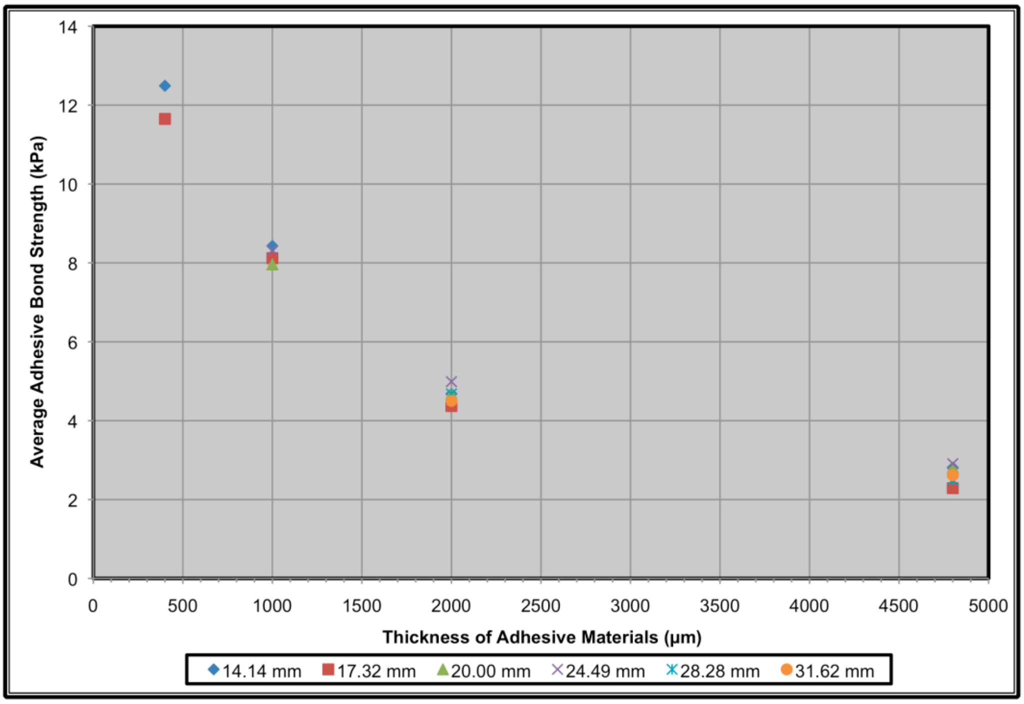
Notes: [1]Percentage area of failure is estimated to the nearest 10% for each types of failure

Results from the pull off test are influenced not only by the properties of the adhesive materials and substrate(s), but also by nature and preparation of the substrates, methods of application of the adhesive materials, temperature, humidity and types of the testing equipments being used (British Standard Institution 2003). Kendall (1971) has conducted a study in order to determine the effect of area of contact and thickness of adhesive materials on the adhesive bond strength based on the pull off test. Gelatine of thickness between 0.04 cm and 0.48 cm was used as elastic adhesive materials and Perspex was used as substrates. Figure 2.16 shows the relationship between adhesion force (measured in unit of Gram-Force) and disc diameter (measured in unit of cm^2) over wide ranges of thicknesses of adhesive materials. Based on the Figure 2.16, disc diameter, which represents the area of contact, and thickness of adhesive materials were found to have a profound influence on the adhesion force. The value of the adhesion force tends to increase with the increasing value of disc diameter and decreasing thickness of adhesive materials. However, by re-plotting the data of Kendall (1971) in the forms of adhesive bond strength against thickness of adhesive materials as shown in Figure 2.17, it was found that the effect of disc diameter and hence the effect of area of contact are almost negligible. Specimens of different disc diameter of 14.14 mm, 17.32 mm, 20.00 mm, 24.49 mm, 28.28 mm and 31.62 mm have shown to result in approximately the same value of adhesive bond strength, provided having the same thickness of adhesive materials. Hence, it can be concluded that the adhesive bond strength is mainly influenced by the thickness of adhesive materials rather than the disc diameter or area of contact.



Notes:
1. 1 Gram-Force = 9.80665 mN
2. Substrates: Perspex
3. Adhesive Materials: Gelatine
4. Conditioning Procedures: Dry conditioning at room temperature
5. Testing Conditions: Deformation rate of 0.6 mm/minute at room temperature

Figure 2.16 Relationship between adhesion force, disc diameter and thickness of adhesive materials (Source: Kendall 1971)



Notes: 1. Substrates: Perspex
2. Adhesive Materials: Gelatine
3. Conditioning Procedures: Dry conditioning at room temperature
4. Testing Conditions: Deformation rate of 0.6 mm/minute at room temperature

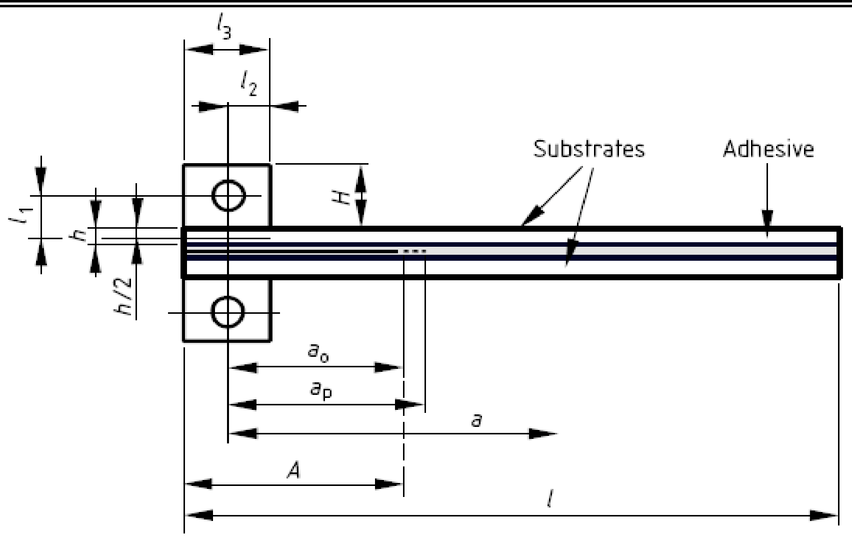
Figure 2.17 Relationship between average adhesive bond strength, thickness of adhesive materials and disc diameter

One of the major problems in designing the adhesion test methods based on the pull off (tension) mode is that unless one of the substrates is highly compliance, then even a small misalignment of the substrates will result in cleavage stresses (Kendall 1971). However, based on the past studies, pull off test has been found useful in comparing the adhesive properties and also providing relative ratings of different types of adhesive materials. Pull off test may be applied using wide ranges of substrates including metals, plastics, woods, concretes and aggregates.

2.3.1.3 Double Cantilever Beam (DCB) Test

Double cantilever beam (DCB) test as shown in Figure 2.18 is based on the Linear-Elastic-Fracture-Mechanics (LEFM) approach and published as ASTM D3433-99 Standard Test Method for Fracture Strength in Cleavage of

Adhesives in Bonded Metal Joints and BS 7991:2001 Determination of the Mode I Adhesive Fracture Energy G_{IC} of Structure Adhesives Using the Double Cantilever Beam (DCB) and Tapered Double Cantilever Beam (TDCB) Specimens. DCB test is widely used for the determination of the fracture resistance of the adhesive and bonded joints of composite materials under mode I tensile loading conditions. Mode I is an opening of the fracture corresponding to the tensile loading, which is in the direction normal to the fracture plane, as shown in Figure 2.19. Other modes of failure (i.e. Mode II and Mode III) correspond to the sliding or in-plane shear mode where the fracture surfaces slide over one another in the direction normal to the crack front, and tearing or anti plane shear mode respectively.



Note:
- A = Distance between the end of the specimen and the tip of the insert film
- a = Crack length distance between the load lone and the tip of the pre-crack
- a_O = Distance between the load line to the tip of the insert film
- a_P = Pre-crack length measured from the load line to the tip of the mode I pre-crack
- B = Width of specimen
- H = Thickness of the load-block
- h = Thickness of substrate beam at a crack length, a
- l = Total length of the specimen
- l_1 = Distance from the centre of the loading pin to the mid-plane of the arm of the substrate beam
- l_2 = Distance from the loading pin centre to the edge of the block
- l_3 = Total length of the load-block

Figure 2.18 Double cantilever beam (DCB) specimen (Source: British Standards Institution 2001)

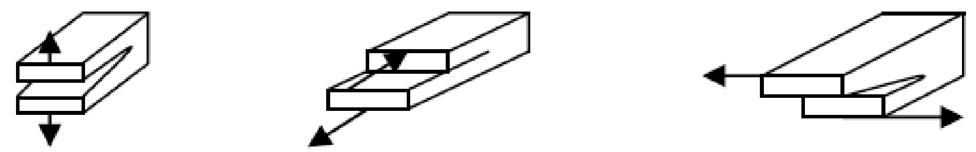


Figure 2.19 Mode of failure (Mode I, Mode II and Mode III)

DCB test measures the mode I adhesive fracture energy, G_C which can be described as the measure of the adhesive strength or toughness of the adhesive materials in the presence of flaws (British Standards Institution 2001). DCB test is well suited for testing adhesive and bonded joints of thin adhesively bonded fibre composite materials, but may also be used when the metallic substrates, which possess a relatively high yield stress, are employed.

DCB test is a testing geometry whereby adhesive materials are applied between two identical substrates, often steel. A crack is initiated first by inserting a wedge into the adhesive materials. This initiates a crack formation in a predefined position of the adhesive materials. The specimen of the DCB test is then loaded by pulling apart the two beams at a certain rate resulting in an increase in the deflection of the two beams as the load increases (Varun 1999). At a certain critical load, the crack begins to propagate resulting in a slight drop in the applied load. At this point, the beams are stopped from moving apart, thus keeping the deflection constant. The slight drop in the applied load and the crack length are carefully followed. The DCB specimen is then consecutively unloaded and then loaded, and the overall procedures are repeated several times leading to the total cleavage of the DCB specimen. Results of the DCB test are collected at various times and consist of load, deflection, crack length and compliance. The obtained results can then be analysed using several different approaches such as Corrected Beam Theory

(CBT), Experimental Compliance Method (ECM) and ASTM Method (British Standard Institution 2001; Varun 1999).

Figures 2.20 and 2.21 show the example of the results of the DCB test using titanium alloys as substrates and polyimide as adhesive materials, as being conducted by Varun (1999). Figure 2.20 shows the captured data of the load versus displacement, and Figure 2.21 shows the results, which have been analysed using different approaches (i.e. Experimental Compliance Method (ECM) and ASTM Method). The value of the maximum strain energy release rate, G was found to be approximately the same although was analysed using different approaches, and the value was in the range of 1610 J/m^2 and 1700 J/m^2 for Sample A, and 1900 J/m^2 and 2100 J/m^2 for Sample B, respectively.

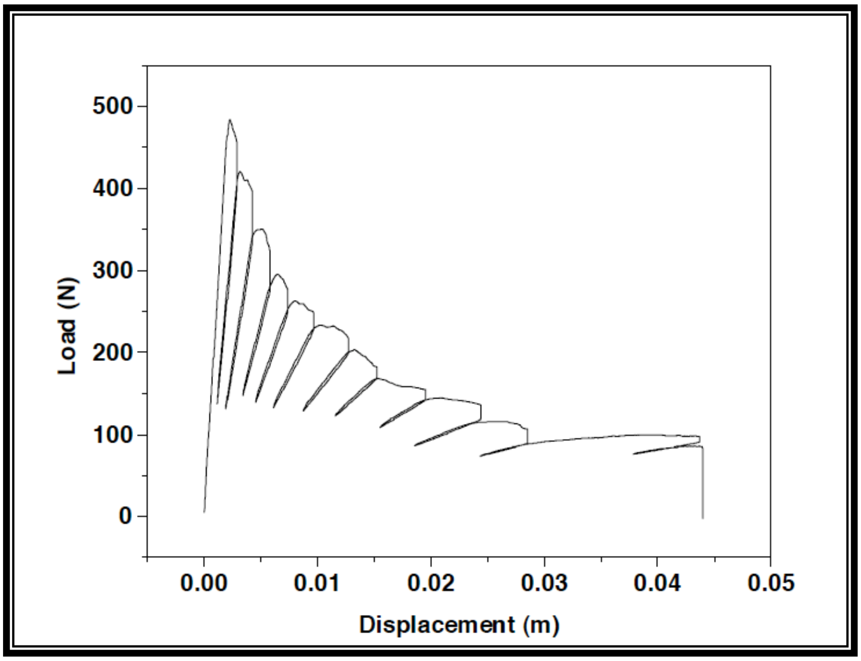


Figure 2.20 Load versus displacement for DCB test using titanium alloys as substrates and polyimide as adhesive materials (Source: Varun 1999)

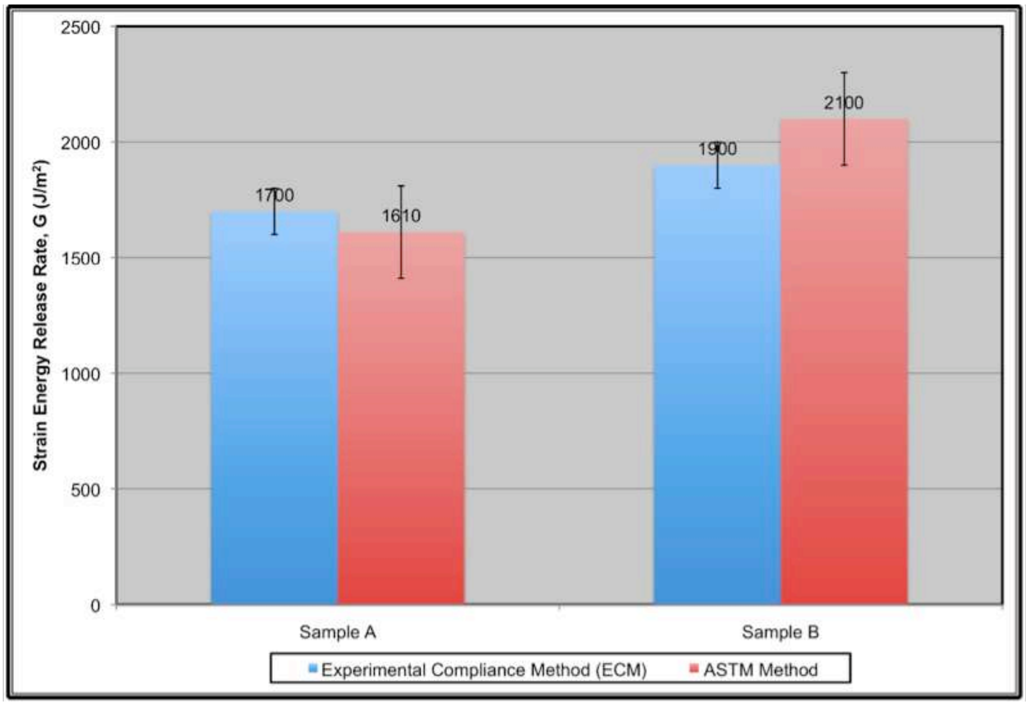
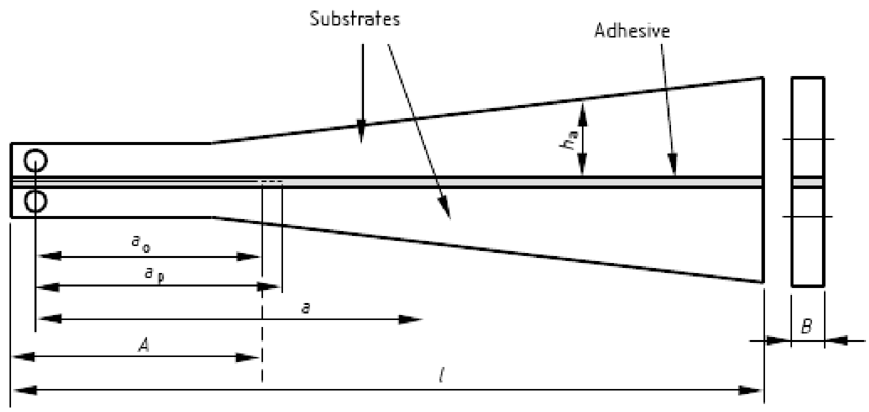


Figure 2.21 Results (strain energy release rate) based on Experimental Compliance Method (ECM) and ASTM Method (Source: Varun 1999)

2.3.1.4 Tapered Double Cantilever Beam (TDCB) Test

Tapered double cantilever beam (TDCB) test as shown in Figure 2.22 was developed by Mostovoy and Ripling (1966). BS 7991:2001 Determination of the Mode I Adhesive Fracture Energy G_{IC} of Structure Adhesives Using the Double Cantilever Beam (DCB) and Tapered Double Cantilever Beam (TDCB) Specimens provides a method based upon Linear-Elastic-Fracture-Mechanics (LEFM) for the determination of the fracture resistance of the adhesive and bonded joints of composite materials under mode I tensile loading conditions, using the TDCB test. The TDCB test is similar to the DCB test, which consists of layer of adhesive materials in between two identical substrates. The two identical substrates however are tapered away from the point where the load is applied. The aim of tapering the substrates is to mitigate the errors in the testing procedures due to inelastic or plastic deformation of the substrates. TDCB test is designed so that, over a large range of crack length values, the rate of change of compliance with the crack length is constant. Thus, this will result in value of adhesive fracture energy, G_C that is independent of the crack length values at any given applied load. Here lies the main advantage of the TDCB test, namely the value of the adhesive fracture energy, G_C may be readily calculated without the knowledge of the crack length and this is particularly useful since the crack tip is often difficult to be defined accurately in the adhesive and bonded joints of composite materials (Kinloch 1982). Also, relatively tough adhesive materials can be tested without the occurrence of plastic deformation of the arms, and the substrates can possess a relatively low yield stress, but again no plastic deformation of the arms is incurred during the test (British Standard Institution 2001). In contrast, DCB test is well suited for testing adhesive and bonded joints of thin adhesively bonded fibre composite materials. Table 2.3 shows the equations used to calculate the adhesive fracture energy, G_C using several different approaches (i.e. Simple Beam Theory (SBT), Corrected Beam Theory (CBT) and Experimental Compliance Method (ECM)) for both DCB test and TDCB test (British Standard Institution 2001).



Note:
A = Distance between the end of the specimen and the tip of the insert film
a = Crack length distance between the load lone and the tip of the pre-crack
a_O = Distance between the load line to the tip of the insert film
a_P = Pre-crack length measured from the load line to the tip of the mode I pre-crack
B = Width of specimen
h_a = Thickness of adhesive layer
l = Total length of the specimen

Figure 2.22 Tapered double cantilever beam (TDCB) specimen (Source: British Standard Institution 2001)

Table 2.3 Equation for adhesive fracture energy, G_C based on Simple Beam Theory (SBT), Corrected Beam Theory (CBT) and Experimental Compliance Method (ECM) (Source: British Standard Institution 2001)

	Types of Approaches		
	Simple Beam Theory (SBT)	Corrected Beam Theory (CBT)	Experimental Compliance Method (ECM)
Double Cantilever Beam (DCB) Test	$G_C = \frac{4P^2}{E_S B^2} \cdot m$	$G_C = \frac{3P\delta}{2B(a+\|\Delta\|)} \cdot F$	$G_C = \frac{nP\delta}{2Ba} \cdot F$
Tapered Double Cantilever Beam (TDCB) Test	$G_C = \frac{4P^2}{E_S B^2} \cdot m$	$G_C = \frac{4P^2 m}{E_S B^2}\left[1 + 0.43\left(\frac{3}{ma}\right)^{\frac{1}{3}}\right]$	$G_C = \frac{P^2 m}{2B} \cdot \frac{dC}{da}$

where:

G_C = *Adhesive fracture energy*
P = *Measure Load*
E_S = *Independently measured flexural or tensile modulus of substrates*
B = *Width of the peel arm*
m = *Specimen geometry factor and is given by* $\frac{3a^2}{h^3} + \frac{1}{h}$
h = *Thickness of substrates at crack length,* a
δ = *Displacement*
a = *Crack length*
Δ = *Crack length correction for a beam that is not perfectly built in*
F = *Large displacement correction*
n = *Slope of a plot of* $\log_{10} C$ *versus* $\log_{10} a$
C = *Compliance and is given by* δ/P

TDCB test can be used to determine the rate of crack growth under various cyclic loading and environmental conditions. Compared to the other adhesion test methods, TDCB test is relatively complex and expensive in terms of the specimen preparation. Thus, various engineering applications would far prefer to deduce the value of the adhesive fracture energy, G_C from the most common and widely used peel test (Hadavinia et al. 2006).

Cui et al. (2010) has conducted a study to determine the fracture resistance of the bitumen as adhesive materials and aluminium alloy as substrates using the TDCB test, as shown in Figure 2.23. Results of the study over various thicknesses of the adhesive layer of bitumen and wide ranges of deformation rates and test temperatures have shown promising potential for the TDCB test to be used in measuring the adhesive bond strength of asphalt mixtures (Figures 2.24 and 2.25).

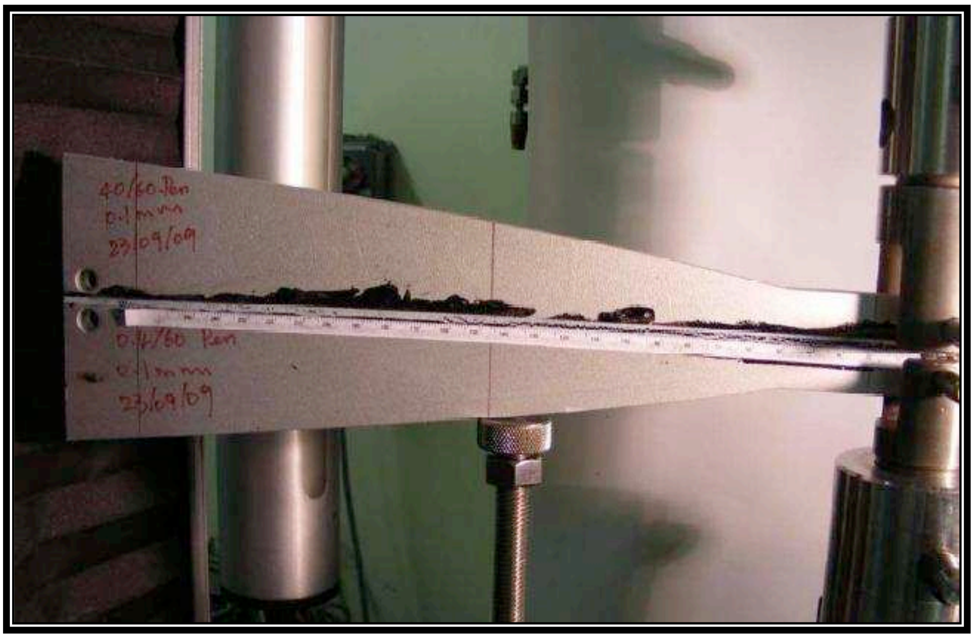


Figure 2.23 TDCB test using bitumen as adhesive materials and aluminium alloy as substrates (Source: Cui et al. 2010)

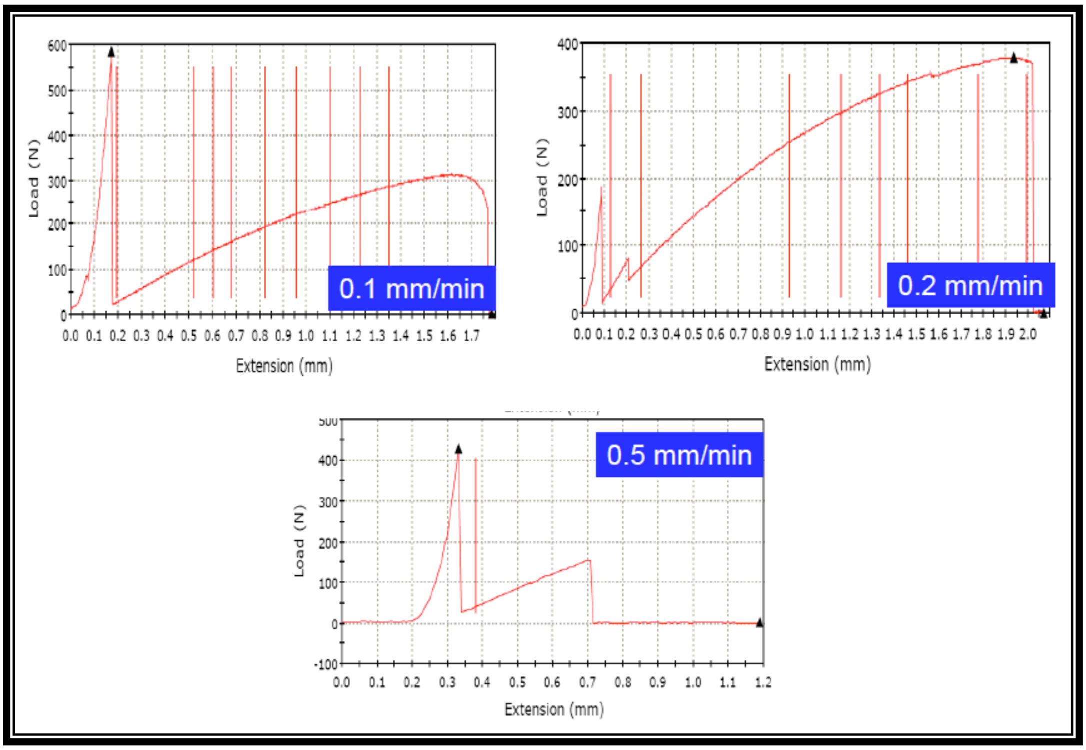


Figure 2.24 Effect of deformation rate on tensile load (Source: Cui et al. 2010)

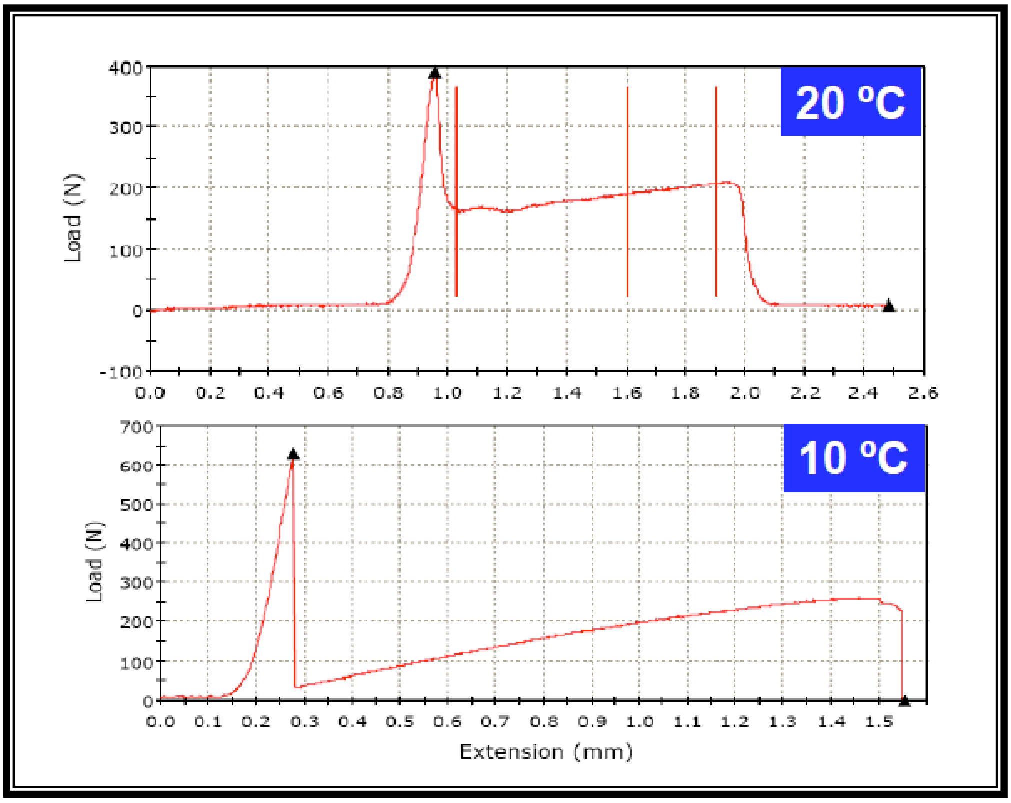


Figure 2.25 Effect of temperature on tensile load (Source: Cui et al. 2010)

2.3.1.5 Impact Wedge Peel (IWP) Test

Impact wedge peel (IWP) test is a standard method for measuring the resistance of structural adhesives to cleavage fracture at a relatively high deformation rate and has been published as ISO 1143:2003 Adhesives-Determination of Dynamic Resistance to Cleavage of High-Strength Adhesive Bonds Under Impact Conditions-Wedge Impact Method (Blackman et al. 2000). Schematic drawing of the IWP test in accordance with the ISO 1143:2003 is shown in Figure 2.26.

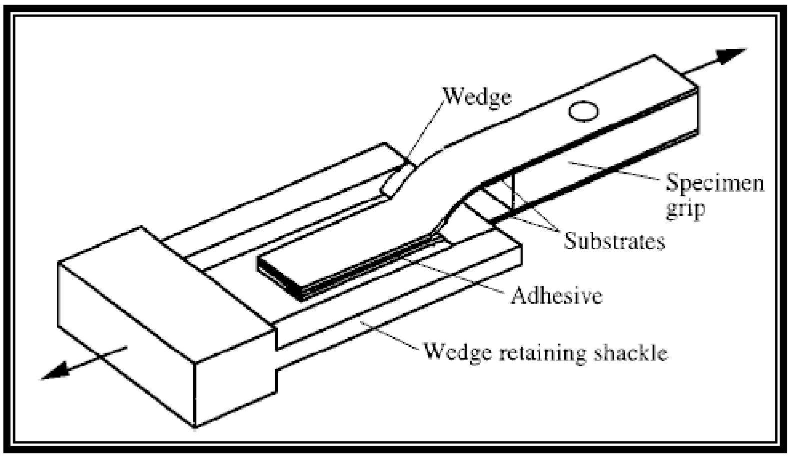


Figure 2.26 Impact wedge peel (IWP) test specimen (Source: Blackman et al. 2000)

The IWP test is a follow-up study and an improved method based on the most common and widely used peel test (Kinloch 1997). In the IWP test, two preformed metal substrates are bonded over a length of 30 mm only at one end to give a tuning fork joint. The free arms of the specimen are clamped and a wedge is driven through the bonded joints of the specimen, as illustrated in Figure 2.26. Based on the ISO 1143:2003, the IWP test specimen should be 90 mm long and 20 mm wide, and made using sheet-metal substrates of between 0.6 mm and 1.7 mm of thickness. The velocity used to drive the wedge is 2 m/s for steel substrates and 3 m/s for aluminium-alloy substrates.

Based on the ISO 1143:2003, the average impact force is calculated from the plot of the measured impact force versus time, disregarding the first 25% and the last 10% of the curve, as shown in Figure 2.27 (Taylor 1996). The energy needed to rupture the IWP test specimen is calculated based on the integration over the same part of the curve (i.e. calculating the area under the curve), and multiply the results of the integration with velocity.

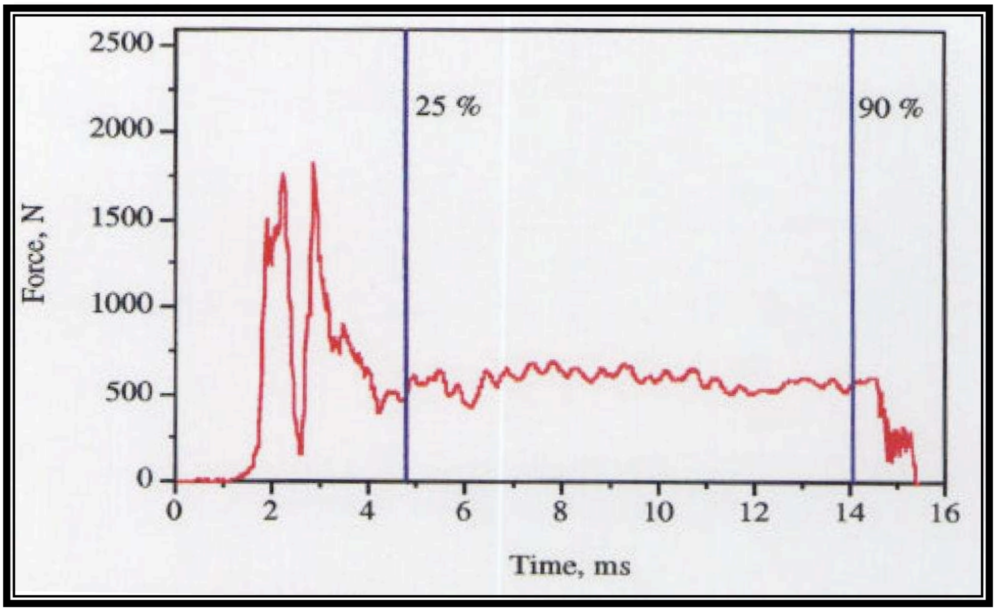


Figure 2.27 Plot to calculate average impact force and energy needed to rupture the IWP test specimen (Source: Taylor 1996)

The value of the average impact force depends on the types of adhesive and substrates. Same as the peel test, plastic deformation of the substrates of the IWP test specimen greatly contributes to the measured impact force and energy needed to cause rupture. Furthermore, in the IWP test the frictional energy losses may also be significant. Hence, the IWP test would not be expected to give a measure of the impact properties of the adhesive materials (Kinloch 1997).

Kinloch et al. (1996) has proved the existence of direct correlations between the results obtained from the IWP test and the values of the adhesive fracture energy, G_C measured using the TDCB test, as shown in Figure 2.28. Based on the Figure 2.28, the correlations between the results obtained from the IWP test and the values of the adhesive fracture energy, G_C measured using the TDCB test demonstrate that the degree of plastic deformation of the substrates increases as the values of the adhesive fracture energy, G_C increases, and this is more pronounced when the mild steel substrates are employed. For the IWP test to be used in measuring the adhesive bond strength of bitumen (bitumen-filler mastic) and aggregates, the substitution of metal substrates used in the IWP test with the aggregates substrates may be very challenging due to the nature of the adhesive and bonded joints and failure mechanisms. Also, the application of the wedge in inducing crack initiation and propagation are not representative of the moisture damage mechanisms of adhesive or cohesive failure.

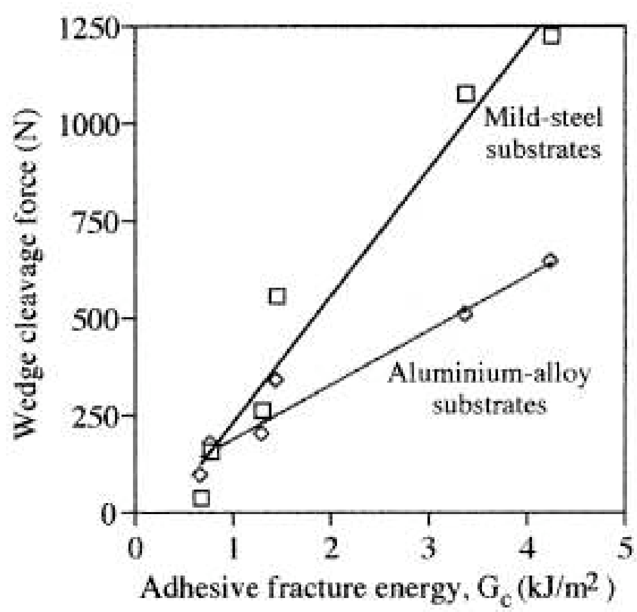


Figure 2.28 Correlations between results obtained from IWP test and values of adhesive fracture energy, G_C measured using TDCB test (Source: Kinloch 1997)

2.3.1.6 Scratching of Thin Films Test

Scratching of thin films test is used by Heavens (1950) in the study related to the adhesion of evaporated coatings. Scratching of thin films test uses a spherically tipped stylus, which is loaded onto thin films and then dragged along the surface as shown in Figure 2.29. At normal loads, shearing occurs in the thin films. The value of load required to induce shearing is calculated based on Equation 2.8, and is expressed in terms of the traction force, F^2. As the load is increased, a point is reached where the thin films are completely removed from the substrates. The load at which the removal of the thin films occurs is taken as the measure of the adhesive bond strength of the thin films and substrates, and can be calculated based on Equation 2.9. Figure 2.30 shows the relationship between traction forces, F^2 that are represented by Equations 2.8 and 2.9, and applied load. As applied load is increased, failure is expected to change from shearing to peeling. Based on Figure 2.30, in the area of shearing, the minimum traction force, F^2 required to produce failure is based on Equation 2.8, and vice versa for the area of peeling.

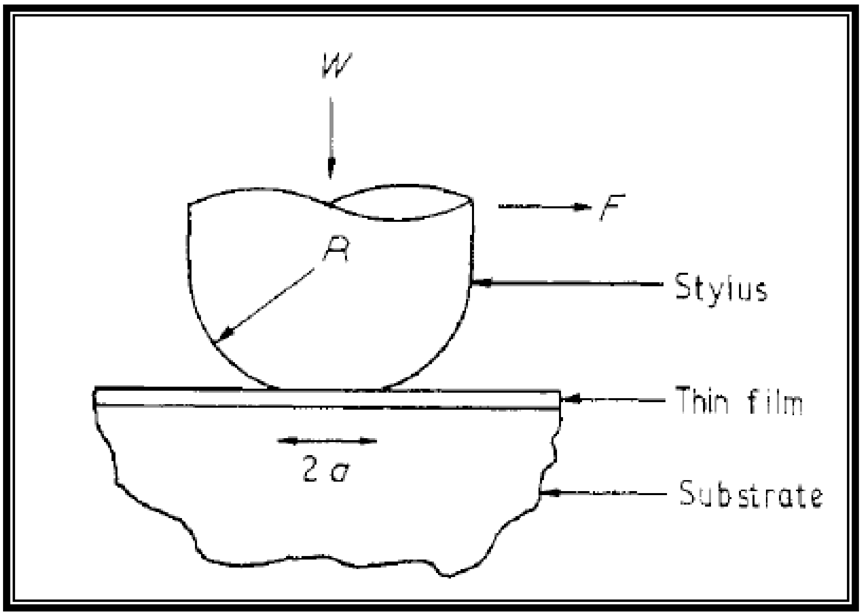


Figure 2.29 Scratching of thin films test specimen (Source: Kendall 1971)

Equation 2.8

$$F^2 \cong \pi^2 S^2 \left(\frac{3}{4}\frac{RW}{G}\right)^{\frac{4}{3}}$$

where:

F^2	=	*Traction force*
S	=	*Shear strength of thin films*
R	=	*Radius of spherically tipped stylus*
W	=	*Applied load*
G	=	*Modulus of spherically tipped stylus and substrate*

Equation 2.9

$$F^2 \cong 6\pi\gamma RW$$

where:

F^2	=	*Traction force*
γ	=	*Interfacial surface energy (i.e. energy or amount of external work needed to create a unit area of new surface between the thin films and substrates)*
R	=	*Radius of spherically tipped stylus*
W	=	*Applied load*

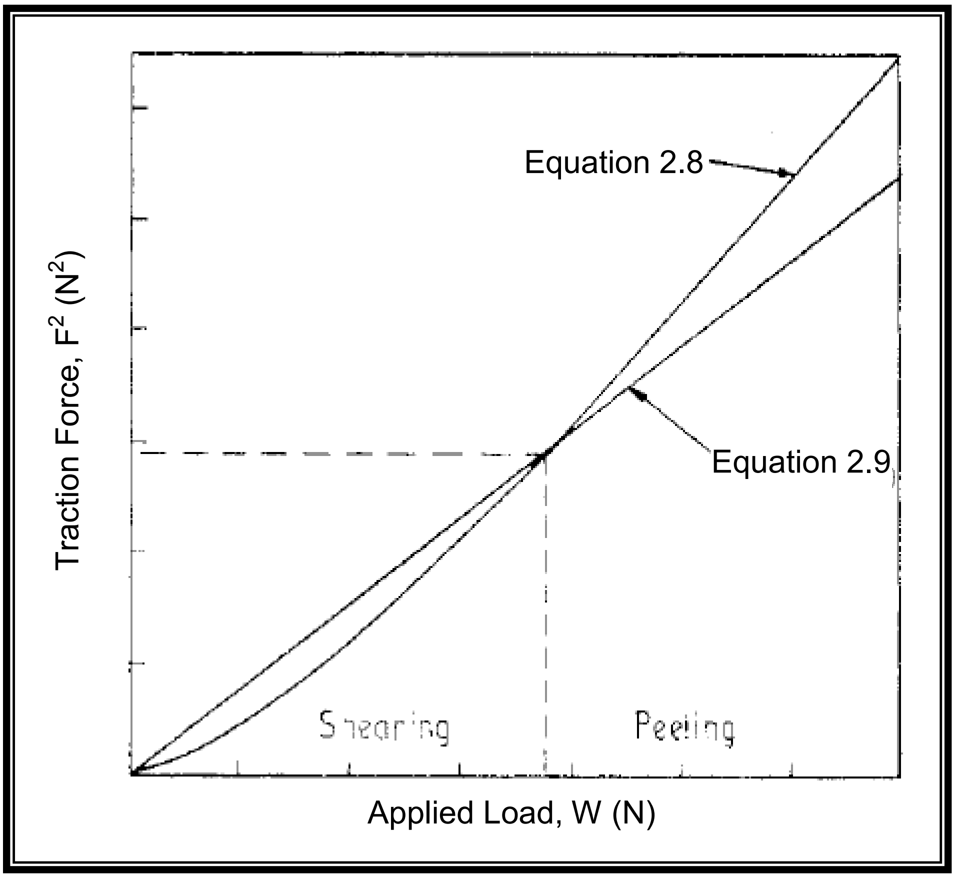


Figure 2.30 Relationship between traction forces, F^2 represented by Equations 2.8 and 2.9, and applied load (Source: Kendall 1971)

However, the behaviour of the scratching of the thin films test can only be explained qualitatively by using the following assumption; only elastic deformation takes place in the spherically tipped stylus and substrates, and the spherically tipped stylus and substrates are assumed as having equal elastic properties. Plastic and viscoelastic deformation in the spherically tipped stylus and the substrates have been neglected.

2.3.2 Adhesion Test Methods Performed on Asphalt Mixtures

In the pavement related areas, there are only a few testing techniques and procedures known to be used for measuring the adhesive bond strength of asphalt mixtures and most of the testing techniques and procedures are used to measure the adhesive bond strength of tack coat, either in the laboratory or in the field. Testing techniques and procedures used to measure the adhesive bond strength of tack coat that can be considered as one of the possible approaches for adoption and applications on the bitumen (bitumen-filler mastic) and aggregates is the University of Texas at El Paso (UTEP) pull off device. The blister test is one of the adhesion test methods that had been considered practical for quantitative evaluation of the adhesive bond strength of bitumen and aggregates (Anderson et al. 1994; Chang 1994). During the Strategic Highway Research Program (SHRP), the blister test that is widely used for determination of the adhesive strength of bonded joints and laminates of various composite materials has been modified for quantitative evaluation of the adhesive bond strength of bitumen and aggregates. However, the complexity of the modified adhesive blister test put a quick end to the development.

In the past few years, there have been some efforts by researchers in developing testing techniques and procedures that can be used to directly measure the adhesive bond strength between bitumen and aggregates, such as published by Copeland (2007), Kanitpong and Bahia (2003), Kanitpong and Bahia (2004) and Kanitpong and Bahia (2005). However, the developed testing techniques and procedures have not enjoyed universal success and acceptance, and not yet established due to poor repeatability of the test results, and limitations in terms of the applicability to measure the adhesive bond strength for wide ranges of asphalt mixture materials under various testing conditions (various conditioning procedures (dry and wet conditionings), deformation rates and test temperatures).

2.3.2.1 University of Texas at El Paso (UTEP) Pull Off Device

University of Texas at El Paso (UTEP) pull off device has been fabricated at the University of Texas at El Paso based on the pull off mechanisms and is used to measure the quality of tack coat in pull off (tension) mode. Tack coat is a thin bituminous layer applied between the existing pavements and the newly constructed pavements in order to promote bonding. Various laboratory testing techniques and procedures are available to identify the quality of tack coat in the laboratory such as published in the ASTM D244-00 Emulsified Asphalts (Viscosity, Sieve Test, Particle Charge Test, Cement Mixing Test, 5-day Settlement Test, Residue). However, there is no testing techniques and procedures that are reliable for quantifying the quality of the applied tack coat in the field and for validation of whether the quality of the tack coat applied in the field met the required standard of the predetermined quality of the tack coat in the laboratory (Tandon & Puentes 2006).

The UTEP pull off device is simple, economical, easy to use and able to determine the quality of tack coat either in the laboratory or in the field within a short duration (Tashman et al. 2006). The UTEP pull off device weighs about 10.4 kg and can be easily levelled with the help of pivoting feet, as shown in Figure 2.31. The UTEP pull off device has a weight key on the top, which provides stability when loads are placed on the top. A 0 Nm to 18 Nm of torque wrench is used to pull the contact plate upward from the tack coated surface until the contact plate separates from the surface. The UTEP pull off device consists of 127 mm diameter of aluminium contact plate. With the UTEP pull off device firmly seated on the tack coated surface, loads of 18 kg are placed on the load rack for 10 minutes. After 10 minutes, the loads are removed and the torque wrench is rotated to detach the aluminium contact plate from the tack coated surface. The torque required to detach the aluminium contact plate is recorded and then converted to stress using calibration factor.

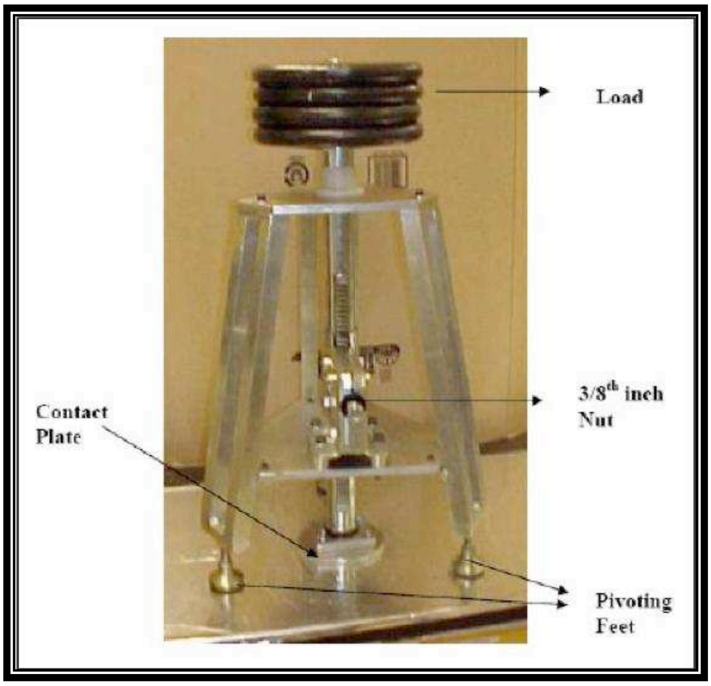


Figure 2.31 UTEP pull off device (Source: Tandon & Puentes 2006)

Typically the strength of the bitumen decreases with the increasing temperature due to its viscoelastic properties. However, based on the study conducted by Tandon and Puentes (2006), the results obtained from the UTEP pull off device indicated the opposite trends. Based on the results, it was found that the strength of the tack coated surface increases with increasing temperature, which can be considered as unusual. The reason for this is due to the area of coverage of the tack coated surface, which was less at lower temperature due to the lower flow ability (higher viscosity), as shown in Figure 2.32.

Figure 2.32 Area of coverage of tack coated surface at low temperature (Source: Tandon & Puentes 2006)

Tandon and Puentes (2006) concluded that the UTEP pull off device has the potential of identifying the quality of tack coated surface. The pull off (tension) mode of the UTEP pull off device is independent of the surface tested and the test setup is handy, reliable and can measure the quality of tack coat in less than 45 minutes after the application of the tack coat in the field. However, the direct adoption and applications of the UTEP pull off device for measuring the adhesive bond strength of bitumen (bitumen-filler mastic) and aggregates are likely to be complicated due to the different testing materials (i.e. tack coated surface and asphalt mixtures as a whole, and bitumen (bitumen-filler mastic) and aggregates). However, some modifications of the UTEP pull off device can result in a promising potential for the development of the laboratory adhesion test method for measuring the adhesive bond strength of bitumen (bitumen-filler mastic) and aggregates.

2.3.2.2 Blister Test

Blister test comprises of a rigid substrate with a hole drilled in the centre and then coated with layer of adhesive materials as shown in Figure 2.33. When pressure is applied through the hole drilled in the centre of the substrate, a blister will be formed. Radius of the formed blister is constant until a critical pressure is reached. At this point, radius of the formed blister increases in size, thus signifying an adhesive failure along the interfaces (Chang 1994). The applied pressure for the blister test can be provided in the form of either dry gas (such as dry nitrogen gas) or water. However, the applied pressure in the form of water was found to be an effective accelerated ageing method for the assessment of the durability of the adhesive and bonded joints and also effectiveness of the adhesive materials (Kinloch 1997). Blister test is well suited for adhesion test method of thin layer materials of adhesive, coatings and paint, especially for the materials, which cannot be readily gripped or if successfully gripped may fail cohesively during the test (Kinloch 1997).

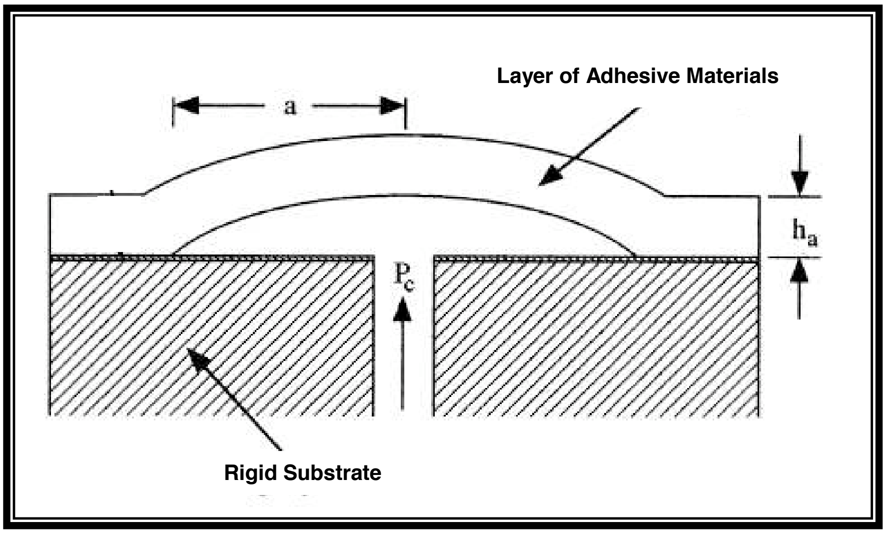


Figure 2.33 Blister test specimen (Source: Kinloch 1997)

In the pavement related areas, blister test has been identified as one of the possible approaches for quantitative evaluation of the adhesive bond strength of bitumen and aggregates in the presence of water or moisture (Anderson et al. 1994; Chang 1994). Based on the study conducted by Chang (1994), the modified adhesive blister test shown in Figure 2.34 was found to be the most appropriate testing technique and procedure for measuring the adhesive bond strength of bitumen and aggregates, either experimentally or analytically. The modified adhesive blister test provides a convenient way to study the effect of immersion of water or moisture, pH and salt concentration on the adhesive bond strength of the asphalt mixtures. The modified adhesive blister test, which has been developed by Chang (1994) uses aluminium, Teflon and aggregates as substrate materials in order to provide different surface characteristics, and also water as a medium for pressure. The modified adhesive blister test consists of coatings of bitumen over the flat substrate materials with a hole drilled though the substrate to permit the application of water pressure to the underside of the coatings. Adhesive bond strength is then measured by measuring the force required to displace the bitumen films from the surface of the substrate materials. Figure 2.35 shows the typical data for the blister test.

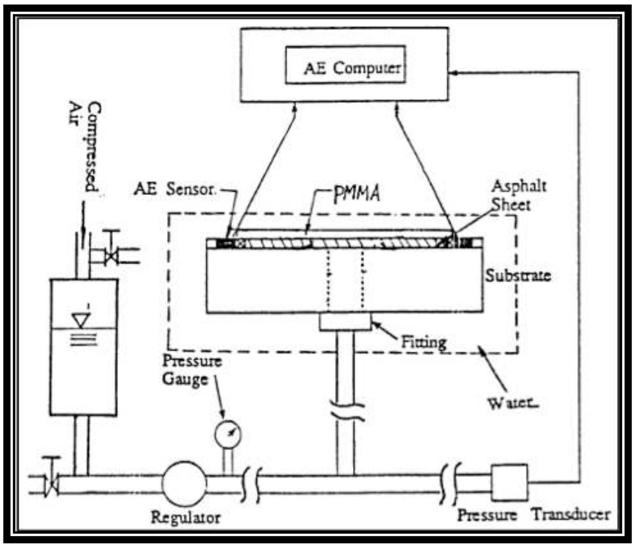


Figure 2.34 Modified adhesive blister test specimen (Source: Chang 1994)

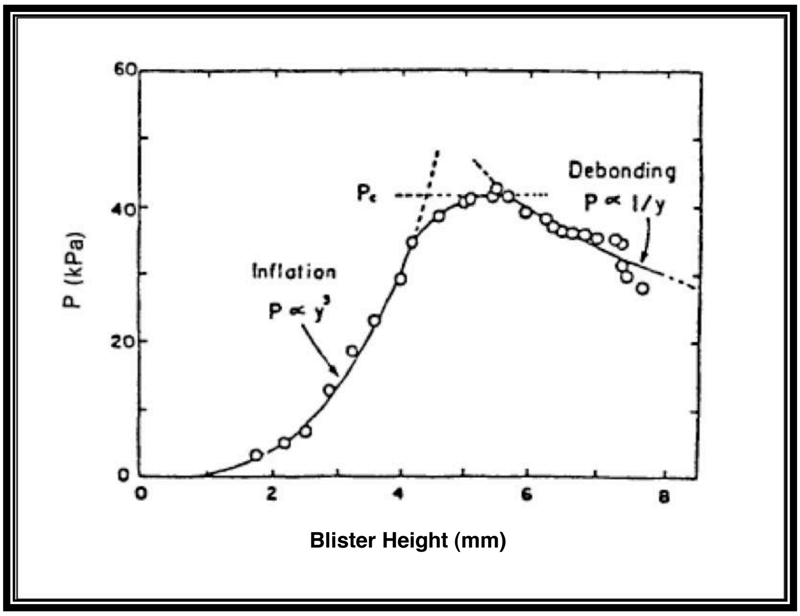


Figure 2.35 Typical data for the blister test (Source: Anderson et al. 1994)

However, after a series of testing for validation, the modified adhesive blister test was found to be very cumbersome and time consuming, and serious concern has been raised with respect to the representative nature of the substrate materials used in the test. A freshly sawn surface of the substrate materials is necessary in order to conduct the test and such surface is not representative of the actual aggregates surface used for pavement construction. Also, the mode of failure for the blister test was found to be cohesive regardless of the types of aggregates, as shown in Table 2.4 (Anderson et al. 1994). Based on the Table 2.4, a series of tests involving two types of aggregates (limestone and Texas chert) and one type of bitumen which have been vacuum saturated, followed by immersion in water for 30 minutes at 60°C (140°F) and then conditioned at either 0°C or 25°C for 18 hours prior to testing, does not shown any evidence of stripping. In the second conditioning procedures (i.e. vacuum saturated, followed by immersion in water for 30 minutes at 60°C (140°F), 18 hours of freeze and then conditioned at 0°C for 4 hours), adhesive failure was produced. However, the adhesive failure occurred for both types of the aggregates; the limestone and the Texas chert aggregates. Thus, it can be concluded that there was no significant difference in the behaviour of the two types of aggregates. Texas chert aggregates are commonly known for the reputation as notorious stripper in the presence of water or moisture while limestone is vice versa. Therefore, the conclusion has been made that the blister test did not differentiate between the two types of aggregates, which have radically different susceptibility to water or moisture. As a consequence, the modified adhesive blister test was not pursued as a standard testing techniques and procedures for measuring the adhesive bond strength of bitumen and aggregates (Anderson et al. 1994).

Table 2.4 Summary of the results of modified adhesive blister test (Source: Anderson et al. 1994)

Conditioning	Aggregates	Bitumen	Types of Failure
Unconditioned	Limestone	Type A	Cohesive
Unconditioned	Texas Chert	Type A	Cohesive
Conditioning[1]	Texas Chert	Type A	Cohesive
Conditioning[1]	Limestone	Type A	Cohesive
Conditioning[2]	Texas Chert	Type A	Adhesive
Conditioning[2]	Limestone	Type A	Adhesive

Notes: Conditioning[1]: Vacuum saturation, 30 minutes immersion at 60°C (140°F), 18 hours condition at either 0°C or 25°C; Conditioning[2]: Vacuum saturation, 30 minutes immersion at 60°C (140°F), 18 hours of freeze, 4 hours condition at 0°C

2.3.2.3 Specially Designed Sliding Plate Viscometer

Specially designed sliding plate viscometer was built to measure rheologically the interaction between aggregates and bitumen and also the contribution of the bitumen to the properties of the asphalt mixtures, in order to improve the prediction of the performance of the asphalt mixtures (Huang et al. 2005). Specially designed sliding plate viscometer is an accelerated test method used to measure the physical and rheological properties of bitumen with various types of aggregates plates, thus to simulate the real world interaction of aggregates and bitumen system. However, the specially designed sliding plate viscometer needs further study for further development as an improved method in predicting the performance of the asphalt mixtures. Figure 2.36 shows the specially designed sliding plate viscometer, which utilised a 25 mm by 40 mm by 6.5 mm of aggregates plates as substrates.

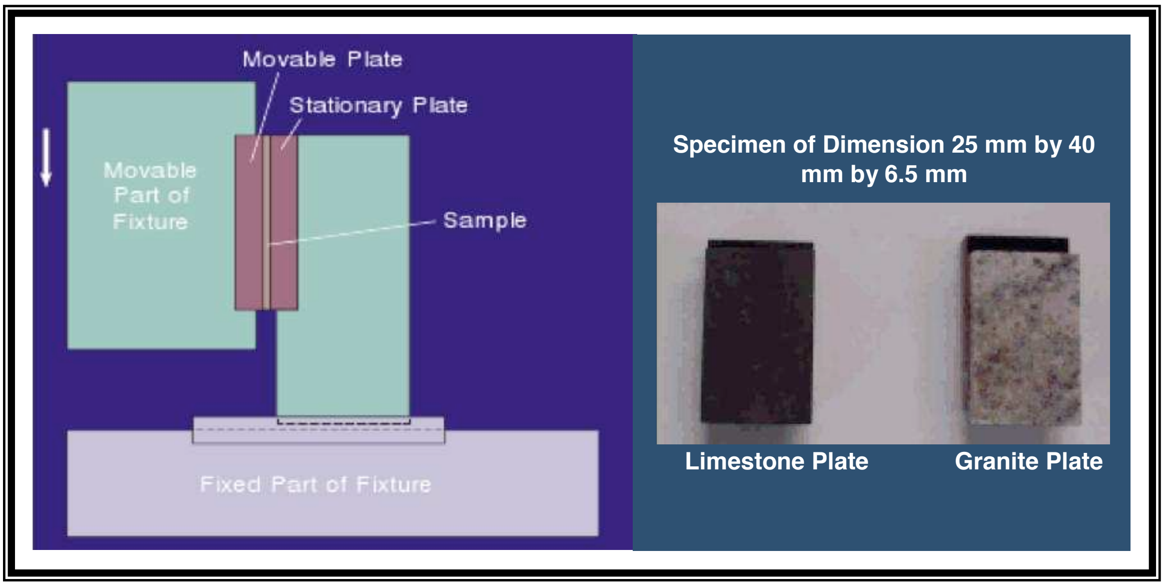


Figure 2.36 Specially designed sliding plate viscometer (Source: Huang 2005)

2.3.2.4 Pneumatic Adhesion Tensile Testing Instrument (PATTI) Device

Pneumatic Adhesion Tensile Testing Instrument (PATTI) device was initially developed by the National Institute of Standards and Technology (NIST) to evaluate the adhesive bond strength of aggregates and bitumen in the presence of water or moisture. The modified version of the PATTI device, known as PATTI 110 has been used by Kanitpong and Bahia (2003), Kanitpong and Bahia (2004) and Kanitpong and Bahia (2005) in measuring the moisture damage mechanisms based on the ASTM D4541 Standard Test Method for Pull-off Strength of Coating using Portable Adhesion Testers. The main features of the PATTI 110 include a portable pneumatic adhesion tester (PATTI 110), a pressure hose, a reaction plate, a piston and a pull stub as loading fixture, as shown in Figure 2.37. Figure 2.38 shows the schematic drawing of the PATTI 110.

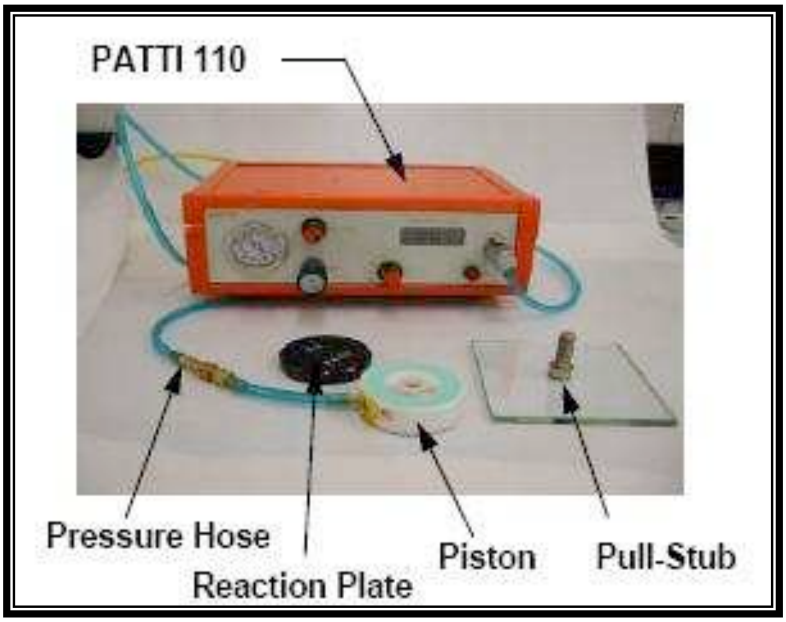


Figure 2.37 Modified Pneumatic Adhesion Tensile Testing Instrument (PATTI) device (Source: Kanitpong & Bahia 2003)

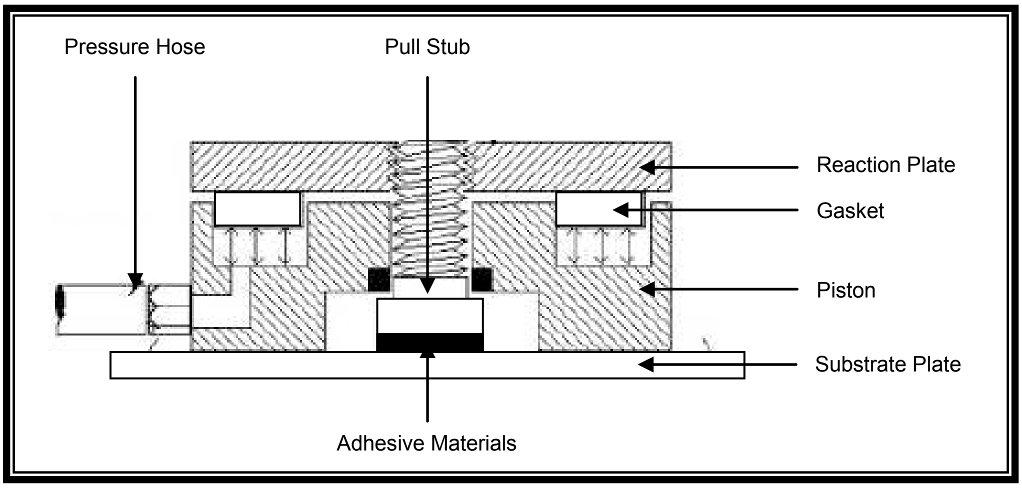


Figure 2.38 Schematic drawing of PATTI 110 (Source: Kanitpong & Bahia 2003)

Based on the studies conducted by Kanitpong and Bahia (2003), Kanitpong and Bahia (2004) and Kanitpong and Bahia (2005), the role of bitumen in moisture damage resistance of asphalt mixtures could be separated into two mechanisms; adhesion and cohesion. In the conducted studies, the relationship of adhesion between aggregates and bitumen and cohesion within bitumen with respect to the moisture damage were evaluated, and two different but interrelated approaches for measuring moisture damage of asphalt mixtures have been proposed. A method known as Tackiness Test of Asphalt using Dynamic Shear Rheometer (DSR) has been employed to measure the cohesion within bitumen and PATTI 110 has been used to measure the adhesion between aggregates and bitumen. Details of the Tackiness Test of Asphalt using Dynamic Shear Rheometer (DSR) is presented in the following section of 2.4.1 Details of the Tackiness Test of Asphalt using Dynamic Shear Rheometer (DSR) and Continuation of the Study of PATTI 110.

PATTI 110 has been used to measure the adhesion between aggregates and bitumen. Bitumen which has been heated to about 135°C was applied to the pull stub, and then pressed onto the surface of the substrate plate measuring an area of 51 mm by 51 mm and thickness of 6.25 mm. Various types of aggregates were used as substrate in order to evaluate the effect of the porosity on the adhesion. Thickness of the bitumen films is 200 μm (0.200 mm). Specimens were then subjected to dry conditioning at room temperature for 24 hours before being further conditioned as follows; 0 hours (i.e. immediately tested) or immersed in distilled water at 25°C for 4, 8 24 or 48 hours respectively.

Testing was then conducted at room temperature by transmitting air pressure to the piston, which is placed over the pull stub at speed of 65.7 kPa/second. Reaction plate is then placed onto the piston and screwed to the pull stub. The air pressure induces an airtight seal and forces the gasket of the piston, and thus the reaction plate to move upwards. When the air pressure exceeds the adhesive bond strength of bitumen and substrate plate (in case for the adhesive failure), and/or the cohesive bond strength of bitumen (in case for the cohesive failure), the failure of the specimen occurs. Air pressure at the failure and the types of failure (adhesive and/or cohesive) were recorded and then converted into the pull off tensile strength in unit of kPa.

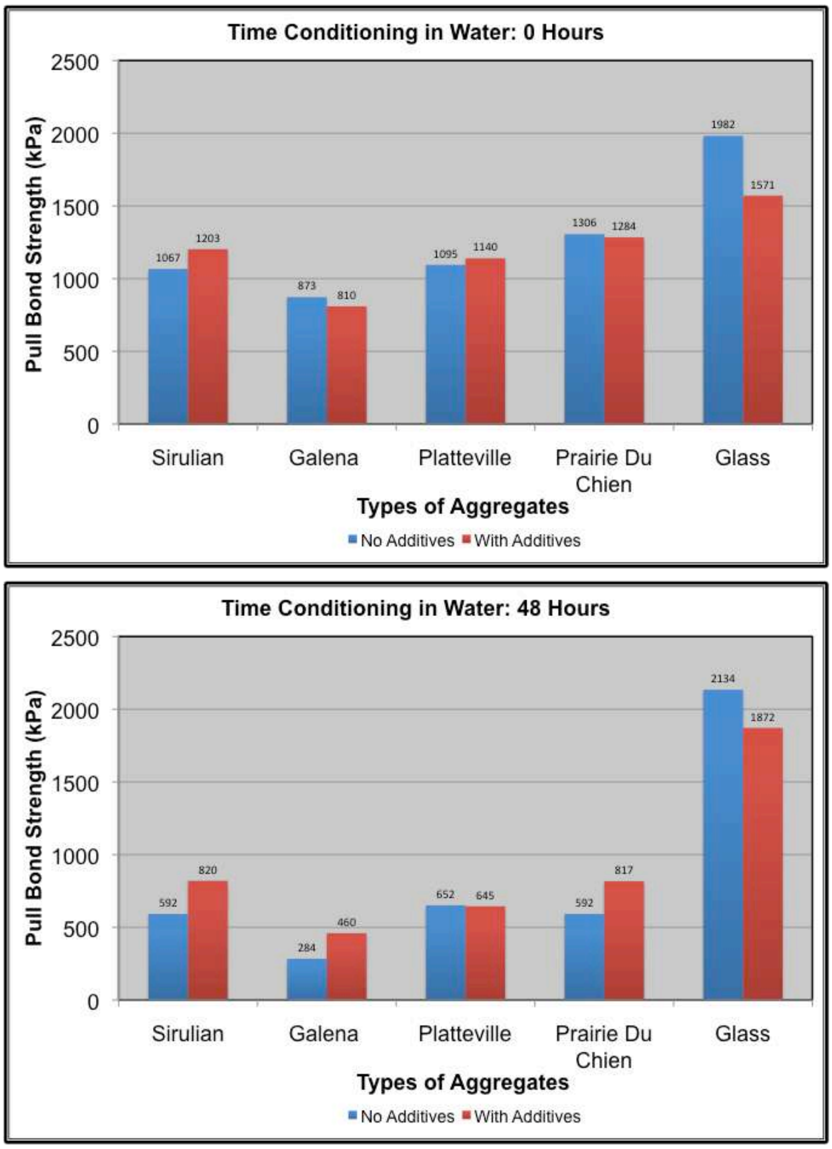
Kanitpong and Bahia (2003) concluded that the pull off tensile strength of the unconditioned specimens correlates with the cohesion of the bitumen, and the pull off tensile strength of the conditioned specimens correlates with both the cohesion of the bitumen and the adhesion between aggregates and bitumen, as shown in Table 2.5. Time conditioning in water has shown to have a significant effect on the types of failure and the pull off tensile strength. The pull off tensile strength tends to decrease as the time conditioning in water increases. The reason for this is due to the assumption that the longer conditioning time in water, the more ability for water to penetrate into the substrate plate of the aggregates, and hence the weaker the adhesive bond strength between aggregates and bitumen. The application of glass as substrate plate was found to result in negligible effect on additives (i.e. antistripping) and time conditioning in water as illustrated in Figure 2.39. Based on the data of the test results shown in Table 2.5 and Figure 2.39, Kanitpong and Bahia (2003) has concluded that the pull off tensile strength was mainly affected by the types of aggregates, additives and time conditioning in water.

Table 2.5 Results of the pull off tensile strength based on time conditioning in water (Source: Kanitpong & Bahia 2003)

Substrates	Antistripping	Pull Off Tensile Strength (kPa)		
		Time Conditioning in Water of 25°C (Hours)		
		0	24	48
Sirulian	No	1067 (C)	638 (A)	592 (A)
	Yes	1203 (C)	901 (A)	820 (A)
Galena	No	873 (C)	350 (A)	284 (A)
	Yes	810 (C)	378 (A)	460 (A)
Platteville	No	1095 (C)	603 (A)	652 (A)
	Yes	1140 (C)	880 (A)	645 (A)
Prairie Du Chien	No	1306 (C)	697 (A)	592 (A)
	Yes	1284 (C)	1126 (A)	817 (A)
Glass	No	1982 (C)[1]	2488 (A)[1]	2134 (C)[1]
	Yes	1571 (C)[1]	1977 (A)[1]	1872 (C)[1]

Notes: C: Cohesive failure; A: Adhesive failure; [1]Data is considered as outlier.

1. Thickness of Adhesive Materials: 200 µm
2. Adhesive Materials: PG 58-28
3. Conditioning Procedures: Dry conditioning at room temperature for 24 hours before being immersed in distilled water at 25°C at specified duration
4. Testing Conditions: Deformation rate of 65.7 kPa/second at room temperature



Notes:
1. Thickness of adhesive layer of bitumen is 200 μm
2. Adhesive Materials: PG 58-28
3. Conditioning Procedures: Dry conditioning at room temperature for 24 hours before being immersed in distilled water at 25°C at 0 hours and 48 hours respectively
4. Testing Conditions: Deformation rate of 65.7 kPa/second at room temperature

Figure 2.39 Response of pull off strength with types of aggregates, additives and time conditioning in water (Source: Kanitpong & Bahia 2003)

However, according to Kanitpong and Bahia (2003), the results of the adhesive bond strength conducted using the PATTI 110 yielded data with poor repeatability due to the uncontrolled variables, especially during specimen preparation. The observed variability of approximately 10% was found in the test results. Kanitpong and Bahia (2003) suggested that modification in controlling the thickness of bitumen films and thermal treatment could improve the repeatability. Also, based on Figure 2.39, the results of the pull off tensile strength of various types of aggregates subjected to dry conditioning (i.e. time conditioning in water of 0 hours) were found to differ significantly, although all specimens have the same adhesive materials (i.e. bitumen) and exhibited the same mode of failure (i.e. cohesive). Theoretically, specimens that exhibited the cohesive mode of failure should result in approximately the same value of the pull off tensile strength, regardless of the substrates. This is due to the fact that cohesion and cohesive failure are mainly influenced by viscosity of adhesive materials and independent of the types of aggregates.

The advantages of the PATTI 110 is due to the ability for conditioning the specimen in water, application of various types of aggregates as substrates and observation of the failure surface in order to define the failure as either adhesive or cohesive. Figure 2.40 shows the types of failure (i.e. adhesive and cohesive failure), which were determined on the basis of visual observations. Based on the results of the adhesive bond strength conducted using the PATTI 110, most of the failures for the unconditioned specimens were cohesive, while most of the failures for the conditioned specimens were adhesive.

Cohesive Failure Adhesive Failure

Figure 2.40 Types of failure of tested specimens using PATTI 110 (Source: Kanitpong & Bahia 2003)

2.4 A Review of Studies on Tensile Behaviour and Failure Characteristics of Asphalt Mixtures

Based on the literature review (i.e. Kanitpong and Bahia (2003), Kanitpong and Bahia (2004), Kanitpong and Bahia (2005) and Tandon and Puentes (2006)), pull off (tension) mode was found to has a promising potential for development of the laboratory adhesion test method for measuring the adhesive bond strength of bitumen (bitumen-filler mastic) and aggregates. In this section, previous studies on tensile behaviour and failure characteristics of asphalt mixtures subjected to axial tensile load (i.e. pull off (tension) mode), such as being conducted by Copeland (2007) and Marek and Herrin (1968) are reviewed, in order to provide some guidelines for development of criteria and procedures for the proposed adhesion test method. Also, details of the Tackiness Test of Asphalt using Dynamic Shear Rheometer (DSR) based on the study conducted by Kanitpong and Bahia (2003), Kanitpong and Bahia (2004) and Kanitpong and Bahia (2005) are presented.

2.4.1 Details of the Tackiness Test of Asphalt Using Dynamic Shear Rheometer (DSR) and Continuation of the Study of PATTI 110

Tackiness Test of Asphalt using Dynamic Shear Rheometer (DSR) has been developed in order to measure the cohesion within bitumen as schematically illustrated in Figure 2.41. Based on Figure 2.41, the measuring system of Dynamic Shear Rheometer (DSR) can be moved upward with a defined speed of 0.01 mm/second and the applied force acting within bitumen is measured. The applied force and the time of separation between bitumen and measuring system of Dynamic Shear Rheometer (DSR) are recorded and plotted. The stickiness or tack factor, C_T of bitumen was then calculated based on the area under the curve of graph of applied force versus time of separation between bitumen and measuring system of Dynamic Shear Rheometer (DSR). Alternatively, the stickiness or tack factor, C_T of bitumen can be calculated based on the following equation.

Equation 2.10

$$C_T = \frac{1}{A}\int (F \times v \times dt)$$

where:

C_T	=	*Stickiness or Tack Factor*
A	=	*Area of contact of the bitumen and substrates*
F	=	*Applied force*
v	=	*Speed of separation*
t	=	*Time*

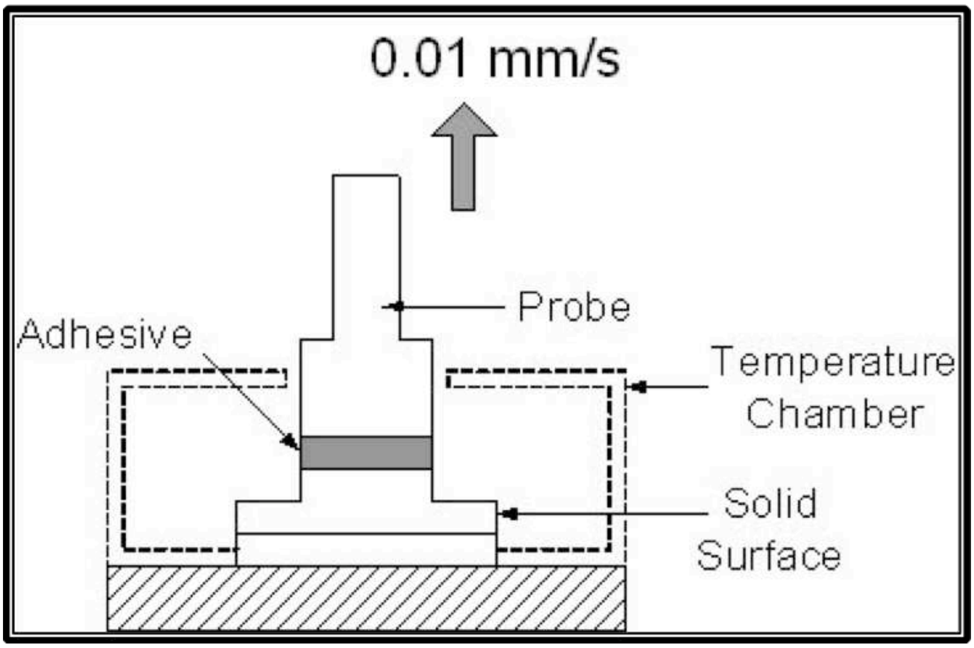


Figure 2.41 Schematic drawing of Tackiness Test of Asphalt using Dynamic Shear Rheometer (DSR) (Source: Kanitpong & Bahia 2003)

For the Tackiness Test of Asphalt using Dynamic Shear Rheometer (DSR), the temperature significantly affects the stickiness or tack factor, C_T of the bitumen. As the temperature increases, the stickiness or tack factor, C_T decreases as shown in Figure 2.42.

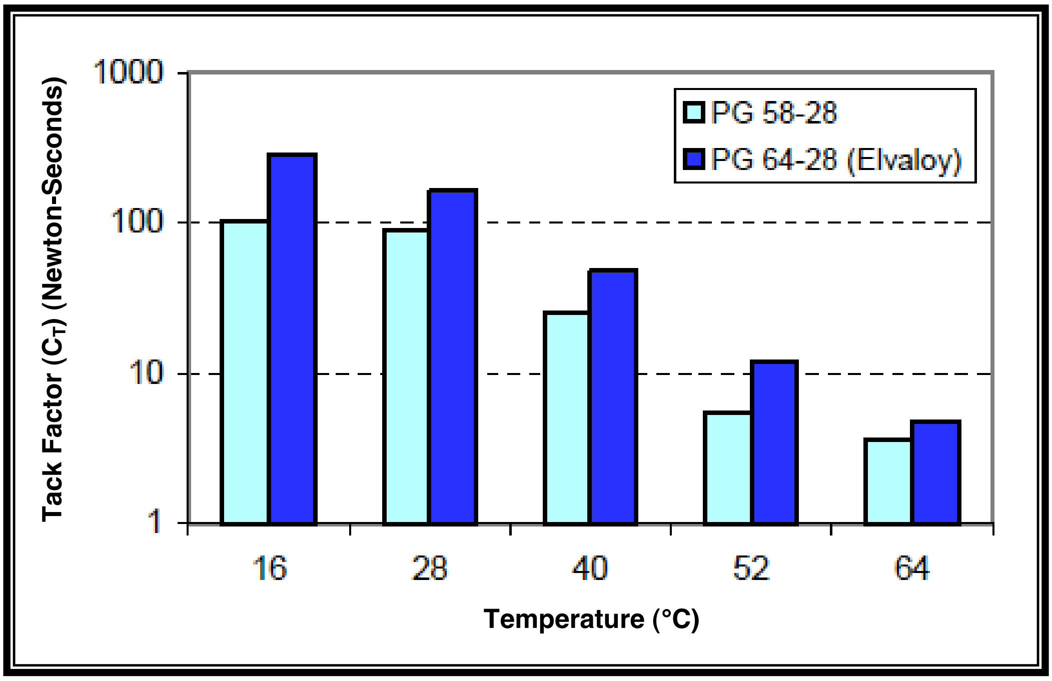


Figure 2.42 Effect of temperature on stickiness or tack factor, C_T of the bitumen (Source: Kanitpong & Bahia 2003)

Copeland (2007) in the study to determine the influence of water or moisture on the adhesive bond strength of the asphalt mixtures has further evaluated the feasibility of the modified version of the Pneumatic Adhesion Tensile Testing Instruments (PATTI) device, known as PATTI 110, and tried to correlate the test results with the combined experimental-numerical model of the function of water or moisture content at the interfaces of the bitumen films and aggregates. Based on Copeland (2007), for the unconditioned specimens, cohesive failure within bitumen is expected; and for the moisture conditioned specimens, the mode of failure is expected to change from cohesive to mixed cohesive and adhesive or entirely adhesive. Hence, hypothesis has been developed proposing that the presence of water or moisture in the pavement structure will result in the reduction of the adhesive bond strength at the interfaces of the bitumen films and aggregates.

In the study, due to the large amount of specimens required, reuse of the aggregates plates is necessary. Hence, the remaining layer of the bitumen on the aggregates plates must be removed or cleaned after each test. It should be noted that the reuse of the aggregates plates will result in discrepancy and inaccuracy of the test results. However, the discrepancy and inaccuracy of the test results can be minimised by selecting the right method for removal of the remaining layer of the bitumen without significant change in the properties of the aggregates plates. Based on the study, chemical cleaning procedures involving chemical solution such as white spirit solvent has been identified as the best method to remove or clean the remaining layer of the bitumen on the aggregates plates instead of heating cleaning procedures. Analysis of variance (ANOVA) has been used to determine the effect of various methods to remove or clean the remaining layer of the bitumen on the aggregates plates, and the results are tabulated in Table 2.6.

Table 2.6 Analysis of variance (ANOVA) (Source: Copeland 2007)

Adhesive Materials	Soak Time (Hours)	ANOVA (p-Value > F)	Comparison of Data of Test Results between Various Cleaning Procedures			
			Baseline & Chemical		Baseline & Heating	
			p-Value	Significant Difference	p-Value	Significant Difference
AAD	0	0.0287	0.2972	NO	0.04331	YES
	24	0.1899			0.1899	NO
AAM	0	0.3275	0.7252	NO	0.1708	NO
	24	0.0361			0.0361	YES
SBS-LG 6295	0	0.2237	0.2237	NO	-	-
PG70-22 6298	0	0.5725	0.5725	NO	-	-

Notes: Baseline is the control specimens using new aggregates (i.e. aggregates that are not subjected to any cleaning procedures)

In the previous study conducted by Kanitpong and Bahia (2003), Kanitpong and Bahia (2004) and Kanitpong and Bahia (2005), procedures for specimen preparation in terms of the controlling of the thickness of bitumen films are operator dependent, thus result in uneven thickness of bitumen films. Copeland (2007) has modified the procedures for specimen preparation by introducing a device to allow for the compression of the specimens and thus enabled a better controlling of the thickness of bitumen films, as shown in Figure 2.43.

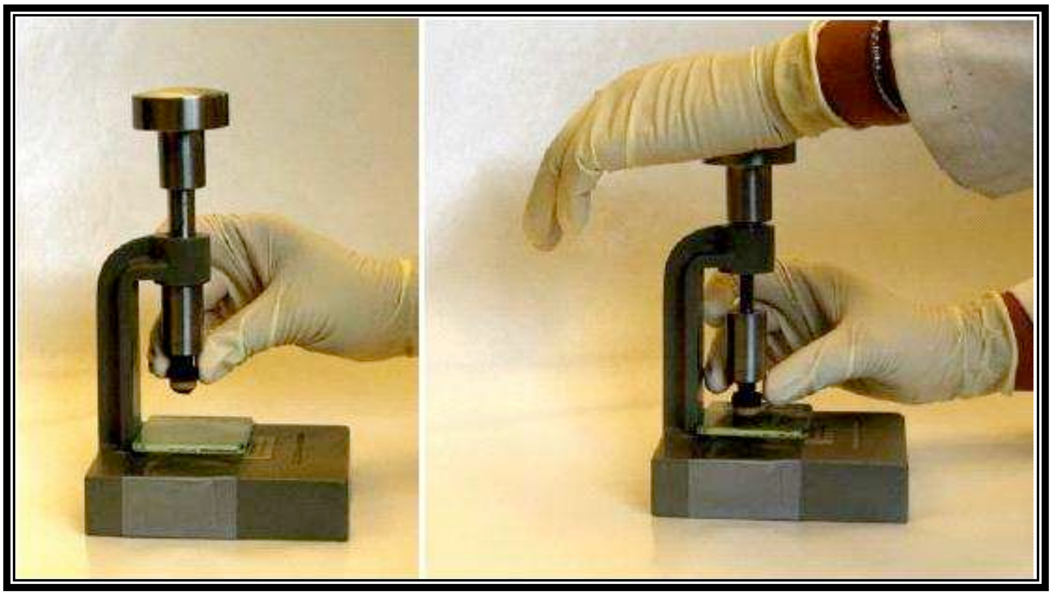
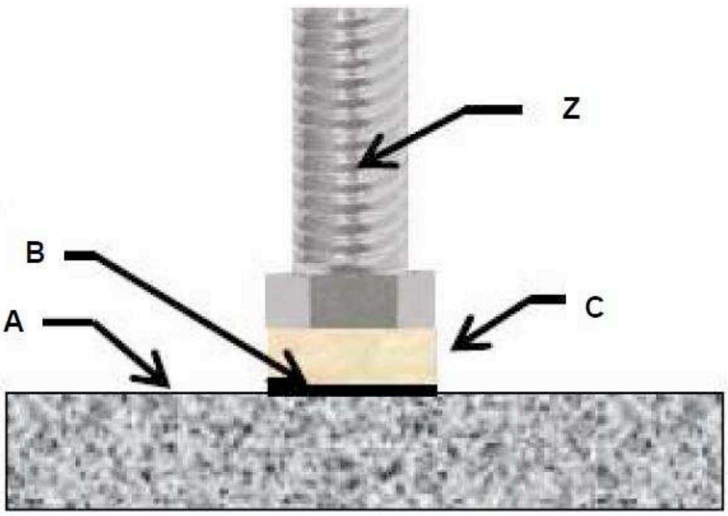


Figure 2.43 Device for controlling the thickness of bitumen films (Source: Copeland 2007)

Based on Copeland (2007), the mode of failure is hypothesised to occur along the interfaces of the bitumen films and aggregates (i.e. adhesively) after subjecting the specimens to conditioning process for certain period of time. However, based on the test results shown in Table 2.7, no definite conclusion can be made since the mode of failure was found to be more the cohesive rather than the mixed cohesive and adhesive or entirely adhesive. In Table 2.7, two types of binder (i.e. binder AAD and AAM) were used as adhesive materials and diabase was used as substrates. Specimens were submerged in water at 25°C for different conditioning time. Types of failure were found to occur within bitumen (as denoted by B), between bitumen and ceramic frit (as denoted by B-C) or between ceramic frit and loading fixture (as denoted by C-Z). None of the test results showed that the failure had occurred along the interfaces of bitumen and aggregates (as denoted by A-B). Figure 2.44 shows the components for the tested specimens.



Component	Description	Thickness
A	Substrates	Varies
B	Adhesive Materials with Beads	200 μm
C	Ceramic	6.35 mm
Z	Pull Stub	Not Available

Figure 2.44 Components for the tested specimens (Source: Copeland 2007)

Table 2.7 Results of the pull off tensile strength based on different conditioning time (Source: Copeland 2007)

Adhesive Materials	Substrates	Submerged in Water at 25°C (Hours)	Mean Pull Off Tensile Strength (kPa)	Standard Deviation	Coefficient of Variation (%)	Failure
AAD	Diabase	0	1069	11.90	7.70	B
AAD	Diabase	0				B
AAD	Diabase	0				B
AAD	Diabase	0				B
AAD	Diabase	24	386	12.76	22.62	B
AAD	Diabase	24				B
AAD	Diabase	24				B
AAD	Diabase	24				B
AAD	Diabase	48	331	2.95	6.10	B
AAD	Diabase	48				B
AAD[1]	Diabase	48				C-Z
AAD	Diabase	48				B
AAD	Diabase	72	359	8.00	15.00	B
AAD	Diabase	72				B
AAD	Diabase	72				B
AAD	Diabase	72				B
AAM	Diabase	0	1910	13.23	4.78	B
AAM	Diabase	0				B
AAM	Diabase	0				B
AAM	Diabase	0				B
AAM	Diabase	16	1151	28.86	17.25	B
AAM[1]	Diabase	16				B-C
AAM	Diabase	16				B
AAM[1]	Diabase	16				B-C
AAM[1]	Diabase	24	1262	Not Available	Not Available	B-C
AAM[1]	Diabase	24				B-C
AAM	Diabase	24				B
AAM[1]	Diabase	24				B-C
AAM	Diabase	48	683	36.20	36.44	B
AAM	Diabase	48				B
AAM	Diabase	48				B
AAM[1]	Diabase	48				C-Z
AAM	Diabase	72	986	26.47	18.50	B
AAM	Diabase	72				B
AAM	Diabase	72				B
AAM	Diabase	72				B
AAM	Diabase	99	917	43.96	33.08	B
AAM	Diabase	99				B
AAM	Diabase	99				B
AAM	Diabase	99				B

Notes: [1]Data is considered as outlier and not included in the calculation; B: Types of failure were found to occur within bitumen; B-C: Types of failure were found to occur between bitumen and ceramic frit; C-Z: Types of failure were found to occur between ceramic frit and loading fixture.

1. Thickness of Adhesive Materials: 200 μm
2. Conditioning Procedures: Dry conditioning at room temperature for 24 hours before being submerged in water at 25°C for the specified duration
3. Testing Conditions: Deformation rate of 65.7 kPa/second at room temperature

Hence, further study on the conditioning process to result in the mode of failure of mixed cohesive and adhesive or entirely adhesive is suggested. Copeland (2007) has also recommended for the evaluation of the properties of the aggregates substrates in term of the porosity and interconnected voids. Although PATTI 110 has promising potential for measuring the moisture damage performance of the asphalt mixtures, the results of the adhesive bond strength yielded data with poor repeatability and a conclusion has been made that a single test method cannot satisfactorily and accurately determine the moisture damage performance. Also, PATTI 110 has limitations in terms of the applicability to measure the adhesive bond strength for wide ranges of asphalt mixture materials under various testing conditions (deformation rates and test temperatures). Test of the PATTI 110 can only be conducted at room temperature. Copeland (2007) has identified that deformation rate and test temperature are among the variables that would contribute to the types of failure (adhesive or cohesive). These were further verified by the study conducted by Marek and Herrin (1968) on the behaviour and failure characteristics of thin films of bitumen subjected to axial tensile load (i.e. pull off (tension) mode). Details of the study conducted by Marek and Herrin (1968) are presented in the following section.

2.4.2 Details of the Study Conducted by Marek and Herrin

Marek and Herrin (1968) conducted a study on the behaviour and failure characteristics of thin films of bitumen subjected to axial tensile load (i.e. pull off (tension) mode) via the tensile testing of 1-inch diameter pair of cylindrical test blocks as shown in Figure 2.45. In the conducted study, various combinations of variables including thickness of adhesive layer of bitumen, deformation rate, test temperature and penetration grade of bitumen have been used, in order to enable for the generalisation of conclusions. The ranges of the selected variables are listed as follows.

1. Thickness of adhesive layer of bitumen is between 20 μm (0.020 mm) and 600 μm (0.600 mm).

2. Deformation rate is between 0.005 inch/minute (0.127 mm/minute) and 1.000 inch/minute (25.400 mm/minute).

3. Test temperature is between 0°C and 60°C.

4. Penetration grade of bitumen is between 50 and 217.

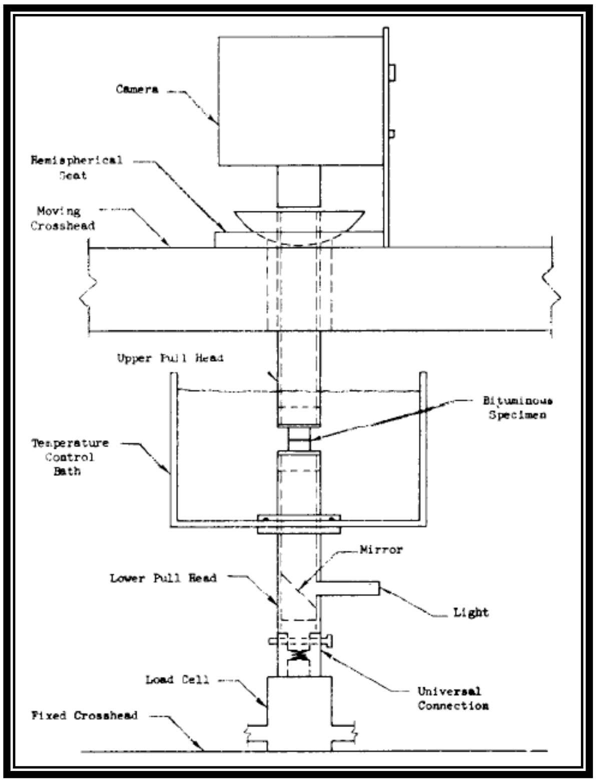


Figure 2.45 Apparatus for tensile testing of thin films of bitumen (Source: Marek & Herrin 1968)

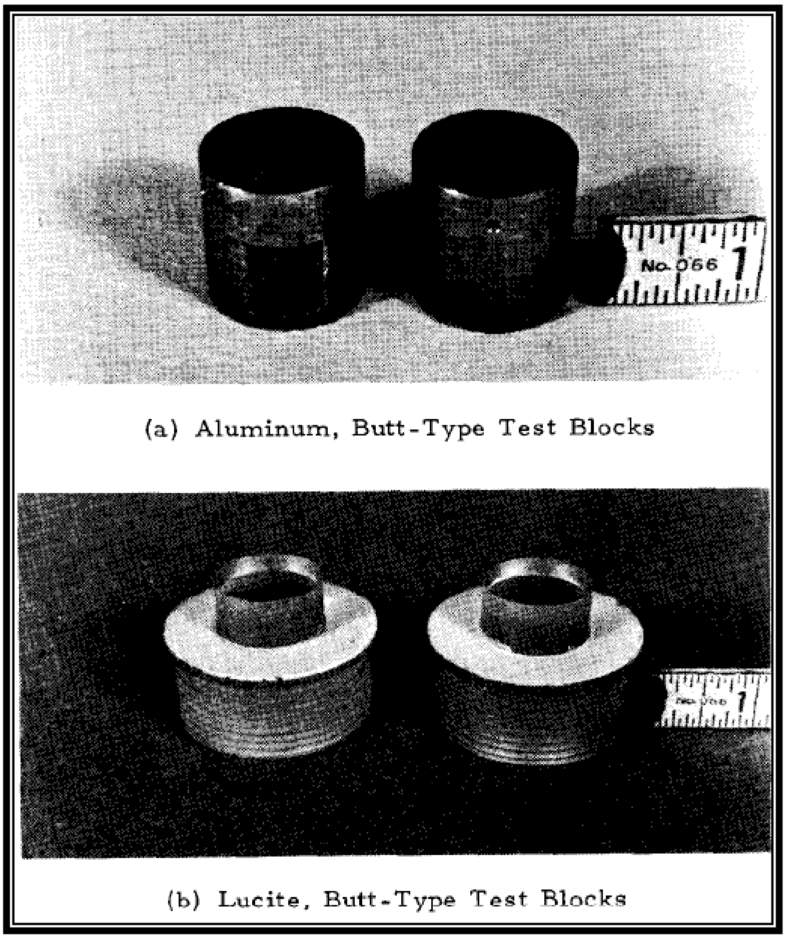
According to Mack (1957) and Wood (1958), the ranges of the selected thickness of adhesive layer of bitumen (i.e. 20 μm (0.020 mm) and 600 μm (0.600 mm)) fall within the ranges of the average thickness of bitumen (bitumen-filler mastic) films in the field compacted mixtures. In the conducted study, bitumen was sandwiched between two 1-inch diameter pair of cylindrical test blocks, made from either aluminium or lucite as shown in Figure 2.46. Pressure was then applied onto the pair of the cylindrical test blocks in order to obtain the required uniform thickness of adhesive layer of bitumen. Excess bitumen at the edges was removed prior to the testing in order to prevent any discrepancy and inaccuracy of the test results. The average thickness of adhesive layer of bitumen was calculated based on the weight difference methods and volume-density calculations, as shown in the Equation 2.11.

Equation 2.11

$$\begin{aligned} Weight\ of\ Bitumen\ (g) &= \left(Weight\ of\ Test\ Blocks\ with\ Bitumen\ (g)\right) \\ &\quad - \left(Weight\ of\ Test\ Blocks\ without\ Bitumen\ (g)\right) \end{aligned}$$

$$\begin{aligned} Bulk\ Density\ of\ Bitumen\ (g/cm^3) &= (Specific\ Gravity\ of\ Bitumen) \\ &\quad \times \left(Bulk\ Density\ of\ Water\ (g/cm^3)\right) \end{aligned}$$

$$Thickness\ of\ Bitumen\ (cm) = \frac{(Weight\ of\ Bitumen\ (g))}{\left(Bulk\ Density\ of\ Bitumen\ (g/cm^3)\right) \times \left(Surface\ Area\ of\ Test\ Blocks\ (cm^2)\right)}$$



(a) Aluminum, Butt-Type Test Blocks

(b) Lucite, Butt-Type Test Blocks

Figure 2.46 Pair of cylindrical test blocks (Source: Marek & Herrin 1968)

Based on the conducted study, thickness of adhesive layer of bitumen, deformation rate, test temperature and penetration grade of bitumen were found to have a profound influence on the maximum tensile bond strength (i.e. maximum tensile load per unit area of contact) and the types of failure (adhesive or cohesive). Based on Figure 2.47, the relationship between the maximum tensile bond strength (i.e. maximum tensile load per unit area of contact) and the thickness of adhesive layer of bitumen on semi logarithmic plot have resulted in three main regions.

1. In the first region where the thickness of adhesive layer of bitumen is less than 20 μm (0.020 mm), the maximum tensile bond strength (i.e. maximum tensile load per unit area of contact) was found to increase

with the increasing value of the thickness of adhesive layer of bitumen. However, no definite conclusion can be made regarding the relationship in the first region due to the limitations in producing thin films of bitumen of uniform thickness of less than 20 μm (0.020 mm). Hence the relationship between the thickness of adhesive layer of bitumen and the maximum tensile bond strength (i.e. maximum tensile load per unit area of contact) in the first region were meaningless.

2. In the second region where the thickness of adhesive layer of bitumen is between 20 μm (0.020 mm) and 200 μm (0.200 mm), linear inverse relationship has been found which depicts a decrease in the maximum tensile bond strength (i.e. maximum tensile load per unit area of contact) as the thickness of adhesive layer of bitumen is increased.

3. In the third region where the thickness of adhesive layer of bitumen is more than 200 μm (0.200 mm), a nearly horizontal line which indicates an almost constant value of the maximum tensile bond strength (i.e. maximum tensile load per unit area of contact) as the thickness of adhesive layer of bitumen is increased was found. This indicates that, under some combinations of the thickness of more than 200 μm (0.200 mm) and other variables, the adhesive layer of bitumen has almost constant and low value of the maximum tensile bond strength (i.e. maximum tensile load per unit area of contact).

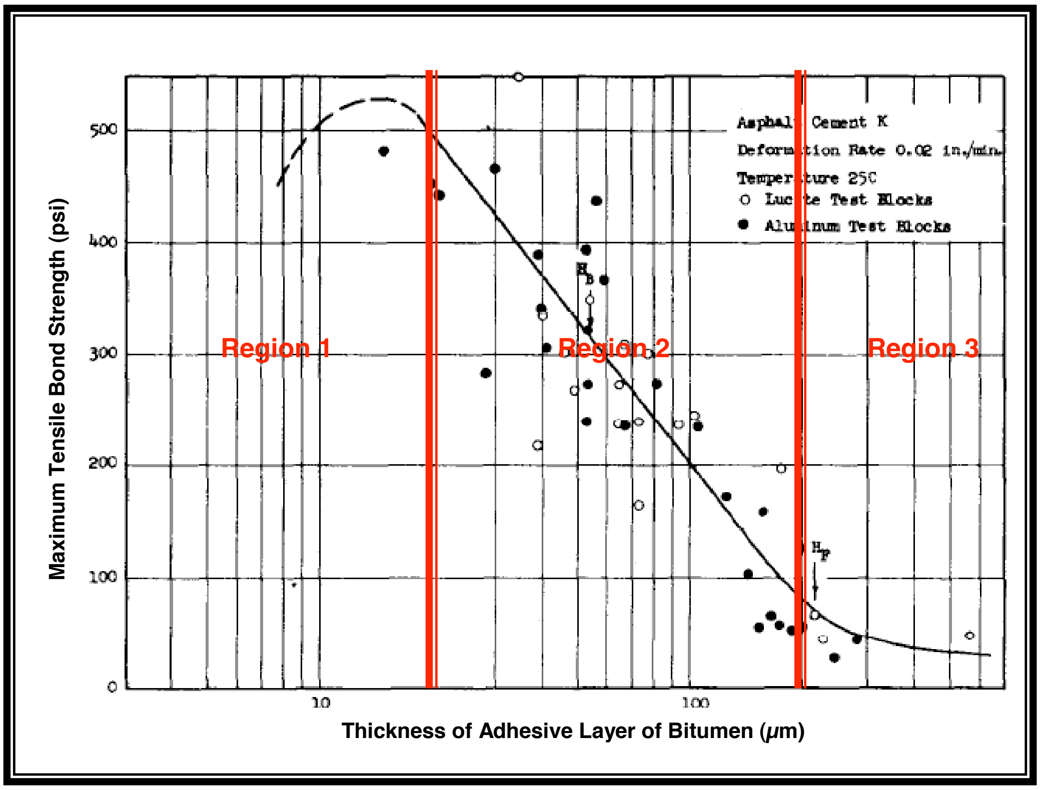


Figure 2.47 Relationship between maximum tensile bond strength and thickness of adhesive layer of bitumen (Source: Marek & Herrin 1968)

Also, based on the Figures 2.48 to 2.50, the value of the maximum tensile bond strength (i.e. maximum tensile load per unit area of contact) was found to increase with the increasing deformation rate, and decrease with the increasing temperature and the increasing value of the penetration grade of bitumen, respectively. Based on the results of the study, Marek and Herrin (1968) has concluded that the value of the maximum tensile bond strength (i.e. maximum tensile load per unit area of contact) was mainly characterised by the thickness of adhesive layer of bitumen, deformation rate, test temperature and penetration grade of bitumen.

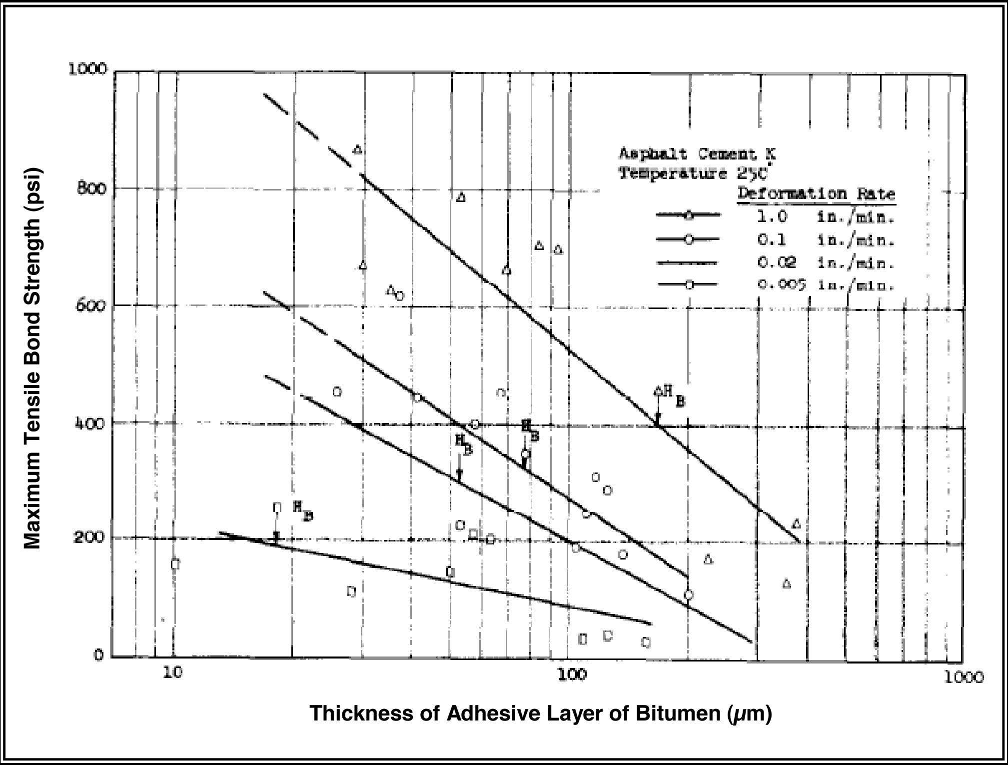


Figure 2.48 Relationship between maximum tensile bond strength and deformation rate (Source: Marek & Herrin 1968)

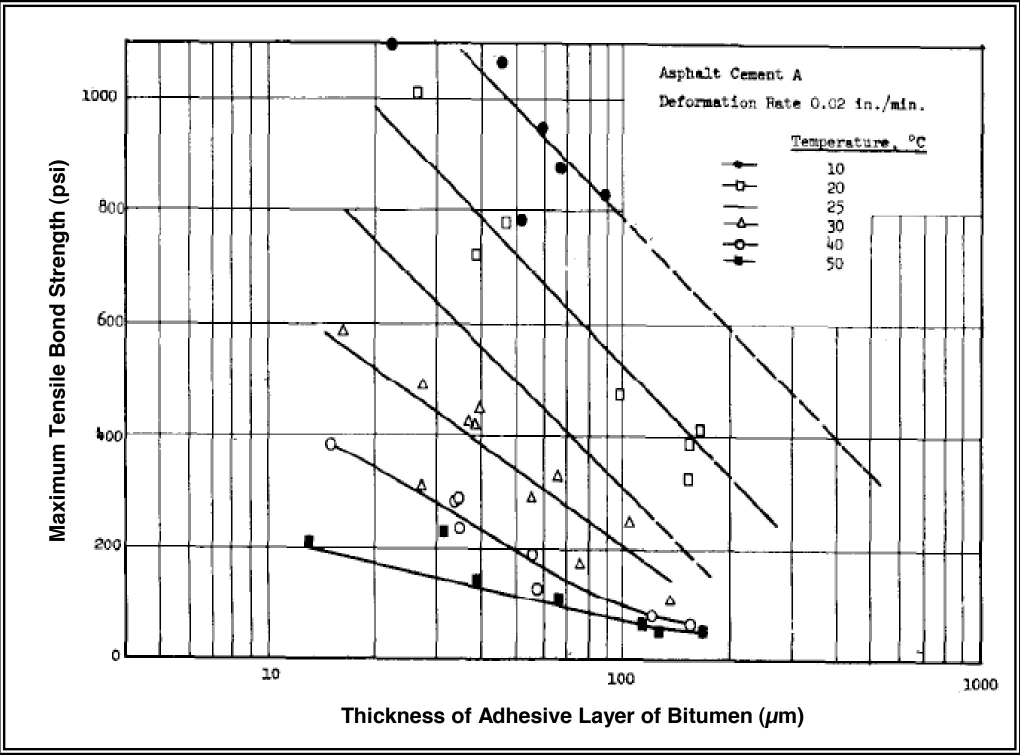


Figure 2.49 Relationship between maximum tensile bond strength and test temperature (Source: Marek & Herrin 1968)

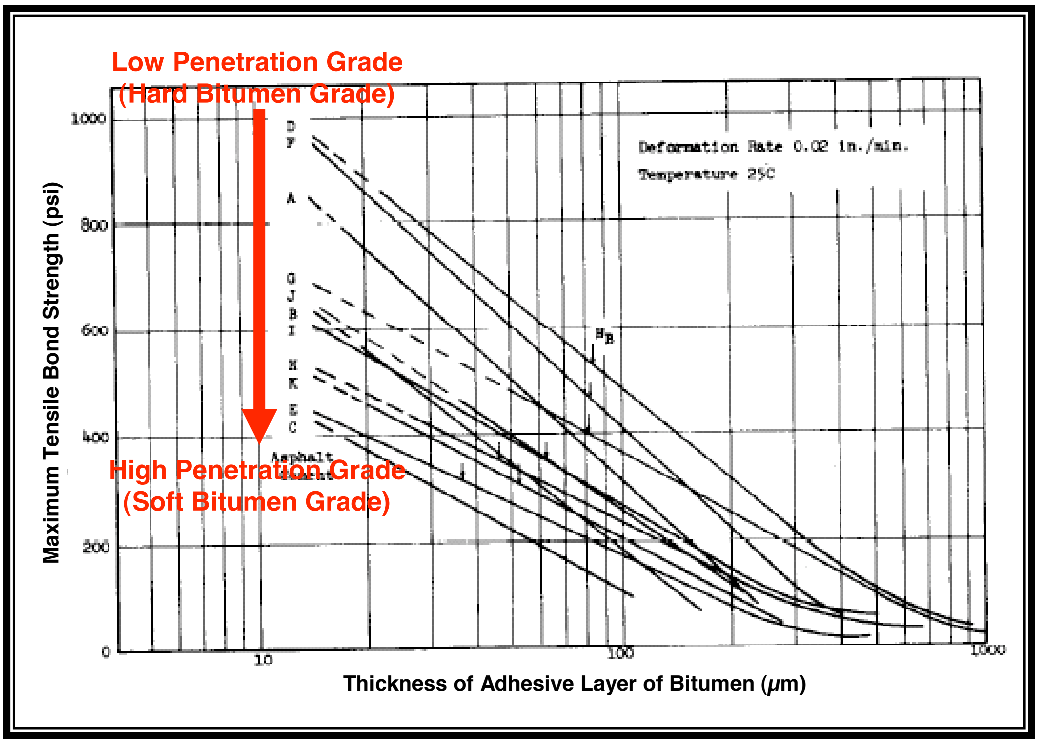
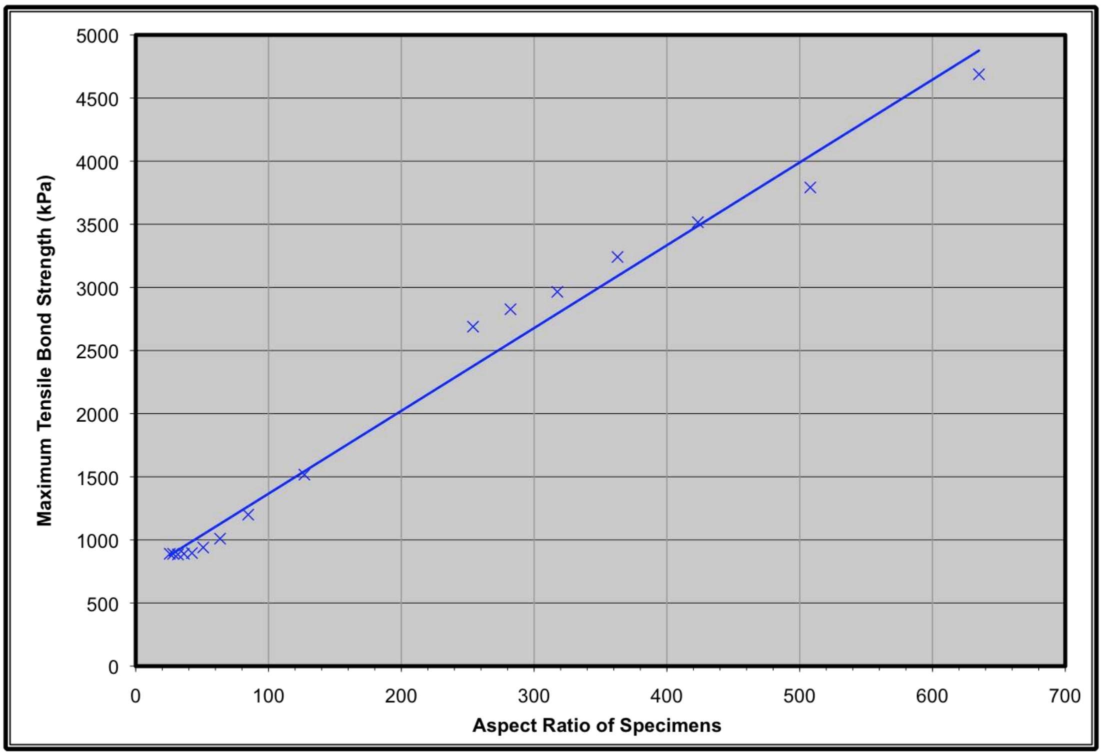


Figure 2.50 Relationship between maximum tensile bond strength and penetration grade of bitumen (Source: Marek & Herrin 1968)

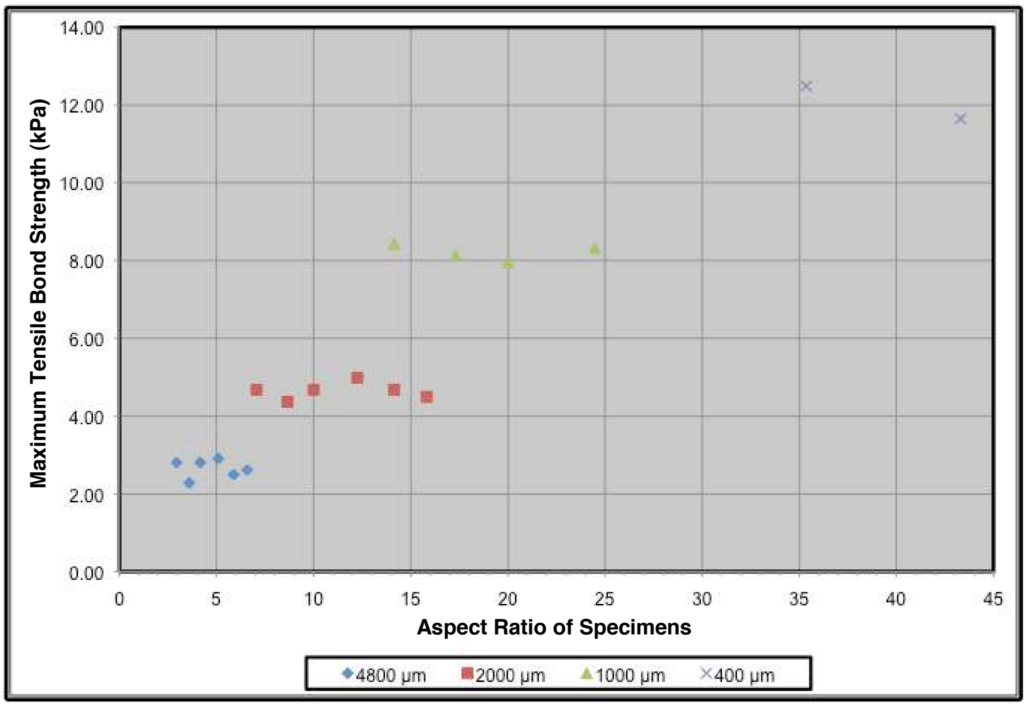
In terms of the aspect ratio of specimens, data of the study conducted by Marek and Herrin (1968) was used in order to develop a relationship between maximum tensile bond strength and aspect ratio of specimens. Aspect ratio of specimens is defined as the ratio of the longest dimension to the shortest dimension, which is referred to diameter of the discs and the thickness of adhesive layer of bitumen respectively. Based on the definition, aspect ratio of specimens can vary in two ways; due to variation of diameter of the discs and fixed thickness of adhesive layer of bitumen or due to variation of thickness of adhesive layer of bitumen and fixed diameter of the discs. Figure 2.51 shows the relationship between maximum tensile bond strength and aspect ratio of specimens based on the fixed diameter of the discs (i.e. 1-inch diameter pair of cylindrical test blocks).



Notes: 1. Diameter of Disc: 1 inch (25.4 mm)
2. Substrates: Aluminium alloy
3. Adhesive Materials: Asphalt cement K (Penetration at 25°C is 52)
4. Conditioning Procedures: Dry conditioning at room temperature for 3 hours prior to testing
5. Testing Conditions: Deformation rate and test temperature of 25.4 mm/minute and 25°C respectively

Figure 2.51 Relationship between maximum tensile bond strength and aspect ratio of specimens (Fixed diameter of the discs) (Source: Marek & Herrin 1968)

Based on Figure 2.51, the maximum tensile bond strength was found to increase as the value of the aspect ratio of specimens was increased. However, the increment of the maximum tensile bond strength can also be correlated with the decreasing value of the thickness of adhesive layer of bitumen. As has been stated before, the value of the maximum tensile bond strength is mainly influenced by the thickness and is expected to decrease with the increasing thickness of adhesive layer of bitumen. Based on Harrison and Harrison (1972) in the study of adhesion using finite element analysis, tensile bond strength was found to be independent of aspect ratio under the following conditions; aspect ratio of more than 10 and Poisson's ratio of adhesive materials of less than 0.49. Kendall (1971) has proved the negligible effect of the aspect ratio of specimens based on the following condition; aspect ratio of specimens is varied due to the variation of the diameter of discs while thickness of adhesive layer of bitumen is remained constant. In the conducted study, gelatine of thickness of 4800 μm (4.800 mm), 2000 μm (2.000 mm), 1000 μm (1.000 mm) and 400 μm (0.400 mm) were used as adhesive materials and Perspex was used as substrates. Figure 2.52 shows the plot of the test results. As long as the thickness of adhesive layer of bitumen is remained constant, the effect of aspect ratio on the maximum tensile bond strength was found to be negligible.



Notes: 1. Substrates: Perspex
2. Adhesive Materials: Gelatine
3. Conditioning Procedures: Dry conditioning at room temperature
4. Testing Conditions: Deformation rate of 0.6 mm/minute at room temperature

Figure 2.52 Relationship between maximum tensile bond strength and aspect ratio of specimens (Fixed thickness of adhesive layer of bitumen) (Source: Kendall 1971)

Based on the conducted study, Marek and Herrin (1968) also classified the failure characteristics of the thin films of bitumen subjected to axial tensile load (i.e. pull off (tension) mode), into three groups, as illustrated in Figure 2.53 and namely as follows; brittle fracture, intermediate failure and flow failure. Brittle fracture, which can be defined as the adhesive mode of failure was characterised by complete and instantaneous separation at the maximum tensile bond strength (i.e. maximum tensile load per unit area of contact). Intermediate failure was characterised by the occurrence of the cobwebbing (multiple strings or strands and limited necking of the thin films of bitumen) and a gradual drop off in the tensile bond strength after the attainment of the maximum (i.e. peak) value of the tensile bond strength. Cobwebbing is a common paint films and coatings term used to describe the spider web effect caused by the tendency of the premature drying of paint films and coatings to forms strings or strands on the substrates. Flow failure was characterised by the formation of single string or strand, and large deformation was observed as the tensile bond strength was gradually dropped off. Marek and Herrin (1968) classified the intermediate and flow failure as the cohesive mode of failure. Based on the test results, Marek and Herrin (1968) concluded that the types of failure for the thin films of bitumen subjected to axial tensile load (i.e. pull off (tension) mode) can generally be classified as cohesive failure rather than adhesive failure. In most instances, the occurrence of the failure was through the adhesive layer of bitumen with the presence of the remaining bitumen on both surfaces of the pair of the cylindrical test blocks. Marek and Herrin (1968) also suggested that for the occurrence of the adhesive mode of failure, high deformation rate and low test temperature must be utilised.

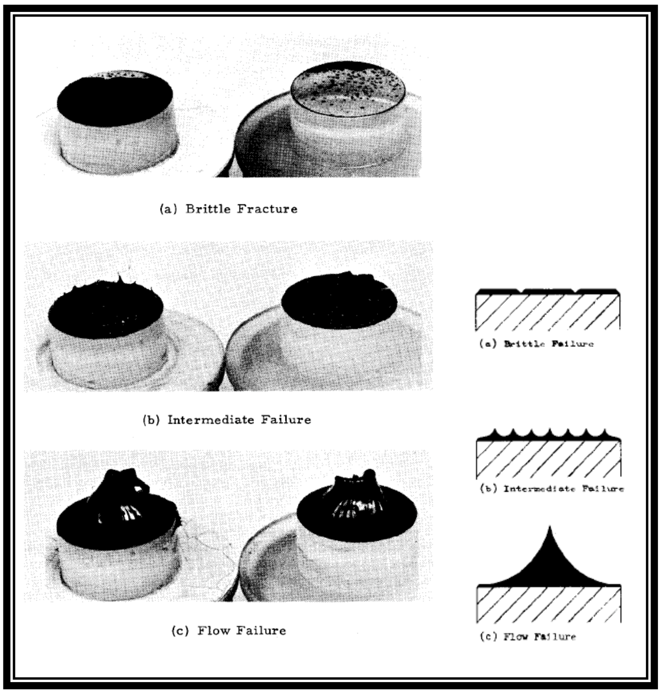


Figure 2.53 Failure characteristics of thin films of bitumen subjected to axial tensile load (Source: Marek & Herrin 1968)

Thin films of bitumen subjected to axial tensile load (i.e. pull off (tension) mode), were found to exhibit cavitations regardless of the failure characteristics as shown in Figure 2.54. The occurrence of the cavitations has been related to the presence of the foreign particles such as dust or mineral matter on the surface of the pair of the cylindrical test blocks and also in the bitumen, entrapment of air voids during procedures for specimen preparation and also roughness of the surface of the pair of the cylindrical test blocks.

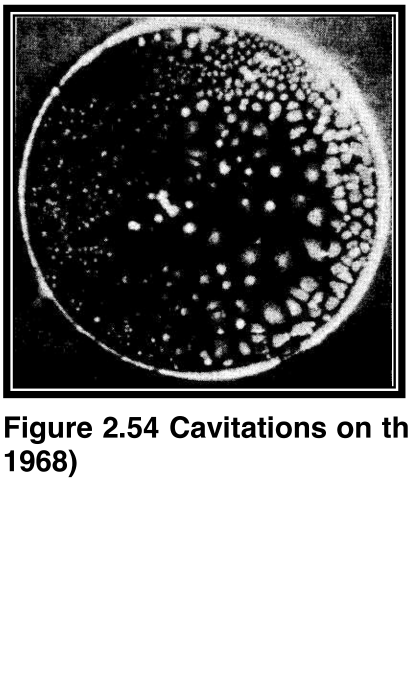


Figure 2.54 Cavitations on thin films of bitumen (Source: Marek & Herrin 1968)

2.5 Summary of Literature Review

In this chapter, asphalt mixtures and moisture damage in asphalt mixtures were defined, and two primary modes of failure for moisture damage were discussed. Moisture damage is mainly characterised by adhesion between bitumen (bitumen-filler mastic) and aggregates and cohesion of bitumen (bitumen-filler mastic). However, many studies have concluded that the failure for the moisture damage of asphalt mixtures is more the adhesive mode of failure rather than the cohesive mode of failure.

In the context of asphalt mixtures, adhesion can be defined as the amount of energy required to break the adhesive bond between bitumen (bitumen-filler mastic) and aggregates. Adhesion between bitumen (bitumen-filler mastic) and aggregates is considered as one of the main fundamental properties of the asphalt mixtures, which can be correlated with the quality, performance and serviceability of the flexible pavements. Adhesive failure between bitumen (bitumen-filler mastic) and aggregates can be identified via the occurrence of stripping in the asphalt mixtures. Four broad theories of adhesion between bitumen (bitumen-filler mastic) and aggregates have been discussed.

Research on the adhesion of the asphalt mixtures is limited and there are no established testing techniques and procedures that can be used to quantify the adhesive bond strength between bitumen (bitumen-filler mastic) and aggregates. Hence, a detailed review of literature on various testing techniques and procedures used to measure the adhesive bond strength, which can be found in numerous areas of scientific literature and international standards were presented. Among the most commonly used testing techniques and procedures are peel test, pull off test, double cantilever beam (DCB) test and tapered double cantilever beam (TDCB) test.

Based on the studies conducted by Copeland (2007), Kanitpong and Bahia (2003), Kanitpong and Bahia (2004), Kanitpong and Bahia (2005) and Marek and Herrin (1968), mode of failure between bitumen and aggregates subjected to axial tensile load (i.e. pull off (tension) mode) was found to be more the cohesive rather than the mixed cohesive and adhesive or entirely adhesive. This is true for both unconditioned specimens and moisture conditioned specimens. Thickness of adhesive layer of bitumen, deformation rate, test temperature and penetration grade of bitumen were found to have a profound influence on the maximum tensile bond strength (i.e. maximum tensile load per unit area of contact) and the types of failure (adhesive or cohesive). Marek and Herrin (1968) suggested that for the occurrence of the adhesive mode of failure, high deformation rate and low test temperature must be utilised.

CHAPTER 3

RESEARCH METHODOLOGY

This chapter provides the background on research methodology and experimental procedures involved in the study. The whole concept of this study was designed in order to develop and establish a laboratory adhesion test method for direct measurement of the adhesive bond strength of bitumen (bitumen-filler mastic) and aggregates, and thus to quantify the adhesive bond strength and failure characteristics of various combinations of asphalt mixture materials over wide ranges of testing conditions. Since the adhesion between bitumen (bitumen-filler mastic) and aggregates is considered as one of the main fundamental properties of the asphalt mixtures and as there is no established testing techniques and procedures that can be used to quantify the adhesive bond strength between bitumen (bitumen-filler mastic) and aggregates, research in this area is crucial and evidently needed. Also, in order to determine the effect of various variables and parameters on the test results, to propose suitable testing conditions and to validate the reliability and efficiency of the proposed adhesion test method, adhesive bond strength and failure characteristics of various combinations of asphalt mixture materials over wide ranges of testing conditions need to be accessed.

The general concept of this study is illustrated in Figure 3.1. This study was divided into three parts based on the specific objectives outlined in Chapter 1 and is described in the following sections.

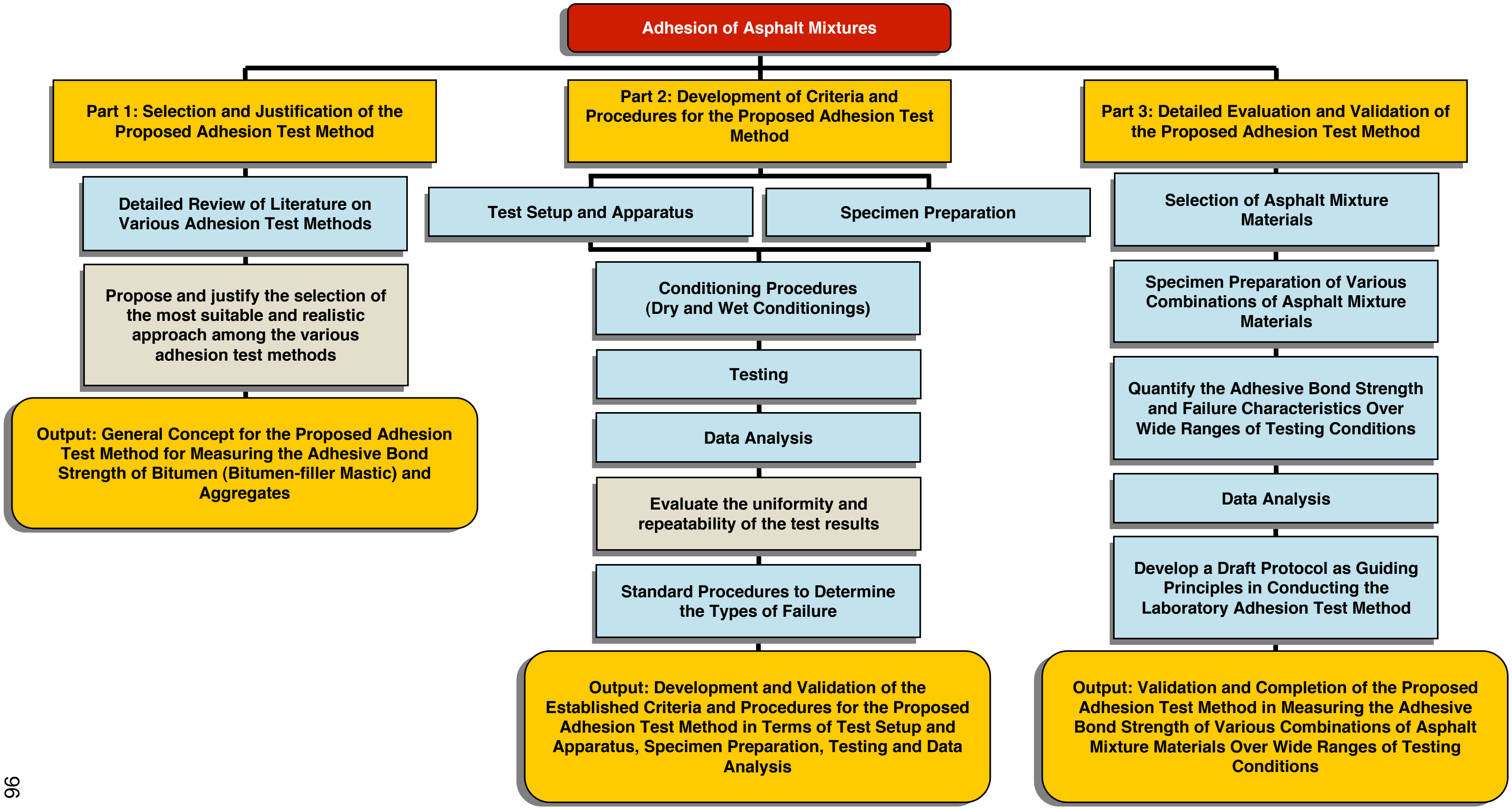


Figure 3.1 Flow chart for general concept of the study

3.1 Part 1: Selection and Justification of the Proposed Adhesion Test Method

In this part, a detailed review of literature on various testing techniques and procedures used to measure the adhesive bond strength in numerous areas of scientific literature and international standards which has been presented in Chapter 2, was assessed in order to select and propose the most suitable and realistic approach for development of laboratory adhesion test method for measuring the adhesive bond strength of bitumen (bitumen-filler mastic) and aggregates. Among the testing techniques and procedures that have been taken into consideration are peel test, pull off test, double cantilever beam (DCB) test and tapered double cantilever beam (TDCB) test. Since there is no established testing techniques and procedures for direct measurement of the adhesive bond strength of bitumen (bitumen-filler mastic) and aggregates in the pavement related areas, this part is considered as crucial and evidently needed. The right selection of the approach is regarded as highly important as it will become the key success for this study.

Justification for the selection of the most suitable and realistic approach was presented in order to support the decision making process. Several factors such as simplicity, practicality, ease of specimen preparation and cost effectiveness of the test setup and apparatus will be taken into account in making the selection. Since the proposed testing techniques and procedures were considered from the various fields of study, which are mostly related to the testing of the adhesive bond strength of coatings of composite materials such as plastic, metals and glasses, modifications of the proposed testing techniques and procedures were expected, in order to suit the asphalt mixtures. Therefore, other factors such as availability of suitable testing equipment in the pavement related areas which is capable to conduct test similar to the proposed testing techniques and procedures and require only minimum modifications on the existing equipments and parts, and compatibility of the proposed testing techniques and procedures with asphalt mixtures need to be taken into consideration too. Also, ability to allow for various conditioning procedures (dry and wet conditionings) must be included as criteria for the selection. At the end of this part, a general concept for the proposed adhesion test method will be developed.

Further details of the proposed adhesion test method, which has been selected and considered as the most suitable and realistic approach, and justification that has been taken into account for the selection will be discussed in Chapter 4. Details of the experimental design for this part are illustrated in Figure 3.2.

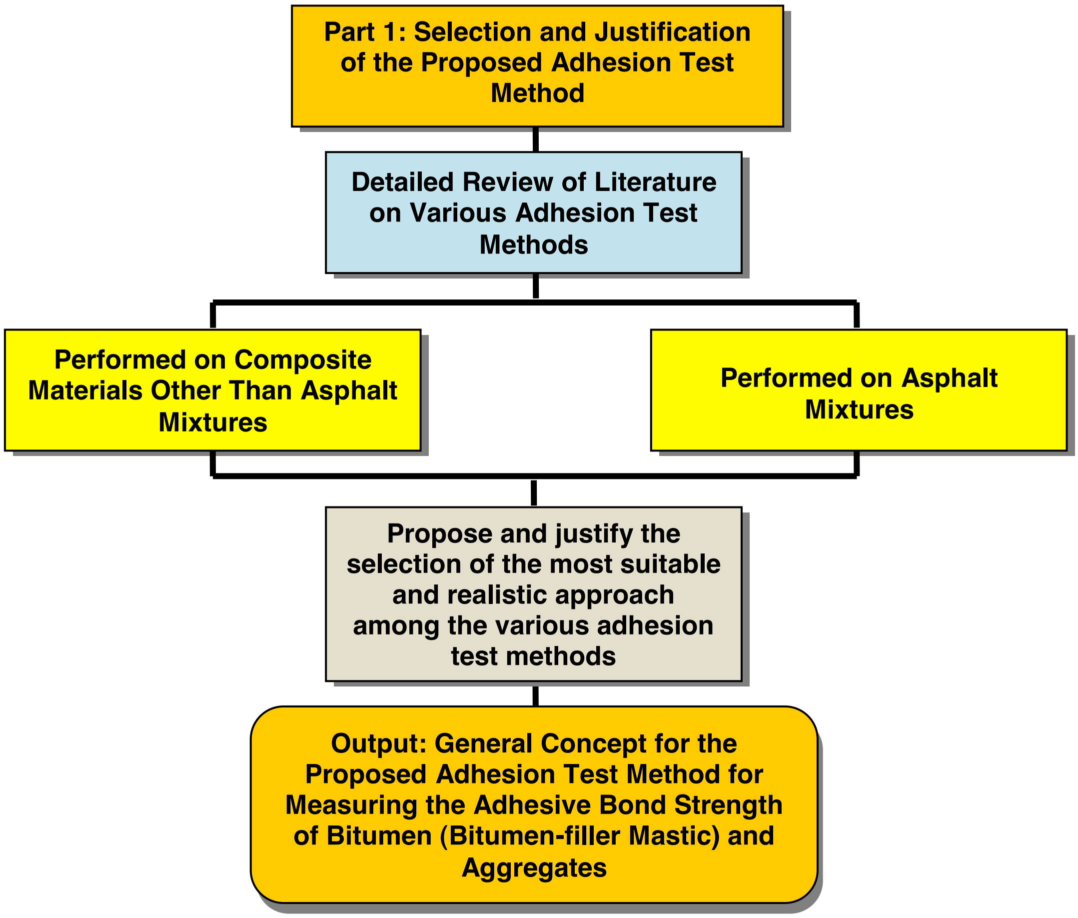


Figure 3.2 Flow chart for Part 1: Selection and Justification of the Proposed Adhesion Test Method

3.2 Part 2: Development of Criteria and Procedures for the Proposed Adhesion Test Method

In this part, a general concept for the proposed adhesion test method from the previous part was subjected to evaluation, mainly based on the trial and error experimental approach, in order to adapt and thus to establish the criteria and procedures for test setup and apparatus, specimen preparation, testing and data analysis. Since the proposed adhesion test method was considered from the various fields of study, which are mainly related to the testing of the adhesive bond strength of coatings of composite materials for aerospace and automotive industries, modifications of the proposed adhesion test method were expected, in order to suit the asphalt mixtures. However, modifications should be conducted based on the following basis; development of the proposed adhesion test method should be as close as possible to the original adaptation of the general concept especially in terms of the fundamental approaches and principles, and at the same time being compatible with asphalt mixtures and existing testing equipments in the pavement related areas. Hence, factors such as availability of suitable testing equipment in the pavement related areas, which is capable to conduct test similar to the original adaptation of the general concept and require only minimum modifications need to be taken into account for the selection. The reasons for the inclusion of these factors are made in order to optimally utilise the existing equipments and parts without the need to introduce the new one (i.e. equipments and parts), and also to allow for the results of the proposed adhesion test method to be directly correlated with the existing data from the currently available tests.

This part will focus on four main sections; test setup and apparatus, specimen preparation, testing and data analysis. In order to achieve the adhesive mode of failure, the procedures for specimen preparation were designed so that the thickness of adhesive layer of bitumen is uniform and as thin as possible. The initial value of the thickness of bitumen (bitumen-filler mastic) films is suggested to be 800 μm (0.800 mm) or less, although based on the results of the micromechanics analysis conducted by Lytton et al. (2005), the thickness of adhesive layer of bitumen of less than 60 μm (0.060 mm) is required to produce the adhesive mode of failure. The initial value of 800 μm (0.800 mm) has been set by taking into consideration the nature of bitumen (bitumen-filler mastic), inaccuracy of the measurements that will result due to the small value of thickness and the procedures involved in the specimen preparation. The value of the thickness of bitumen (bitumen-filler mastic) films will be reduced accordingly in order to result in the adhesive mode of failure, and the final value will be determined at the end of this part. Hence, the ability of the specimen to cater for various thicknesses of the adhesive layer of bitumen (bitumen-filler mastic) should be taken into consideration. Although in the actual pavement structure the thickness of adhesive layer of bitumen (bitumen-filler mastic) coatings the aggregates vary considerably and both adhesive and cohesive failure could occur in the asphalt mixtures, this study focused only on the adhesive interaction between bitumen (bitumen-filler mastic) and aggregates. In this part, conventional 70/100 penetration grade of bitumen was used as adhesive materials and substrates were aluminium alloy, acts as control substrates. The emphasis on using aggregates as substrates is not regarded as highly important until established criteria and procedures for the proposed adhesion test method have been developed.

Development of the test setup and apparatus and testing was conducted based on the following considerations; ability of the test setup and apparatus to uniformly distribute the applied loads throughout the coated surface of the specimen and thus to produce adhesive mode of failure and capability on conducting the adhesion test method over wide ranges of testing conditions (i.e. deformation rates and test temperatures). Based on the literature review and analysis of the past studies, deformation rate and test temperature are among the variables that would contribute to the types of failure (adhesive or cohesive) (Copeland 2007; Marek & Herrin 1968). Therefore, the proposed adhesion test method should be able to measure the adhesive bond strength of various combinations of asphalt mixture materials over wide ranges of testing conditions (i.e. deformation rates and test temperatures). Also, standard procedures to determine the types of failure of the specimen as either adhesive or cohesive were established. The simplest and easiest method to differentiate between the adhesive and cohesive failure is via visual observation and calculation based on the percentage area of adhesive and cohesive failure, which is the most commonly used approach in various adhesion test methods.

In the last section of Part 2: Development of Criteria and Procedures for the Proposed Adhesion Test Method, data analysis will be conducted in order to determine the uniformity and repeatability of the test results in terms of thickness of adhesive layer of bitumen, total percentage area of adhesive failure, maximum tensile bond strength and tensile energy required to produce failure per unit volume. At the end of this part, established criteria and procedures for the proposed adhesion test method in terms of test setup and apparatus, specimen preparation, testing and data analysis will be developed. Since the development of the criteria and procedures for the proposed adhesion test method is mainly based on the trial and error experimental approach, the possibility of not meeting the expected requirements in any of the four main sections as listed above is high. Therefore, modifications are highly expected and may even require for a total change to a new approach for the adhesion test method. Details of the experimental design for this part are illustrated in Figure 3.3.

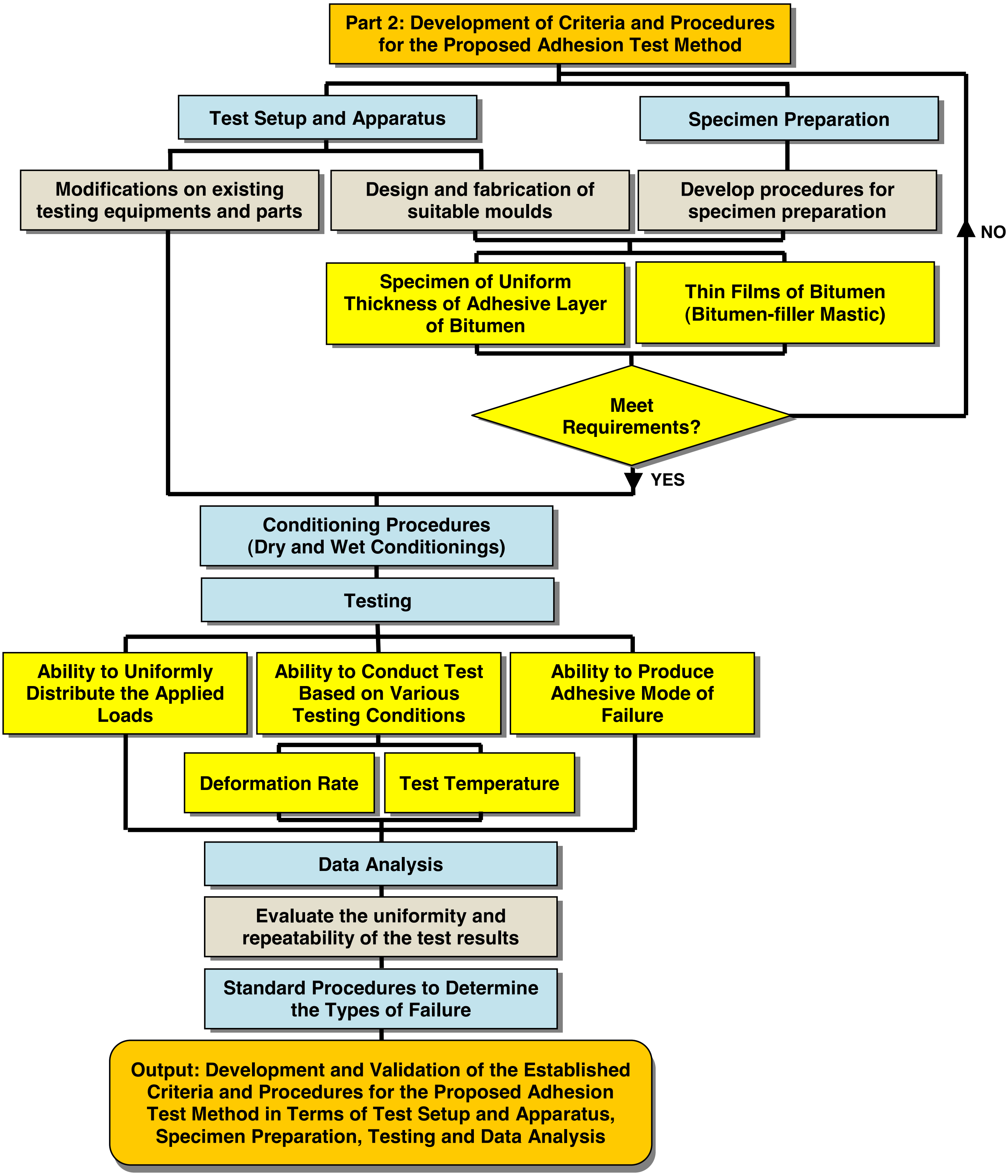


Figure 3.3 Flow chart for Part 2: Development of Criteria and Procedures for the Proposed Adhesion Test Method

3.3 Part 3: Detailed Evaluation and Validation of the Proposed Adhesion Test Method

This part is a continuation from the previous part where the established criteria and procedures for the proposed adhesion test method in terms of test setup and apparatus, specimen preparation, testing and data analysis will be subjected to further evaluation in quantifying the test results (i.e. thickness of adhesive layer of bitumen, total percentage area of adhesive failure, maximum tensile bond strength and tensile energy required to produce failure per unit volume) of various combinations of asphalt mixture materials (i.e. bitumen (bitumen-filler mastic) and aggregates) over wide ranges of thicknesses of adhesive layer of bitumen, aspect ratio of specimens, testing conditions (i.e. deformation rates and test temperatures) and conditioning procedures (dry and wet conditionings).

In the previous part, the development of criteria and procedures for the proposed adhesion test method was conducted generally without emphasis on using various combinations of asphalt mixture materials (i.e. bitumen (bitumen-filler mastic) and aggregates) and various conditioning procedures (dry and wet conditionings). Therefore, in this part, various combinations of asphalt mixture materials (i.e. bitumen (bitumen-filler mastic) and aggregates) were used in order to determine the effect of various variables and parameters on the test results, to propose suitable testing conditions and to validate the reliability and efficiency of the laboratory adhesion test method. In order to consider wide ranges of asphalt mixture materials, at least two types of aggregates and/or bitumen (bitumen-filler mastic) of distinct properties that will reflect the ranges of typically used asphalt mixtures need to be utilised. Aluminium alloy (control substrates), granite and two types of limestone were used as substrates, and conventional 70/100 penetration grade of bitumen was used as control adhesive materials. Various types of mineral filler (i.e. hydrated lime, limestone and gritstone filler) were used in the study in order to produce various types of bitumen-filler mastic. Aluminium alloy was selected as control substrates due to the value of Young's Modulus of approximately 70 GPa, which is close to the typical value of aggregates and also due to the corrosion resistance properties (Harvey 2000). Aggregates (i.e. Dene Limestone, Ivonbrook Limestone and Mount Sorrel Granite) were selected due to availability and distinct properties in terms of the classification as acidic (i.e. hydrophilic) or basic (i.e. hydrophobic).

Testing of the adhesive bond strength of various combinations of asphalt mixture materials (i.e. bitumen (bitumen-filler mastic) and aggregates) was conducted over wide ranges of thicknesses of adhesive layer of bitumen, aspect ratio of specimens, testing conditions (i.e. deformation rates and test temperatures) and conditioning procedures (dry and wet conditionings). Since the conditioning procedures are very subjective and there is no rule of thumb that can be reliably applied to simulate the conditioning process in the actual pavement structure, the following conditioning procedures were used; specimens were subjected to dry or wet conditionings at 25°C for 24 hours prior to testing. Total conditioning time of 24 hours was found to be the optimum time required for the development of the full adhesive bond strength between bitumen and aggregates, based on the study conducted in Chapter 5. Also, 25°C was selected as the test temperature for the conditioning procedures for the purpose of comparative analysis with the previous studies of Copeland (2007), Kanitpong and Bahia (2003) and Marek and Herrin (1968).

The selection of testing conditions (i.e. deformation rates and test temperatures) was conducted based on the following assumptions; deformation rate should not be too high in order to prevent any discrepancy and inaccuracy of the test results, and also should not be too low to result in cobwebbing (multiple strings or strands and limited necking of the thin films of bitumen), and the selection of test temperatures is suggested to be approximately within the ranges of the average pavement temperature (i.e. between 10°C and 60°C).

Results of the study will be subjected to comparative analysis in order to determine the effect of various variables and parameters on the test results, to propose suitable testing conditions and to validate the reliability and efficiency of the proposed adhesion test method. Also, at the end of this part, a draft protocol will be developed as guiding principles in conducting the laboratory adhesion test method. The final output for this part, which is also the final output for the whole study, is the validation and completion of the proposed adhesion test method in measuring the adhesive bond strength of various combinations of asphalt mixture materials (i.e. bitumen (bitumen-filler mastic) and aggregates) over wide ranges of testing conditions. Details of the experimental design for this part are illustrated in Figure 3.4.

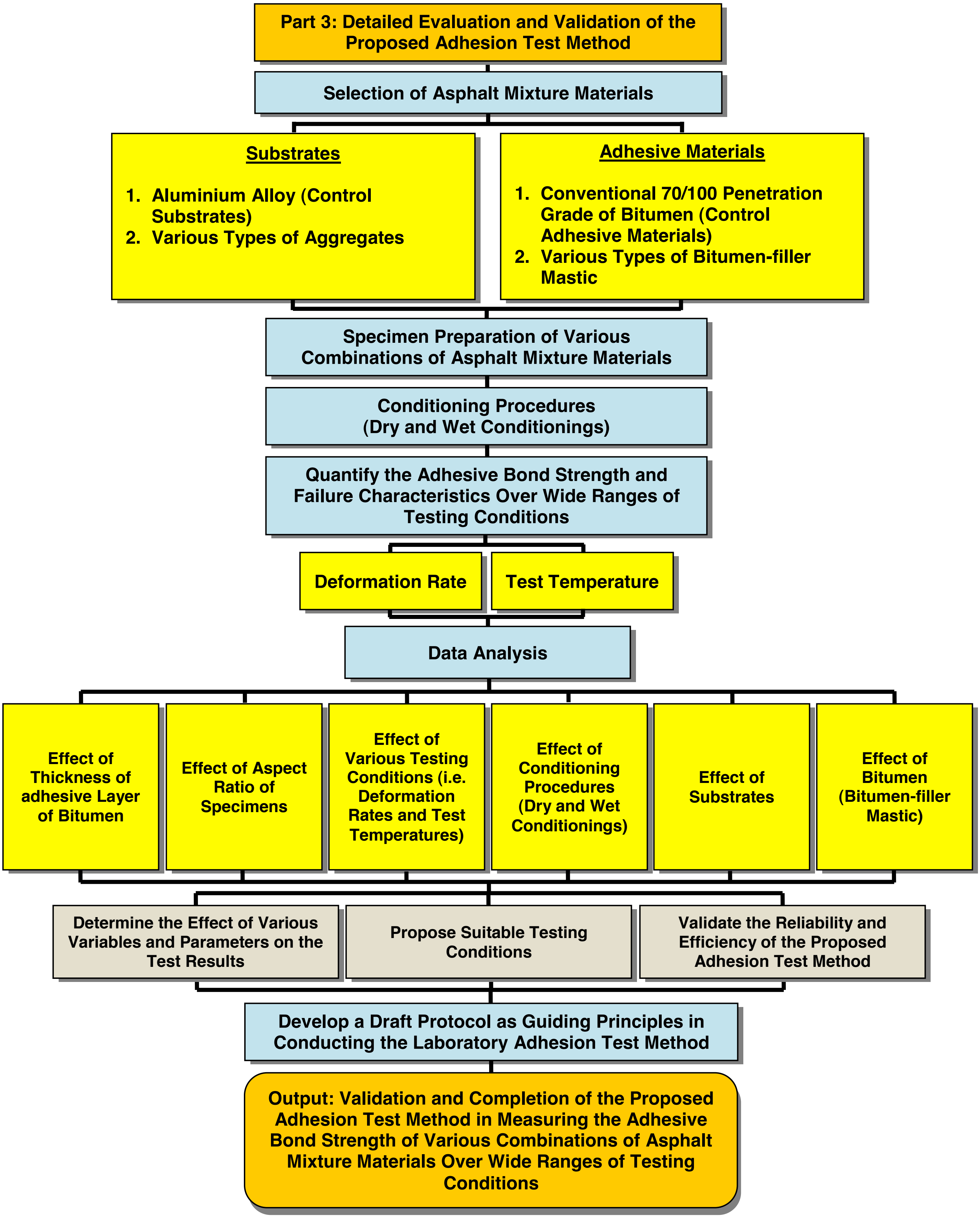


Figure 3.4 Flow chart for Part 3: Detailed Evaluation and Validation of the Proposed Adhesion Test Method

CHAPTER 4

PART 1: SELECTION AND JUSTIFICATION OF THE PROPOSED ADHESION TEST METHOD

4.1 General Background

Various testing techniques and procedures used to measure the adhesive bond strength in numerous areas of scientific literature and international standards were reviewed in Chapter 2. Among the testing techniques and procedures that have been taken into consideration are peel test, pull off test, double cantilever beam (DCB) test and tapered double cantilever beam (TDCB) test. In this part, out of all the testing techniques and procedures that have been reviewed, the most suitable and realistic approach will be selected for development of laboratory adhesion test method in measuring the adhesive bond strength of bitumen (bitumen-filler mastic) and aggregates. Justification for the selection of the most suitable and realistic approach was presented at the end of this part, in order to support the decision making process. Since there is no established testing techniques and procedures for direct measurement of the adhesive bond strength of bitumen (bitumen-filler mastic) and aggregates in the pavement related areas, this part is considered as crucial and evidently needed. The right selection of the approach is regarded as highly important as it will become the key success for this study.

4.2 Selection of the Proposed Adhesion Test Method

Based on the testing techniques and procedures that have been reviewed in Chapter 2, the approaches that seem to be suitable and promising for development of laboratory adhesion test method for asphalt mixtures were summarised in Table 4.1. Blister test was not included since a study conducted by Anderson et al. (1994) has shown the complexity and ineffectiveness of the blister test.

Table 4.1 Summary of various adhesion test methods

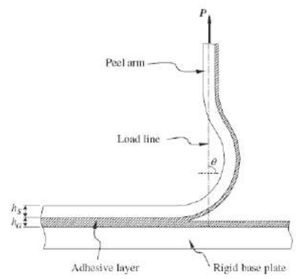
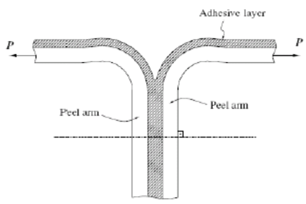
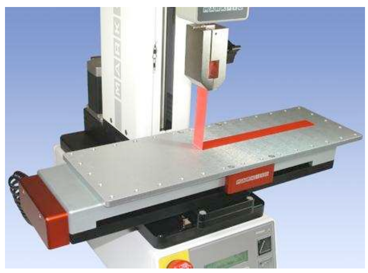
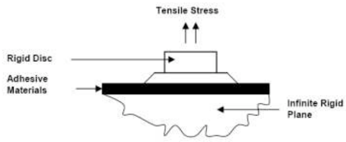
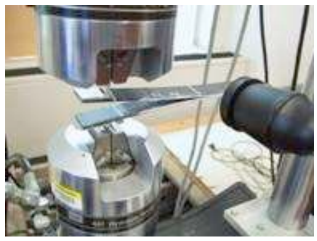
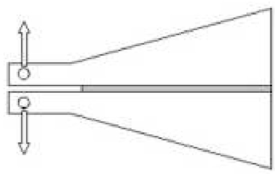
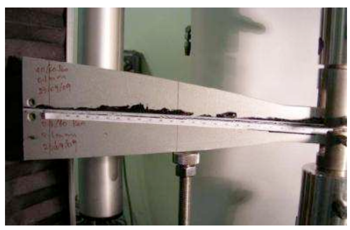
Adhesion test Methods	Brief Descriptions	Figures
Peel Test	• Simple adhesion test method conducted by peeling a thin flexible adhesively bonded peel arm at a specified angle and deformation rate. • Typically used to measure the adhesive bond strength of bonded joints and laminates of composite materials in aerospace and automotive industries. • Based upon Elastic-Plastic-Fracture-Mechanics (EPFM) approach. • Various standards have been developed (differ in the way that the load is applied but remain the same in the basic principles). • Useful for predicting the initial capability of adhesive materials. • Resulted in complex deformation behaviour of the tested specimens (i.e. cannot measure the fundamental aspect of adhesion between adhesive layer and substrates, and cannot directly assess the cohesive strength of adhesive materials). • Results are dependent on various factors such as peel angle, thickness and mechanical properties of peel arm, deformation rate and test temperature. • Results usually reflect the stress and strain of specimens failing under conditions of extensive yielding. • Include a large degree of plastic deformation of peel arm.	 **Fixed Arm**  **T Peel** 

Table 4.1 Summary of various adhesion test methods (continued)

Adhesion test Methods	Brief Descriptions	Figures
Pull Off Test	• Conducted by measuring the tensile stress required to detach or fracture the coatings of adhesive materials in direction perpendicular to substrates. • Typically used to measure the mechanical tensile strength of paint films, varnishes, mortars and concretes. • Various testing techniques and procedures have been developed. • Useful in comparing properties and providing relative ratings of different types of adhesive materials. • Can be applied using wide ranges of substrates (i.e. metals, plastics, woods and aggregates). • Results are influenced by the procedures for specimen preparation, deformation rate and test temperature. • Possible problems are due to unevenly distributed tensile stress throughout the coated surfaces.	 
Double Cantilever Beam (DCB) Test	• Conducted by pulling apart two beams of identical substrates with layer of adhesive materials in between, at certain deformation rate. • A crack is initiated first by inserting a wedge into the adhesive materials (i.e. presence of flaws). • Typically used to measure the fracture resistance of adhesive and bonded joints of a thin adhesively bonded fibre composite materials under mode I tensile loading conditions. • Based upon Linear-Elastic-Fracture-Mechanics (LEFM) approach. • Results need to be analysed using several different approaches such as Corrected Beam Theory (CBT), Experimental Compliance Method (ECM) and ASTM Method.	 
Tapered Double Cantilever Beam (TDCB) Test	• TDCB test is similar to the DCB test in terms of basic principles and procedures for specimen preparation, except the two beams of identical substrates are tapered away from the point where the load is applied. • Results are independent of crack length values. • Useful for measuring tough adhesive materials without the occurrence of plastic deformation of the arms. • Can be used to determine the rate of crack growth under various cyclic loading and environmental conditions. • Based upon Linear-Elastic-Fracture-Mechanics (LEFM) approach. • Relatively complex and expensive in terms of specimen preparation.	 

Based on the summarised data in Table 4.1 and considerations of the factors such as ease of specimen preparation, cost effectiveness of test setup and apparatus, availability of suitable testing equipment and compatibility with asphalt mixtures, adhesion test method based on the pull off (tension) mode was found to be the most suitable and realistic approach for development of laboratory adhesion test method for asphalt mixtures.

4.3 Justification for the Selection of the Proposed Adhesion Test Method

Adhesion test method based on the pull off (tension) mode presents a simple, practical and reliable approach in determining the adhesive bond strength of bitumen (bitumen-filler mastic) and aggregates, especially in terms of the procedures for specimen preparation and testing. Also, the adhesion test method based on the pull off (tension) mode can be applied using wide ranges of substrates including metals, plastics, woods, concretes and aggregates. Hence, the application of the non aggregates materials as substrates can be applied during the preliminary study, before being replaced by aggregates once the established criteria and procedures for the proposed adhesion test method have been developed. The reason for this is due to the simplicity and cost effectiveness of the non aggregates materials compared to the aggregates.

Availability of suitable testing equipment in pavement related areas such as INSTRON servo hydraulic frame and Ductilometer testing apparatus, which are capable of applying loads in tension and compression, and also capable of conducting tests over wide ranges of deformation rates and test temperatures is also one of the reasons for the selection of the pull off (tension) mode.

INSTRON servo hydraulic frame has been used before as standard testing equipment for measuring the adhesive bond strength of multi layer specimens of asphalt mixtures, as published in the in-house standard of LOP 9.28 Tensile Bond Testing (TBT) Laboratory Operations Procedures-Test Methods/Testing (NTEC 2007) (Appendix A). However, the measured adhesive bond strength is only based on the interaction between multi layer specimen, rather than the individual components of the asphalt mixture materials (i.e. bitumen (bitumen-filler mastic) and aggregates).

Ductilometer testing apparatus is generally used to measure the tensile properties of bitumen in a temperature controlled water bath. The following testing conditions are available for the Ductilometer testing apparatus.

1. Ranges of deformation rates are 2.5 mm/minute and 140 mm/minute.

2. Ranges of test temperatures of the temperature controlled water bath are -10°C and 60°C.

3. Ranges of tensile load that can be recorded are 1 N and 300 N with an accuracy of ± 0.1 N.

Figures 4.1 and 4.2 show the INSTRON servo hydraulic frame and Ductilometer testing apparatus respectively.

Figure 4.1 INSTRON servo hydraulic frame

Figure 4.2 Ductilometer testing apparatus

Pull off (tension) mode has been selected by Kanitpong and Bahia (2003) and Marek and Herrin (1968) as the preferred mode in measuring the adhesive bond strength of asphalt mixtures, and has been described in detail in Chapter 2. Based on Kanitpong and Bahia (2003), the advantages of using pull off (tension) mode are due to the ability to condition the specimens in water and applications of various types of aggregates as substrates. However, the developed adhesion test methods of Kanitpong and Bahia (2003) and Marek and Herrin (1968) are not yet established due to poor repeatability of the test results and limitations in terms of the applicability to measure the adhesive bond strength of wide ranges of asphalt mixture materials under various testing conditions. Study conducted by Kanitpong and Bahia (2003) is still being carried out.

Based on Harvey (2000), combinations of traffic loads, which create shear stress on the pavement surface, and pavement cracks were found to result in mode I tensile loading conditions, as illustrated in Figure 4.3. Also, by considering two aggregates coated with thin films of bitumen and adhered to each other as shown in Figure 4.4 (a), the bond between bitumen and aggregates can thus be idealised as a butt joint (Figure 4.4 (b)). Failure for the bonded butt joint can occur adhesively between bitumen and aggregates, cohesively within bitumen, or mixed of adhesive and cohesive. The adhesive bond strength measured based on the pull off (tension) mode will be representative of the maximum tensile load that the bonded bitumen and aggregates can sustain until failure or fracture occurs.

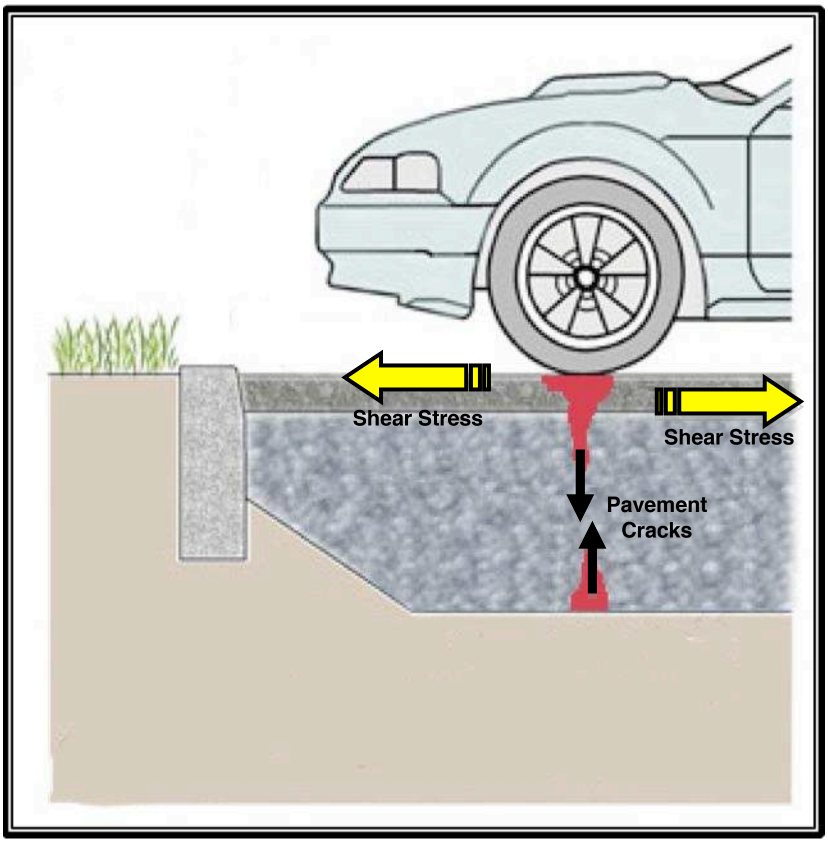


Figure 4.3 Mode I tensile loading conditions due to combinations of traffic loads and pavement cracks

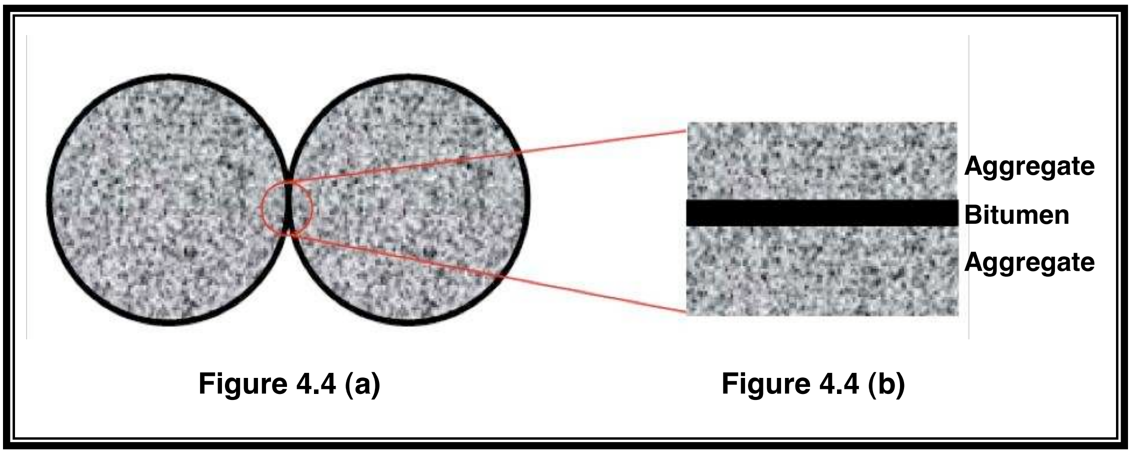


Figure 4.4 Idealised butt joint of bitumen and aggregates (Source: Copeland 2007)

4.4 Conclusions

Based on the summarised data in Table 4.1 and the aforementioned justifications, the pull off (tension) mode has been identified and selected as the most suitable and realistic approach for the development of laboratory adhesion test method in measuring the adhesive bond strength of bitumen (bitumen-filler mastic) and aggregates. Pull off (tension) mode seems to be the best approach to describe the adhesive bond strength and failure characteristic of the asphalt mixtures. Although there were adhesion test methods based on the pull off (tension) mode that have been developed by Kanitpong and Bahia (2003) and Marek and Herrin (1968), the development of the proposed adhesion test method in this study will start from scratch, rather than continuing the work that had been done. One of the reasons is due to the in-house testing equipment of Kanitpong and Bahia (2003) and Marek and Herrin (1968) that are not widely available.

In the next part, the general concept based on the pull off (tension) mode will be subjected to evaluation, mainly based on the trial and error experimental approach. Development of criteria and procedures for the proposed adhesion test method will be conducted based on the consideration of both INSTRON servo hydraulic frame and Ductilometer testing apparatus, and the most suitable and practical testing equipment will be selected upon completion of the next part.

CHAPTER 5

PART 2: DEVELOPMENT OF CRITERIA AND PROCEDURES FOR THE PROPOSED ADHESION TEST METHOD

5.1 General Background

In this part, a general concept for the proposed adhesion test method based on the pull off (tension) mode, which has been identified as the most suitable and realistic approach in the previous part was subjected to evaluation, mainly based on the trial and error experimental approach. The main objective of this part was to establish the criteria and procedures for the proposed adhesion test method in terms of test setup and apparatus, specimen preparation, testing and data analysis, and then to evaluate the uniformity and repeatability of the test results in terms of thickness of adhesive layer of bitumen, total percentage area of adhesive failure, maximum tensile bond strength and tensile energy required to produce failure per unit volume, in order to validate the established criteria and procedures. Since there is no established adhesion test method based on the pull off (tension) mode for asphalt mixtures and the development of the criteria and procedures is mainly based on the trial and error experimental approach, modifications are highly expected.

Based on the literature review and analysis of the past studies, there are various testing techniques and procedures that can be applied to conduct the adhesion test method based on the pull off (tension) mode, as have been described by British Standard Institution (1999), British Standard Institution (2003), Copeland (2007), DFD® Instruments (n.d.), Kanitpong and Bahia

(2003), Kanitpong and Bahia (2004), Kanitpong and Bahia (2005), Kendall (1971) and Marek and Herrin (1968). In this part, the INSTRON servo hydraulic frame and Ductilometer testing apparatus have been selected as possible testing equipments based on the consideration of the following factors; simplicity, practicality, availability, compatibility with asphalt mixtures and ability to conduct the adhesion test method based on the pull off (tension) mode over wide ranges of deformation rates and test temperatures. Throughout this part, development of criteria and procedures for the proposed adhesion test method was conducted based on the consideration of both the INSTRON servo hydraulic frame and Ductilometer testing apparatus, and the most suitable and practical testing equipment will be selected upon the completion of this part. Details of the INSTRON servo hydraulic frame and Ductilometer testing apparatus were given in the Chapter 4.

In order to simplify the analysis until the established criteria and procedures have been developed, the following variables and parameters were fixed throughout this part.

1. Conventional 70/100 penetration grade of bitumen was used as adhesive material.

2. Aluminium alloy was used as control substrates.

3. Specimens were subjected to dry conditioning for 24 hours prior to testing.

In this part, both of the INSTRON servo hydraulic frame and Ductilometer testing apparatus were subjected to preliminary and subsequent study.

Preliminary study was conducted based on the trial and error experimental approach in order to achieve a general overview on the development of criteria and procedures for the proposed adhesion test method, by taking several possible assumptions into consideration. The emphasis of the preliminary study is focused on the uniformity and repeatability of the test results. The thickness of adhesive layer of bitumen of 800 μm (0.800 mm) or less has been set for the preliminary study.

Results of the preliminary study were then used as a point of reference in the subsequent study in order to finalise the value of the thickness of adhesive layer of bitumen that will result in the adhesive mode of failure. Also, adhesion test method was conducted over wide ranges of testing conditions (i.e. deformation rates and test temperatures) in order to generally observe the effect on the test results. At the end of this part, the selection of the most suitable and practical testing equipment between the INSTRON servo hydraulic frame and Ductilometer testing apparatus was made before progressing into the next part.

5.2 Preliminary Study Using INSTRON Servo Hydraulic Frame

This section focuses on the initial development of criteria and procedures for the proposed adhesion test method based on the INSTRON servo hydraulic frame. Laboratory work was divided into two parts, which focused on specimen preparation and testing. However, design and fabrication of suitable moulds and testing rig (i.e. test setup and apparatus) were required in the first place, in order to suit the INSTRON servo hydraulic frame. At the end of this part, data analysis was conducted in order to evaluate the uniformity and repeatability of the test results.

5.2.1 Test Setup and Apparatus

Pair of aluminium alloy plates measuring an area of 100 mm by 100 mm with thickness of 16 mm for each plate as shown in Figure 5.1, was used as control substrates. Each pair of plates was labelled in order to provide consistency in pairing and thus accuracy in determining the uniformity and repeatability of the test results. In order to determine the thickness of adhesive layer of bitumen, the combined thickness of the top and bottom of each pair of plates was measured and recorded. Prior to the specimen preparation, surface of the pair of plates needs to be cleaned in order to ensure the cleanliness and thus full adhesive bond strength between adhesive layer of bitumen and substrates. Chemical solution such as white spirit solvent can be used to clean the surface of the pair of plates.

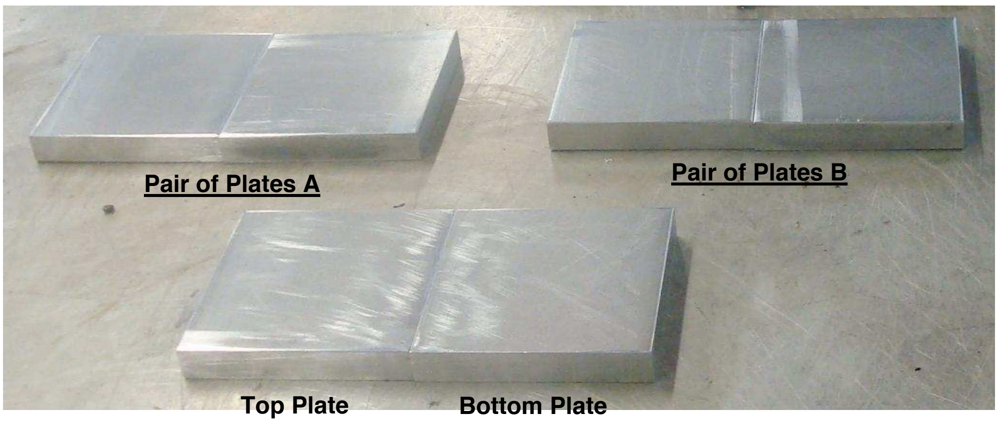


Figure 5.1 Pair of aluminium alloy plates

Design and fabrication of the testing rig was suggested to be rigid in order to ensure equal axial tensile load distribution onto the specimens and thus to result in failure of the tested specimens based on the pull off (tension) mode only (i.e. to exclude the effect of peel and shear mode). The rigid testing rig consisted of two parts; top plate with two vertical hollow rods and base plate with two vertical solid rods, which can be slide into each other as shown in Figure 5.2. The arrangement of the rigid testing rig will thus ensure equal axial tensile load distribution onto the specimens by preventing any lateral movement of either the tested specimens or the top and base plate of the rigid testing rig. Also, in order to ensure the evenly distributed axial tensile load, universal joint attachment has been used to attach the rigid testing rig to the upper part of the INSTRON servo hydraulic frame and thus to transfer the load in perpendicular direction onto the specimens. Carver clamps have been used to secure the rigid testing rig to the lower part of the INSTRON servo hydraulic frame. Two Linear Variable Differential Transducer (LVDT) have been used to measure the vertical pull off displacement of the specimens. The captured data of the vertical pull off displacement and tensile load was analysed via the built-in software of the INSTRON servo hydraulic frame. The test setup and apparatus using the rigid testing rig is shown in Figure 5.3.

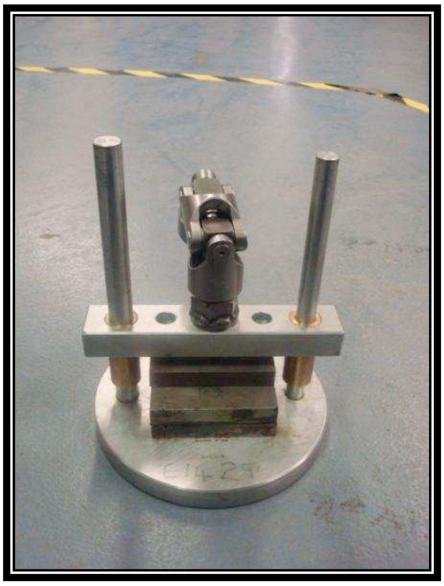


Figure 5.2 Rigid testing rig

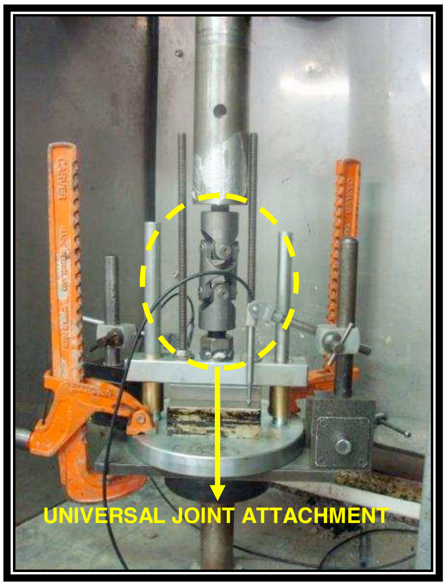


Figure 5.3 Test setup and apparatus

5.2.2 Specimen Preparation

Procedures for specimen preparation were designed with the emphasis on the uniformity and repeatability of the thickness of adhesive layer of bitumen. In this preliminary study, the thickness of adhesive layer of bitumen has been set to 800 μm (0.800 mm). In order to achieve the required thickness of adhesive layer of bitumen, approach using volume-density calculations was suggested in order to determine the amount of bitumen required for specimen preparation, as shown in Equation 5.1. However, after several trials of specimen preparation, this approach was found to be tedious, not practical and time consuming. The amount of the bitumen required needs to be carefully poured and weighed, and thus allows for the temperature of the bitumen to drop drastically to room temperature. Also, by pouring and measuring the correct amount of bitumen, there is no guarantee that fully coated area of contact between bitumen and substrates can be achieved.

Equation 5.1

$$Bulk\ Density\ of\ Bitumen\ (g/cm^3) = (Specific\ Gravity\ of\ Bitumen) \times \left(Bulk\ Density\ of\ Water\ (g/cm^3)\right)$$

$$Weight\ of\ Bitumen\ (g) = \left(Bulk\ Density\ of\ Bitumen\ (g/cm^3)\right) \times \left[\left(Area\ of\ Contact\ (cm^2)\right) \times \left(Required\ Thickness\ of\ Bitumen\ (cm)\right)\right]$$

Therefore, it is suggested that the bitumen be poured until fully coated surface of the bottom plate is achieved, followed by the placement of the top plate, and the required thickness of adhesive layer of bitumen is then achieved via compression. Hence, a method to limit the downward vertical movement of the top plate up to the required thickness of adhesive layer of bitumen is required. Youtcheff and Aurilio (1997) had suggested that glass beads be used to control the thickness of adhesive layer of bitumen in the study of the evaluation and modelling of pneumatic adhesion test. However, in the follow-up study conducted by Kanitpong and Bahia (2003) on the same pneumatic adhesion test which is later known as PATTI 110, two pieces of metal blocks have been used to control the thickness of adhesive layer of bitumen, as shown in Figure 5.4.

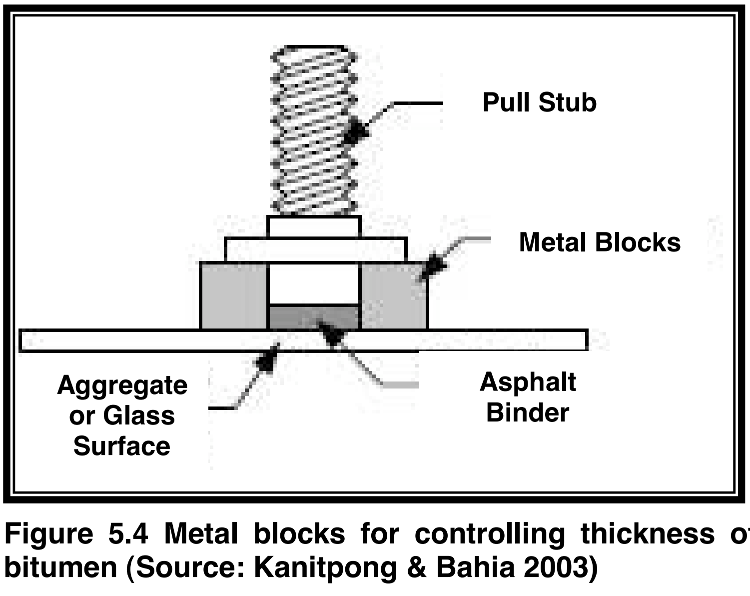


Figure 5.4 Metal blocks for controlling thickness of adhesive layer of bitumen (Source: Kanitpong & Bahia 2003)

Based on the considerations of the factors such as simplicity, practicality and ease of specimen preparation, steel balls have been suggested to limit the downward vertical movement of the top plate. Steel balls of approximately 0.794 mm (1/32 inch) diameter have been selected to provide thickness of adhesive layer of bitumen of 800 μm (0.800 mm). Selection of the 800 μm (0.800 mm) as the required thickness was made based on the accuracy of micrometer that can measure up to 10 μm (0.010 mm) only.

Several methods have been suggested and implemented in order to place the steel balls on the bottom plate, which includes welding of the steel balls, distributing the steel balls randomly prior to pouring of the bitumen and attaching the steel balls either by glue or tape. The later method of attaching the steel balls has been selected due to the simplicity, consistency and flexibility. A set of four steel balls is required to be positioned at approximately 20 mm from each corner of the bottom plate, as illustrated in Figure 5.5. Double-sided tape has been used to attach the steel balls onto the bottom plate. In case of any detachment of the steel balls from the double-sided tape despite careful measures that have been taken, a distance of 20 mm from each corner is assumed to be sufficient to keep the steel balls in between the pair of plates. Also, by positioning the steel balls at fixed positions, consistency and accuracy for comparative analysis between specimens can be achieved. The effect of the presence of the steel balls and double-sided tape in the adhesive layer of bitumen was found to be minimal based on an unreported X-ray analysis of the position of the steel balls and the data of test results, and can be neglected since the proportion of the occupied area is small.

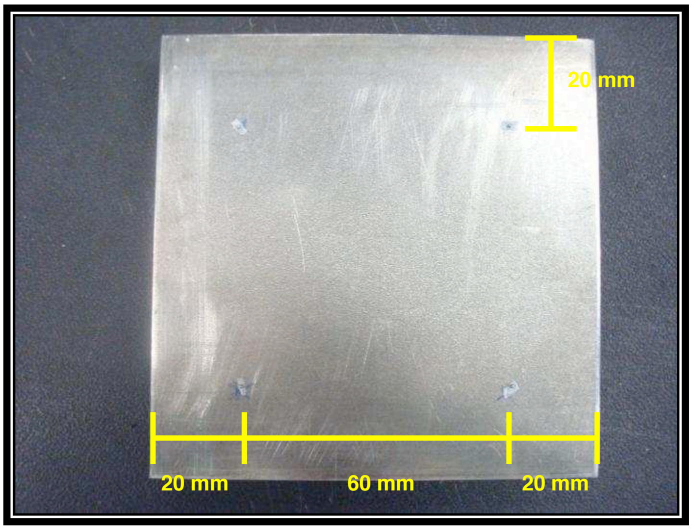


Figure 5.5 Set of four steel balls positioned at 20 mm from each corner

Square mould of approximately 105 mm by 105 mm with height of 30 mm was used to confine the pair of plates during the specimen preparation and thus prevent any lateral movement between the top and bottom plates (Figure 5.6). Grease needs to be applied on the inner surface of the mould in order to prevent sticking between the mould and the pair of plates.

Figure 5.6 Square mould to confine the pair of plates

In order to achieve the required thickness of adhesive layer of bitumen and also the full adhesive bond strength between adhesive layer of bitumen and substrates, the pair of plates need to be compressed after bitumen has been poured. Several methods have been suggested which includes using hand pump compressor and the INSTRON servo hydraulic frame. By taking into consideration the factors such as simplicity and practicality, hand pump compressor was found to be the most effective and less time consuming method. Although the downward vertical movement of the top plate has been limited due to the presence of the steel balls and thus maximum amount of load and total time required for the compression can be applied, there should be standard procedures for the compression in order to prevent any excessive loading which can result in the steel balls to be pushed out from the specimens or in the worse case, break. Standard procedures for the compression were suggested as follows; load of 6000 psi (41.37 MPa) is applied for 5 minutes before the thickness is measured using micrometer to ensure the required thickness of adhesive layer of bitumen of 800 µm (0.800 mm) has been achieved. If the required thickness of adhesive layer of bitumen is not achieved, specimen is subjected to another compression of 6000 psi (41.37 MPa) for 1 minute before being measured, and this is continued until the required thickness of adhesive layer of bitumen is achieved. A tolerance of ±50 µm (±0.050 mm) is allowed in the measurement. It is suggested that the compression can be applied up to a maximum number of four times. As the temperature of the adhesive layer of bitumen drops over time, there is an increased resistance to further compression. Further compression beyond this point is wasteful and can even be detrimental to the adhesive layer of bitumen and also to the steel balls, in some cases. Also, in order to ensure the evenly distributed load from the hand pump compressor, a steel ball has been used to transfer the load in a direction perpendicular to the specimen, as shown in Figure 5.7. The compressed pair of plates that have achieved the required thickness of adhesive layer of bitumen were then subjected to conditioning procedures prior to testing.

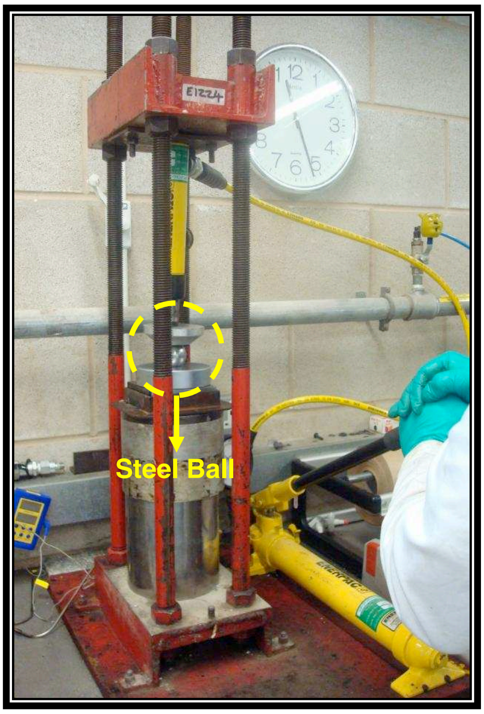


Figure 5.7 Hand pump compressor and a steel ball

5.2.2.1 Standard Procedures for Specimen Preparation based on the INSTRON Servo Hydraulic Frame

The whole procedure for the specimen preparation can be summarised as follows, and illustrated in Figure 5.8.

1. Surface of the pair of plates is cleaned by hand with chemical solution (i.e. white spirit solvent) in order to ensure the cleanliness, and then followed by acetone (ethyl acetate) in order to remove the remaining chemical solution of the white spirit solvent. (Note: Minimum rubbing should be applied to the surface in order to ensure no significant change in the properties of the surface).

2. Pair of plates is heated to approximately 80°C for at least 30 minutes in order to ensure no significant drop of temperature of bitumen during the specimen preparation.

3. A set of four steel balls of approximately 0.794 mm (1/32 inch) diameter is positioned at approximately 20 mm from each corner of the bottom plate using double-sided tape.

4. Bottom plate is positioned into the square mould of approximately 105 mm by 105 mm with height of 30 mm. Grease should be applied on the inner surface of the square mould before being introduced with the bottom plate.

5. Bitumen which has been heated to approximately 160°C for at least two hours is then poured onto the bottom plate until fully coated surface is achieved.

6. Top plate is then loaded onto the bottom plate, which has just been covered with the bitumen.

7. The confined pair of plates is then subjected to compression of load of 6000 psi (41.37 MPa) for 5 minutes via hand pump compressor.

8. The confined pair of plates is measured using micrometer to ensure the required thickness of adhesive layer of bitumen has been achieved. A tolerance of ±50 μm (±0.050 mm) has been allowed in the measurement.

9. If the required thickness of adhesive layer of bitumen is not achieved, the confined pair of plates is subjected to another compression of 6000 psi (41.37 MPa) for 1 minute before being measured, and this is continued until the required thickness of adhesive layer of bitumen is

achieved. It is suggested that the compression can be applied up to a maximum number of four times.

10. The confined pair of plates is removed from the square mould. Any excess bitumen at the edges needs to be removed before the pair of plates being introduced back into the square mould for conditioning procedures prior to testing.

1. Surface of the pair of plates is cleaned by hand with chemical solution

2. A set of four steel balls of 0.794 mm (1/32 inch) diameter is positioned using double-sided tape

3. Square mould of 105 mm by 105 mm with height of 30 mm is used to confine the pair of plates

4. Bitumen is poured onto the bottom plate and the top plate is then loaded

5. Confined pair of plates is subjected to compression via hand pump compressor

6. Excess bitumen at the edges is removed before the pair of plates being introduced back into the square mould for conditioning procedures prior to testing

Figure 5.8 Procedures for specimen preparation for preliminary study using INSTRON servo hydraulic frame

5.2.2.2 Conditioning Procedures for Preliminary Study Using INSTRON Servo Hydraulic Frame

Throughout this study, specimens were subjected to dry conditioning at 25°C for 24 hours prior to testing. Total conditioning time of 24 hours was found to be the optimum time required for the development of the full adhesive bond strength between adhesive layer of bitumen and substrates of aluminium alloy plates, based on the test results presented in Table 5.1 and also tabulated in Figure 5.9. In Table 5.1 and Figure 5.9, the adhesive bond strength was represented by the value of the maximum tensile bond strength. The following assumptions were made in determining the optimum time required for the development of the full adhesive bond strength.

Due to the application of non-porous material as substrates, and also due to the curing process of bitumen, adhesive bond strength is expected to increase with the increasing total conditioning time. This increase is expected to taper off once the completion of the curing process of bitumen has been achieved. Beyond this point, the adhesive bond strength is expected to reach steady state conditions, which can be related to the development of the full adhesive bond strength between adhesive layer of bitumen and substrates. However, in the application of aggregates as substrates, which are porous materials, and also in the presence of water or moisture in the conditioning procedures (i.e. wet conditioning), due to the curing process of bitumen, adhesive bond strength is expected to increase with the increasing total conditioning time. However, due to the effect of water or moisture, the increasing value of the adhesive bond strength is expected to taper off and thus experienced a decrease. The longer the total conditioning time, the more decrease of adhesive bond strength could be expected.

Table 5.1 Results of conditioning procedures for preliminary study using INSTRON servo hydraulic frame

Deformation Rate (mm/minute)	Test Temperature (°C)	Conditioning Time at 25°C Prior to Testing (hours)	Maximum Tensile Load (kN)	Maximum Tensile Bond Strength (kPa)
20 (0.333 mm/s)	25	6	3.60	360
			3.51	350
			4.09	410
			4.34	430
			3.96	400
			3.72	370
			4.00	400
			3.98	400
			4.10	410
			4.21	420
		12	5.81	580
			6.08	610
			6.22	620
			7.04	700
			6.71	670
			5.92	590
			5.80	580
			6.11	610
			6.73	670
			6.94	690
		24	8.01	800
			8.61	860
			8.19	820
			9.56	960
			9.99	1000
			8.72	870
			8.13	810
			7.32	730
			7.98	800
			8.60	860
		48	7.61	760
			8.13	810
			8.02	800
			7.82	780
			8.81	880
			8.02	800
			7.77	780
			7.91	790
			8.16	820
			8.43	840

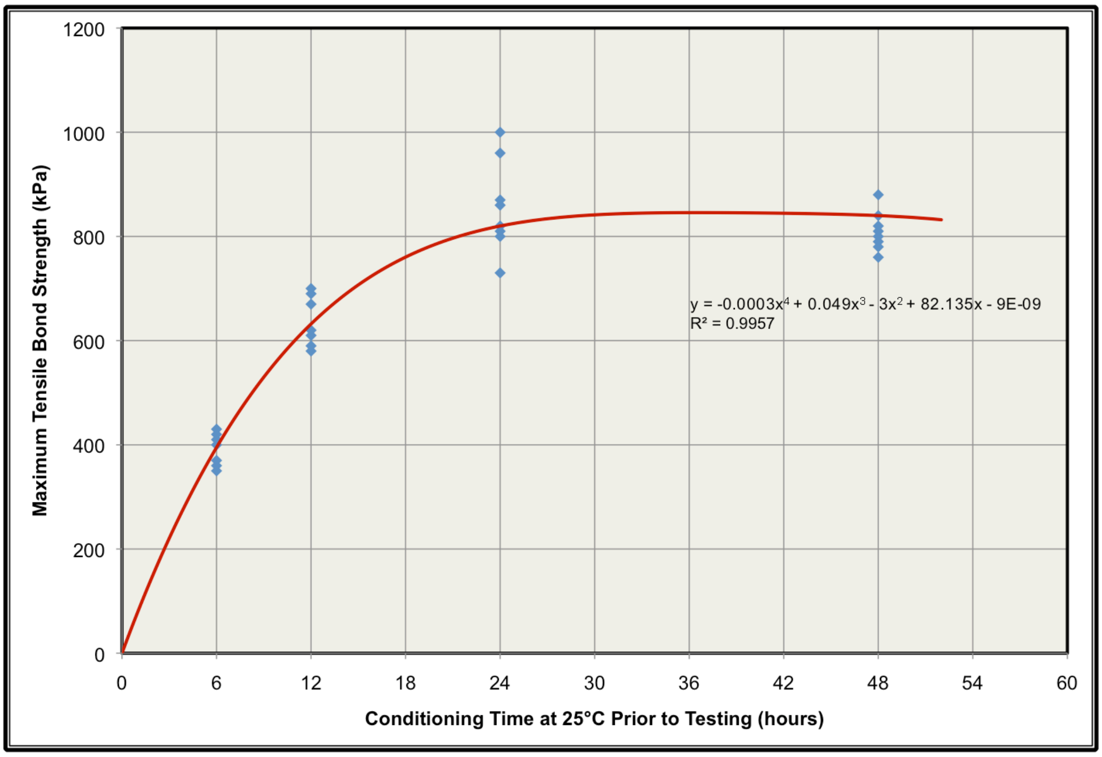


Figure 5.9 Results of conditioning procedures for preliminary study using INSTRON servo hydraulic frame

Based on the results shown in Table 5.1 and Figure 5.9, the value of the maximum tensile bond strength (i.e. maximum tensile load per unit area of contact) was found to increase as the total conditioning time was increased. However, as expected, this increase tapered off when reaching the total conditioning time of 24 hours and reaches steady state conditions which can be related to the development of the full adhesive bond strength. Hence, total conditioning time of 24 hours has been selected as the standard conditioning procedures for the preliminary study using INSTRON servo hydraulic frame based on non-aggregates and non-porous material (i.e. aluminium alloy) as substrates.

This is also supported by the study conducted by Copeland (2007). Based on the study, the optimum time required for the conditioning procedures is suggested to be greater than 8 hours but less than 24 hours. Discrepancy and inaccuracy of the test results especially in terms of the maximum tensile bond strength were found to be high as the total conditioning time exceeds 24 hours. According to Copeland (2007), total conditioning time of more than 24 hours is considered as too severe. Hence, total conditioning time of 24 hours has been concluded as the standard conditioning procedures throughout the study for both dry and wet conditionings, regardless of the substrates, and the temperature for the conditioning procedures was dependent on the test temperature.

5.2.3 Testing Using INSTRON Servo Hydraulic Frame

A total of 60 specimens was tested at a fixed deformation rate and test temperature of 20 mm/minute and 25°C respectively, in order to evaluate the uniformity and repeatability of the test results. Data of the test results in terms of thickness of adhesive layer of bitumen and total percentage area of adhesive failure, and also the calculated maximum tensile bond strength (i.e. maximum tensile load per unit area of contact) and tensile energy required to produce failure per unit volume was analysed and presented in the next section. Once the data analysis of the uniformity and repeatability of the test results has been completed, further evaluation was undertaken in the subsequent study.

5.2.4 Data Analysis of Preliminary Study Using INSTRON Servo Hydraulic Frame

In this section, data analysis was conducted in order to evaluate the uniformity and repeatability of the test results in terms of thickness of adhesive layer of bitumen, total percentage area of adhesive failure, maximum tensile bond strength (i.e. maximum tensile load per unit area of contact) and tensile energy required to produce failure per unit volume. The general procedures for analysis basically consist of performing descriptive statistics to determine the average, standard deviation and coefficient of variation (COV). Microsoft Excel and CurveExpert 1.4 were used for performing the statistical analysis and calculating the area under the curve of the graph of tensile load versus pull off displacement, which represents the tensile energy required to produce failure, respectively.

Coefficient of variation was used to evaluate and compare the variation between data sets and calculated as in Equation 5.2. A value for indicating too much variation seems to be subject dependent. Based on Math Central (n.d.), 25% or less is acceptable for the cut-off value of coefficient of variation. Since there is no exact cut-off value, a predefined cut-off threshold value of 7% was used in this study to control the consistency level of the data sets. Based on the literature found in the pavement related areas, which had used the coefficient of variation as part of the data analysis, the selection of 7% as cut-off value seems to be reasonable (Kandhal 1989; Wu & Hossain 2003; Zhang 2005).

Equation 5.2

$$\text{Coefficient of Variation (COV)} = \frac{s}{\overline{X}} \times 100\%$$

where:

s = *Standard deviation*
$\overline{X}$ = *Average*

Table 5.2 shows the data of the test results in terms of thickness of adhesive layer of bitumen, total percentage area of adhesive failure, maximum tensile bond strength (i.e. maximum tensile load per unit area of contact) and tensile energy required to produce failure per unit volume.

Table 5.2 Data of the test results (Preliminary study using INSTRON servo hydraulic frame)

Pair of Plates	Data Sets	Thickness of Adhesive Layer of Bitumen (μm)	Total Percentage Area of Adhesive Failure (%)	Maximum Tensile Bond Strength (kPa)	Tensile Energy Required to Produce Failure Per Unit Volume (kJ/m^3)
A	1	810	15	800	871
	2	100[1]	50[1]	860[1]	8140[1]
	3	800	45	820	970
	4	900[1]	15[1]	960[1]	806[1]
	5	800	15	860	1056
	6	830	15	920	1055
	7	820	15	780	1152
	8	790	30	880	1073
	9	840	25	900	852
	10	810	15	1010	906
	11	800	25	1000	1422
	12	540[1]	15[1]	840[1]	1633[1]
	13	910[1]	20[1]	520[1]	665[1]
	14	820	25	920	765
	15	830	20	740	990
	16	810	30	950	1065
	17	790	30	840	1024
	18	790	25	800	978
	19	810	20	690	742
	20	800	30	900	1168
B	1	1030[1]	15[1]	430[1]	783[1]
	2	1030[1]	20[1]	910[1]	926[1]
	3	820	5	870	1108
	4	810	35	810	634
	5	820	25	850	910
	6	830	30	930	952
	7	840	25	740	956
	8	800	35	750	972
	9	810	30	980	1149
	10	920[1]	35[1]	860[1]	981[1]
	11	840	10	730	1352
	12	830	15	800	914
	13	810	25	860	654
	14	820	15	800	904
	15	840	20	900	750
	16	800	25	830	690
	17	830	20	810	1254
	18	810	20	900	1193
	19	840	25	930	979
	20	810	15	840	1014

Table 5.2 Data of the test results (Preliminary study using INSTRON servo hydraulic frame) (continued)

Pair of Plates	Data Sets	Thickness of Adhesive Layer of Bitumen (μm)	Total Percentage Area of Adhesive Failure (%)	Maximum Tensile Bond Strength (kPa)	Tensile Energy Required to Produce Failure Per Unit Volume (kJ/m^3)
C	1	790	35	910	1016
	2	1070[1]	35[1]	640[1]	927[1]
	3	800	15	830	946
	4	820	10	970	1052
	5	810	45	860	821
	6	820	25	850	800
	7	840	25	810	987
	8	820	25	780	960
	9	1020[1]	35[1]	900[1]	1000[1]
	10	830	15	960	772
	11	830	15	770	973
	12	830	20	820	1075
	13	820	25	840	1146
	14	820	25	950	1056
	15	830	15	880	870
	16	800	15	710	917
	17	810	15	870	1311
	18	950[1]	15[1]	940[1]	952[1]
	19	820	20	730	801
	20	820	25	850	1106
Average		**816**	**23**	**851**	**982**
Standard Deviation		**15**	**8**	**77**	**171**
Coefficient of Variation (%)		**2**	**37**	**9**	**17**

Notes:
1. [1]Data is considered as outlier and not included in the calculation
2. Substrates: Aluminium alloy
3. Adhesive Materials: Conventional 70/100 penetration grade of bitumen
4. Conditioning Procedures: Dry conditioning at 25°C for 24 hours prior to testing
5. Testing Conditions: Deformation rate and test temperature of 20 mm/minute and 25°C respectively

Thickness of adhesive layer of bitumen was measured using micrometer. However, there is limitation in the measurement since the micrometer being used can only measure to the nearest 10 μm (0.010 mm). Hence, data of the thickness of adhesive layer of bitumen in this study was rounded to nearest 10 μm (0.010 mm). Standard procedures in determining the types of failure of the specimens as either adhesive or cohesive were developed based on the BS EN ISO 4624:2003 Paints and Varnishes-Pull-Off Test for Adhesion which had used the simplest, easiest and commonly used method; visual observation.

Appendix B shows the procedures in determining the types of failure of specimens subjected to the laboratory adhesion test method using INSTRON servo hydraulic frame.

Maximum tensile bond strength was calculated as follows; maximum tensile load divided by the unit area of contact. CurveExpert 1.4 has been used to calculate the area under the curve of graph of tensile load versus pull off displacement, which represents the tensile energy required to produce failure. Figure 5.10 shows the example of the graphs of tensile load versus pull off displacement of the original and corrected curve of the specimen tested using Ductilometer testing apparatus. The same graphs were plotted for the specimens tested using INSTRON servo hydraulic frame. The curvature of the initial part of the original curve was attributed to the initial seating and adjustment of the apparatus (i.e. moulds and testing rig) and also testing equipments. Hence, corrected curve is required in order to eliminate these effects. For the corrected curve, correction was determined by projecting the linear portion of the curve (i.e. positive slope) to the pull off displacement axis and horizontal shift based on the distance between the intersection and the origin was then applied. Based on Harvey and Cebon (2005), the value of the tensile energy required to produce failure can be best described by the area under the curve of graph of tensile load versus pull off displacement divided by the unit volume of the adhesive layer of bitumen (i.e. tensile energy required to produce failure per unit volume). Any variation in the thickness of adhesive layer of bitumen, which might affect the test results, will be taken into account when using the definition of the tensile energy required to produce failure per unit volume.

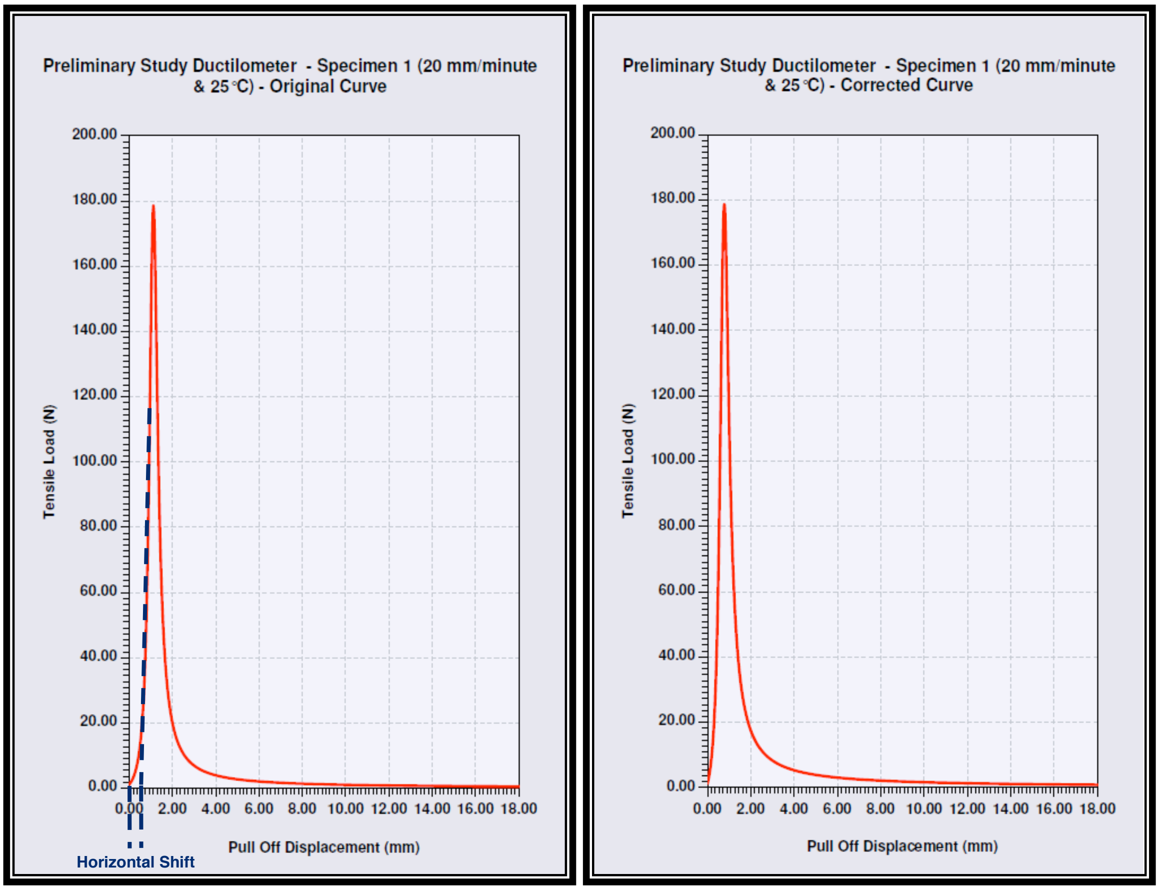


Figure 5.10 Graphs of tensile load versus pull off displacement

5.2.4.1 Analysis of Thickness of Adhesive Layer of Bitumen

The pair of the compressed plates was designed to result in the thickness of adhesive layer of bitumen of 800 μm (0.800 mm) with a tolerance of ±50 μm (±0.050 mm). Analysis of thickness of adhesive layer of bitumen is basically a sensitivity study to determine the uniformity and repeatability between data sets and also to determine if significant difference exists between the measured and the theoretical thickness of 800 μm (0.800 mm). Based on Table 5.2, thickness of adhesive layer of bitumen that lies outside the tolerance of ±50 μm (±0.050 mm) was considered as outlier and thus not included in the analysis. The average, standard deviation and coefficient of variation of the data sets were 816 μm (0.816 mm), 15 and 2% respectively. Small percentage of coefficient of variation of 2% as compared to the predefined cut-off value of 7% indicates the excellent uniformity and repeatability of the thickness of adhesive layer of bitumen between data sets.

The average, standard deviation and coefficient of variation of the thickness of adhesive layer of bitumen within the individual pair of plates (i.e. pair of plates A, B and C) were as follows; 809 μm (0.809 mm), 15 and 2% for pair of plates A, 821 μm (0.821 mm), 14 and 2% for pair of plates B and 818 μm (0.818 mm), 13 and 2% for pair of plates C. Also, in terms of the uniformity of the average thickness of adhesive layer of bitumen between the individual pair of plates (i.e. pair of plates A, B and C), the standard deviation of 6 and the small percentage of coefficient of variation of 1% indicated the excellent uniformity. Therefore, it can be concluded that there is no significant difference in the thickness of adhesive layer of bitumen within and between the individual pair of plates (i.e. pair of plates A, B and C).

Analysis was then required to determine whether there is evidence of significant difference in the average measured thickness of adhesive layer of bitumen from the theoretical value of 800 μm (0.800 mm). A hypothesis test involving One-Sample t-Test procedure, at level of significance, α of 0.05, was conducted based on the data of the measured thickness of adhesive layer of bitumen in Table 5.2. Data of the measured thickness of adhesive layer of bitumen was assumed to be normally distributed.

The following hypotheses were then established.

1. The null hypothesis, H_0

   H_0: $\overline{X}$ = 800 μm (0.800 mm)

   The average measured thickness of adhesive layer of bitumen is 800 μm (0.800 mm).

2. The alternative hypothesis, H_1

   H_1: $\overline{X} \neq$ 800 μm (0.800 mm)

   The average measured thickness of adhesive layer of bitumen is not 800 μm (0.800 mm).

The alternative hypothesis, H_1 is a non-directional or two-tailed which answers the question of interest; whether there is evidence of significant difference in the average measured thickness of adhesive layer of bitumen from the theoretical value of 800 μm (0.800 mm). For a given sample size, n of 50, the test statistic follows a t-distribution with (50-1) degrees of freedom. Based on MINITAB statistical analysis, the t-statistic, T was found to be 7.90 (Figure 5.11). The decision rule for rejecting H_0 based on the p-value approach is as follows.

Reject H_0 if p-value is smaller than level of significance, α.

Otherwise, fail to reject H_0.

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Welcome to Minitab, press F1 for help.

One-Sample T: (Measured Thickness of Adhesive Layer of Bitumen)

Test of mu = 800 vs mu not = 800

Variable	N	Mean	StDev	SE Mean
(Meas. Thick.)	50	816.40	14.675	2.075

Variable	95.0% CI	T	P
(Meas. Thick.)	(12.23, 20.57)	7.90	0.000

Figure 5.11 MINITAB statistical analysis

Therefore, based on the value of t-statistic, T of 7.90 and also p-value of 0.000, the null hypothesis, H_0 is rejected at the level of significance, α of 0.05. The analysis shows that significant statistical difference exists in the average measured thickness of adhesive layer of bitumen from the theoretical value of 800 μm (0.800 mm). The average measured thickness of adhesive layer of bitumen was 816 μm (0.816 mm), in contrast to the theoretical value of 800 μm (0.800 mm). Hence, improved procedures for specimen preparation is required in order to achieve the thickness as close as possible to the required thickness of adhesive layer of bitumen (i.e. theoretical value). The problem can be attributed to the high resistance of the adhesive layer of bitumen to compression. As the thickness of adhesive layer of bitumen is relatively high, the rate of the temperature drops over time is increased; hence the increased resistance to compression.

5.2.4.2 Analysis of Total Percentage Area of Adhesive Failure

Types of failure (adhesive or cohesive failure) were determined via visual observation of the top and bottom of each pair of plates, and then calculated based on the percentage area of adhesive failure, as shown in Appendix B.

Figure 5.12 shows the top and bottom of a pair of plates after being subjected to testing.

Top Plate Bottom Plate

Figure 5.12 Top and bottom of pair of plates after being subjected to testing

Based on Table 5.2, the mode of failure can be classified as cohesive due to small value of the average total percentage area of adhesive failure of 23%. The total percentage area of adhesive failure was in the range of 5% and 45%, which is too low to be considered as sufficient for the occurrence of the adhesive mode of failure. In this study, adhesive mode of failure was characterised by the value of the total percentage area of adhesive failure of more than 90%.

Large percentage of coefficient of variation of 37% as compared to the predefined cut-off value of 7% indicated the high variability or distribution of the total percentage area of adhesive failure, which can be attributed to the large area of contact between bitumen and substrates. Also, the average, standard deviation and coefficient of variation of the total percentage area of adhesive failure within the individual pair of plates (i.e. pair of plates A, B and C) were as follows; 24%, 8 and 35% for pair of plates A, 22%, 8 and 38% for pair of plates B and 22%, 9 and 40% for pair of plates C. Based on Harrison and Harrison (1972) in the study of adhesion using finite element analysis, axial tensile load distribution was found to be uniform up to a radius of five times the thickness of bitumen films from edges of the specimen. Holownia (1972) has shown that for specimen with lower value of Poisson's ratio of adhesive materials (i.e. 0.49 or less), the axial tensile load distribution at the centre was less parabolic and more uniform. However, since the area of contact between bitumen and substrates in this study is too large (i.e. 10,000 mm^2), the axial tensile load distribution was assumed to be non-uniform and hence resulted in high variability or distribution of the total percentage area of adhesive failure. Also, large area of contact between bitumen and substrates was found to exhibit cavitations as shown in Figure 5.13. The occurrence of the cavitations has been related to the presence of the foreign particles such as dust or mineral matter on the surface of the pair of plates and also in the bitumen, and entrapment of air voids during specimen preparation. Hence, large area of contact will increase the probability for the occurrence of the cavitations.

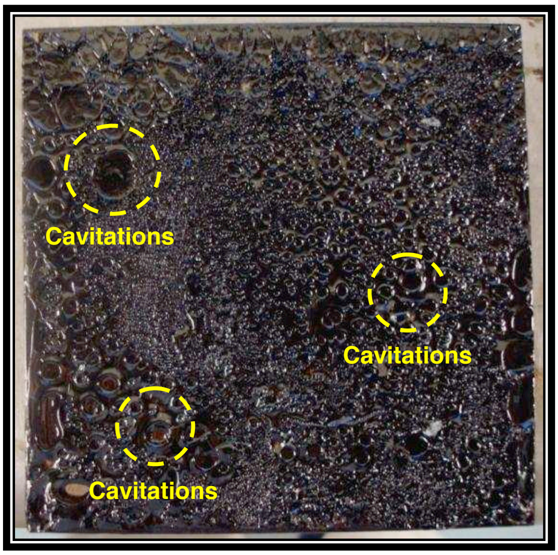

Figure 5.13 Cavitations

Results of the total percentage area of adhesive failure have been grouped as in Table 5.3, and a plot has been deduced as shown in Figure 5.14. Based on Figure 5.14, data of the total percentage area of adhesive failure was found to be skewed to the right, which indicates the cohesive mode of failure. Based on the study conducted by Kanitpong and Bahia (2003), most of the failures for the unconditioned specimens (i.e. dry conditioning) were cohesive. Also, based on the study conducted by Lytton et al. (2005), cohesive failure is expected to occur for the thickness of adhesive layer of bitumen of more than 60 μm (0.060 mm). In this section, both of the assumptions made by Kanitpong and Bahia (2003) and Lytton et al. (2005) for the occurrence of the cohesive mode of failure were applied.

Table 5.3 Results based on grouped total percentage area of adhesive failure

Total Percentage Area of Adhesive Failure (%)	Number of Specimens, n	Percentage (%)
0	0	0
5	1	2
10	2	4
15	14	28
20	7	14
25	15	30
30	6	12
35	3	6
40	2	4
45	0	0
50	0	0
55	0	0
60	0	0
65	0	0
70	0	0
75	0	0
80	0	0
85	0	0
90	0	0
95	0	0
100	0	0

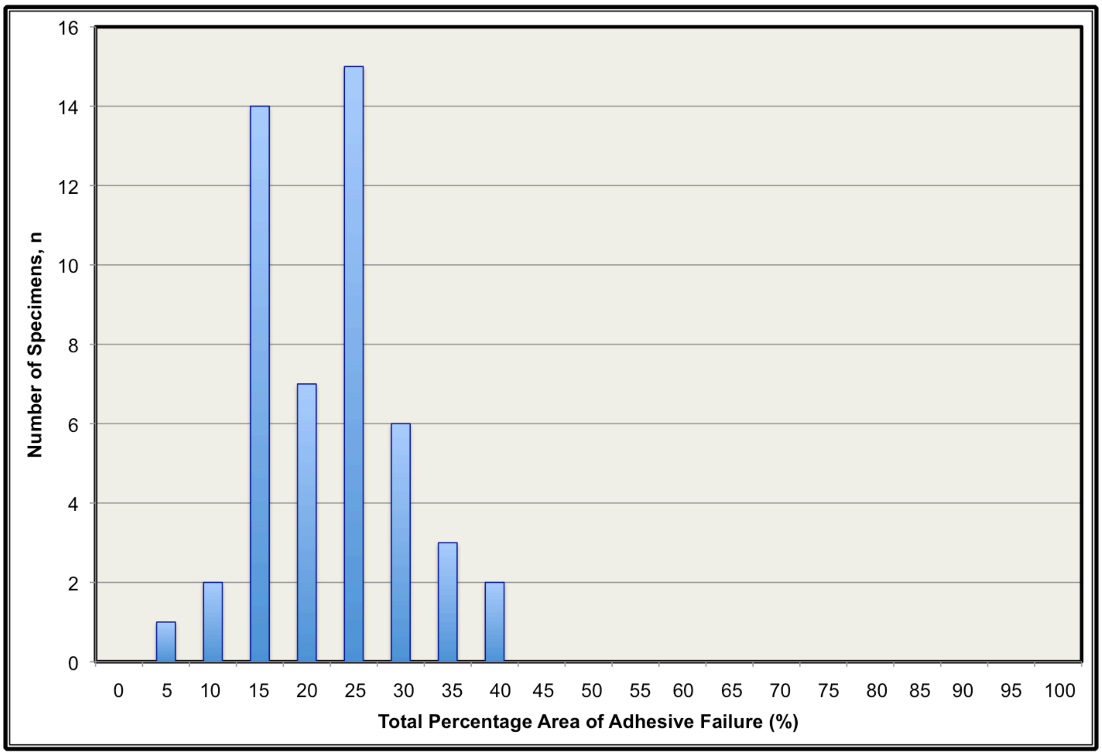

Figure 5.14 Histogram of total percentage area of adhesive failure

Cobwebbing (multiple strings or strands and limited necking of the thin films of bitumen) was found to occur during the testing, as shown in Figure 5.15. Cobwebbing is a common paint films and coatings term used to describe the spider web effect caused by the tendency of the premature drying of paint films and coatings to forms strings or strands on the substrates. The occurrence of the cobwebbing can be related to the high thickness of adhesive layer of bitumen, as compared to the suggested value of 60 μm (0.060 mm) by Lytton et al. (2005).

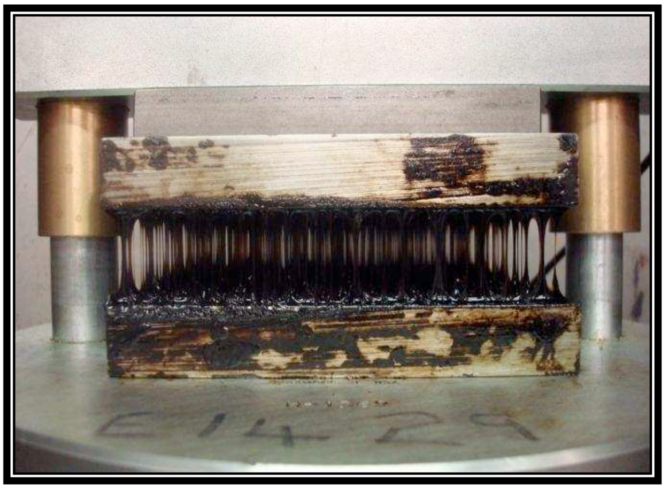

Figure 5.15 Cobwebbing

5.2.4.3 Analysis of Maximum Tensile Bond Strength

Based on Table 5.2, the value of the maximum tensile bond strength (i.e. maximum tensile load per unit area of contact) for each pair of plates was in the range of 690 kPa and 1010 kPa, with values for average, standard deviation and coefficient of variation of 851 kPa, 77 and 9% respectively. Also, the average, standard deviation and coefficient of variation of the maximum tensile bond strength within the individual pair of plates (i.e. pair of plates A, B and C) were as follows; 863 kPa, 89 and 10% for pair of plates A, 843 kPa, 71 and 8% for pair of plates B and 846 kPa, 75 and 9% for pair of plates C. A slightly large percentage of coefficient of variation as compared to the predefined cut-off value of 7%, indicates the high variability or distribution of the measured maximum tensile bond strength. This was further verified based on the spread of the distribution of the data sets for the measured maximum tensile bond strength (i.e. within the range of approximately 650 kPa and 1050 kPa), as shown in Figure 5.16. High variability or distribution of the measured maximum tensile bond strength was attributed to the large area of contact between bitumen and substrates, and hence to the non-uniform of the axial tensile load distribution, and also the occurrence of cavitations.

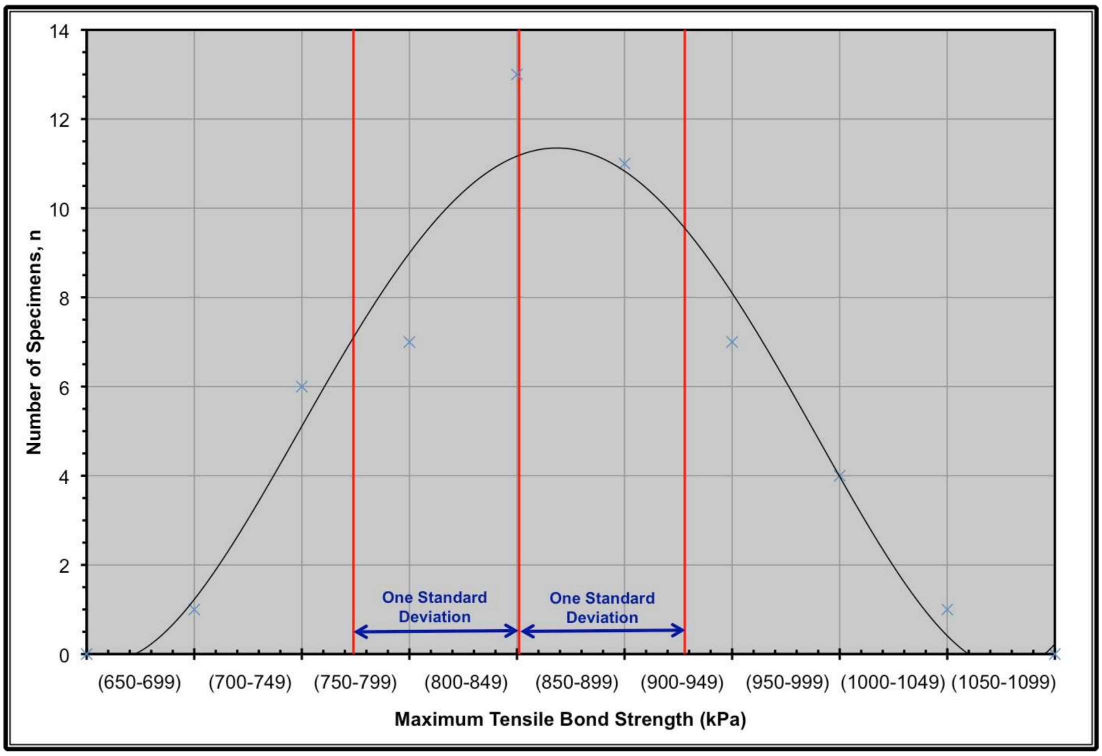

Figure 5.16 Distribution of maximum tensile bond strength

The average value of the maximum tensile bond strength (i.e. 851 kPa) was in the range of the expected value, based on the test results of the adhesion test method conducted using PATTI 110, as shown in Table 2.5 (Kanitpong & Bahia 2003). Based on the study conducted by Kanitpong and Bahia (2003), the average value of 1982 kPa was obtained under the following conditions; glass was used as substrates, bitumen grade PG 58-28 was used as adhesive materials and specimens were subjected to dry conditioning at 25°C for 24 hours prior to testing. However, it should be noted that the thickness of adhesive layer of bitumen in the study conducted by Kanitpong and Bahia (2003) was 200 μm (0.200 mm), thus justify the differences. For the conventional 70/100 penetration grade of bitumen with thickness of 800 μm (0.800 mm), a lower value of maximum tensile bond strength compared to the 1982 kPa is expected.

5.2.4.4 Analysis of Tensile Energy Required to Produce Failure Per Unit Volume

As has been stated before, tensile energy required to produce failure per unit volume was calculated based on the area under the curve of graph of tensile load versus pull off displacement divided by the unit volume of the adhesive layer of bitumen. CurveExpert 1.4 has been used for computation of the area under the curve of graph of tensile load versus pull off displacement. Hence, the values of the tensile energy required to produce failure per unit volume is subjected to estimation errors due to the curve fitting procedures (i.e. uncertainty that presents in a curve that is fitted to the data sets). Also, since the tensile energy required to produce failure per unit volume is governed by the tensile load and pull off displacement, and also indirectly by the thickness of adhesive layer of bitumen and total percentage area of adhesive failure, the variation between data sets is expected to be high. Even a small in the value of the governed parameters can lead to significant variation of the tensile energy required to produce failure per unit volume between data sets.

Based on Table 5.2, the average, standard deviation and coefficient of variation of the data sets for the tensile energy required to produce failure per unit volume were 982 kJ/m^3, 171 and 17% respectively. The average, standard deviation and coefficient of variation within the individual pair of plates (i.e. pair of plates A, B and C) were as follows; 1006 kJ/m^3, 167 and 17% for pair of plates A, 964 kJ/m^3, 206 and 21% for pair of plates B and 977 kJ/m^3, 142 and 15% for pair of plates C. Large percentage of coefficient of variation as compared to the predefined cut-off value of 7% indicates the high variability or distribution of the measured tensile energy required to produce failure per unit volume. Also, based on Figure 5.17, the values of the data sets tend to be flatter and more spread out.

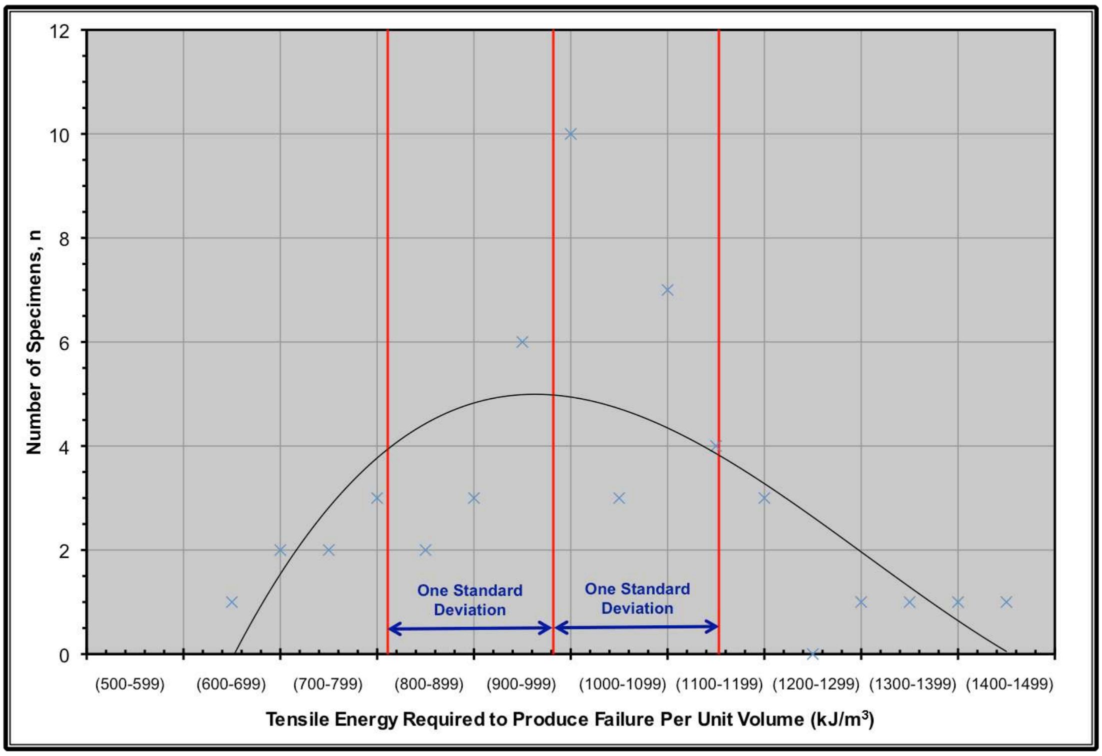

Figure 5.17 Distribution of tensile energy required to produce failure per unit volume

5.2.5 Summary of Preliminary Study Using INSTRON Servo Hydraulic Frame

In this section, initial development of criteria and procedures for the proposed adhesion test method based on the INSTRON servo hydraulic frame was conducted. Total conditioning time of 24 hours was found to be the optimum time required for the conditioning procedures, and this is also supported by the study conducted by Copeland (2007). Although data of the thickness of adhesive layer of bitumen was found to result in excellent uniformity and repeatability between data sets, the average measured thickness of 816 µm (0.816 mm) has shown significant statistical difference from the theoretical value of 800 μm (0.800 mm). Mode of failure for the tested specimens can be classified as cohesive due to the small value of the average total percentage area of adhesive failure of 23%. The occurrence of the cohesive mode of failure can be attributed to the conditioning procedures (i.e. dry conditioning) and the high thickness of adhesive layer of bitumen. Also, high variability or distribution of the measured parameters can be attributed to the large area of contact between bitumen and substrates (i.e. 10,000 mm^2) and the occurrence of the cobwebbing. However, the average value of the maximum tensile bond strength (i.e. 851 kPa) was in the range of the expected value, based on the study conducted by Kanitpong & Bahia (2003).

5.3 Subsequent Study Using INSTRON Servo Hydraulic Frame

In this section, results of the preliminary study were used as point of reference in order to refine the criteria and procedures for the proposed adhesion test method based on the INSTRON servo hydraulic frame. The following recommendations were suggested in order to finalise the value of the thickness of adhesive layer of bitumen that will result in the adhesive mode of failure and at the same time maintaining the uniformity and repeatability of the test results.

1. Reduce the thickness of adhesive layer of bitumen

   In the preliminary study, high thickness of adhesive layer of bitumen of 800 μm (0.800 mm) was found to result in high resistance to compression during the specimen preparation and hence, result in the significant statistical difference between the average measured thickness of adhesive layer of bitumen and the theoretical value of 800 μm (0.800 mm). Also the mode of failure was found to be cohesive with the occurrence of cobwebbing. Hence, several values of thickness of adhesive layer of bitumen have been randomly prepared and tested in order to find the optimum value that can result in the adhesive mode of failure and at the same time maintaining the uniformity and repeatability of the test results. Based on Marek and Herrin (1968), too low thickness of adhesive layer of bitumen could compromise the precision in the measurements, and even a very small difference of thickness of adhesive layer of bitumen between data sets could result in significant difference in the test results. Conclusion has been made that the optimum thickness of adhesive layer of bitumen that can result in the adhesive mode of failure and at the same time maintaining the uniformity and repeatability of the test results was 50 μm (0.050 mm).

2. Reduce the area of contact between bitumen and substrates

Large area of contact between bitumen and substrates was found to exhibit cavitations and the occurrence of cobwebbing during testing, and the most importantly the non-uniform distribution of the axial tensile load. Area of contact between bitumen and substrates was found to result in negligible effect on the test results, as shown in the next part (i.e. Part 3: Detailed Evaluation and Validation of the Proposed Adhesion Test Method). Specimens of different area of contact between bitumen and substrates have shown to result in approximately the same value of the test results, provided having the same thickness of adhesive layer of bitumen. Hence, pair of plates used in the preliminary study was modified as illustrated in Figures 5.18 and 5.19. The modification of the pair of plates was made in order to allow for the insertion of the 25 mm diameter (i.e. 490.87 mm^2 area of contact) of the aluminium alloy discs into top and bottom plates. Circle (i.e. discs) has been selected as the preferred geometry for the area of contact between bitumen and substrates in order to eliminate the edge effect. Also, modification has been made to prevent any lateral movement between top and bottom plates by introducing four rods inserted at each corner of the pair of plates. Although considered as simple and practical, the previous setup of the pair of plates which required the positioning of the steel balls was found to be time consuming and the risk of the steel balls being pushed out from the specimens is still present.

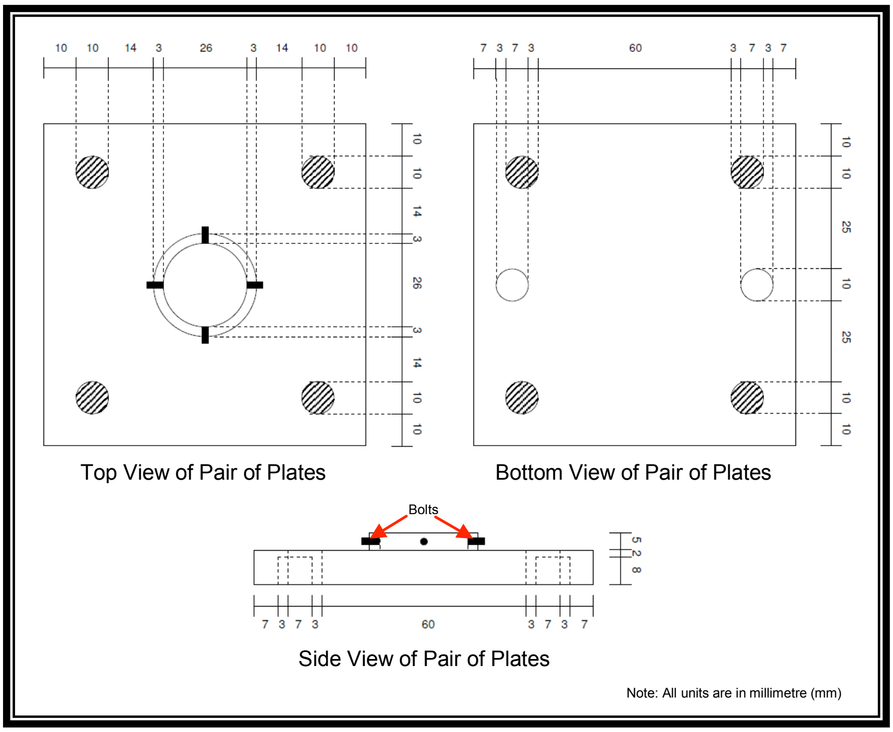

Figure 5.18 Schematic drawing of modified pair of plates

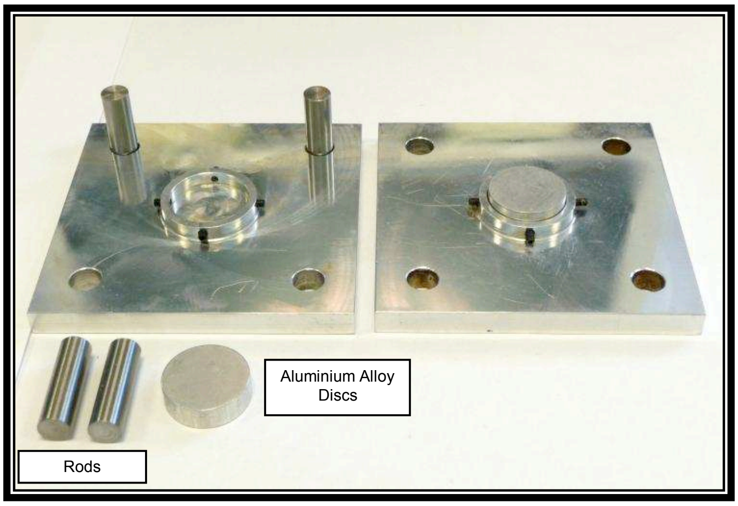

Figure 5.19 Modified pair of plates and aluminium alloy discs

Spacers have been used to control the thickness of adhesive layer of bitumen of 50 μm (0.050 mm). Instead of using hand pump compressor as in the preliminary study, a compression device as shown in Figure 5.20, has been developed and used in order to achieve the required thickness of adhesive layer of bitumen and also the full adhesive bond strength between adhesive layer of bitumen and substrates. The compression device consists of a micrometer, which can be used to compress the pair of plates up to the required thickness of adhesive layer of bitumen. Modified procedures for specimen preparation are presented in Appendix C. The same rigid testing rig and hence the same setup for the testing using the INSTRON servo hydraulic frame as in the preliminary study, were applied.

Figure 5.20 Compression device

In this section, data analysis was conducted in order to validate the value of the thickness of adhesive layer of bitumen that will result in the adhesive mode of failure and also to evaluate the uniformity and repeatability of the test results. Also, limited number of specimens was subjected to various testing conditions (i.e. deformation rates and test temperatures) in order to generally observe the effect on the test results and also to validate the capability of the proposed adhesion test method, before progressing into the next part. Table 5.4 shows the experimental matrix for the subsequent study. Specimens were subjected to dry conditioning at specified test temperature for 24 hours prior to testing. The selection of testing conditions (i.e. deformation rates and test temperatures) was made based on the literature review and analysis of the past studies. The selection of test temperatures was suggested to be approximately within the ranges of the average pavement temperature (i.e. between 10°C and 60°C).

Table 5.4 Experimental matrix for subsequent study

		Test Temperature (°C)				
		10	15	20	25	30
Deformation Rate (mm/minute)	10	5 Specimens	5 Specimens	5 Specimens	5 Specimens	5 Specimens
	20	5 Specimens	5 Specimens	5 Specimens	60 Specimens	5 Specimens
	30	5 Specimens	5 Specimens	5 Specimens	5 Specimens	5 Specimens

5.3.1 Data Analysis of Subsequent Study Using INSTRON Servo Hydraulic Frame

Data analysis of the subsequent study was conducted in the same way as that for the preliminary study, in which evaluation of the uniformity and repeatability of the test results was performed. In addition to that, analysis was also conducted in order to observe the effect of various testing conditions (i.e. deformation rates and test temperatures) on the test results. Table 5.5 shows the data of the test results of the subsequent study, in which a total of 60 specimens was subjected to testing at fixed deformation rate and test temperature of 20 mm/minute and 25°C respectively.

Table 5.5 Data of the test results (Subsequent study using INSTRON servo hydraulic frame)

Pair of Plates	Data Sets	Thickness of Adhesive Layer of Bitumen (µm)	Total Percentage Area of Adhesive Failure (%)	Maximum Tensile Bond Strength (kPa)	Tensile Energy Required to Produce Failure Per Unit Volume (kJ/m^3)
A	1	50	100	1310	2190
	2	50	100	1280	1835
	3	60	100	1340	2004
	4	50	100	1240	2011
	5	50	100	1320	1794
	6	50	90	1250	1949
	7	50	100	1350	1986
	8	50	100	1320	1838
	9	50	100	1320	2462
	10	50	100	1330	2219
	11	50	100	1310	1882
	12	50	95	1260	1602
	13	50	100	1210	1968
	14	50	100	1330	1841
	15	50	100	1340	2188
	16	50	90	1270	2135
	17	50	100	1330	1846
	18	50	100	1370	1854
	19	50	95	1320	1913
	20	50	100	1280	2347
B	1	50	100	1310	2653
	2	50	100	1280	1954
	3	50	100	1300	1626
	4	60	100	1420	2132
	5	50	100	1280	2294
	6	50	100	1290	1854
	7	50	100	1360	1721
	8	50	100	1310	2945
	9	50	100	1240	3075
	10	50	100	1360	2381
	11	50	100	1300	1827
	12	50	100	1230	1922
	13	50	100	1230	2449
	14	50	100	1350	2082
	15	50	100	1280	2842
	16	50	95	1230	2237
	17	50	100	1410	1773
	18	50	100	1390	2231
	19	50	100	1400	2949
	20	60	100	1420	2402

Table 5.5 Data of the test results (Subsequent study using INSTRON servo hydraulic frame) (continued)

Pair of Plates	Data Sets	Thickness of Adhesive Layer of Bitumen (μm)	Total Percentage Area of Adhesive Failure (%)	Maximum Tensile Bond Strength (kPa)	Tensile Energy Required to Produce Failure Per Unit Volume (kJ/m^3)
C	1	50	100	1300	2599
	2	50	100	1250	2079
	3	50	100	1260	2471
	4	50	100	1270	1699
	5	50	100	1530	2682
	6	50	100	1210	2681
	7	50	100	1270	2838
	8	50	100	1150	2115
	9	50	100	1220	1824
	10	50	100	1370	2289
	11	50	100	1280	1891
	12	50	100	1390	2024
	13	50	100	1180	1883
	14	50	100	1350	2008
	15	50	100	1160	2307
	16	50	100	1380	2350
	17	50	100	1390	2432
	18	50	100	1380	3063
	19	50	100	1320	1860
	20	50	100	1230	2360
Average		**51**	**99**	**1306**	**2178**
Standard Deviation		**2**	**2**	**70**	**373**
Coefficient of Variation (%)		**4**	**2**	**5**	**17**

Notes:
1. Substrates: Aluminium alloy
2. Adhesive Materials: Conventional 70/100 penetration grade of bitumen
3. Conditioning Procedures: Dry conditioning at 25°C for 24 hours prior to testing
4. Testing Conditions: Deformation rate and test temperature of 20 mm/minute and 25°C respectively

5.3.1.1 Analysis of Thickness of Adhesive Layer of Bitumen

In the subsequent study, the pair of plates was designed to result in the thickness of adhesive layer of bitumen of 50 μm (0.050 mm). Spacers and compression device have been used to control the thickness of adhesive layer of bitumen. Based on Table 5.5, the average measured thickness of adhesive layer of bitumen was 51 μm (0.051 mm) with the value of standard deviation and coefficient of variation of 2 and 4% respectively. The average, standard deviation and coefficient of variation of the measured thickness of adhesive layer of bitumen within the individual pair of plates (i.e. pair of plates A, B and C) were as follows; 51 μm (0.051 mm), 2 and 4% for pair of plates A, 51 μm (0.051 mm), 3 and 6% for pair of plates B and 50 μm (0.050 mm), 0 and 0% for pair of plates C. Small percentage of coefficient of variation indicates the excellent uniformity and repeatability of the thickness of adhesive layer of bitumen between data sets. Also, in terms of the uniformity of the average thickness of adhesive layer of bitumen between the individual pair of plates (i.e. pair of plates A, B and C), the standard deviation of 1 and the small percentage of coefficient of variation of 1% justified the excellent uniformity.

Analysis was then conducted in order to determine if significant difference exists between the average measured thickness of adhesive layer of bitumen of 51 μm (0.051 mm) and the theoretical value of 50 μm (0.050 mm). A hypothesis test involving One-Sample t-Test procedure, at level of significance, α of 0.05, was conducted. Data of the measured thickness of adhesive layer of bitumen was assumed to be normally distributed.

The following hypotheses were then established.

1. The null hypothesis, H_0

   H_0: $\overline{X}$ = 50 μm (0.050 mm)

   The average measured thickness of adhesive layer of bitumen is 50 μm (0.050 mm).

2. The alternative hypothesis, H_1

   H_1: $\overline{X} \neq$ 50 μm (0.050 mm)

   The average measured thickness of adhesive layer of bitumen is not 50 μm (0.050 mm).

Based on MINITAB statistical analysis, the t-statistic, T was found to be 1.76 (Figure 5.21). The decision rule for rejecting H_0 based on the p-value approach is as follows.

Reject H_0 if p-value is smaller than level of significance, α.

Otherwise, fail to reject H_0.

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One-Sample T: (Measured Thickness of Adhesive Layer of Bitumen)

Test of mu = 50 vs mu not = 50

Variable	N	Mean	StDev	SE Mean
(Meas. Thick.)	60	50.50	2.198	0.284

Variable	95.0% CI	T	P
(Meas. Thick.)	(-0.07, 1.07)	1.76	0.083

Figure 5.21 MINITAB statistical analysis

Therefore, based on the value of t-statistic, T of 1.76 and also p-value of 0.083, the null hypothesis, H_0 failed to be rejected at the level of significance, α of 0.05. Therefore, it can be concluded that there is sufficient evidence of no significant statistical difference in the average measured thickness of adhesive layer of bitumen from the theoretical value of 50 μm (0.050 mm). Based on the test results, the modified test setup and apparatus and also the procedures for specimen preparation have been proven capable of producing specimen as close as possible to the required thickness of adhesive layer of bitumen (i.e. theoretical value). The problem attributed to the high resistance of adhesive layer of bitumen to compression due to the increased rate of the temperature drops over time, was then eliminated.

5.3.1.2 Analysis of Total Percentage Area of Adhesive Failure

Figure 5.22 shows the aluminium alloy discs with respect to the top and bottom plates after being subjected to testing. Again, the adhesive mode of failure was characterised by the value of the total percentage area of adhesive failure of more than 90%.

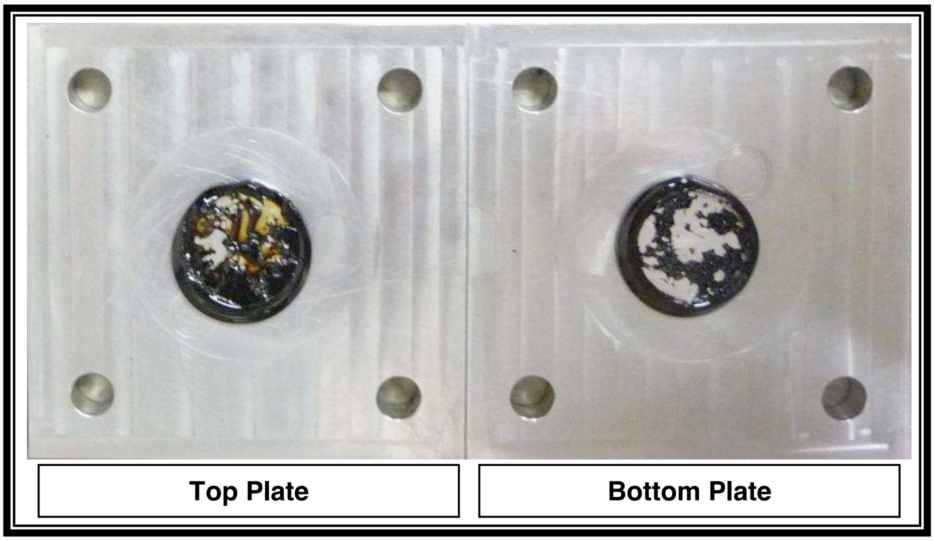


Figure 5.22 Aluminium alloy discs after being subjected to testing

Based on Table 5.5, the average, standard deviation and coefficient of variation of the data sets for the total percentage area of adhesive failure were 99%, 2 and 2% respectively. The total percentage area of adhesive failure was in the range of 90% and 100%, which is considered as sufficient to indicate the occurrence of the adhesive mode of failure. Also, the average, standard deviation and coefficient of variation of the total percentage area of adhesive failure within the individual pair of plates (i.e. pair of plates A, B and C) were as follows; 99%, 3 and 3% for pair of plates A, 100%, 1 and 1% for pair of plates B and 100%, 0 and 0% for pair of plates C.

Data of the test results in terms of total percentage area of adhesive failure seems to disagree with the statement of Kanitpong and Bahia (2003), which indicated that most of the failures for the unconditioned specimens (i.e. dry conditioning) were cohesive. Marek and Herrin (1968) has concluded that the types of failure (i.e. adhesive or cohesive) can be influenced by various factors such as thickness of adhesive layer of bitumen, deformation rate, test temperature, conditioning procedures and penetration grade of bitumen. Hence, based on the test results of this section, it can be concluded that for the unconditioned specimens (i.e. dry conditioning), the occurrence of the adhesive mode of failure can still be achieved provided that the thickness of adhesive layer of bitumen is thin enough.

Small percentage of coefficient of variation indicated the excellent uniformity and repeatability of the data sets for the total percentage area of adhesive failure. The occurrence of cavitations and cobwebbing was minimised due to the reduction of the thickness of adhesive layer of bitumen and the area of contact between bitumen and substrates. Small area of contact between bitumen and substrates (i.e. 490.87 mm^2) will thus allow for the evenly distributed axial tensile load.

Results of the total percentage area of adhesive failure have been grouped as in Table 5.6, and a plot has been deduced as shown in Figure 5.23. Based on Figure 5.23, data of the total percentage area of adhesive failure was found to be skewed to the left, which indicates the mode of failure to be adhesive.

Table 5.6 Results based on grouped total percentage area of adhesive failure

Total Percentage Area of Adhesive Failure (%)	Number of Specimens, n	Percentage (%)
0	0	0
5	0	0
10	0	0
15	0	0
20	0	0
25	0	0
30	0	0
35	0	0
40	0	0
45	0	0
50	0	0
55	0	0
60	0	0
65	0	0
70	0	0
75	0	0
80	0	0
85	0	0
90	2	3
95	3	5
100	55	92

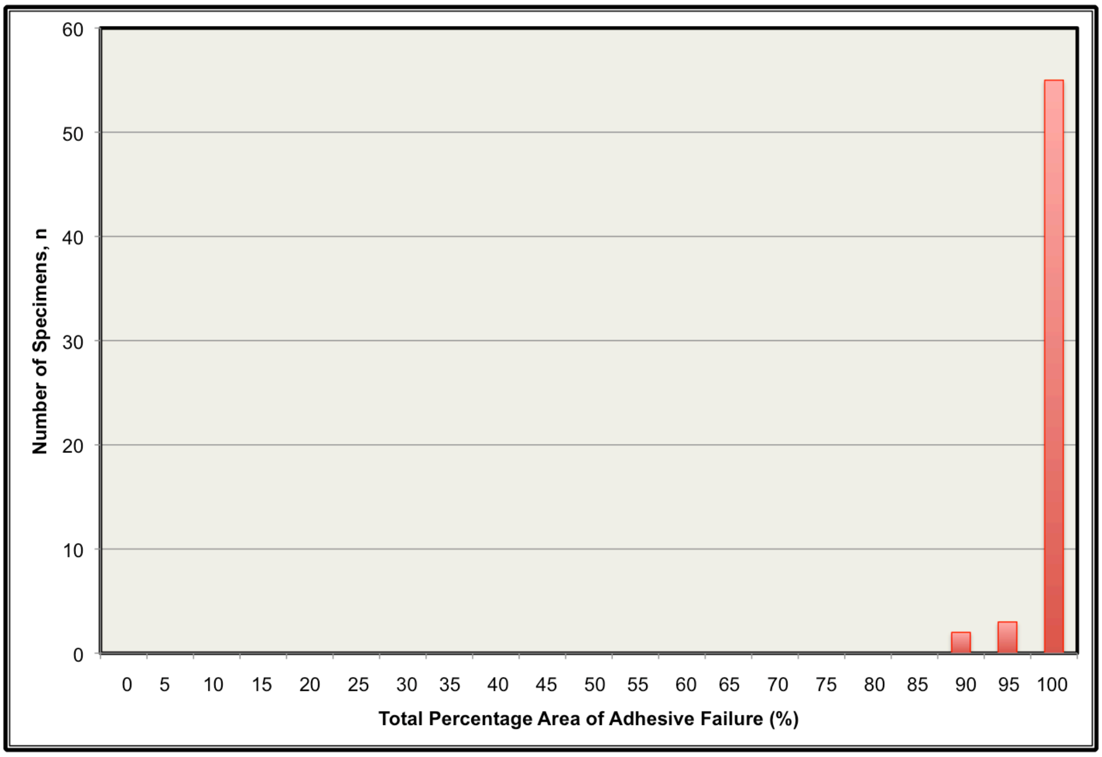


Figure 5.23 Histogram of total percentage area of adhesive failure

5.3.1.3 Analysis of Maximum Tensile Bond Strength

Based on Table 5.5, the value of the maximum tensile bond strength (i.e. maximum tensile load per unit area of contact) for each pair of plates was in the range of 1150 kPa and 1530 kPa, with values for average, standard deviation and coefficient of variation of 1306 kPa, 70 and 5% respectively. The average, standard deviation and coefficient of variation within the individual pair of plates (i.e. pair of plates A, B and C) were as follows; 1304 kPa, 41 and 3% for pair of plates A, 1320 kPa, 65 and 5% for pair of plates B and 1295 kPa, 95 and 7% for pair of plates C. The average value of the maximum tensile bond strength (i.e. 1306 kPa) was higher compared to the average value of the maximum tensile bond strength of the preliminary study, which was attributed to the reduction of the thickness of adhesive layer of bitumen. Also, the small percentage of coefficient of variation indicated the excellent uniformity and repeatability of the data sets, as shown in Figure 5.24. Based on the Figure 5.24, data of the maximum tensile bond strength was concentrated within small ranges of the one-standard deviation above and below the average maximum tensile bond strength.

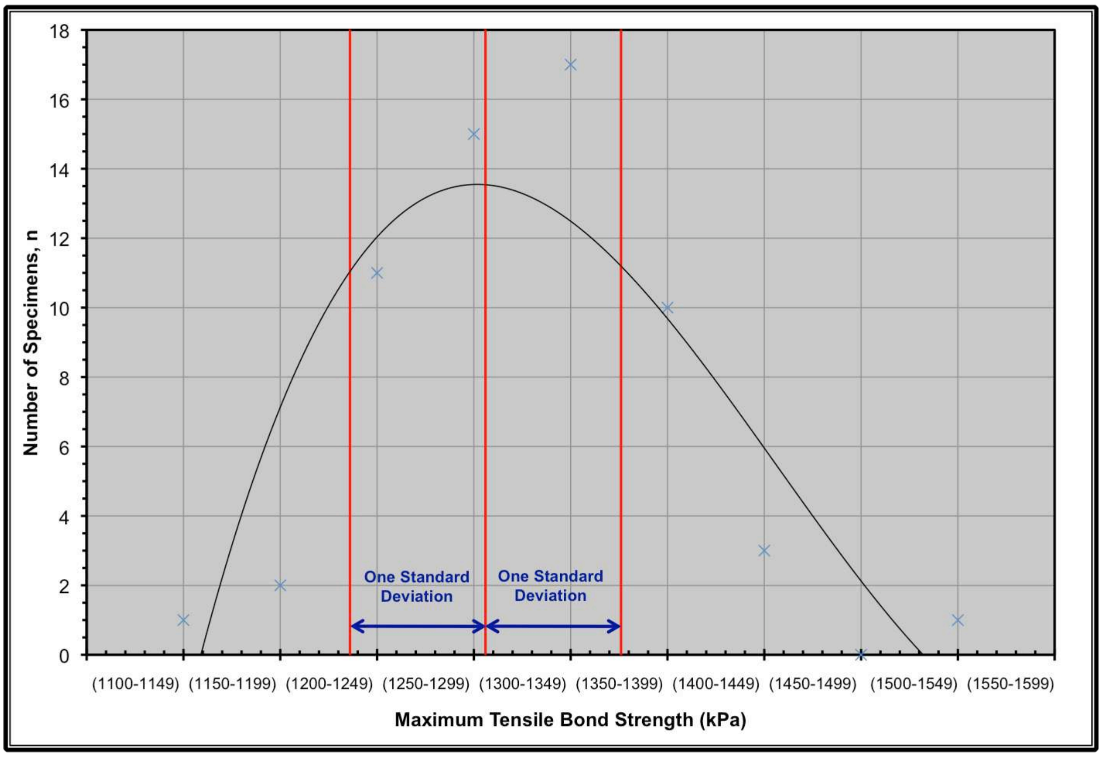


Figure 5.24 Distribution of maximum tensile bond strength

5.3.1.4 Analysis of Tensile Energy Required to Produce Failure Per Unit Volume

Based on Table 5.5, the average, standard deviation and coefficient of variation of the data sets for the tensile energy required to produce failure per unit volume were 2178 kJ/m^3, 373 and 17% respectively. Also, the average, standard deviation and coefficient of variation within the individual pair of plates (i.e. pair of plates A, B and C) were as follows; 1993 kJ/m^3, 208 and 10% for pair of plates A, 2267 kJ/m^3, 442 and 19% for pair of plates B and 2273 kJ/m^3, 374 and 16% for pair of plates C. Based on Figure 5.25, the values of the tensile energy required to produce failure per unit volume tend to be flatter and more spread out. Again, the high variability of the data sets can be attributed to the estimation errors due to the curve fitting procedures (i.e. uncertainty that presents in a curve that is fitted to the data sets) and the parameters that governed the values of the tensile energy required to produce failure per unit volumes such as tensile load, pull off displacement, thickness of adhesive layer of bitumen and total percentage area of adhesive failure.

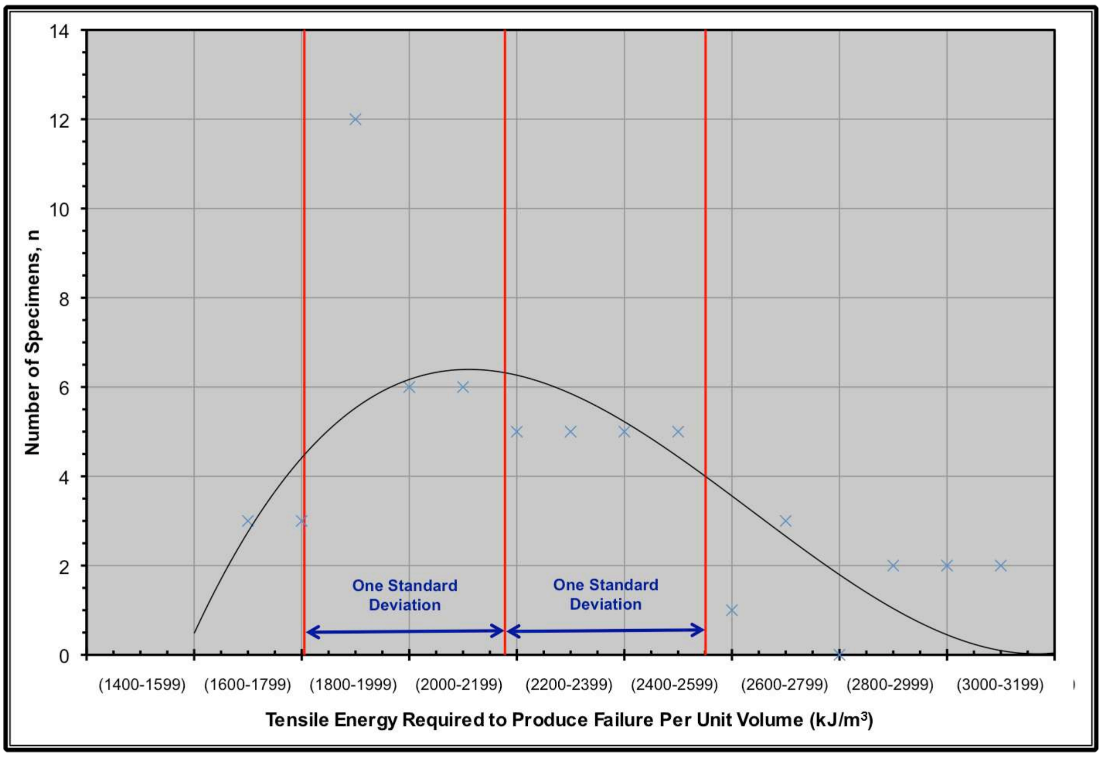


Figure 5.25 Distribution of tensile energy required to produce failure per unit volume

5.3.1.5 Analysis to Determine the Effect of Various Testing Conditions on the Test Results

Basically, the main objective of this section was to determine whether the established criteria and procedures of the proposed adhesion test method capable of testing the asphalt mixture materials over wide ranges of deformation rates and test temperatures, before going into a more detailed study in the next part (i.e. Part 3: Detailed Evaluation and Validation of the Proposed Adhesion Test Method). Also, in this section, data of the test results was useful and vital in predicting the ranges of suitable testing conditions. Since this section was supposed to be a pilot study prior to the next part, a limited number of specimens was prepared and tested, based on the experimental matrix shown in the Table 5.4. A total of 5 specimens, which have been conditioned based on the established standard conditioning procedures were tested at each combination of deformation rate and test temperature.

Theoretically, the value of the maximum tensile bond strength is expected to increase with the increasing deformation rate and decreasing test temperature. Also, the mode of failure is expected to change from cohesive to adhesive with the increasing deformation rate and decreasing test temperature. Table 5.7 shows the data of the test results. Tensile energy required to produce failure per unit volume was not included in order to simplify the analysis. Based on the test results, the following plots have been deduced; Figures 5.26 to 5.28. The error bar represents the one-standard deviation above and below the average maximum tensile bond strength for Figures 5.26 and 5.27, and the one-standard deviation above and below the average total percentage area of adhesive failure for Figure 5.28, respectively. Coefficient of determination, R^2 is a measure of the global fit of the linear relationship between variables, in which coefficient of determination, R^2 of 1 indicates that the fitted linear relationship explains all the variability and coefficient of determination, R^2 of 0 indicates no linear relationship exists between variables (i.e. the higher the value of coefficient of determination, R^2, the stronger the linear relationship between variables). Generally, coefficient of determination, R^2 greater than 0.8 are considered to show a reasonable fits for the data sets.

Table 5.7 Data of the test results over wide ranges of deformation rates and test temperatures (Subsequent study using INSTRON servo hydraulic frame)

Deformation Rate (mm/minute)	Test Temperature (°C)	Data Sets	Thickness of Adhesive Layer of Bitumen (μm)	Total Percentage Area of Adhesive Failure (%)	Maximum Tensile Bond Strength (kPa)
10 (0.167 mm/s)	10	1	50	100	1410
		2	50	100	1490
		3	50	100	1410
		4	50	100	1380
		5	50	100	1440
	15	1	50	100	1330
		2	50	100	1340
		3	50	100	1410
		4	50	100	1380
		5	50	100	1360
	20	1	50	100	1310
		2	50	95	1250
		3	50	100	1280
		4	50	100	1240
		5	50	100	1260
	25	1	50	100	1130
		2	50	90	1160
		3	50	100	1190
		4	50	90	1200
		5	50	100	1170
	30	1	50	90	1140
		2	50	80	1120
		3	50	85	1110
		4	50	85	1120
		5	50	90	1110
20 (0.333 mm/s)	10	1	50	100	1590
		2	50	100	1580
		3	50	100	1530
		4	50	100	1490
		5	50	100	1530
	15	1	50	100	1460
		2	50	100	1450
		3	50	100	1440
		4	50	100	1500
		5	50	100	1430
	20	1	50	100	1390
		2	50	100	1420
		3	50	100	1370
		4	50	100	1400
		5	50	100	1360
	25	1	50	100	1330
		2	50	100	1320
		3	50	100	1300
		4	50	95	1290
		5	50	100	1330
	30	1	50	90	1180
		2	50	90	1170
		3	50	100	1150
		4	50	95	1190
		5	50	95	1180

Table 5.7 Data of the test results over wide ranges of deformation rates and test temperatures (Subsequent study using INSTRON servo hydraulic frame) (continued)

Deformation Rate (mm/minute)	Test Temperature (°C)	Data Sets	Thickness of Adhesive Layer of Bitumen (μm)	Total Percentage Area of Adhesive Failure (%)	Maximum Tensile Bond Strength (kPa)
30 (0.500 mm/s)	10	1	50	100	1610
		2	50	100	1630
		3	50	100	1630
		4	50	100	1630
		5	50	100	1610
	15	1	50	100	1550
		2	50	100	1530
		3	50	100	1520
		4	50	100	1490
		5	50	100	1500
	20	1	50	100	1500
		2	50	100	1480
		3	50	100	1450
		4	50	100	1510
		5	50	100	1460
	25	1	50	100	1400
		2	50	100	1350
		3	50	100	1330
		4	50	100	1350
		5	50	100	1360
	30	1	50	100	1190
		2	50	100	1210
		3	50	90	1150
		4	50	100	1140
		5	50	100	1200

Notes:
1. Substrates: Aluminium alloy
2. Adhesive Materials: Conventional 70/100 penetration grade of bitumen
3. Conditioning Procedures: Dry conditioning at specified test temperature for 24 hours prior to testing

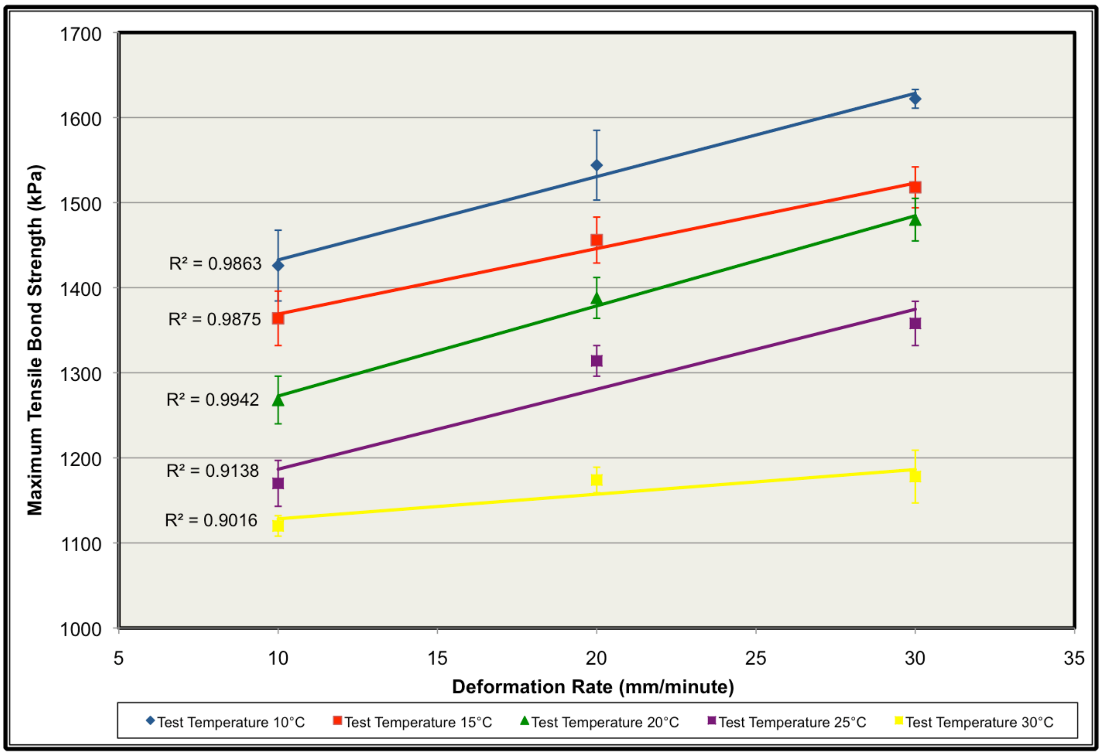


Figure 5.26 Relationship between maximum tensile bond strength and deformation rate over wide ranges of test temperatures

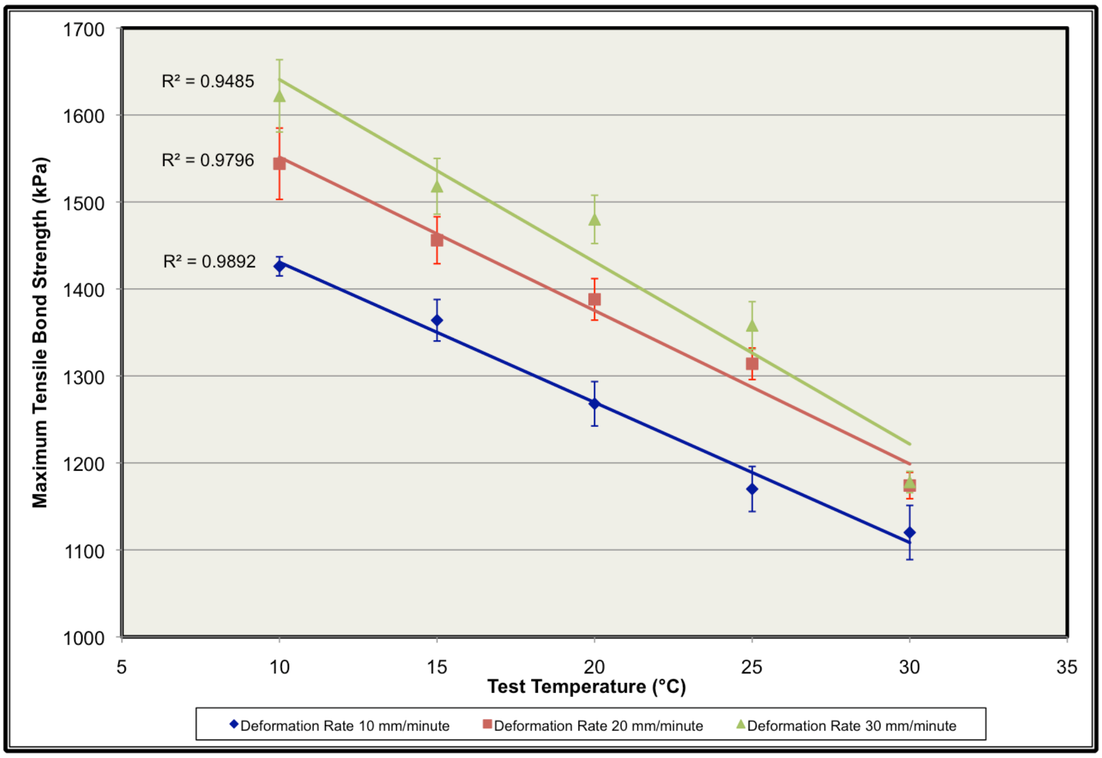


Figure 5.27 Relationship between maximum tensile bond strength and test temperature over wide ranges of deformation rates

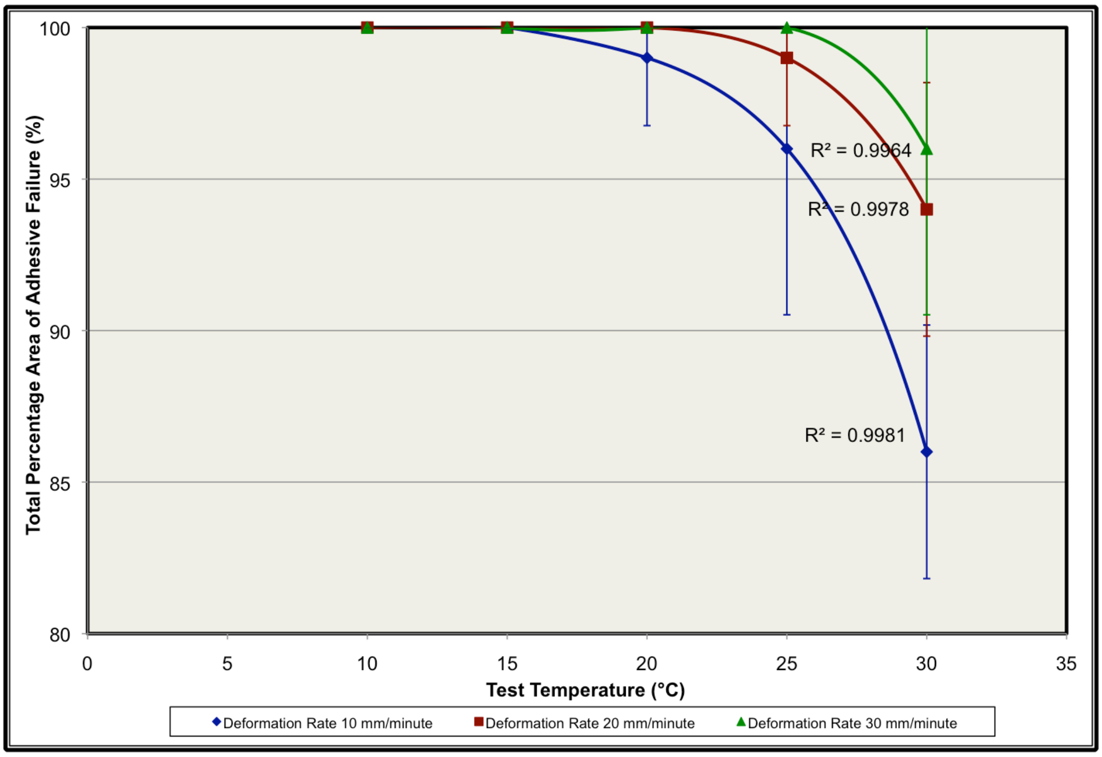


Figure 5.28 Relationship between total percentage area of adhesive failure and test temperature over wide ranges of deformation rates

Based on Figure 5.26, the maximum tensile bond strength was found to increase with the increasing deformation rate for all specified test temperatures. At a certain value of deformation rate, the maximum tensile bond strength of low-test temperature was higher compared to the maximum tensile bond strength of high-test temperature. However, the maximum tensile bond strength of specimens tested at the combined deformation rate and test temperature of 30 mm/minute and 30°C, did not follow the expected trend, and thus can be considered as outlier.

Also, based on Figure 5.27, the maximum tensile bond strength was found to decrease with the increasing test temperature. As the test temperature was increased, there was a decrease in the bitumen stiffness (i.e. bitumen becomes soft and starts to flow), and thus justified the reduction of the maximum tensile bond strength. Also, as the test temperature was increased, the value of the maximum tensile bond strength over wide ranges of deformation rates was found to converge to a constant value. The value of the maximum tensile bond strength was assumed to be independent of deformation rate at high test temperature.

Based on Figure 5.28, deformation rate and test temperature were found to have a profound influence on the types of failure of specimens of either adhesive or cohesive, apart from the thickness of adhesive layer of bitumen. Mode of failure was found to be more the adhesive as the value of the deformation rate was increased and the value of the test temperature was decreased respectively. Also, at high test temperature of more than 25°C, high variability or distribution of the data sets was observed, as represented by the error bar of one-standard deviation above and below the average total percentage area of adhesive failure.

5.3.2 Summary of Subsequent Study Using INSTRON Servo Hydraulic Frame

In this section, pair of plates was modified by allowing two 25 mm diameter (i.e. 490.87 mm^2 area of contact) of aluminium alloy discs as substrates to be inserted into top and bottom plates. The modification of the pair of plates was made in order to allow for the reduction of the thickness of adhesive layer of bitumen and also the reduction of the area of contact between bitumen and substrates. Thickness of adhesive layer of bitumen of 50 µm (0.050 mm) was found to be the optimum value that can result in the adhesive mode of failure and at the same time maintaining the uniformity and repeatability of the test results. Based on the study, a conclusion has been made that for the unconditioned specimens (i.e. dry conditioning), the occurrence of the adhesive mode of failure can still be achieved provided that the thickness of adhesive layer of bitumen is thin enough. Due to the reduction of the area of contact between bitumen and substrates, the occurrence of cavitations and cobwebbing was minimised. Modified test setup and apparatus and also the procedures for specimen preparation have been proven capable of producing specimen as close as possible to the required thickness of adhesive layer of bitumen (i.e. theoretical value). The ranges of suitable testing conditions in terms of deformation rate and test temperature for the proposed adhesion test method were suggested to be 10 mm/minute and 20 mm/minute, and 15°C and 25°C respectively. However, no definite conclusions can be made yet regarding the ranges of suitable testing conditions, due to the limited number of the tested specimens. Data of the test results in this section was used as guiding principles and point of reference in the next part.

5.4 Preliminary Study Using Ductilometer Testing Apparatus

This section focuses on the initial development of criteria and procedures for the proposed adhesion test method based on the Ductilometer testing apparatus. Details of the Ductilometer testing apparatus were given in the Chapter 4. Laboratory works were conducted in the same way as that for the INSTRON servo hydraulic frame, which include the design and fabrication of suitable moulds (i.e. test setup and apparatus), and development of procedures for specimen preparation and testing. Since there is a limitation in the range of the tensile load that can be recorded using the Ductilometer testing apparatus (i.e. up to 300 N), design and fabrication of suitable moulds need to cater for this limitation. At the end of this section, data analysis was conducted in order to evaluate the uniformity and repeatability of the test results.

5.4.1 Test Setup and Apparatus

Pair of aluminium alloy plates as shown in Figure 5.29 was used as control substrates, providing area of contact between bitumen and substrates of 20 mm by 10 mm. Schematic drawing of the pair of plates is shown in Figure 5.30. During the specimen preparation, the pair of plates was held together by bolt mounted on the base plate. The base plate is made of non-corrosive metal sheet. Each pair of plates was labelled in order to provide consistency in pairing and thus accuracy in determining the uniformity and repeatability of the test results. In order to determine the thickness of adhesive layer of bitumen, the combined length of the left-side and right-side of each pair of plates was measured and recorded. Prior to the specimen preparation, surface of the area of contact between bitumen and substrates needs to be cleaned in order to ensure the cleanliness and thus full adhesive bond strength between adhesive layer of bitumen and substrates.

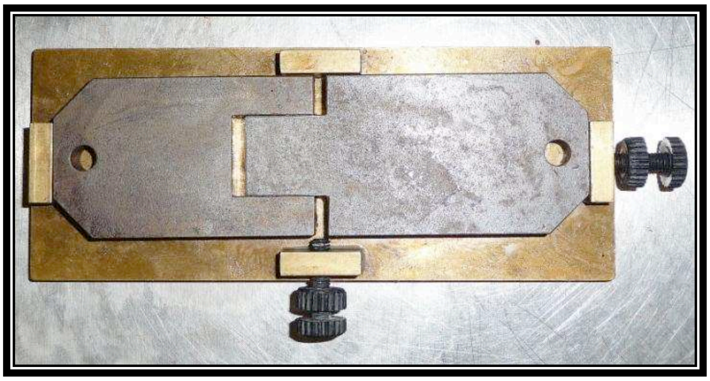


Figure 5.29 Pair of aluminium alloy plates held together by bolt mounted on base plate

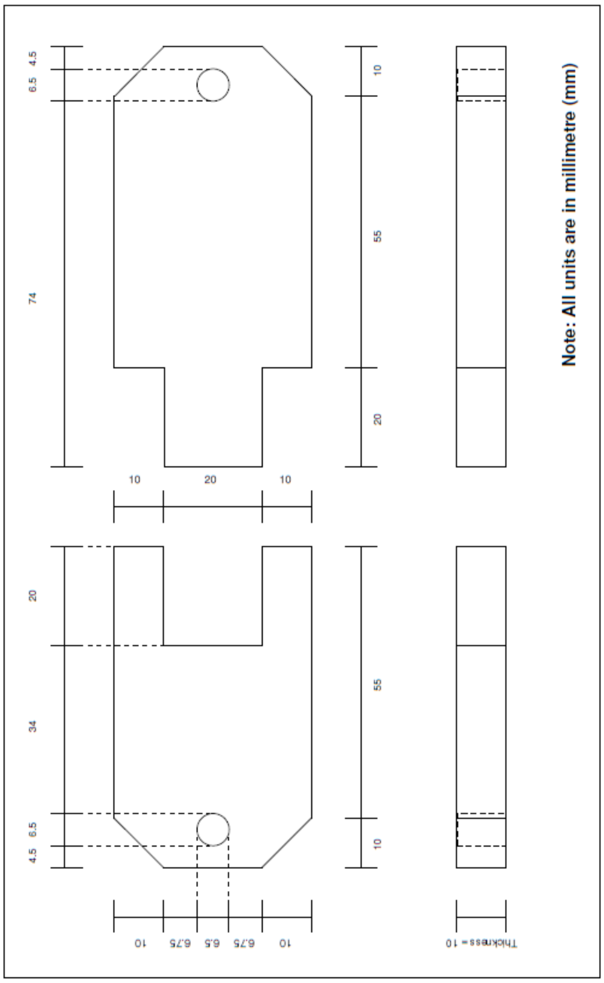


Figure 5.30 Schematic drawing of pair of plates

5.4.2 Specimen Preparation

Procedures for specimen preparation were designed with the same emphasis as for the INSTRON servo hydraulic frame (i.e. uniformity and repeatability of the thickness of adhesive layer of bitumen). In this preliminary study, the thickness of adhesive layer of bitumen has been set to 520 μm (0.520 mm). Steel balls of 0.520 mm diameter have been used to control the thickness of adhesive layer of bitumen. Development of the procedures for specimen preparation was mainly based on the BS EN 13398:2003 Methods of Test for Petroleum and Its Products-BS 2000-516:Bitumen and Bituminous Binders-Determination of the Elastic Recovery of Modified Bitumen.

During the procedure for specimen preparation, thin coat of release agent (mixture of one part of glycerine and one part of dextrine) was applied to the base plate and the sides of the lateral walls of the pair of plates, in order to ensure that no other adhesive bond strength except on the area of contact between bitumen and substrates was developed. It is suggested that the amount of bitumen to be poured is about two-third filled of the gap, in order to minimise the amount of excess bitumen. In order to achieve the required thickness of adhesive layer of bitumen and also the full adhesive bond strength between adhesive layer of bitumen and substrates, the bolt mounted on the base plate was tightened to clamp the specimen. In this case, since no excessive loading was observed and the horizontal movement of the specimen was limited due to the presence of the steel balls, the specimen can be left in the clamped position for any amount of time. Excess bitumen was removed and the specimen was then subjected to conditioning procedures prior to testing. Throughout this section, specimens were subjected to dry conditioning at 25°C for 24 hours prior to testing. However, testing was conducted in wet conditioning in a temperature-controlled water bath at test temperature of 25°C. Reason for the selection of the wet conditioning instead of the dry conditioning as in the previous section was due to the inability of the Ductilometer testing apparatus in conducting test in dry conditioning over wide ranges of test temperatures.

The whole procedure for the specimen preparation can be summarised as follows, and illustrated in Figure 5.31.

1. Pair of plates is cleaned by hand with chemical solution (i.e. white spirit solvent) in order to ensure the cleanliness, and then followed by acetone (ethyl acetate) in order to remove the remaining chemical solution of the

white spirit solvent. (Note: Minimum rubbing should be applied to the surface in order to ensure no significant change in the properties of the surface).

2. Pair of plates is heated to approximately 80°C for at least 30 minutes in order to ensure no significant drop of temperature of bitumen during the specimen preparation.

3. Base plate and the sides of the lateral walls of the pair of plates are applied with a thin coat of release agent (mixture of one part of glycerine and one part of dextrine).

4. Pair of plates is held loosely on the base plate. Ensure that the distance between the pair of plates is sufficient for the bitumen to be poured into the gap.

5. Steel balls of 0.520 mm diameter are positioned at both sides between the pair of plates.

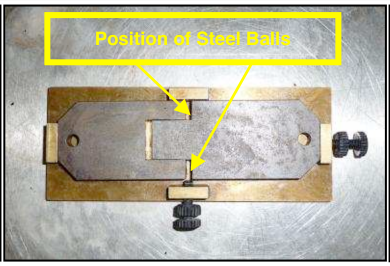
6. Bitumen which has been heated to approximately 160°C for at least two hours is then poured into the gap of the pair of plates, up to about two-third filled.

7. Pair of plates is then subjected to compression by tightening the bolt mounted on the base plate and left in the clamped position for 30 minutes.

8. The clamped pair of plates is measured using micrometer to ensure the required thickness of adhesive layer of bitumen has been achieved.

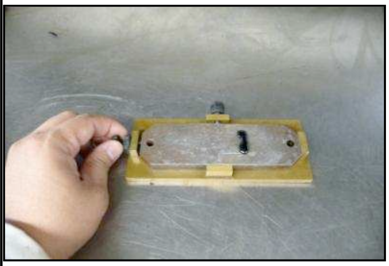
9. The pair of plates is removed from the base plate. Any excess bitumen needs to be removed using heated knife, before the pair of plates being introduced back to the clamped position for conditioning procedures prior to testing.

1. Thin coat of release agent is applied on the base plate and the sides of the lateral walls of the pair of plates

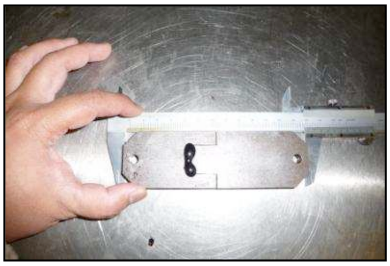


2. Pair of plates is held loosely on the base plate. Steel balls are positioned at both sides between the pair of plates

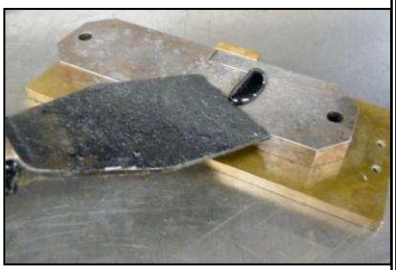
3. Bitumen is poured into the gap of the pair of plates



4. Pair of plates is subjected to compression by tightening the bolt mounted on the base plate and left in the clamped position for 30 minutes



5. The clamped pair of plates is measured



6. Excess bitumen is removed using heated knife prior to conditioning procedures

Figure 5.31 Procedures for specimen preparation for preliminary study using Ductilometer testing apparatus

5.4.3 Testing Using Ductilometer Testing Apparatus

Development of the criteria and procedures for the testing was mainly based on the BS EN 13398:2003 Methods of Test for Petroleum and Its Products-BS 2000-516:Bitumen and Bituminous Binders-Determination of the Elastic

Recovery of Modified Bitumen. However, several testing variables and parameters were adjusted in order to suit the proposed adhesion test method.

In order to evaluate the uniformity and repeatability of the test results, a total of 60 specimens was tested at a fixed deformation rate and test temperature of 20 mm/minute and 25°C respectively. Data of the test results was analysed and presented in the next section.

5.4.4 Data Analysis of Preliminary Study Using Ductilometer Testing Apparatus

Data analysis of preliminary study using Ductilometer testing apparatus was conducted in the same way as that for the INSTRON servo hydraulic frame, in which evaluation of the uniformity and repeatability of the test results was performed. Table 5.8 shows the data of the test results in terms of thickness of adhesive layer of bitumen, total percentage area of adhesive failure, maximum tensile bond strength (i.e. maximum tensile load per unit area of contact) and tensile energy required to produce failure per unit volume. A standard procedure in determining the types of failure of the specimens as either adhesive or cohesive was given in Appendix B.

Table 5.8 Data of the test results (Preliminary study using Ductilometer testing apparatus)

Pair of Plates	Data Sets	Thickness of Adhesive Layer of Bitumen (μm)	Total Percentage Area of Adhesive Failure (%)	Maximum Tensile Bond Strength (kPa)	Tensile Energy Required to Produce Failure Per Unit Volume (kJ/m^3)
A	1	560	40	1080	1526
	2	560	60	1030	968
	3	560	100	1090	994
	4	540	80	1150	1254
	5	520	55	1010	1359
	6	540	70	1020	1499
	7	580	45	960	1125
	8	560	90	960	1163
	9	600	50	840	1011
	10	640	65	840	871
	11	580	70	940	931
	12	580	95	1060	1500
	13	500	100	1210	1461
	14	660	20	890	647
	15	560	60	870	940
	16	540	65	980	1042
	17	560	60	750	801
	18	560	70	1080	1193
	19	580	45	980	928
	20	580	50	940	996
B	1	580	90	1040	1521
	2	560	70	1060	1092
	3	600	30	950	1135
	4	560	50	1050	1182
	5	620	40	980	1084
	6	560	70	1040	1395
	7	620	40	840	835
	8	580	90	880	1126
	9	580	35	890	1064
	10	600	25	790	1123
	11	580	40	820	1042
	12	520	70	1100	1554
	13	540	90	1110	1325
	14	580	50	990	1091
	15	580	60	890	982
	16	580	45	770	971
	17	580	40	680	649
	18	560	40	1030	1169
	19	540	30	1040	1423
	20	560	40	960	1169

Table 5.8 Data of the test results (Preliminary study using Ductilometer testing apparatus) (continued)

Pair of Plates	Data Sets	Thickness of Adhesive Layer of Bitumen (μm)	Total Percentage Area of Adhesive Failure (%)	Maximum Tensile Bond Strength (kPa)	Tensile Energy Required to Produce Failure Per Unit Volume (kJ/m^3)
C	1	520	75	1160	1703
	2	580	70	1040	1115
	3	560	70	1030	1244
	4	540	50	1120	1444
	5	620	20	940	1231
	6	640	45	950	1223
	7	580	100	970	1273
	8	620	60	880	1024
	9	620	75	920	997
	10	680	40	790	1064
	11	540	70	1020	1567
	12	540	90	1200	1362
	13	560	85	1150	1143
	14	580	50	920	1011
	15	560	40	880	1225
	16	580	55	770	863
	17	540	50	750	949
	18	600	40	970	1023
	19	580	45	970	1005
	20	540	40	1090	1316
Average		**574**	**58**	**969**	**1149**
Standard Deviation		**34**	**21**	**120**	**228**
Coefficient of Variation (%)		**6**	**36**	**12**	**20**

Notes:
1. Substrates: Aluminium alloy
2. Adhesive Materials: Conventional 70/100 penetration grade of bitumen
3. Conditioning Procedures: Dry conditioning at 25°C for 24 hours prior to testing
4. Testing Conditions: Deformation rate and test temperature of 20 mm/minute and 25°C respectively
5. Testing was conducted in wet conditioning in a temperature-controlled water bath at specified test temperature

5.4.4.1 Analysis of Thickness of Adhesive Layer of Bitumen

The pair of plates was designed to result in the thickness of adhesive layer of bitumen of approximately 520 μm (0.520 mm). Analysis of the thickness of adhesive layer of bitumen was conducted in the same way as that for the INSTRON servo hydraulic frame, in order to determine the uniformity and repeatability between data sets and also to determine if significant difference exists between the measured and the theoretical thickness of 520 μm (0.520 mm).

The average, standard deviation and coefficient of variation of the data sets were 574 μm (0.574 mm), 34 and 6% respectively. The average, standard deviation and coefficient of variation of the thickness of adhesive layer of bitumen within the individual pair of plates (i.e. pair of plates A, B and C) were as follows; 568 μm (0.568 mm), 36 and 6% for pair of plates A, 574 μm (0.574 mm), 25 and 4% for pair of plates B and 579 μm (0.579 mm), 41 and 7% for pair of plates C. Also, in terms of the uniformity of the average thickness of adhesive layer of bitumen between the individual pair of plates (i.e. pair of plates A, B and C), the standard deviation of 6 and the small percentage of coefficient of variation of 6% indicated the excellent uniformity. Therefore, it can be concluded that there is no significant difference in the thickness of adhesive layer of bitumen within and between the individual pair of plates (i.e. pair of plates A, B and C).

Analysis was then conducted in order to determine if significant difference exists between the average measured thickness of adhesive layer of bitumen of 574 μm (0.574 mm) and the theoretical value of 520 μm (0.520 mm). A hypothesis test involving One-Sample t-Test procedure, at level of significance, α of 0.05, was conducted.

The following hypotheses were then established.

1. The null hypothesis, H_0

   H_0: $\overline{X}$ = 520 μm (0.520 mm)

   The average measured thickness of adhesive layer of bitumen is 520 μm (0.520 mm).

2. The alternative hypothesis, H_1

   H_1: $\overline{X} \neq$ 520 μm (0.520 mm)

   The average measured thickness of adhesive layer of bitumen is not 520 μm (0.520 mm).

Based on MINITAB statistical analysis, the t-statistic, T was found to be 12.07 (Figure 5.32). The decision rule for rejecting H_0 based on the p-value approach is as follows.

Reject H_0 if p-value is smaller than level of significance, α.

Otherwise, fail to reject H_0

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One-Sample T: (Measured Thickness of Adhesive Layer of Bitumen)

Test of mu = 520 vs mu not = 520

Variable	N	Mean	StDev	SE Mean
(Meas. Thick.)	60	573.67	34.443	4.447

Variable	95.0% CI	T	P
(Meas. Thick.)	(44.77, 62.56)	12.07	0.000

Figure 5.32 MINITAB statistical analysis

Therefore, based on the value of t-statistic, T of 12.07 and also p-value of 0.000, the null hypothesis, H_0 is rejected at the level of significance, α of 0.05. The analysis shows that significant statistical difference exists in the average measured thickness of adhesive layer of bitumen from the theoretical value of 520 μm (0.520 mm). Hence, improved procedures for specimen preparation are required in order to achieve the thickness as close as possible to the required thickness of adhesive layer of bitumen (i.e. theoretical value). Again, the problem can be attributed to the high thickness of adhesive layer of bitumen, which has resulted in high resistance to compression.

5.4.4.2 Analysis of Total Percentage Area of Adhesive Failure

Figure 5.33 shows the left-side and right-side of a pair of plates after being subjected to testing. Based on Table 5.8, the average, standard deviation and coefficient of variation of the data sets were 58%, 21 and 36% respectively. The total percentage area of adhesive failure was in the range of 20% and 100%, which justified the large percentage of coefficient of variation, and thus indicated the high variability or distribution of the data sets. Also, the average, standard deviation and coefficient of variation of the total percentage area of adhesive failure within the individual pair of plates (i.e. pair of plates A, B and C) were as follows; 65%, 21 and 32% for pair of plates A, 52%, 21 and 40% for pair of plates B and 59%, 20 and 35% for pair of plates C.

Since the area of contact between bitumen and substrates is small (i.e. 200 mm^2), the high variability or distribution of the data sets was attributed to the thickness of adhesive layer of bitumen. High thickness of adhesive layer of bitumen was found to result in cobwebbing, as shown in Figure 5.34. Due to the occurrence of cobwebbing and also the testing that was conducted in wet conditioning in a temperature-controlled water bath, determination of the types of failure of the specimens becomes more complicated. The presence of water has resulted in some of the strings or strands of the cobwebbing re-adhering to the surface of the area of contact between bitumen and substrates, and thus influenced the observation of the types of failure. Results of the total percentage area of adhesive failure have been grouped as in Table 5.9, and a plot has been deduced as shown in Figure 5.35. Based on Figure 5.35, the mode of failure can be classified as mixed cohesive and adhesive.

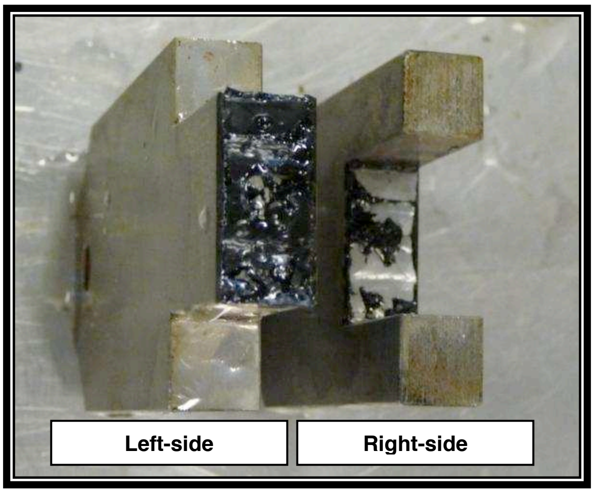

Figure 5.33 Left-side and right-side of a pair of plates after being subjected to testing

Figure 5.34 Cobwebbing

Table 5.9 Results based on grouped total percentage area of adhesive failure

Total Percentage Area of Adhesive Failure (%)	Number of Specimens, n	Percentage (%)
0	0	0
5	0	0
10	0	0
15	0	0
20	2	3
25	1	2
30	2	3
35	1	2
40	11	18
45	5	8
50	7	12
55	2	3
60	5	8
65	2	3
70	9	15
75	2	3
80	1	2
85	1	2
90	5	8
95	1	2
100	3	5

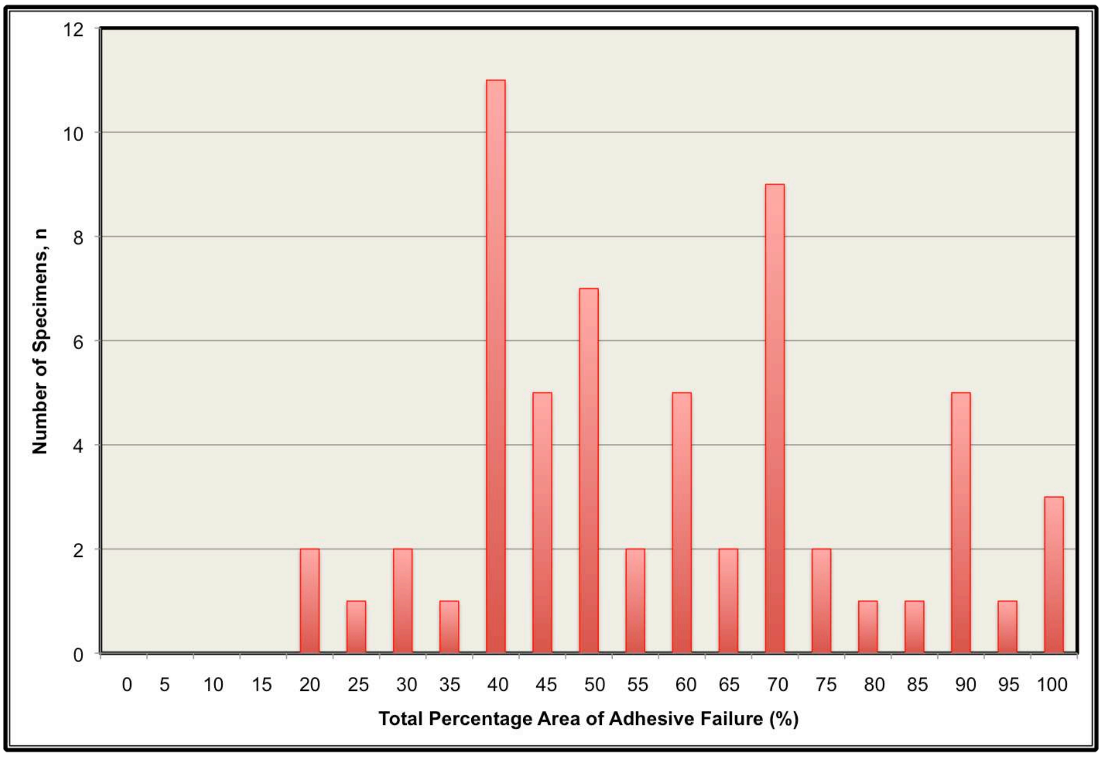

Figure 5.35 Histogram of total percentage area of adhesive failure

5.4.4.3 Analysis of Maximum Tensile Bond Strength

Based on Table 5.8, the value of the maximum tensile bond strength (i.e. maximum tensile load per unit area of contact) for each pair of plates was in the range of 680 kPa and 1210 kPa, with values for average, standard deviation and coefficient of variation of 969 kPa, 120 and 12% respectively. The average, standard deviation and coefficient of variation of the measured maximum tensile bond strength within the individual pair of plates (i.e. pair of plates A, B and C) were as follows; 981 kPa, 113 and 11% for pair of plates A, 946 kPa, 120 and 13% for pair of plates B and 976 kPa, 128 and 13% for pair of plates C. Large percentage of coefficient of variation as compared to the predefined cut-off value of 7% indicates the high variability or distribution of the measured maximum tensile bond strength. This was further verified based on the spread of the distribution of the data sets for the measured maximum tensile bond strength (i.e. within the range of approximately 650 kPa and 1350 kPa), as shown in Figure 5.36. Based on Figure 5.36, the values of the measured maximum tensile bond strength tend to be flatter and more spread out. Again, high variability or distribution of the measured maximum tensile bond strength was attributed to the thickness of adhesive layer of bitumen and the occurrence of cobwebbing. The average value of the maximum tensile bond strength (i.e. 969 kPa) was in the range of the expected value, as compared to the data of the test results of the preliminary study using INSTRON servo hydraulic frame (i.e. 851 kPa with the average thickness of adhesive layer of bitumen of 816 μm).

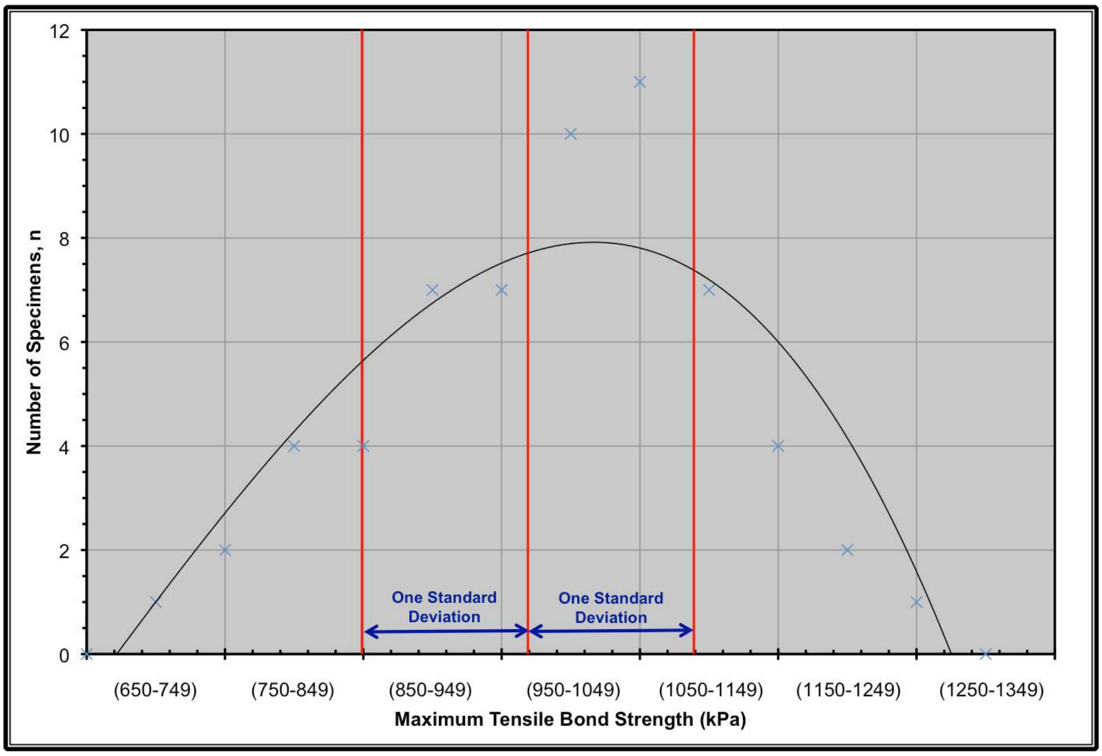

Figure 5.36 Distribution of maximum tensile bond strength

5.4.4.4 Analysis of Tensile Energy Required to Produce Failure Per Unit Volume

As has been stated before, tensile energy required to produce failure per unit volume was calculated based on the area under the curve of graph of tensile load versus pull off displacement per unit volume of the adhesive layer of bitumen. Based on Table 5.8, the average, standard deviation and coefficient of variation of the data sets for the tensile energy required to produce failure per unit volume were 1149 kJ/m^3, 228 and 20% respectively. Also, the average, standard deviation and coefficient of variation of the tensile energy required to produce failure per unit volume within the individual pair of plates (i.e. pair of plates A, B and C) were as follows; 1110 kJ/m^3, 253 and 23% for pair of plates A, 1147 kJ/m^3, 219 and 19% for pair of plates B and 1189 kJ/m^3, 215 and 18% for pair of plates C. Also, based on the Figure 5.37, the data of the tensile energy required to produce failure per unit volume tend to be flatter and more spread out.

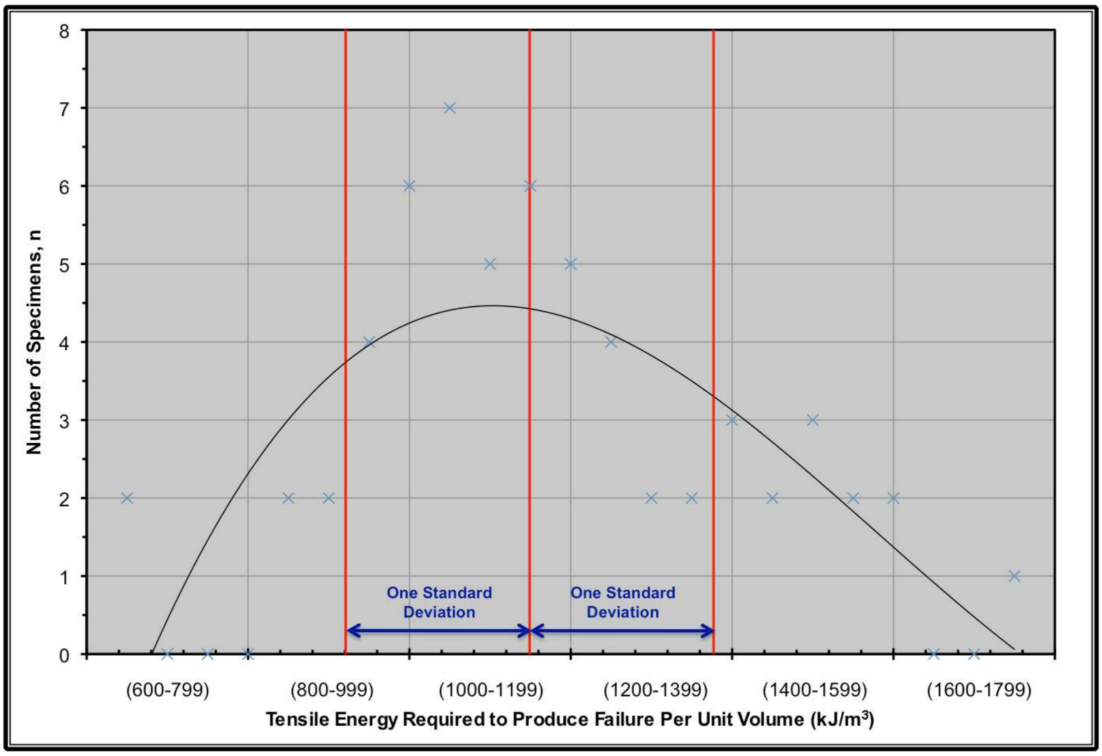

Figure 5.37 Distribution of tensile energy required to produce failure per unit volume

5.4.5 Summary of Preliminary Study Using Ductilometer Testing Apparatus

In this section, initial development of criteria and procedures for the proposed adhesion test method based on the Ductilometer testing apparatus was conducted, mainly based on the BS EN 13398:2003 Methods of Test for Petroleum and Its Products-BS 2000-516: Bitumen and Bituminous Binders-Determination of the Elastic Recovery of Modified Bitumen. Design and fabrication of moulds, which consist of pair of aluminium alloy plates providing area of contact between bitumen and substrates of 20 mm by 10 mm were established. However, it should be noted that there is limitation in the range of the tensile load that can be recorded using the Ductilometer testing apparatus (i.e. up to 300 N) and also inability of the Ductilometer testing apparatus in conducting test in dry conditioning over wide ranges of test temperatures. Data of the test results was found to be highly variable and mode of failure for the tested specimens can be classified as mixed cohesive and adhesive. However, the average value of the maximum tensile bond strength (i.e. 969 kPa) was in the range of the expected value, as compared to the data of the test results of preliminary study using INSTRON servo hydraulic frame.

5.5 Subsequent Study Using Ductilometer Testing Apparatus

Results of the preliminary study in the previous section were used as point of reference in order to refine the criteria and procedures for the proposed adhesion test method based on the Ductilometer testing apparatus. Reduction of the thickness of adhesive layer of bitumen of 520 μm (0.520 mm) was suggested in order to allow for the occurrence of the adhesive mode of failure and at the same time maintaining the uniformity and repeatability of the test results. However, it should be noted that as the thickness of adhesive layer of bitumen is reduced, the value of the maximum tensile bond strength is expected to increase. Since there is limitation in the range of the tensile load that can be measured using the Ductilometer testing apparatus (i.e. up to 300 N), modification of the pair of plates was required in the first place. The modification of the pair of plates was made by reducing the area of contact between bitumen and substrates to 10 mm by 10 mm (i.e. 100 mm^2), as shown in Figure 5.38. The optimum thickness of adhesive layer of bitumen that can result in the adhesive mode of failure and at the same time maintaining the uniformity and repeatability of the test results was selected to be 50 μm (0.050 mm). The selection was made by referring to the data of the test results of the subsequent study using INSTRON servo hydraulic frame.

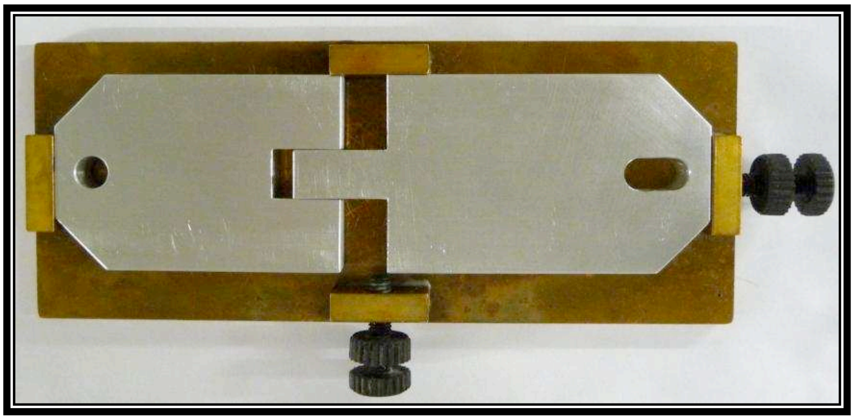

Figure 5.38 Modified pair of plates

Procedures for specimen preparation were still based on the procedures that have been developed in the previous section, and the only modification was the reduction of the area of contact between bitumen and substrates of the pair of plates. In this section, data analysis was conducted in order to validate the value of the selected thickness of adhesive layer of bitumen in resulting the adhesive mode of failure and also to evaluate the uniformity and repeatability of the test results. Also, further evaluation was undertaken in order to generally observe the effect of various testing conditions (i.e. deformation rates and test temperatures) on the test results and to validate the capability of the Ductilometer testing apparatus in conducting adhesion test method. Table 5.10 shows the experimental matrix for the subsequent study. Specimens were subjected to dry conditioning at specified test temperature for 24 hours prior to testing. Again, testing was conducted in wet conditioning in a temperature-controlled water bath at specified test temperature.

Table 5.10 Experimental matrix for subsequent study

		Test Temperature (°C)				
		10	15	20	25	30
Deformation Rate (mm/minute)	10	5 Specimens	5 Specimens	5 Specimens	5 Specimens	5 Specimens
	20	5 Specimens	5 Specimens	5 Specimens	60 Specimens	5 Specimens
	30	5 Specimens	5 Specimens	5 Specimens	5 Specimens	5 Specimens

5.5.1 Data Analysis of Subsequent Study using Ductilometer Testing Apparatus

Table 5.11 shows the data of the test results of the subsequent study, in which a total of 60 specimens was subjected to testing at fixed deformation rate and test temperature of 20 mm/minute and 25°C respectively.

Table 5.11 Data of the test results (Subsequent study using Ductilometer testing apparatus)

Pair of Plates	Data Sets	Thickness of Adhesive Layer of Bitumen (μm)	Total Percentage Area of Adhesive Failure (%)	Maximum Tensile Bond Strength (kPa)	Tensile Energy Required to Produce Failure Per Unit Volume (kJ/m^3)
A	1	50	90	1310	2185
	2	50	100	1410	2865
	3	50	100	1250	2835
	4	50	100	1300	1681
	5	60	95	1260	1835
	6	50	100	1320	2068
	7	50	100	1460	2058
	8	50	100	1190	2140
	9	50	95	1250	2665
	10	60	90	1310	2077
	11	50	100	1320	1508
	12	50	100	1380	1706
	13	50	100	1200	1618
	14	50	100	1410	1703
	15	50	100	1440	2123
	16	50	100	1380	2140
	17	50	100	1240	1683
	18	60	90	1340	1912
	19	50	100	1280	1872
	20	50	100	1340	1945
B	1	60	90	1380	1578
	2	50	100	1380	2203
	3	50	90	1370	2863
	4	50	100	1350	2078
	5	50	100	1330	2583
	6	50	95	1310	1848
	7	50	100	1290	1660
	8	50	100	1250	1990
	9	50	100	1330	2072
	10	50	95	1350	2058
	11	60	100	1240	1882
	12	50	100	1320	1553
	13	50	95	1370	1404
	14	50	100	1340	1575
	15	50	100	1260	1547
	16	60	100	1310	1680
	17	60	95	1280	1537
	18	50	100	1340	2598
	19	50	95	1380	2458
	20	50	100	1250	1628

Table 5.11 Data of the test results (Subsequent study using Ductilometer testing apparatus) (continued)

Pair of Plates	Data Sets	Thickness of Adhesive Layer of Bitumen (μm)	Total Percentage Area of Adhesive Failure (%)	Maximum Tensile Bond Strength (kPa)	Tensile Energy Required to Produce Failure Per Unit Volume (kJ/m^3)
C	1	50	100	1330	1355
	2	60	100	1310	1372
	3	50	100	1380	1690
	4	60	95	1320	1720
	5	50	100	1180	2028
	6	50	100	1250	1872
	7	50	100	1410	1718
	8	50	95	1260	1279
	9	50	100	1430	1410
	10	50	100	1460	1518
	11	50	95	1230	1973
	12	50	100	1300	1665
	13	50	85	1350	2545
	14	50	100	1400	2045
	15	50	100	1260	1893
	16	50	100	1230	1762
	17	60	95	1320	1820
	18	50	100	1280	1525
	19	50	100	1330	1352
	20	50	100	1360	1855
Average		**52**	**98**	**1320**	**1897**
Standard Deviation		**4**	**4**	**66**	**390**
Coefficient of Variation (%)		**7**	**4**	**5**	**21**

Notes:
1. Substrates: Aluminium alloy
2. Adhesive Materials: Conventional 70/100 penetration grade of bitumen
3. Conditioning Procedures: Dry conditioning at 25°C for 24 hours prior to testing
4. Testing Conditions: Deformation rate and test temperature of 20 mm/minute and 25°C respectively
5. Testing was conducted in wet conditioning in a temperature-controlled water bath at specified test temperature

5.5.1.1 Analysis of Thickness of Adhesive Layer of Bitumen

Pair of plates was designed to result in the thickness of adhesive layer of bitumen of 50 μm (0.050 mm). Based on the data of the test results in Table 5.11, the average measured thickness of adhesive layer of bitumen was 52 μm (0.052 mm) with the value of standard deviation and coefficient of variation were 4 and 7% respectively.

The average, standard deviation and coefficient of variation of the thickness of adhesive layer of bitumen within the individual pair of plates (i.e. pair of plates A, B and C) were as follows; 52 μm (0.052 mm), 4 and 7% for pair of plates A, 52 μm (0.052 mm), 4 and 8% for pair of plates B and 52 μm (0.052 mm), 4 and 7% for pair of plates C. Also, in terms of the uniformity of the average thickness of adhesive layer of bitumen between the individual pair of plates (i.e. pair of plates A, B and C), the standard deviation of 0 and the small percentage of coefficient of variation of 0% indicated the excellent uniformity and repeatability.

Analysis was then conducted in order to determine if significant difference exists between the average measured thickness of adhesive layer of bitumen of 52 μm (0.052 mm) and the theoretical value of 50 μm (0.050 mm). A hypothesis test involving One-Sample t-Test procedure, at level of significance, α of 0.05, was then conducted. Data of the measured thickness of adhesive layer of bitumen was assumed to be normally distributed.

The following hypotheses were then established.

1. The null hypothesis, H_0

   H_0: $\overline{X}$ = 50 μm (0.050 mm)

   The average measured thickness of adhesive layer of bitumen is 50 μm (0.050 mm).

2. The alternative hypothesis, H_1

   H_1: $\overline{X} \neq$ 50 μm (0.050 mm)

   The average measured thickness of adhesive layer of bitumen is not 50 μm (0.050 mm).

Based on MINITAB statistical analysis, the t-statistic, T was found to be 3.44 (Figure 5.39). The decision rule for rejecting H_0 based on the p-value approach is as follows.

Reject H_0 if p-value is smaller than level of significance, α.

Otherwise, fail to reject H_0.

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One-Sample T: (Measured Thickness of Adhesive Layer of Bitumen)

Test of mu = 50 vs mu not = 50

Variable	N	Mean	StDev	SE Mean
(Meas. Thick.)	60	51.67	3.758	0.485

Variable	95.0% CI	T	P
(Meas. Thick.)	(0.70, 2.64)	3.44	0.001

Figure 5.39 MINITAB statistical analysis

Based on the value of t-statistic, T of 3.44 and also p-value of 0.001, the null hypothesis, H_0 is rejected at the level of significance, α of 0.05. Therefore, it can be concluded that there is sufficient evidence of significant statistical difference in the average measured thickness of adhesive layer of bitumen from the theoretical value of 50 µm (0.050 mm). Although the procedures for specimen preparation had been improved by reducing the thickness of adhesive layer of bitumen and also the area of contact between bitumen and substrates, it is still insufficient in achieving the thickness as close as possible to the required thickness (i.e. theoretical value). The problem can be attributed to the insufficient pressure being applied to clamp the specimen. During the specimen preparation, pair of plates was clamped by manually tightening (i.e. by hand) the bolt mounted on the base plate, in order to achieve the required thickness of adhesive layer of bitumen and also the full adhesive bond strength between adhesive layer of bitumen and substrates.

5.5.1.2 Analysis of Total Percentage Area of Adhesive Failure

Based on Table 5.11, the average, standard deviation and coefficient of variation of the data sets for the total percentage area of adhesive failure were 98%, 4 and 4% respectively. The total percentage area of adhesive failure was in the range of 85% and 100%, which is considered as sufficient to indicate the occurrence of the adhesive mode of failure. Also, the average, standard deviation and coefficient of variation of the total percentage area of adhesive failure within the individual pair of plates (i.e. pair of plates A, B and C) were as follows; 98%, 4 and 4% for pair of plates A, 98%, 3 and 4% for pair of plates B and 98%, 4 and 4% for pair of plates C. Small percentage of coefficient of variation indicated the excellent uniformity and repeatability of the data sets for the total percentage area of adhesive failure. The occurrence of cobwebbing was minimised due to the reduction of thickness of adhesive layer of bitumen, and hence the problem related to the strings or strands of the cobwebbing that re-adhere to the surface of the area of contact between bitumen and substrates during the testing was solved. Results of the total percentage area of adhesive failure have been grouped as in Table 5.12, and a plot has been deduced as shown in Figure 5.40. Based on Figure 5.40, data of the total percentage area of adhesive failure was found to be skewed to the left, which indicates the mode of failure to be adhesive.

Table 5.12 Results based on grouped total percentage area of adhesive failure

Total Percentage Area of Adhesive Failure (%)	Number of Specimens, n	Percentage (%)
0	0	0
5	0	0
10	0	0
15	0	0
20	0	0
25	0	0
30	0	0
35	0	0
40	0	0
45	0	0
50	0	0
55	0	0
60	0	0
65	0	0
70	0	0
75	0	0
80	0	0
85	1	2
90	5	8
95	11	18
100	43	72

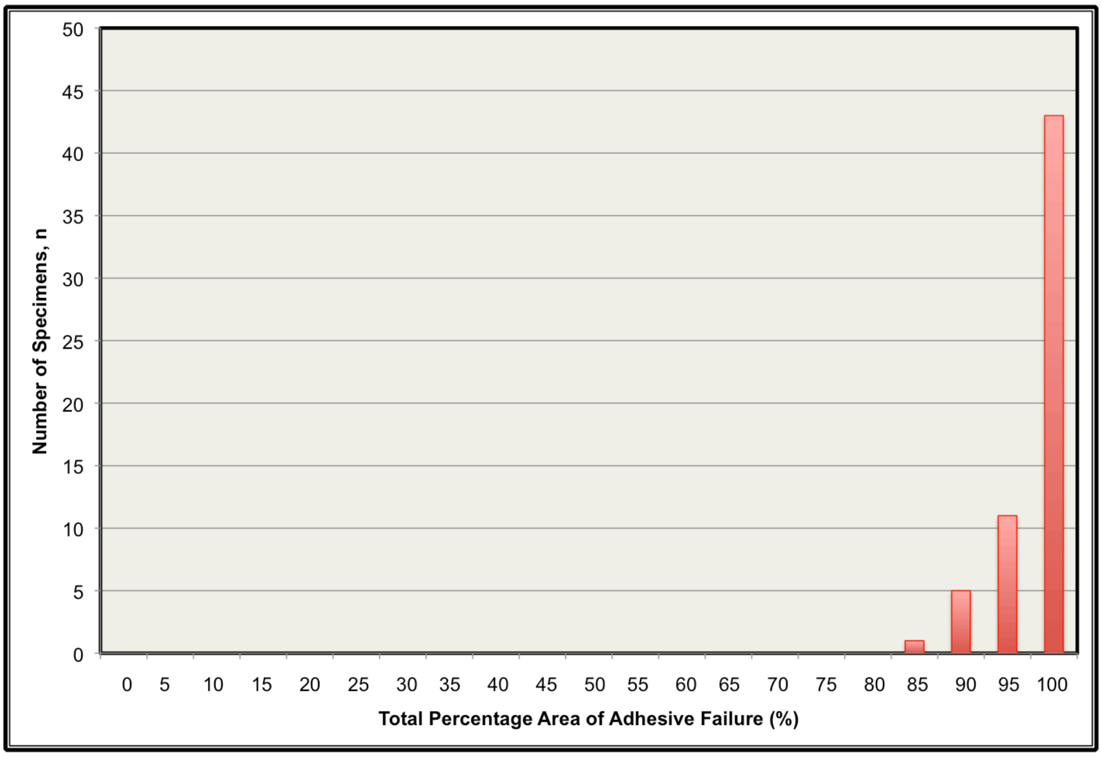


Figure 5.40 Histogram of total percentage area of adhesive failure

5.5.1.3 Analysis of Maximum Tensile Bond Strength

Based on Table 5.11, the value of the maximum tensile bond strength (i.e. maximum tensile load per unit area of contact) for each pair of plates was in the range of 1180 kPa and 1460 kPa, with values for average, standard deviation and coefficient of variation of 1320 kPa, 66 and 5% respectively. The average value of the maximum tensile bond strength (i.e. 1320 kPa) was higher compared to the average value of the maximum tensile bond strength of the preliminary study, which was attributed to the reduction of the thickness of adhesive layer of bitumen. Also, the average, standard deviation and coefficient of variation of the measured maximum tensile bond strength within the individual pair of plates (i.e. pair of plates A, B and C) were as follows; 1320 kPa, 77 and 6% for pair of plates A, 1322 kPa, 46 and 4% for pair of plates B and 1320 kPa, 73 and 6% for pair of plates C. The small percentage of coefficient of variation indicated the excellent uniformity and repeatability of the data sets, as verified in Figure 5.41.

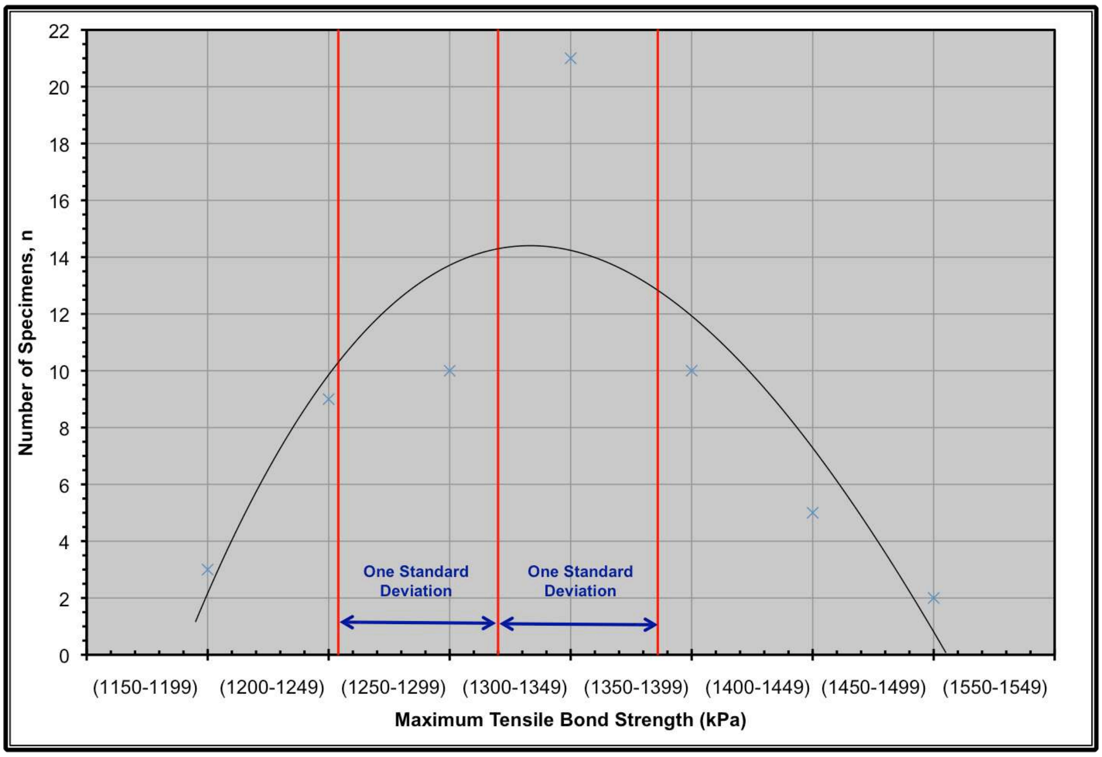


Figure 5.41 Distribution of maximum tensile bond strength

5.5.1.4 Analysis of Tensile Energy Required to Produce Failure Per Unit Volume

Based on Table 5.11, the average, standard deviation and coefficient of variation of the data sets for the tensile energy required to produce failure per unit volume were 1897 kJ/m^3, 390 and 21% respectively. The average, standard deviation and coefficient of variation of the tensile energy required to produce failure per unit volume within the individual pair of plates (i.e. pair of plates A, B and C) were as follows; 2031 kJ/m^3, 383 and 19% for pair of plates A, 1940 kJ/m^3, 421 and 22% for pair of plates B and 1720 kJ/m^3, 307 and 18% for pair of plates C. As has been stated before, the high variability of the data sets can be attributed to the estimation errors due to the curve fitting procedures (i.e. uncertainty that presents in a curve that is fitted to the data sets) and the parameters that governed the values of the tensile energy required to produce failure per unit volumes. Based on Figure 5.42, the values of the data sets tend to be flatter and spread out within the large range of 1250 kJ/m^3 and 3050 kJ/m^3.

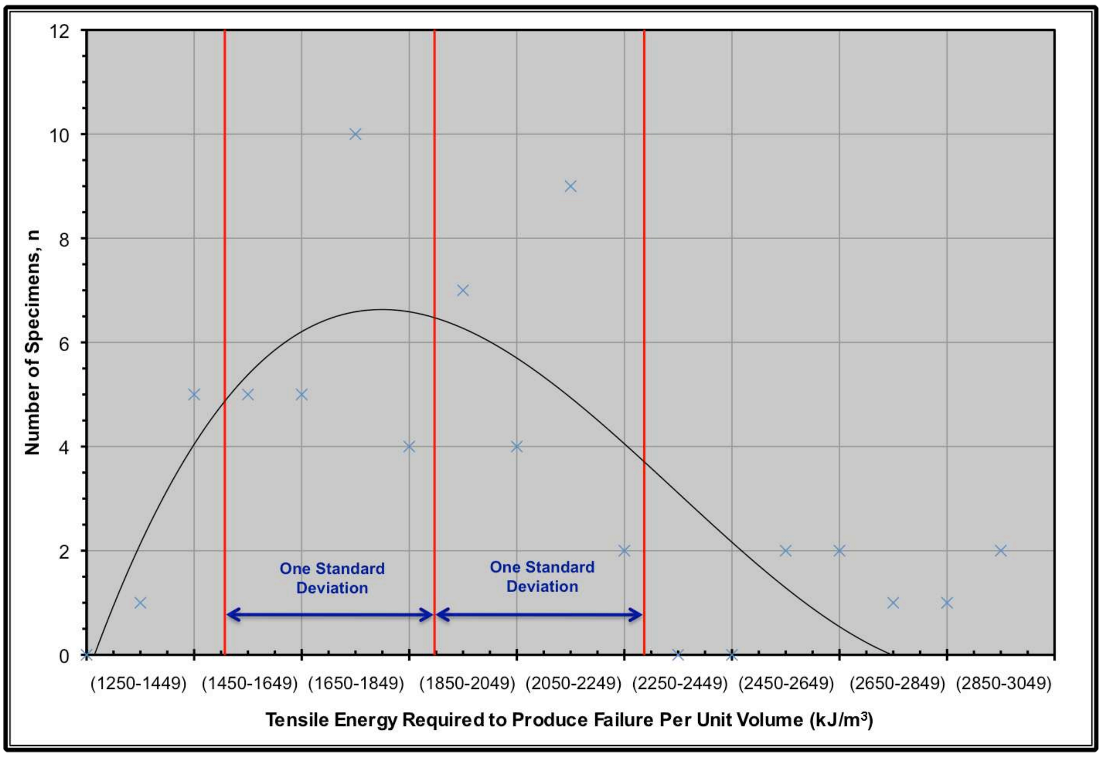


Figure 5.42 Distribution of tensile energy required to produce failure per unit volume

5.5.1.5 Analysis to Determine the Effect of Various Testing Conditions on the Test Results

Table 5.13 shows the data of the test results over wide ranges of deformation rates and test temperatures. Tensile energy required to produce failure per unit volume was not included in order to simplify the analysis. Based on the data of the test results, the following plots have been deduced, as shown in Figures 5.43 to 5.45. Again, the error bar represents the one-standard deviation above and below the average maximum tensile bond strength for Figures 5.43 and 5.44, and the one-standard deviation above and below the average total percentage area of adhesive failure for Figure 5.45, respectively.

Table 5.13 Data of the test results over wide ranges of deformation rates and test temperatures (Subsequent study using Ductilometer testing apparatus)

Deformation Rate (mm/minute)	Test Temperature (°C)	Data Sets	Thickness of Adhesive Layer of Bitumen (μm)	Total Percentage Area of Adhesive Failure (%)	Maximum Tensile Bond Strength (kPa)
10 (0.167 mm/s)	10	1	50	100	1550
		2	50	100	1530
		3	60	100	1590
		4	50	100	1470
		5	50	100	1690
	15	1	50	100	1440
		2	50	100	1430
		3	50	100	1480
		4	50	100	1380
		5	60	100	1490
	20	1	50	100	1330
		2	50	100	1390
		3	50	100	1320
		4	60	100	1400
		5	50	100	1470
	25	1	50	100	1270
		2	50	90	1280
		3	50	100	1290
		4	60	95	1300
		5	60	90	1310
	30	1	60	80	1180
		2	50	95	1150
		3	50	85	1130
		4	50	90	1180
		5	60	100	1190
20 (0.333 mm/s)	10	1	50	100	1620
		2	50	100	1690
		3	50	100	1610
		4	50	100	1740
		5	50	100	1700
	15	1	50	100	1510
		2	60	100	1520
		3	50	100	1550
		4	60	100	1610
		5	50	100	1490
	20	1	50	100	1450
		2	60	100	1440
		3	50	100	1420
		4	50	100	1340
		5	50	100	1380
	25	1	50	100	1350
		2	50	100	1340
		3	50	90	1290
		4	50	100	1340
		5	60	100	1310
	30	1	50	100	1230
		2	60	90	1280
		3	50	100	1290
		4	50	95	1270
		5	60	90	1110

Table 5.13 Data of the test results over wide ranges of deformation rates and test temperatures (Subsequent study using Ductilometer testing apparatus) (continued)

Deformation Rate (mm/minute)	Test Temperature (°C)	Data Sets	Thickness of Adhesive Layer of Bitumen (μm)	Total Percentage Area of Adhesive Failure (%)	Maximum Tensile Bond Strength (kPa)
30 (0.500 mm/s)	10	1	50	100	1770
		2	50	100	1730
		3	50	100	1680
		4	60	100	1720
		5	50	100	1890
	15	1	50	100	1620
		2	50	100	1730
		3	50	100	1520
		4	60	100	1680
		5	50	100	1620
	20	1	50	100	1520
		2	60	100	1550
		3	50	100	1519
		4	50	100	1490
		5	50	100	1470
	25	1	50	100	1340
		2	50	100	1450
		3	50	95	1420
		4	50	100	1410
		5	50	100	1320
	30	1	50	90	1380
		2	50	100	1330
		3	60	95	1280
		4	50	100	1320
		5	50	100	1320

Notes:
1. Substrates: Aluminium alloy
2. Adhesive Materials: Conventional 70/100 penetration grade of bitumen
3. Conditioning Procedures: Dry conditioning at specified test temperature for 24 hours prior to testing
4. Testing was conducted in wet conditioning in a temperature-controlled water bath at specified test temperature

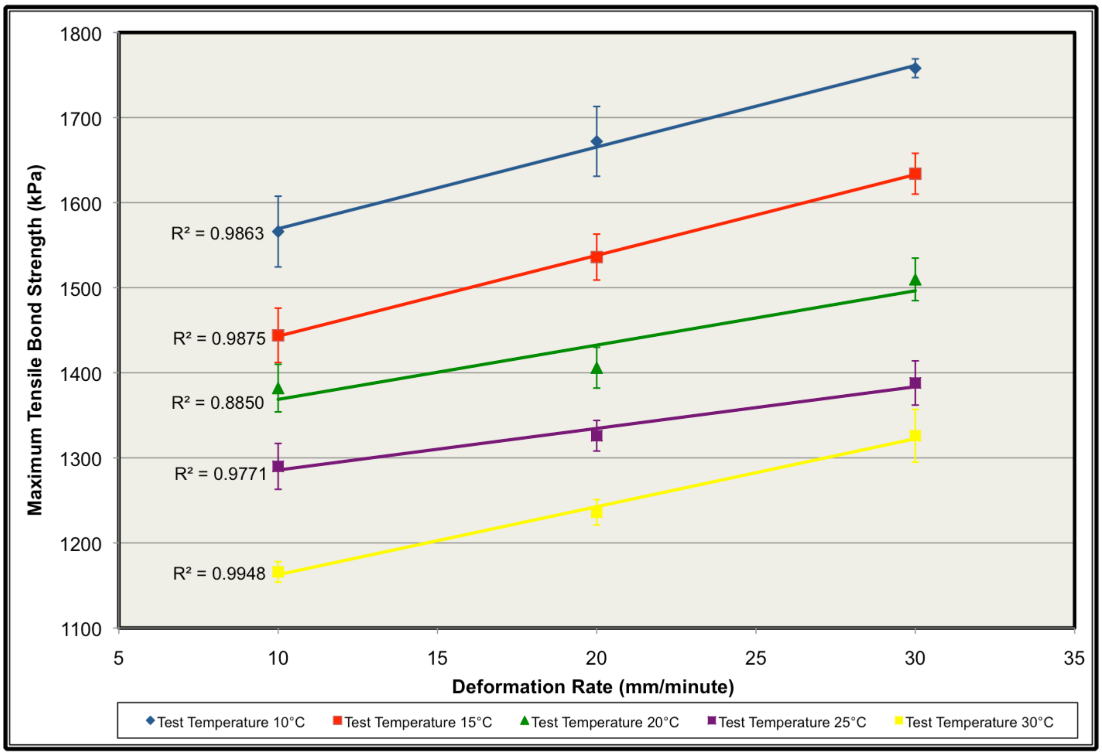


Figure 5.43 Relationship between maximum tensile bond strength and deformation rate over wide ranges of test temperatures

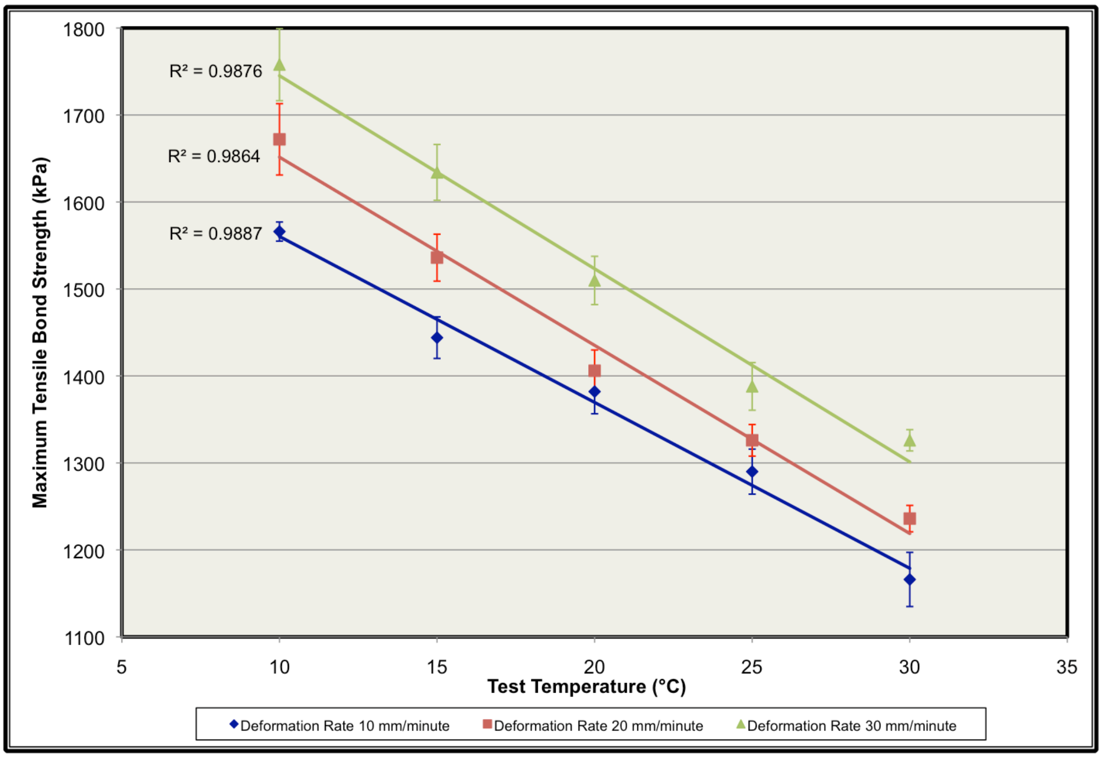


Figure 5.44 Relationship between maximum tensile bond strength and test temperature over wide ranges of deformation rates

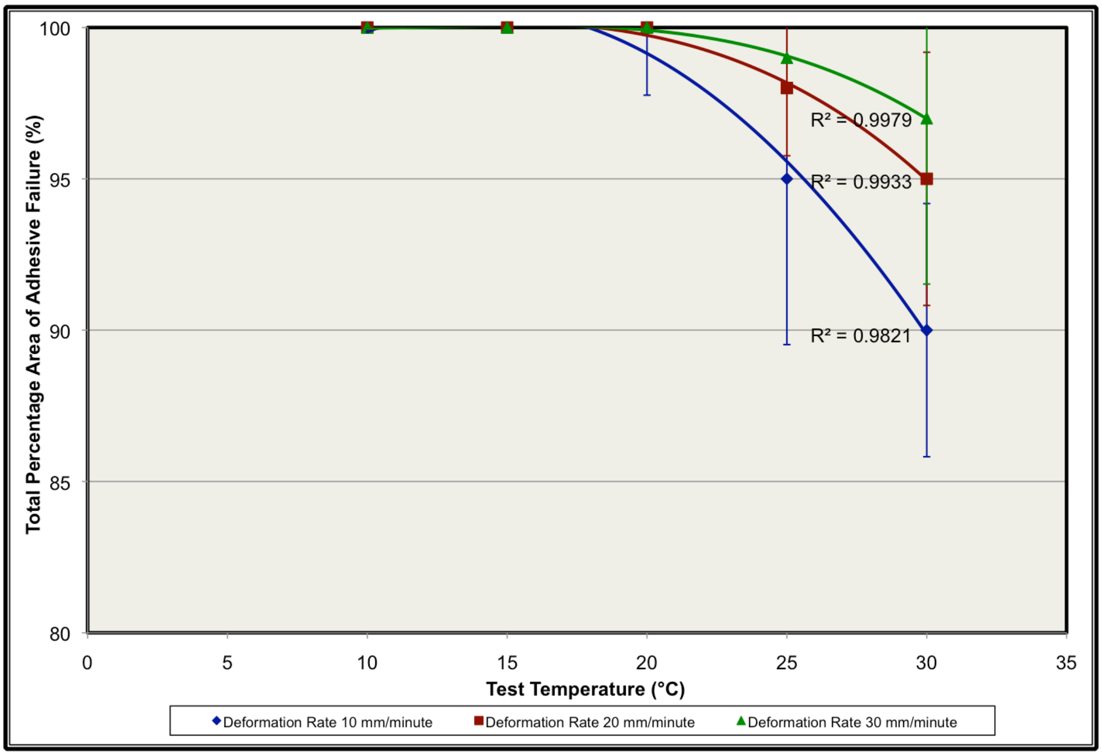


Figure 5.45 Relationship between total percentage area of adhesive failure and test temperature over wide ranges of deformation rates

Based on Figure 5.43, as expected, the maximum tensile bond strength was found to increase with the increasing deformation rate for all specified test temperatures. At a certain value of deformation rate, the maximum tensile bond strength of low-test temperature was higher compared to the maximum tensile bond strength of high-test temperature. A directly proportional relationship exists between maximum tensile bond strength and deformation rate with constant shift due to the increasing or decreasing of the test temperature. The values of the coefficient of determination, R^2 greater than 0.80 indicate a reasonable fits for the data sets.

Also, based on Figure 5.44, an inversely proportional relationship was observed, which indicated that as the test temperature is increased, the maximum tensile bond strength will decrease. Again, constant shift due to the increasing or decreasing deformation rate was found with the value of coefficient of determination, R^2 of more than 0.95. Based on Figure 5.45, deformation rate and test temperature were found to have a profound influence on the types of failure of specimens of either adhesive or cohesive, apart from the thickness of adhesive layer of bitumen. As the test temperature was increased or the deformation rate was decreased, mode of failure was expected to change from adhesive to cohesive. Again, at high test temperature of more than 25°C, high variability or distribution of the data sets was observed, as represented by the error bar of one-standard deviation above and below the average total percentage area of adhesive failure.

5.5.2 Summary of Subsequent Study using Ductilometer Testing Apparatus

In this section, the modifications that have been conducted following the completion of the preliminary study were the reduction of the thickness of adhesive layer of bitumen to 50 µm (0.050 mm) and the area of contact between bitumen and substrates to 100 mm^2. Procedures for specimen preparation were still based on the previously developed procedures. However, data of the thickness of adhesive layer of bitumen was found to result in significant statistical difference from the theoretical value of 50 µm (0.050 mm), which indicated the need for further improvements in terms of the procedures for specimen preparation. Due to the reduction of thickness of adhesive layer of bitumen, the occurrence of cobwebbing was minimised and the problem related to the strings or strands of the cobwebbing that re-adhere to the surface of the area of contact between bitumen and substrates during the testing, was solved. The ranges of suitable testing conditions in terms of deformation rate and test temperature was found to be the same as the subsequent study using INSTRON servo hydraulic frame (i.e. within 10 mm/minute and 20 mm/minute, and 15°C and 25°C, respectively)

5.6 Conclusions

In this chapter, two testing equipments (i.e. INSTRON servo hydraulic frame and Ductilometer testing apparatus), which have been identified as capable to conduct the adhesion test method based on the pull off (tension) mode, were subjected to evaluation. Development of criteria and procedures in terms of test setup and apparatus, specimen preparation, testing and data analysis for both of the INSTRON servo hydraulic frame and Ductilometer testing apparatus were established. Based on the preliminary and subsequent study, data of the test results was summarised and presented in Table 5.14.

Table 5.14 Summary of data of the test results of Part 2: Development of Criteria and Procedures for the Proposed Adhesion Test Method

	Area of Contact between Bitumen and Substrates (mm^2)	Thickness of Adhesive Layer of Bitumen		Average Total Percentage Area of Adhesive Failure (%)	Average Maximum Tensile Bond Strength (kPa)	Average Tensile Energy Required to Produce Failure Per Unit Volume (kJ/m^3)
		Average (μm)	Evidence of Significant Statistical Difference			
Preliminary Study - INSTRON Servo Hydraulic Frame	10,000	816 (COV = 2%)	YES	23 (COV = 37%) Cohesive Failure	851 (COV = 9%)	982 (COV = 17%)
Subsequent Study - INSTRON Servo Hydraulic Frame	490.87	51 (COV = 4%)	NO	99 (COV = 2%) Adhesive Failure	1306 (COV = 5%)	2159 (COV = 20%)
Preliminary Study - Ductilometer Testing Apparatus[1]	200	574 (COV = 6%)	YES	58 (COV = 36%) Mixed Failure	969 (COV = 12%)	1149 (COV = 20%)
Subsequent Study - Ductilometer Testing Apparatus[1]	100	52 (COV = 7%)	YES	98 (COV = 4%) Adhesive Failure	1320 (COV = 5%)	1897 (COV = 21%)

Notes:
1. [1]Testing was conducted in wet conditioning in a temperature-controlled water bath at specified test temperature
2. COV: Coefficient of Variation
3. Substrates: Aluminium alloy
4. Adhesive Materials: Conventional 70/100 penetration grade of bitumen
5. Conditioning Procedures: Dry conditioning at 25°C for 24 hours prior to testing
6. Testing Conditions: Deformation rate and test temperature of 20 mm/minute and 25°C respectively

The optimum thickness of adhesive layer of bitumen that can result in the adhesive mode of failure and at the same time maintaining the uniformity and repeatability of the test results was selected to be 50 μm (0.050 mm). Based on Marek and Herrin (1968), too low thickness of adhesive layer of bitumen could compromise the precision in the measurement and even a very small difference of thickness between data sets could result in significant difference in the test results. Hence, 50 μm (0.050 mm) was considered as sufficient to result in adhesive mode of failure with excellent uniformity and repeatability of the test results. However, there is limitation in the measurement since the micrometer being used can only measure to the nearest 10 μm (0.010 mm). It is suggested that the measurement of the thickness of adhesive layer of bitumen be improved via a more precise measurement tools.

Also, total conditioning time of 24 hours has been considered as the standard conditioning procedures for both dry and wet conditionings, regardless of the substrates. Temperature for the conditioning procedures was dependent on the test temperature. Based on Copeland (2007), the optimum time required for the conditioning procedures is suggested to be within 8 hours to 24 hours. A standard procedure in determining the types of failure of the specimens was developed, as presented in Appendix B. Adhesive mode of failure was characterised by the value of the total percentage area of adhesive failure of more than 90%.

Based on the data of the test results in Table 5.14, INSTRON servo hydraulic frame was found to be the most suitable and practical testing equipment compared to the Ductilometer testing apparatus. Test setup and apparatus and procedures for specimen preparation based on the subsequent study using INSTRON servo hydraulic frame were proven capable of producing specimen as close as possible to the required thickness of adhesive layer of bitumen (i.e. theoretical value). In terms of the uniformity and repeatability of the test results, except for the tensile energy required to produce failure per unit volume, all parameters have resulted in coefficient of variation of less than 7%. High variability of the data sets for tensile energy required to produce failure per unit volume can be correlated with the estimation errors due to the curve fitting procedures (i.e. uncertainty that presents in a curve that is fitted to the data sets) and also the various parameters that governed the values of the tensile energy required to produce failure per unit volumes such as tensile load, pull off displacement, thickness of adhesive layer of bitumen and total percentage area of adhesive failure.

Other factors favouring INSTRON servo hydraulic frame as testing equipment were the limitation in the tensile load that can be measured using Ductilometer testing apparatus and inability of the Ductilometer testing apparatus in conducting test in dry conditioning over wide ranges of test temperatures. Also, design of the test setup and apparatus based on the subsequent study using INSTRON servo hydraulic frame was found practical, as outlined below.

1. Ability to allow for the insertion of various types of aggregates as substrates in the form of 25 mm diameter discs.

2. Ability to condition the specimen in water.

3. Ability to cater for various thicknesses of adhesive layer of bitumen.

4. Ability to uniformly distribute axial tensile load throughout the coated surface of specimen.

5. Eliminate edge effect due to the selection of circle (i.e. disc) as geometry for area of contact between bitumen and substrates.

Based on the comparison of the data of the test results with the data of the literature review and analysis of the past studies conducted by Copeland (2007), Kanitpong and Bahia (2003) and Marek and Herrin (1968), as summarised in Table 5.15, it can be concluded that the value of the average maximum tensile bond strength of 1306 kPa was comparable to the other studies. Difference in the testing variables and parameters such as penetration grade of bitumen, testing conditions (i.e. deformation rates and test temperatures) and thickness of adhesive layer of bitumen justified the differences in the maximum tensile bond strength. Adhesion test method conducted by Marek and Herrin (1968) using a 1-inch diameter pair of cylindrical test blocks was found to be the closely matched to the proposed adhesion test method, in terms of the area of contact between bitumen and substrates, thickness of adhesive layer bitumen, deformation rate and test temperature. However, average maximum tensile bond strength of 4075 kPa of Marek and Herrin (1968) was higher compared to the 1306 kPa of the conducted study, which can be attributed to the difference in the penetration grade of bitumen. It should be noted that the asphalt cement K used by Marek and Herrin (1968) has lower penetration grade of bitumen (i.e. 52) and thus justify the high value of the tensile bond strength. Hence, criteria and procedures for the proposed adhesion test method were established based on the subsequent study using INSTRON servo hydraulic frame. Draft of standard criteria and procedures for laboratory adhesion test method using INSTRON servo hydraulic frame is presented in Appendix C. Also, based on the study of the adhesion test method conducted over wide ranges of testing conditions, the ranges of suitable deformation rate and test temperature were suggested to be 10 mm/minute and 20 mm/minute, and 15°C and 25°C respectively. However, no definite conclusion can be made yet due to the limited number of tested specimens.

Table 5.15 Comparison of data of test results with data of literature review and analysis of past studies

	Brief Descriptions		Area of Contact between Bitumen and Substrates (mm^2)	Average Thickness of Adhesive Layer of Bitumen (μm)	Average Maximum Tensile Bond Strength (kPa)
Data of Conducted Study – Subsequent Study Using INSTRON Servo Hydraulic Frame	Substrates	Aluminium alloy	490.87	50	1306
	Adhesive Materials	Conventional 70/100 penetration grade of bitumen (Penetration at 25°C is 68)			
	Testing Conditions	Deformation rate and test temperature of 20 mm/minute and 25°C			
Source: Marek and Herrin (1968)	Substrates	Aluminium alloy	506.71	60	4075
	Adhesive Materials	Asphalt cement K (Penetration at 25°C is 52)			
	Testing Conditions	Deformation rate and test temperature of 25.4 mm/minute and 25°C			
Source: Kanitpong and Bahia (2003)	Substrates	Glass	126.68	200	1982
	Adhesive Materials	PG 58-28			
	Testing Conditions	Deformation rate of 65.7 kPa/second at room temperature			
Source: Copeland (2007)	Substrates	Diabase	1520.53	200	1910
	Adhesive Materials	AAM (PG64-16)			
	Testing Conditions	Deformation rate of 65.7 kPa/second at room temperature			

Notes: All data of the test results were obtained from specimens subjected to dry conditioning

CHAPTER 6

PART 3: DETAILED EVALUATION AND VALIDATION OF THE PROPOSED ADHESION TEST METHOD

6.1 General Background

This part is a continuation from the previous part (i.e. Part 2: Development of Criteria and Procedures for the Proposed Adhesion Test Method) where the established criteria and procedures in terms of test setup and apparatus, specimen preparation, testing and data analysis will be subjected to further evaluation. The main objective of this part was to further evaluate the established criteria and procedures in quantifying the adhesive bond strength and failure characteristics of various combinations of asphalt mixture materials (i.e. bitumen (bitumen-filler mastic) and aggregates) over wide ranges of thicknesses of adhesive layer of bitumen, aspect ratio of specimens, testing conditions (i.e. deformation rates and test temperatures) and conditioning procedures (i.e. dry and wet conditionings). Results of the study will be subjected to comparative analysis in order to determine the effect of various variables and parameters on the test results, to propose suitable testing conditions and to validate the reliability and efficiency of the proposed adhesion test method.

In the previous part, the development of criteria and procedures for the proposed adhesion test method was conducted generally without emphasis on using various combinations of asphalt mixture materials and various conditioning procedures. Aluminium alloy, conventional 70/100 penetration grade of bitumen and dry conditioning for 24 hours prior to testing were the selected asphalt mixture materials (i.e. substrates and adhesive materials) and conditioning procedures for the previous part, respectively. Hence, in order to achieve the main objective of this part and also to enable for the generalisation of conclusions for the whole study, various combinations of asphalt mixture materials over wide ranges of testing conditions were evaluated.

Based on the conclusions of the previous part, INSTRON servo hydraulic frame was selected as standard testing equipment, and the draft of standard criteria and procedures for the laboratory adhesion test method are presented in Appendix C. However, the pair of plates which allow for the insertion of various types of aggregates as substrates in the forms of 25 mm diameter discs, had drawbacks due to difficulty in removing the excess bitumen (i.e. overfilling) at the edges of the specimens, as shown in Figure 6.1. Based on Marek and Herrin (1968), excess bitumen at the edges of the specimens should be removed prior to testing in order to prevent any discrepancy and inaccuracy. However, different conclusions have been drawn regarding the effect of the amount of poured bitumen (i.e. under filling, sufficiently filled or overfilling) on the test results. Hence, a simple analysis regarding this issue was conducted.

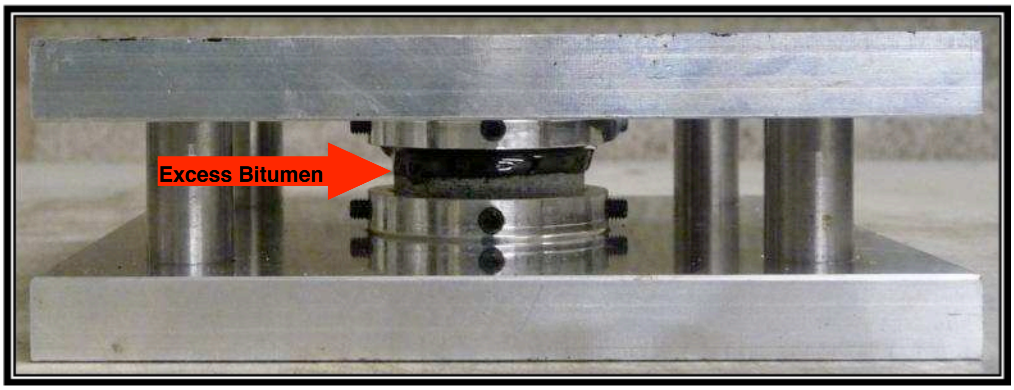


Figure 6.1 Excess bitumen (i.e. overfilling) at the edges of specimen

6.1.1 Analysis of Treatment Effect in Terms of the Amount of Poured Bitumen

Analysis was required to determine whether there is a treatment effect in terms of the amount of poured bitumen on the test results. The following treatments based on the different amount of poured bitumen were considered in the analysis; under filling, sufficiently filled and overfilling. The terms of under filling and overfilling referred to the amount of poured bitumen that has been reasonably filled below and beyond the sufficiently filled, respectively. One-way analysis of variance (ANOVA) was conducted with level of significance, α of 0.05 in order to analyse and compare the average maximum tensile bond strength (i.e. maximum tensile load per unit area of contact) of the considered treatments.

Maximum tensile bond strength was calculated as follows; maximum tensile load divided by the unit area of contact. Since the actual area of contact between bitumen and substrates is realistically difficult to be measured, the following assumptions were made; area of contact between bitumen and substrates is considered constant, regardless of the considered treatments (i.e. under filling, sufficiently filled or overfilling), and the discrepancy and inaccuracy due to the area of contact between bitumen and substrates subjected to the under filling and overfilling can be minimised by pouring the bitumen to reasonably fill below and beyond the sufficiently filled, respectively.

Data of the test results based on the considered treatments were presented in Tables 6.1 to 6.3. The average maximum tensile bond strength for the considered treatments of under filling, sufficiently filled and overfilling were

1273 kPa, 1305 kPa and 1314 kPa respectively. The following hypotheses were then established.

1. The null hypothesis, H_0

   H_0: $\overline{X}_{\text{Under Filling}} = \overline{X}_{\text{Sufficiently Filled}} = \overline{X}_{\text{Overfilling}}$

   There is no difference in terms of the average maximum tensile bond strength among the considered treatments.

2. The alternative hypothesis, H_1

   H_1: Not all the average maximum tensile bond strength are equal.

Microsoft Excel statistical analysis has been used to perform ANOVA test of equality of population means with level of significance, α of 0.05 and Figure 6.2 summarises the output of the analysis.

Table 6.1 Data of the test results (Under filling)

A	B	C	D	E	F = (E/(490.87)) X 1000
Pair of Plates	Data Sets	Thickness of Adhesive Layer of Bitumen (μm)	Total Percentage Area of Adhesive Failure (%)	Maximum Tensile Load (N)	Maximum Tensile Bond Strength (kPa)
A	1	50	100	648	1320
	2	50	100	623	1270
	3	50	100	584	1190
	4	50	100	629	1281
B	1	50	100	619	1260
	2	50	100	623	1270
	3	50	100	611	1245
	4	50	100	639	1302
C	1	50	100	633	1290
	2	50	100	637	1297
	3	50	100	625	1274
	4	50	100	628	1280
Average		50	100	625	1273
Standard Deviation		0	0	16	33
Coefficient of Variation (%)		0	0	3	3

Notes:
1. Area of Contact between Bitumen and Substrates: 490.87 mm^2
2. Substrates: Aluminium alloy
3. Adhesive Materials: Conventional 70/100 penetration grade of bitumen
4. Conditioning Procedures: Dry conditioning at 25°C for 24 hours prior to testing
5. Testing Conditions: Deformation rate and test temperature of 20 mm/minute and 25°C respectively

Table 6.2 Data of the test results (Sufficiently filled)

A	B	C	D	E	F = (E/(490.87)) X 1000
Pair of Plates	Data Sets	Thickness of Adhesive Layer of Bitumen (µm)	Total Percentage Area of Adhesive Failure (%)	Maximum Tensile Load (N)	Maximum Tensile Bond Strength (kPa)
A	1	50	100	655	1334
	2	50	100	631	1285
	3	50	100	657	1339
	4	50	100	588	1198
B	1	50	100	628	1279
	2	50	100	616	1254
	3	50	100	638	1300
	4	50	100	674	1374
C	1	50	100	638	1299
	2	50	100	683	1392
	3	50	100	634	1292
	4	50	100	643	1309
Average		50	100	640	1305
Standard Deviation		0	0	26	52
Coefficient of Variation (%)		0	0	4	4

Notes:
1. Area of Contact between Bitumen and Substrates: 490.87 mm^2
2. Substrates: Aluminium alloy
3. Adhesive Materials: Conventional 70/100 penetration grade of bitumen
4. Conditioning Procedures: Dry conditioning at 25°C for 24 hours prior to testing
5. Testing Conditions: Deformation rate and test temperature of 20 mm/minute and 25°C respectively

Table 6.3 Data of the test results (Overfilling)

A	B	C	D	E	F = (E/(490.87)) X 1000
Pair of Plates	Data Sets	Thickness of Adhesive Layer of Bitumen (µm)	Total Percentage Area of Adhesive Failure (%)	Maximum Tensile Load (N)	Maximum Tensile Bond Strength (kPa)
A	1	50	100	647	1318
	2	50	100	676	1377
	3	50	100	665	1355
	4	50	100	652	1329
B	1	50	80	628	1280
	2	50	95	637	1297
	3	50	100	652	1329
	4	50	100	638	1300
C	1	50	100	652	1329
	2	50	100	632	1287
	3	50	100	612	1247
	4	50	100	646	1316
Average		50	98	645	1314
Standard Deviation		0	6	17	35
Coefficient of Variation (%)		0	6	3	3

Notes:
1. Area of Contact between Bitumen and Substrates: 490.87 mm^2
2. Substrates: Aluminium alloy
3. Adhesive Materials: Conventional 70/100 penetration grade of bitumen
4. Conditioning Procedures: Dry conditioning at 25°C for 24 hours prior to testing
5. Testing Conditions: Deformation rate and test temperature of 20 mm/minute and 25°C respectively

Analysis of Variance (One-Way)

SUMMARY

Groups	Sample Size	Sum	Mean	Variance
Under filling	12	15 279	1273	1080.75
Sufficiently filled	12	15 655	1305	2699.72
Overfilling	12	15 764	1314	1200.24

ANOVA

Source of Variation	SS	df	MS	F	p-value	F crit
Between Groups	10791.1667	2	5395.5833	3.2499	0.0515	3.2849
Within Groups	54787.8333	33	1660.2374			
Total	65579.0000	35				

Figure 6.2 One-way analysis of variance (ANOVA) for treatment effect in terms of the amount of poured bitumen

The p-value approach was used in making direct conclusion about the null hypothesis, H_0. The p-value is the probability of obtaining an F distribution as large as or larger than the one obtained, given that the null hypothesis, H_0 is true. The decision rule for rejecting H_0 based on the p-value approach is as follows.

Reject H_0 if p-value is smaller than level of significance, α.

Otherwise, fail to reject H_0.

Therefore, based on Figure 6.2 the p-value or probability of obtaining an F distribution of 3.2499 or larger, when the null hypothesis, H_0 is true is 0.0515. Since the p-value is larger than the specified level of significance, α of 0.05, the null hypothesis, H_0 failed to be rejected. However, since the p-value of 0.0515 is only slightly larger than the specified level of significance, α of 0.05, further analysis was required.

Unpaired (i.e. Independent) Two-Samples t-Test procedure, at level of significance, α of 0.05, was conducted based on the following three pair of data sets of the considered treatments; under filling and sufficiently filled, under filling and overfilling, and sufficiently filled and overfilling. Table 6.4 summarises the output of the analysis of the Unpaired (i.e. Independent) Two-Samples t-Test procedure. The decision rule in determining the existence of significant statistical difference in the pair of data sets of the considered treatments is as follows.

If two-tailed p-value is smaller than level of significance, α, significant statistical difference exists in the pair of data sets.
Otherwise, there is no significant statistical difference in the pair of data sets.

Based on the summary of statistical analysis of Unpaired (i.e. Independent) Two-Samples t-Test procedure shown in Table 6.4, it can be concluded that there is sufficient evidence of significant statistical difference in the pair of data sets of under filling and overfilling. However, the pair of data sets for the following considered treatments; under filling and sufficiently filled, and sufficiently filled and overfilling has shown no significant statistical difference in the average measured maximum tensile bond strength. Hence, improved procedures for specimen preparation are required in order to ensure the uniformity and consistency of the amount of poured bitumen. It is suggested that a reasonable amount of bitumen be poured until fully coated surface of the bottom plate is achieved, in order to result in overfilling. Since the two-tailed p-value for the pair of data sets of sufficiently filled and overfilling is larger than the level of significance, α, which indicated high level of no significant statistical difference, the amount of poured bitumen is suggested to be within this range (i.e. the amount of poured bitumen is the sufficiently filled and overfilling).

Table 6.4 Summary of statistical analysis of Unpaired (i.e. Independent) Two-Samples t-Test procedure

	Pair of Data Sets of Considered Treatments					
	Pair 1		Pair 2		Pair 3	
	Under Filling	Sufficiently Filled	Under Filling	Overfilling	Sufficiently Filled	Overfilling
Mean	1273 kPa	1305 kPa	1273 kPa	1314 kPa	1305 kPa	1314 kPa
Standard Deviation	33	52	33	35	52	35
SE Mean	9.49	15.00	9.49	10.00	15.00	10.00
95.0% CI	(-68.14, 5.48)		(-69.01, -11.82)		(-46.47, 28.30)	
t-statistic, t	1.7653		2.9315		0.5039	
Two-tailed p-value	0.0914		0.0077		0.6194	
Significant Difference	NO		YES		NO	

6.1.2 Selection of Asphalt Mixture Materials

In order to consider wide ranges of asphalt mixture materials, at least two types of aggregates and/or bitumen (bitumen-filler mastic) of distinct properties that will reflect the ranges of typically used asphalt mixtures need to be utilised. In this study, aluminium alloy, granite and two types of limestone were used as substrates, and conventional 70/100 penetration grade of bitumen was used as control adhesive materials. Three types of mineral filler (i.e. Hydrated Lime, Limestone and Gritstone) were selected in order to produce various types of bitumen-filler mastic. Aluminium alloy was selected as control substrates due to the value of Young's Modulus of approximately 70 GPa, which is close to the typical value of aggregates and also due to the corrosion resistance properties (Harvey 2000). Aggregates (i.e. Dene Limestone, Ivonbrook Limestone and Mount Sorrel Granite) were selected due to availability and distinct properties in terms of the classification as acidic (i.e. hydrophilic) or basic (i.e. hydrophobic). Aggregates were prepared into discs of 25 mm diameter (i.e. 490.87 mm^2 area of contact) and 8 mm thickness via the cutting of the 25 mm diameter cylindrical core specimens extracted from a boulder, as shown in Figure 6.3. In order to minimise the discrepancy and inaccuracy of the test results especially in terms of the uniformity of the thickness of adhesive layer of bitumen, aggregates discs should be precisely cut to 8 mm thickness.

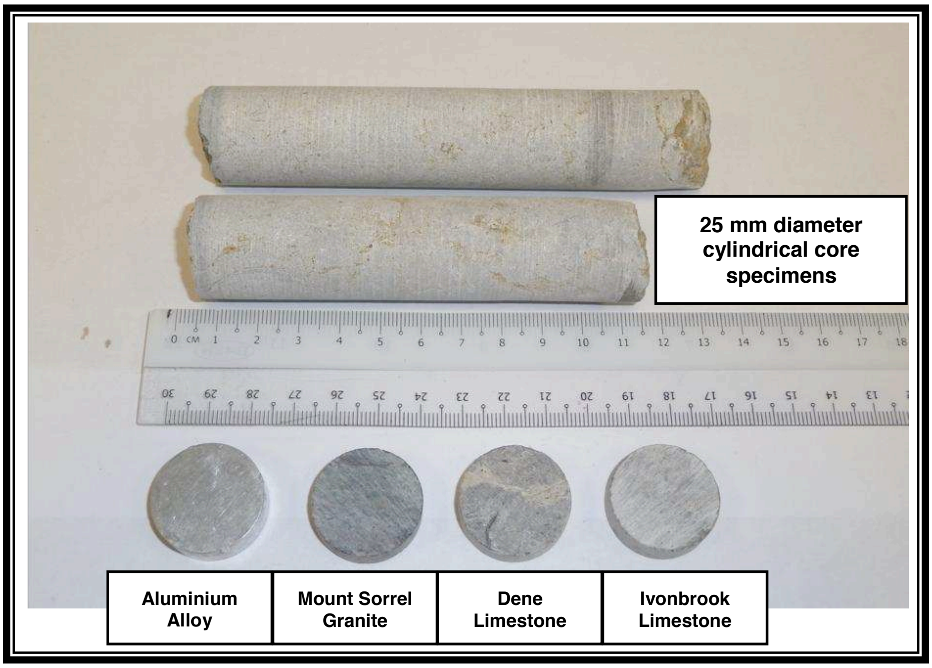


Figure 6.3 Cylindrical core specimens and aggregates discs

Surface of area of contact of aggregates discs (i.e. Dene Limestone, Ivonbrook Limestone and Mount Sorrel Granite) was not treated or modified in any way, and the aggregates discs were used as they were cut. However, surface of area of contact of aluminium alloy discs was polished in order to closely match the roughness, R_a of the surface of area of contact of aggregates discs, which lies in the range of 2.43 μm and 2.54 μm. The reason for this is to minimise the differences in properties among the substrates since adhesive bond strength can vary with roughness of the aggregates surface. Analysis of the surface profile using Mitutoyo-Surftest SV-600 was conducted in order to verify the roughness, R_a of each substrate, and Figure 6.4 shows the result of the roughness, R_a of the surface of Ivonbrook Limestone. Also, each substrate in the forms of discs was subjected to water absorption test based on the BS EN 1097-6:2000 Tests for Mechanical and Physical Properties of Aggregates Determination of Particle Density and Water Absorption. Based on the literature review and analysis of the past studies, large percentage of water absorption could be an indicator of possible moisture susceptibility of the substrates. However, since the results of water absorption is very subjective, it is only used as a rough evaluation in determining the adhesive bond strength and failure characteristics of the asphalt mixtures. Table 6.5 shows the properties of the aggregates discs.

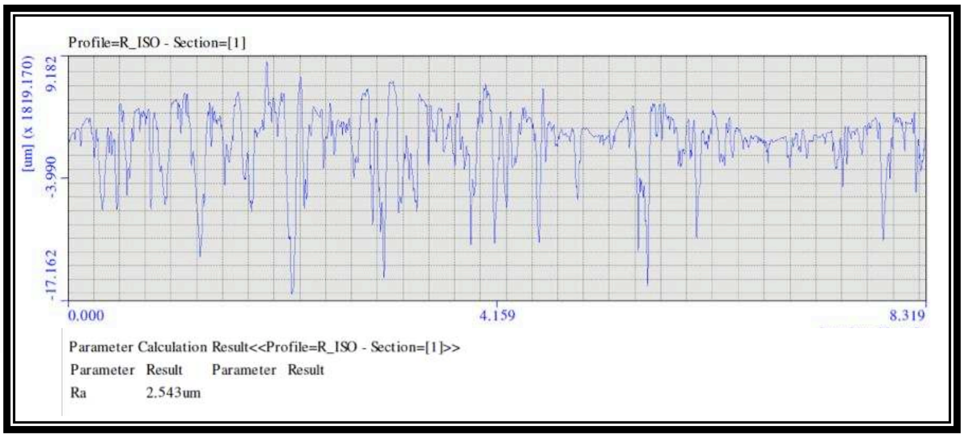


Figure 6.4 Result of the roughness, R_a of the surface of Ivonbrook Limestone

Table 6.5 Properties of aggregates discs

Substrates	Roughness of Surface of Area of Contact, R_a (μm)	Water Absorption (%)[1]	Classification of Aggregates (Acidic or Basic)
Aluminium Alloy	2.41	0.05	-
Dene Limestone	2.43	0.15	Basic
Ivonbrook Limestone	2.54	0.40	Basic
Mount Sorrel Granite	2.46	0.30	Acidic

Notes: [1]Based on BS EN 1097-6:2000 Tests for Mechanical and Physical Properties of Aggregates. Determination of Particle Density and Water Absorption

Empirical rheological tests were performed on the bitumen (bitumen-filler mastic), which includes penetration test (BS EN 1426:2007 Bitumen and Bituminous Binders. Determination of Needle Penetration) and Ring and Ball Method Softening Point Test (BS EN 1427:2007 Bitumen and Bituminous Binders. Determination of the Softening Point. Ring and Ball Method). Mineral filler used in this study satisfied the requirement of 70% to 100% of particles passing a 63-μm sieve based on BS EN 13043:2002 Aggregates for Bituminous Mixtures and Surface Treatments for Roads, Airfields and Other Trafficked Areas. In order to achieve the thickness of adhesive layer of bitumen of 50 μm (0.050 mm), mineral filler was further sieved through a 45-μm sieve (British Standard Sieve Series Mesh No. 350) before being homogenously distributed within bitumen in order to produce various types of bitumen-filler mastic. Procedures for mixing the bitumen-filler mastic are based on the in-house standard of LOP 11.24 Blending Bitumen and Filler Laboratory Operation Procedures – Equipment Usage, as presented in Appendix D. Tables 6.6 shows the properties of the bitumen (bitumen-filler mastic).

Table 6.6 Properties of bitumen (bitumen-filler mastic)

Adhesive Materials	Filler	Penetration at 25°C (d.mm)[1]	Softening Point (°C)[2]	Classification of Filler (Acidic or Basic)
Conventional 70/100 Penetration Grade of Bitumen	-	68	49.8	-
	20% by Volume of Hydrated Lime	38	61.7	Basic
	20% by Volume of Limestone	45	58.4	Basic
	20% by Volume of Gritstone	48	56.7	Acidic

Note: [1]Based on BS EN 1426:2007 Bitumen and Bituminous Binders. Determination of Needle Penetration; [2]Based on BS EN 1427:2007 Bitumen and Bituminous Binders. Determination of the Softening Point. Ring and Ball Method

6.2 Analysis to Determine the Effect of Thickness of Adhesive Layer of Bitumen on the Test Results

In the previous chapter (i.e. Chapter 5), the optimum thickness of adhesive layer of bitumen for the proposed adhesion test method was suggested to be 50 μm (0.050 mm), in order to result in the adhesive mode of failure and also to achieve the uniformity and repeatability of the test results. However, in this section, various thicknesses of adhesive layer of bitumen were prepared and tested in order to study the effect on the test results. In order to cater for the various thicknesses of adhesive layer of bitumen, spacers of different thicknesses were fabricated. Based on the literature review and analysis of the past studies, thickness of adhesive layer of bitumen was found to have a profound influence on the adhesive bond strength and failure characteristics (i.e. tensile bond strength and types of failure of specimens).

In this section, aluminium alloy and conventional 70/100 penetration grade of bitumen were used as control substrates and adhesive materials respectively. A total of 100 specimens of various thicknesses in the range of 50 μm (0.050 mm) and 990 μm (0.990 mm) were subjected to dry conditioning at 25°C for

24 hours before being tested at fixed deformation rate and test temperature of 20 mm/minute and 25°C respectively. Table 6.7 shows the data of the test results.

Table 6.7 Results based on different thickness of adhesive layer of bitumen

Thickness of Adhesive Layer of Bitumen (μm)	Total Percentage Area of Adhesive Failure (%)	Maximum Tensile Bond Strength (kPa)	Tensile Energy Required to Produce Failure Per Unit Volume (kJ/m^3)
990	15	660	806
980	25	780	654
970	35	720	972
960	25	810	765
940	20	760	750
930	30	730	952
920	20	520	990
900	45	860	821
890	15	840	946
880	15	750	1152
860	15	830	1055
850	30	810	1149
840	15	720	871
830	15	800	904
830	10	690	1052
810	30	820	1065
790	30	800	1024
690	35	910	1016
680	40	730	764
660	40	990	1352
660	20	1000	847
640	45	840	1422
640	40	840	923
620	20	860	931
620	40	740	535
600	30	860	835
600	50	780	711
580	70	840	631
580	40	880	671
580	30	830	764
580	40	890	671
560	40	860	1226
560	60	970	501
560	50	850	882
560	40	880	869
540	50	950	649
540	40	920	1016
540	40	980	1016
530	50	810	1059
530	60	1010	1161
360	80	880	1467
350	100	890	870
350	80	851	1516
350	100	820	1927
320	85	870	1873
320	90	820	1304
320	100	790	1135
320	90	810	2156
320	100	920	1400
320	100	860	1627
280	100	870	1522
280	90	910	1704

Table 6.7 Results based on different thickness of adhesive layer of bitumen (continued)

Thickness of Adhesive Layer of Bitumen (μm)	Total Percentage Area of Adhesive Failure (%)	Maximum Tensile Bond Strength (kPa)	Tensile Energy Required to Produce Failure Per Unit Volume (kJ/m^3)
250	100	990	1321
250	100	890	2035
250	100	1020	2819
240	100	970	1713
240	100	1000	2057
240	90	940	1798
230	100	1020	1674
230	100	980	1240
130	100	1170	1253
130	100	1140	1100
130	100	1150	1550
120	90	1160	2107
120	100	1180	1917
120	100	1210	1610
120	95	1150	1711
120	100	1220	1646
120	100	1190	2097
120	100	1170	1913
120	100	1100	1858
120	100	1170	1763
120	85	1140	1925
120	100	1150	2173
100	90	1170	1746
100	100	1180	1880
100	100	1210	1815
100	100	1190	1759
100	100	1230	2139
100	85	1170	1705
100	100	1200	1861
100	100	1180	2117
100	100	1220	1960
90	100	1230	1784
90	100	1220	2039
90	100	1280	1845
90	95	1210	1504
90	100	1250	1823
60	100	1290	2306
60	100	1330	2342
60	100	1300	2004
60	100	1240	1963
60	90	1290	2149
50	100	1300	2096
50	100	1310	2108
50	90	1250	2325
50	100	1270	1824
50	95	1330	2173
50	100	1290	2526
50	100	1340	3165

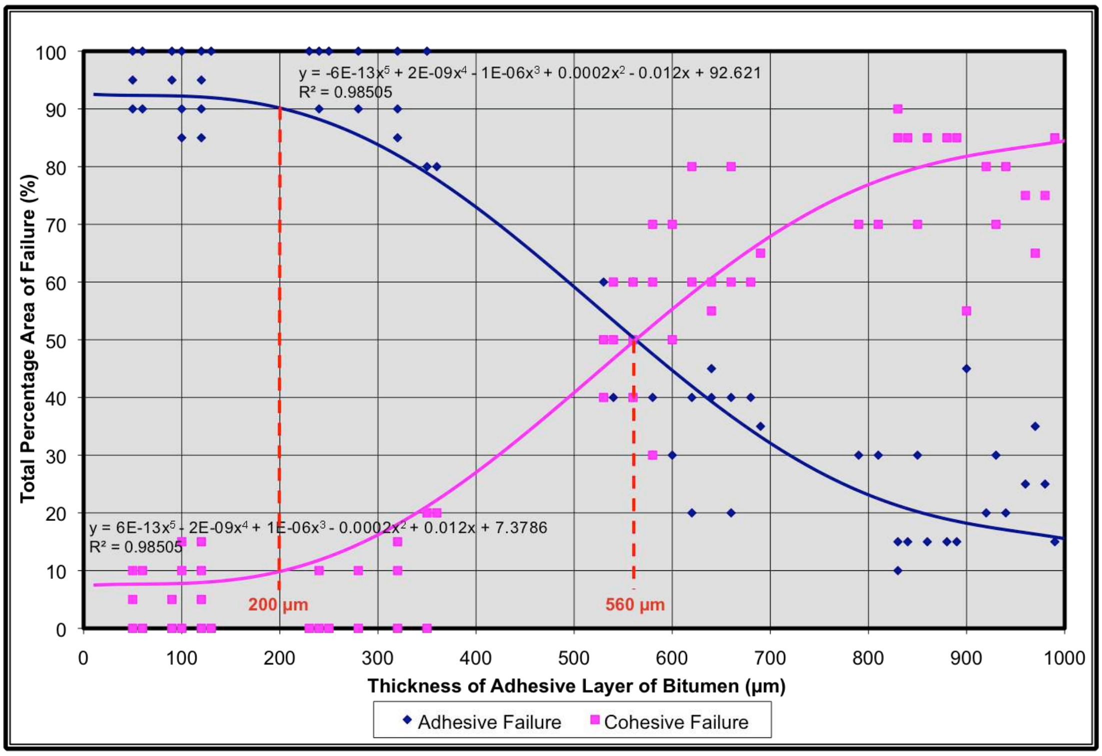
Notes:
1. Substrates: Aluminium alloy
2. Adhesive Materials: Conventional 70/100 penetration grade of bitumen
3. Conditioning Procedures: Dry conditioning at 25°C for 24 hours prior to testing
4. Testing Conditions: Deformation rate and test temperature of 20 mm/minute and 25°C respectively

6.2.1 Relationship between Total Percentage Area of Failure and Thickness of Adhesive Layer of Bitumen

Based on the data of the test results in Table 6.7, best-fit curve has been plotted to determine the relationship between total percentage area of adhesive failure and various thicknesses of adhesive layer of bitumen, as illustrated in Figure 6.5. Curve of the cohesive failure was then derived by reflecting the curve of the adhesive failure through the horizontal axis of symmetry at the value of the total percentage area of failure of 50% (i.e. x-axis at total percentage area of failure of 50%). The equation for the best-fit curve and the value of the coefficient of determination, R^2 were included. Based on GraphPad Software (n.d.), the value of the coefficient of determination, R^2 can be used to quantify the goodness of fit of nonlinear regression. Higher value of the coefficient of determination, R^2 is considered to show a reasonable fits for the data sets.

Based on Figure 6.5, the types of failure of either adhesive or cohesive can be modelled as a S-shaped curve with respect to the thickness of adhesive layer of bitumen. As has been stated before, adhesive mode of failure was characterised by the value of the total percentage area of adhesive failure of more than 90%. Hence, based on the specified asphalt mixture materials and testing conditions, transition of the mode of failure of either adhesive or mixed cohesive and adhesive is expected to occur at the thickness of 200 µm (0.200 mm) (i.e. with respect to the value of the total percentage area of adhesive failure of 90%). Mode of failure was found to change from adhesive to mixed cohesive and adhesive, and then expected to become entirely cohesive as the thickness of adhesive layer of bitumen is continuously increased.

Based on the micromechanics analysis conducted by Lytton et al. (2005), transition of the mode of failure was expected to occur at a lower thickness of about 60 μm (0.060 mm). Hence, in order to generalise the conclusion over wide ranges of asphalt mixture materials and testing conditions, and also to take into account the worst-case scenario that might possibly occur, factor of safety of 2.0 has been applied to the thickness of 200 μm (0.200 mm). Hence, by considering the factor of safety of 2.0, thickness of 100 μm (0.100 mm) was deduced as point of transition of the mode of failure of either adhesive or mixed cohesive and adhesive for this study. Difference in the penetration grade of bitumen and also difference in the testing conditions (i.e. deformation rate and test temperature) from the study conducted by Lytton et al. (2005) were the possible factors that contribute to the difference in the point of transition of the mode of failure.



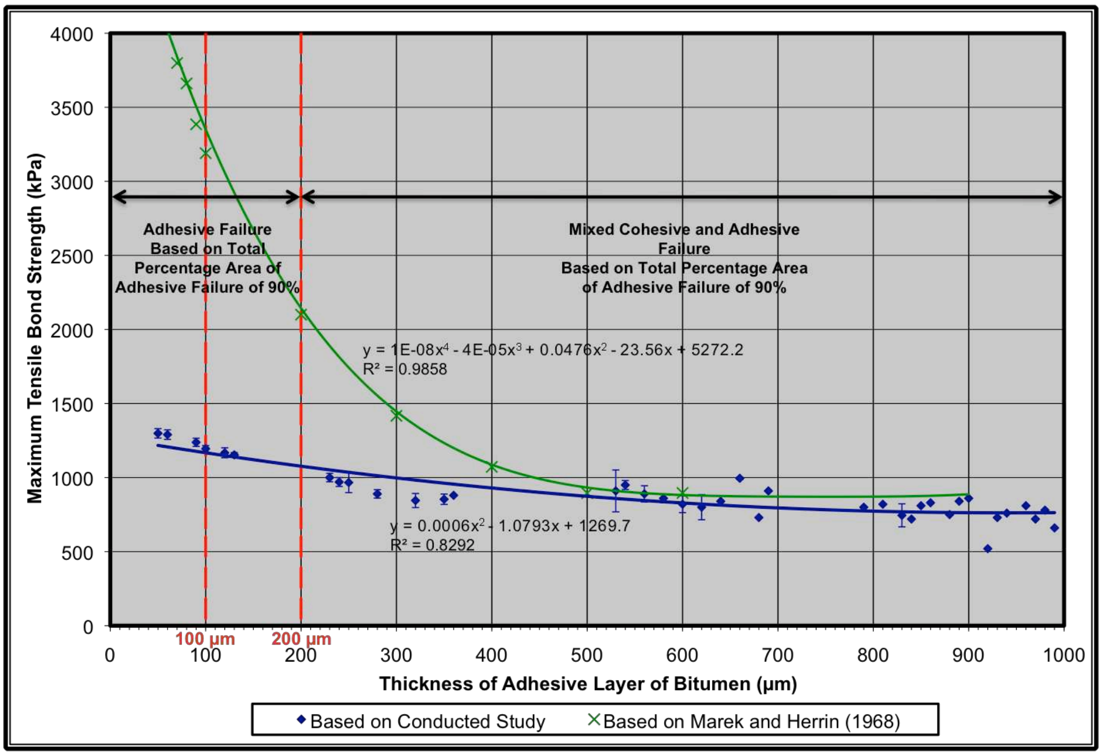
Notes:
1. 200 μm is the thickness of adhesive layer of bitumen related to the total percentage area of adhesive failure of 90%
2. 560 μm is the thickness of adhesive layer of bitumen related to the total percentage area of failure of 50% adhesive and 50% cohesive

Figure 6.5 Relationship between total percentage area of failure and thickness of adhesive layer of bitumen

6.2.2 Relationship between Maximum Tensile Bond Strength and Thickness of Adhesive Layer of Bitumen

Figure 6.6 shows the relationship between maximum tensile bond strength and thickness of adhesive layer of bitumen, based on the data of the test results in Table 6.7 and also based on the data of the study conducted by Marek and Herrin (1968). Data of the test results in Table 6.7 has been grouped according to the thickness of adhesive layer of bitumen, and the values of the average maximum tensile bond strength have been tabulated in Figure 6.6. Curve with the value of coefficient of determination, R^2 of approximately 0.83 was found to be the best to correlate the maximum tensile bond strength and thickness of adhesive layer of bitumen. Generally, coefficient of determination, R^2 greater than 0.8 is considered to show a reasonable fits for the data sets (GraphPad Software n.d.). Data of the study of Marek and Herrin (1968) has been found useful for the purpose of comparative analysis. Curve for the data of the study of Marek and Herrin (1968) has been plotted based on the estimated average values of the maximum tensile bond strength over various thicknesses of the adhesive layer of bitumen, based on Figures 2.47 and 2.48. In Figure 6.6, the error bar represents the one-standard deviation above and below the average maximum tensile bond strength.

Generally, maximum tensile bond strength is expected to decrease with the increasing thickness of adhesive layer of bitumen. This is also supported by the study conducted by Harvey and Cebon (2005), which had evaluated the adhesive behaviour of bitumen using butt joint test. Based on the data of Marek and Herrin (1968), for thickness between 50 μm (0.050 mm) and 300 μm (0.300 mm), a linear inverse relationship has been found which depicts a decrease in the maximum tensile bond strength as the thickness is increased. However, beyond the thickness of 300 μm (0.300 mm), an almost constant value of maximum tensile bond strength of approximately 880 kPa was observed. Data of the test results in Table 6.7 also shows the same curve shape for thickness of more than 600 μm (0.600 mm), in which a nearly horizontal line has been plotted. However, the constant value of approximately 750 kPa was lower compared to the constant value of approximately 880 kPa of Marek and Herrin (1968), which can be attributed to the differences in the penetration grade of bitumen and deformation rate. As expected, hard bitumen grade (i.e. low penetration grade of bitumen) of Marek and Herrin (1968) was found to result in higher value of tensile bond strength as compared to the soft bitumen grade (i.e. high penetration grade of bitumen) of the conducted study. These constant values of tensile bond strength can be correlated with the cohesive bond strength of bitumen (bitumen-filler mastic). Thus, a separate study, which is out of scope of this study, is required in order to evaluate the relationship between the constant value of tensile bond strength within this region and the cohesive bond strength of bitumen (bitumen-filler mastic). Dynamic Shear Rheometer (DSR) and ductility test are suggested for accessing the cohesive bond strength of bitumen (bitumen-filler mastic). Kanitpong and Bahia (2003) has developed a method known as Tackiness Test of Asphalt using Dynamic Shear Rheometer (DSR) in order to measure the cohesive bond strength of bitumen. Details of the developed method are presented in Chapter 2 (Section 2.4.1).



Notes:
1. 200 µm is the thickness of adhesive layer of bitumen related to the total percentage area of adhesive failure of 90% (i.e. based on Figure 6.5)
2. 100 µm is considered as a point of transition of the mode of failure and is derived as follows: Factor of safety of 2.0 is applied to thickness of adhesive layer of bitumen related to the total percentage area of adhesive failure of 90% (i.e. 200 µm). Hence, (200 µm)/(Factor of safety of 2.0) = 100 µm.

Based on Conducted Study
1. Substrates: Aluminium alloy
2. Adhesive Materials: Conventional 70/100 penetration grade of bitumen (Penetration at 25°C is 68)
3. Conditioning Procedures: Dry conditioning at 25°C for 24 hours prior to testing
4. Testing Conditions: Deformation rate and test temperature of 20 mm/minute and 25°C respectively

Based on Marek and Herrin (1968)
1. Substrates: Aluminium alloy
2. Adhesive Materials: Asphalt cement K (Penetration at 25°C is 52)
3. Conditioning Procedures: Dry conditioning at room temperature for 3 hours prior to testing
4. Testing Conditions: Deformation rate and test temperature of 25.4 mm/minute and 25°C respectively

Figure 6.6 Relationship between maximum tensile bond strength and thickness of adhesive layer of bitumen

As has been stated before, adhesive mode of failure was characterised by the value of the total percentage area of adhesive failure of more than 90%. Based on Figure 6.5, transition of the mode of failure of either adhesive or mixed cohesive and adhesive is expected to occur at the thickness of 200 µm (0.200 mm). Thus, the relationship between maximum tensile bond strength and thickness of adhesive layer of bitumen as shown in Figure 6.6 can be divided into two main regions (i.e. adhesive failure and mixed cohesive and adhesive failure), and is described as follows.

1. In the first region where the thickness is less than 200 μm (0.200 mm), a linear inverse relationship can be used to correlate maximum tensile bond strength and thickness of adhesive layer of bitumen. The value of the maximum tensile bond strength within this region can be used to represent the adhesive bond strength between adhesive layer of bitumen and substrates.

2. In the second region where the thickness is more than 200 μm (0.200 mm), a fitted line with a slope of almost zero can be used to represent the relationship between maximum tensile bond strength and thickness of adhesive layer of bitumen. As has been stated before, an almost constant value of the maximum tensile bond strength within this region can be correlated with the cohesive bond strength of bitumen (bitumen-filler mastic). However, further study is required for validation since the mode of failure within this region also consists of mixed cohesive and adhesive, apart from entirely cohesive.

3. In order to ensure the occurrence of the adhesive mode of failure (i.e. total percentage area of adhesive failure of more than 90%) and also to take into account the worst-case scenario that might possibly occur during specimen preparation and testing, factor of safety of 2.0 was applied to the thickness of 200 μm (0.200 mm). Hence, thickness of 100 μm (0.100 mm) was deduced as the final thickness for the occurrence of the adhesive mode of failure in this study.

Based on the data of Marek and Herrin (1968), which also had been described in details in Chapter 2, as the thickness of adhesive layer of bitumen is decreased beyond 20 μm (0.020 mm), there is a sharp decrease in the maximum tensile bond strength. It should be noted that as the thickness of adhesive layer of bitumen becomes too low, precision and accuracy in the procedures for specimen preparation become very significant, as the small errors and differences in the value of the thickness of adhesive layer of bitumen could vary markedly the value of the tensile bond strength. Selection of the thickness of adhesive layer of bitumen was suggested to be more than 40 μm (0.040 mm) in order to prevent any discrepancy and inaccuracy of the test results.

6.2.3 Relationship between Average Maximum Tensile Bond Strength and Total Percentage Area of Adhesive Failure

Based on the data of the test results in Table 6.7, total percentage area of adhesive failure have been grouped as in Table 6.8, and a plot has been deduced as shown in Figure 6.7. As expected, the value of the maximum tensile bond strength was found to increase with the increasing value of the total percentage area of adhesive failure. The increment of the maximum tensile bond strength is expected to begin at an initial value of 745 kPa and increases at a continuously increasing slope. In the range of 0% and 50% of the total percentage area of adhesive failure, the maximum tensile bond strength experienced a slow rate of increasing. However, beyond the total percentage area of adhesive failure of 70%, a significant increase was observed.

Table 6.8 Results of average maximum tensile bond strength based on grouped total percentage area of adhesive failure

Total Percentage Area of Adhesive Failure (%)	Average Maximum Tensile Bond Strength (kPa)
100	1135
95	1230
90	1044
85	1060
80	866
70	840
60	990
50	848
45	850
40	871
35	815
30	808
25	795
20	785
15	767
10	690

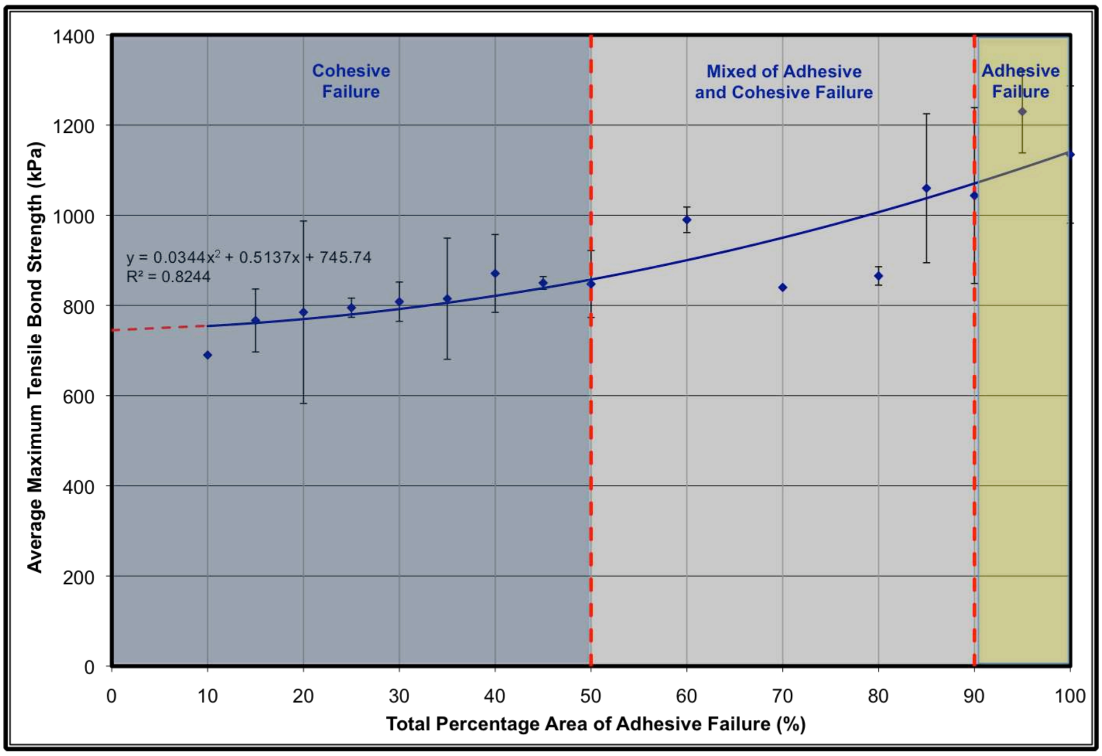


Figure 6.7 Relationship between average maximum tensile bond strength and total percentage area of adhesive failure

The value of the maximum tensile bond strength at 0% of the total percentage area of adhesive failure (i.e. mode of failure of entirely cohesive) of 745 kPa can be assumed as the cohesive bond strength of bitumen (bitumen-filler mastic). This was further verified by comparing with the constant value of approximately 750 kPa obtained in Figure 6.6, which can also be correlated with the cohesive bond strength of bitumen (bitumen-filler mastic). Based on the previously defined occurrence of the adhesive mode of failure (i.e. total percentage area of adhesive failure of more than 90%), the average maximum tensile bond strength of approximately 1070 kPa was found. The value of the maximum tensile bond strength of 1070 kPa can be used to represent the adhesive bond strength between adhesive layer of bitumen and substrates. Again, this was further verified by comparing with the plot in Figure 6.6.

Also, based on Figure 6.7, the value of the maximum tensile bond strength in the range of 0% and 50% of the total percentage area of adhesive failure were found to result in an almost constant value (i.e. average of 800 kPa and coefficient of variation of 7%). Hence, it can be concluded that the cohesive mode of failure is characterised by the value of the total percentage area of adhesive failure of less than 50%. For the range of 50% and 90% of the total percentage area of adhesive failure, mode of failure can be classified as mixed cohesive and adhesive.

6.2.4 Relationship between Tensile Energy Required to Produce Failure Per Unit Volume and Thickness of Adhesive Layer of Bitumen

As has been described in Chapter 5, CurveExpert 1.4 was used to calculate the area under the curve of the graph of tensile load versus pull off displacement, which represents the tensile energy required to produce failure. The curve for graph of tensile load versus pull off displacement was found to behave differently over wide ranges of thicknesses of adhesive layer of bitumen, which was assumed to depend on the mode of failure (i.e. entirely cohesive, mixed cohesive and adhesive or entirely adhesive) and also the occurrence of cobwebbing and cavitations. Figure 6.8 shows the typical graphs of tensile load versus pull off displacement for thickness of adhesive layer of bitumen of 50 μm (0.050 mm), 200 μm (0.200 mm) and 500 μm (0.500 mm) respectively. As the thickness of adhesive layer of bitumen is increased, there is a decrease in the value of the maximum tensile load, and also the attainment of the maximum value of the tensile load occurs at a larger value of the pull off displacement.

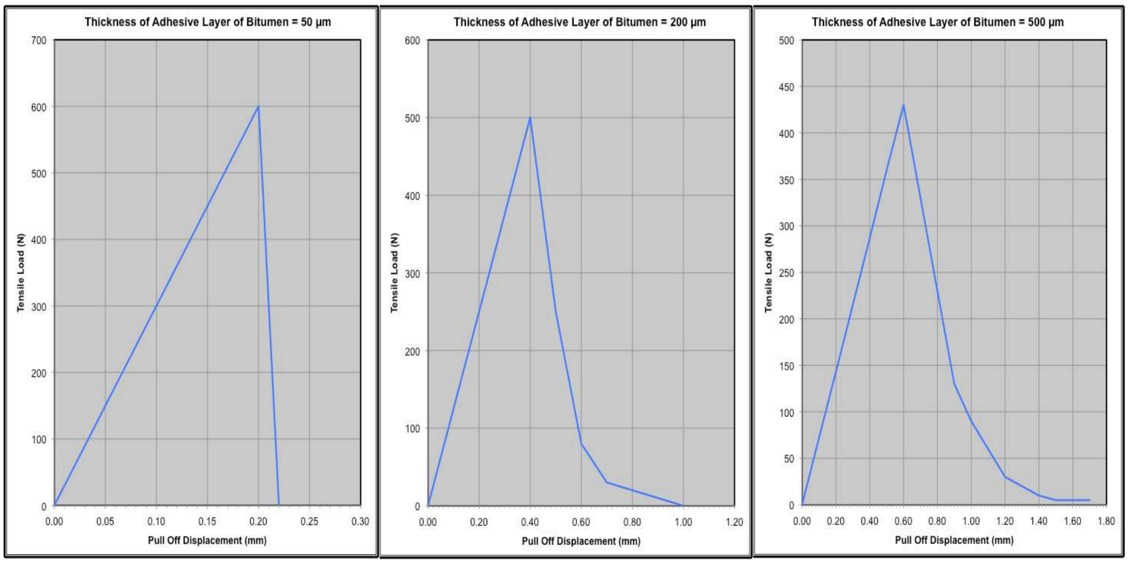


Figure 6.8 Typical graphs of tensile load versus pull off displacement for various thicknesses of adhesive layer of bitumen

Tensile energy required to produce failure per unit volume was found to result in the same curve shape as the maximum tensile bond strength, with respect to the various thicknesses of adhesive layer of bitumen, as shown in Figure 6.9. However, the values of the tensile energy required to produce failure per unit volume tend to be scattered considerably, which can be attributed to the estimation errors due to the curve fitting procedures (i.e. uncertainty that presents in a curve that is fitted to the data sets) and the parameters that governed the values of the tensile energy required to produce failure per unit volumes such as tensile load, pull off displacement and thickness of adhesive layer of bitumen. Low value of coefficient of determination, R^2 of approximately 0.70 as indicated in Figure 6.9, justified the high variability or distribution of the measured tensile energy required to produce failure per unit volume.

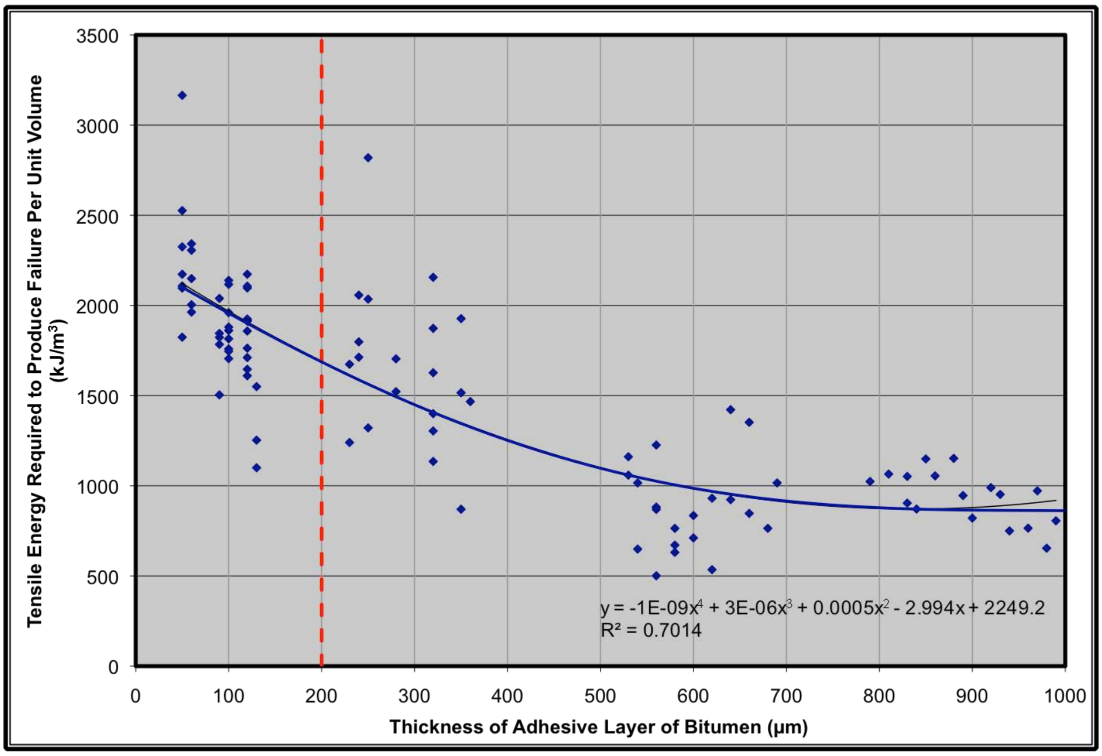


Figure 6.9 Relationship between tensile energy required to produce failure per unit volume and thickness of adhesive layer of bitumen

Relationship between tensile energy required to produce failure per unit volume and thickness of adhesive layer of bitumen can be described as follows, and thickness of 200 μm (0.200 mm) was again chosen as point of transition of the mode of failure.

1. For thickness less than 200 μm (0.200 mm), a linear inverse relationship has been found which depicts a decrease in the tensile energy required to produce failure per unit volume as the thickness is increased. The value of the parameter within this region can be correlated with the amount of energy required to break the adhesive bond between adhesive layer of bitumen and substrates.

2. As the thickness is increased beyond 200 μm (0.200 mm), the value of the tensile energy required to produce failure per unit volume experienced a slow rate of decreasing before reaches steady state conditions of approximately 860 kJ/m^3, which can be correlated to the intermolecular force developed within adhesive layer of bitumen (i.e. cohesion). Cohesion is mainly influenced by viscosity, and hence a test method such as developed by Kanitpong and Bahia (2003) (i.e. Tackiness Test of Asphalt using Dynamic Shear Rheometer (DSR)) as described in details in Chapter 2 (Section 2.4.1) is suggested for further study. Tackiness Test of Asphalt using Dynamic Shear Rheometer (DSR) and also ductility test can be used to measure the cohesive bond strength of bitumen.

Also, a plot has been deduced as shown in Figure 6.10, in order to observe the relationship between various definitions of tensile energy required to produce failure (i.e. tensile energy required to produce failure per unit area of contact and tensile energy required to produce failure per unit volume) and thickness of adhesive layer of bitumen. As the area of contact between bitumen and substrates is constant throughout the study, the curve of the tensile energy required to produce failure per unit area of contact can be used to represent the area under the curve of graph of tensile load versus pull off displacement.

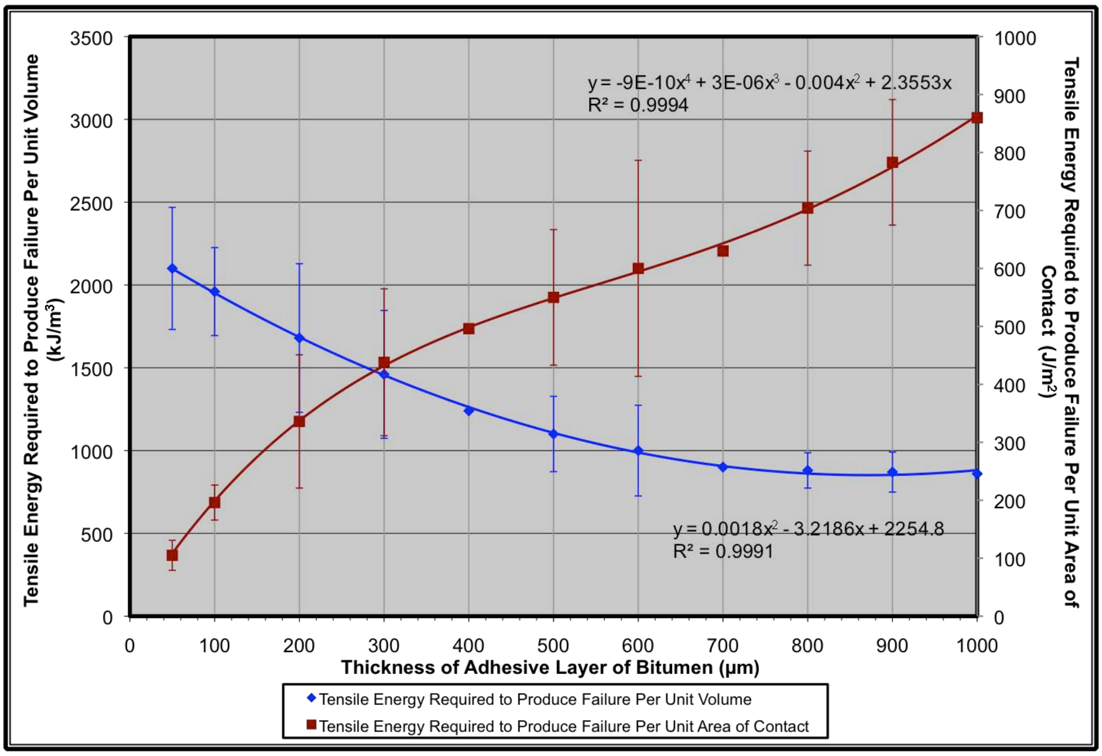


Figure 6.10 Relationship between various definitions of tensile energy required to produce failure and thickness of adhesive layer of bitumen

Based on Figure 6.10, the curve of the tensile energy required to produce failure per unit area of contact and the curve of the tensile energy required to produce failure per unit volume were found to response differently with respect to the thickness of adhesive layer of bitumen. As the thickness of adhesive layer of bitumen is increased, the value of the tensile energy required to produce failure per unit area of contact increases. However, based on Harvey and Cebon (2005), the value of the tensile energy required to produce failure was best described by the area under the curve of graph of tensile load versus pull off displacement divided by the unit volume of the adhesive layer of bitumen (i.e. tensile energy required to produce failure per unit volume). Any variation in the thickness of adhesive layer of bitumen, which might affect the test results, will be taken into account when using the definition of the tensile energy required to produce failure per unit volume. Also, as the volume of the adhesive layer of bitumen deforms during the testing and there is no clearly defined mode of failure for the tested specimen (i.e. failure mechanisms could occur due to adhesive failure along the interfaces, cohesive failure through the adhesive layer of bitumen, or combination of both), energy per unit volume describes the fracture or failure mechanisms better than energy per unit area (Harvey & Cebon 2005).

6.3 Analysis to Determine the Effect of Aspect Ratio of Specimens on the Test Results

Aspect ratio of specimens is defined as the ratio of the longest dimension to the shortest dimension. In this study, the longest and the shortest dimension are represented by the diameter of the discs and the thickness of adhesive layer of bitumen respectively. Some studies have suggested the importance of aspect ratio of specimens on the test results. However, based on Harrison and Harrison (1972) in the study of adhesion using finite element analysis, tensile bond strength was found to be independent of aspect ratio under the following conditions; aspect ratio of more than 10 and Poisson's ratio of adhesive materials of less than 0.49.

Some studies have shown that the test results in terms of the types of failure of specimens and tensile bond strength were mainly influenced by the thickness of adhesive layer of bitumen rather than the area of contact between bitumen and substrates, which in this study can be correlated with the diameter of the discs. Based on the following conditions; constant thickness of adhesive layer of bitumen and other testing conditions, as the diameter of the discs is increased, there is an increase in the aspect ratio of specimens. Hence, aspect ratio of specimens is expected to result in negligible effect on the test results as long as the thickness of adhesive layer of bitumen remained approximately constant.

In this section, a study was conducted in order to determine the effect of aspect ratio of specimens in terms of the diameter of the discs and the thickness of adhesive layer of bitumen on the test results. A total of 60 specimens of various combinations of diameter of the discs and thickness of adhesive layer of bitumen were tested at fixed deformation rate and test temperature of 20 mm/minute and 25°C respectively. In order to cater for various diameter of the discs, modification and fabrication of the pair of plates were specially made for this study. Aluminium alloy, conventional 70/100 penetration grade of bitumen and dry conditioning for 24 hours prior to testing were the selected asphalt mixture materials (i.e. substrates and adhesive materials) and conditioning procedures respectively. Table 6.9 shows the summarised data of the test results.

A total of 3 specimens at each combination of diameter of the discs (i.e. 10 mm, 25 mm, 50 mm and 100 mm) and thickness of adhesive layer of bitumen (i.e. 50 μm, 100 μm, 250 μm, 500 μm and 800 μm) have resulted in the ranges of aspect ratio of specimens between 12.50 and 2000. Based on the summarised data of the test results, a plot has been deduced in order to determine the relationship between average maximum tensile bond strength and aspect ratio of specimens, as presented in Figure 6.11. The error bar represents the one-standard deviation above and below the average maximum tensile bond strength. Generally, without considering the thickness of adhesive layer of bitumen, no specific conclusion can be made regarding the relationship of the two parameters. Based on the plot, no particular trend could be observed. However, by taking into account the thickness of adhesive layer of bitumen, in the region where the thickness is less than 500 μm (0.500 mm), the value of the average maximum tensile bond strength of specimens having approximately the same thickness, was found to be approximately constant. Hence, within this region (i.e. thickness less than 500 μm (0.500 mm), maximum tensile bond strength was concluded to be independent of the aspect ratio of specimens.

Table 6.9 Results based on different aspect ratio of specimens

A	B	C=(A*1000)/B	D	E
Diameter of Discs (mm)	Average Thickness of Adhesive Layer of Bitumen (μm)	Aspect Ratio of Specimens	Average Total Percentage Area of Adhesive Failure (%)	Average Maximum Tensile Bond Strength (kPa)
10	50	200.00	100	1311
	100	100.00	96	1205
	250	40.00	94	985
	500	20.00	55	886
	800	12.50	33	804
25	50	500.00	100	1297
	100	250.00	98	1217
	250	100.00	96	992
	500	50.00	60	885
	800	31.25	35	803
50	50	1000.00	99	1306
	100	500.00	96	1201
	250	200.00	94	983
	500	100.00	51	891
	800	62.50	29	822
100	50	2000.00	99	1281
	100	1000.00	95	1197
	250	400.00	91	1003
	500	200.00	48	902
	800	125.00	25	852

Notes:
1. Substrates: Aluminium alloy
2. Adhesive Materials: Conventional 70/100 penetration grade of bitumen
3. Conditioning Procedures: Dry conditioning at 25°C for 24 hours prior to testing
4. Testing Conditions: Deformation rate and test temperature of 20 mm/minute and 25°C respectively

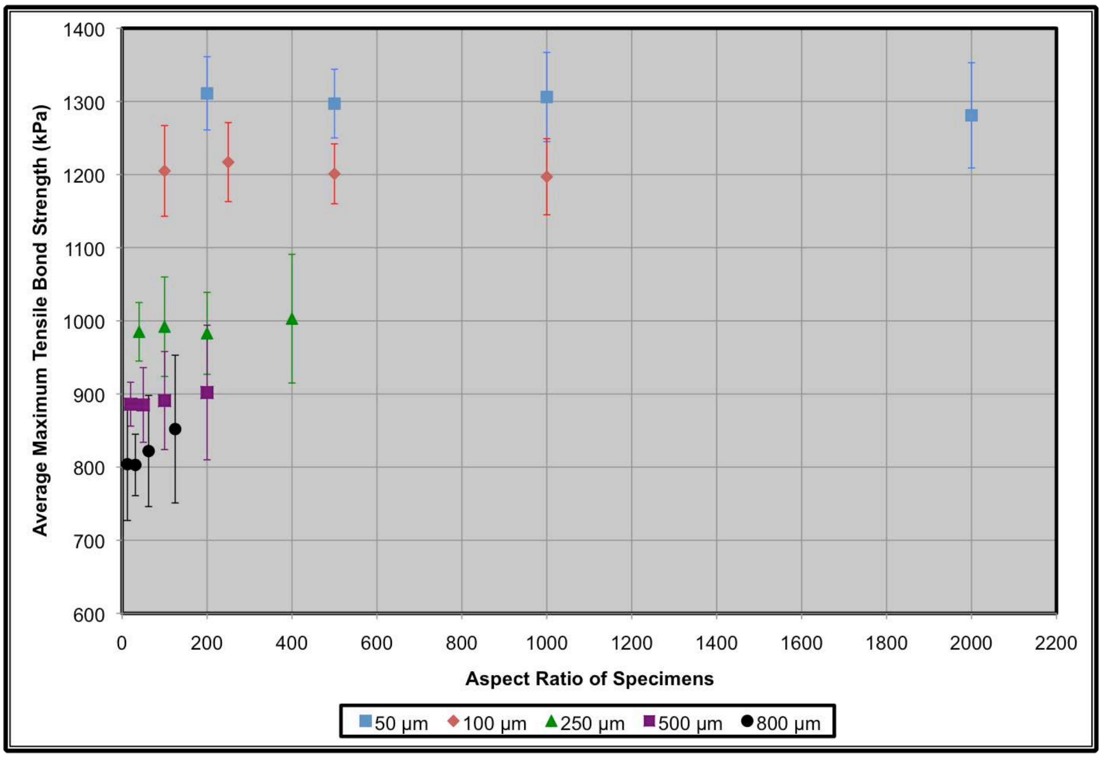


Figure 6.11 Relationship between average maximum tensile bond strength and aspect ratio of specimens

Also, based on the Figure 6.12, which shows the relationship between total percentage area of adhesive failure and aspect ratio of specimens within the ranges of 0 and 500, without considering the thickness of adhesive layer of bitumen, no specific conclusion can be deduced. The types of failure of either adhesive or cohesive were found to be independent of the aspect ratio of specimens. However, by taking into account the thickness of adhesive layer of bitumen, the total percentage area of adhesive failure was found to be in the ranges of 90% and 100% for thickness of adhesive layer of bitumen of less than 250 µm (0.250 mm). Data of the test results has thus shown that the types of failure of either adhesive or cohesive was mainly influenced by the thickness of adhesive layer of bitumen rather than the aspect ratio of specimens, which in this case was correlated with the diameter of the discs.

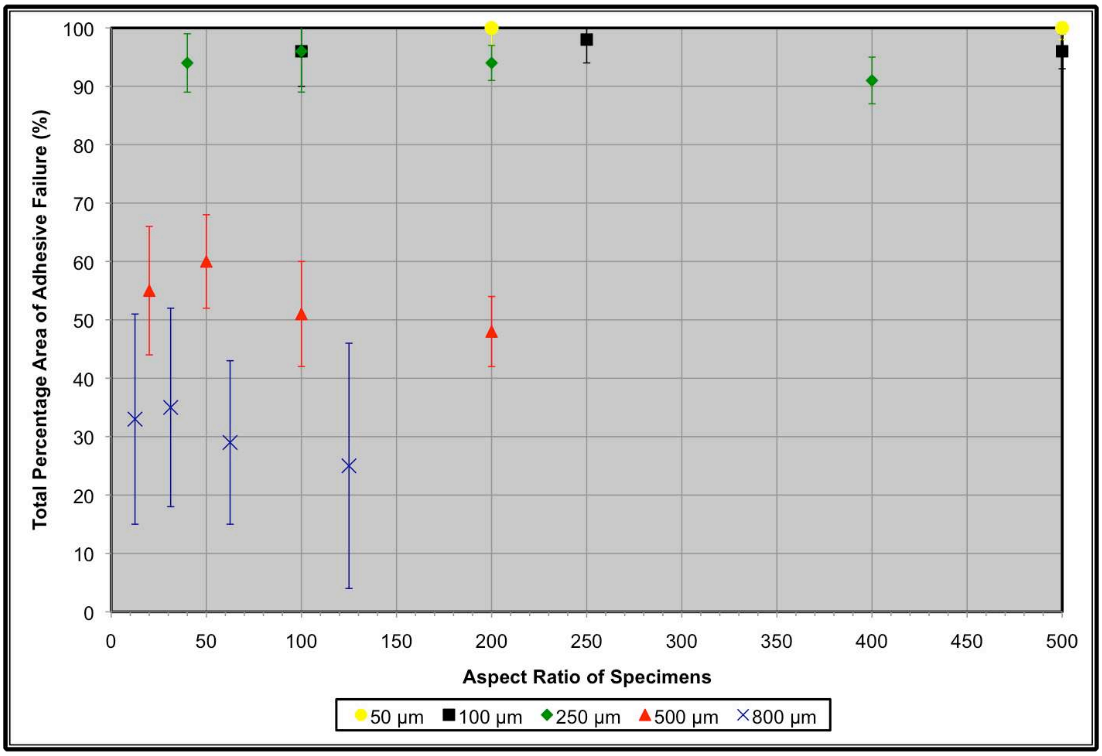


Figure 6.12 Relationship between total percentage area of adhesive failure and aspect ratio of specimens

A plot has been deduced in order to further verify the dependency of the maximum tensile bond strength on the thickness of adhesive layer of bitumen, as illustrated in Figure 6.13. Based on the plot, for thickness of less than 500 µm (0.500 mm), as the diameter of the discs was increased which indicated the increment of the aspect ratio of specimens, the value of the average maximum tensile bond strength was found to be approximately constant. However, for thickness of 800 µm (0.800 mm), the value of the average maximum tensile bond strength was found to slightly increase with the increasing diameter of the discs (i.e. aspect ratio of specimens). High thickness of adhesive layer of bitumen of more than 800 µm (0.800 mm) was expected to behave differently (i.e. tensile bond strength increase with the increasing diameter of the discs) as a result of cohesive mode of failure and also the occurrence of cobwebbing and cavitations.

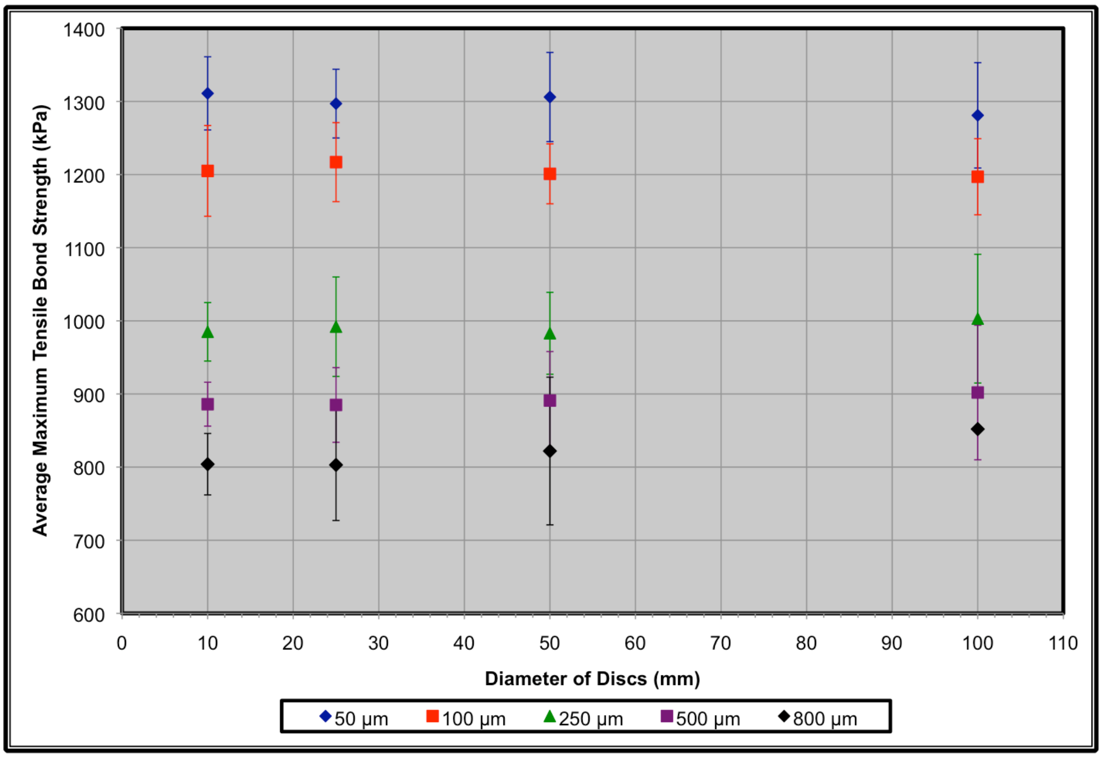
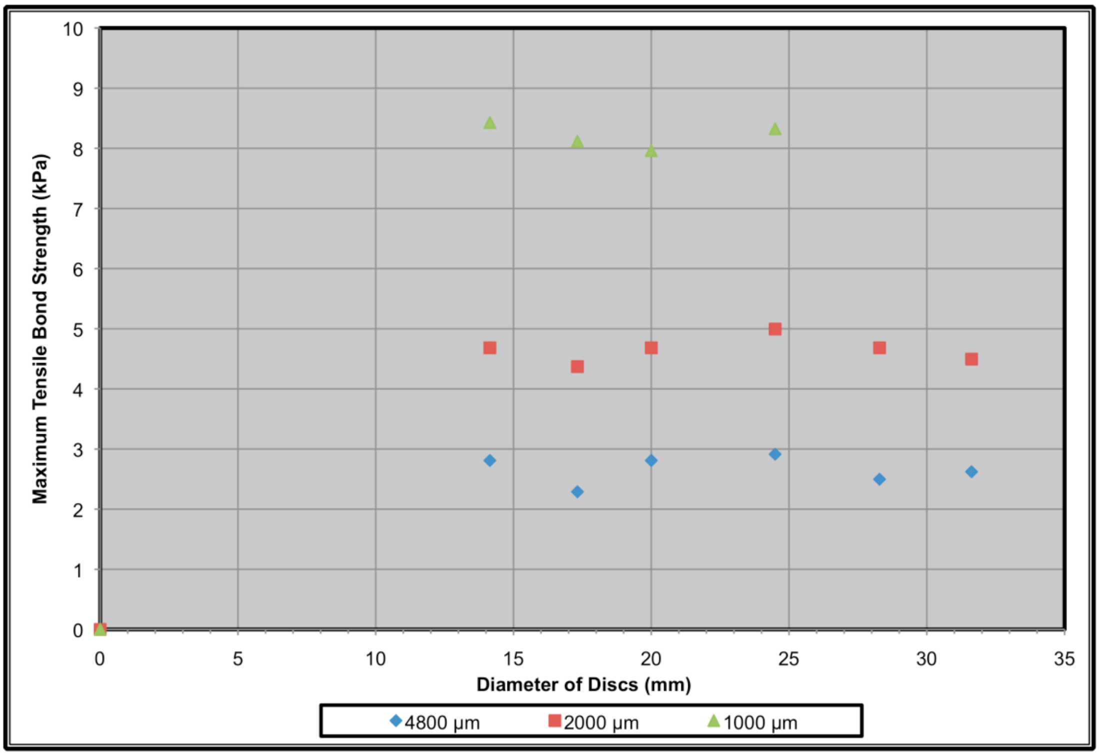


Figure 6.13 Relationship between average maximum tensile bond strength and diameter of discs

Hence, for thickness of less than 500 µm (0.500 mm), a conclusion can be made that aspect ratio of specimens has negligible effect on the tensile bond strength as long as the thickness of adhesive layer of bitumen remained constant. This supports the study conducted by Harrison and Harrison (1972), which indicated that the tensile bond strength was independent of aspect ratio of specimens for the value of more than 10 and Poisson's ratio of adhesive materials of less than 0.49. For thickness of more than 800 µm (0.800 mm), no definite conclusion can be made due to the limited number of the tested specimens. Further study is suggested for assessing the effect of aspect ratio of specimens beyond the thickness of 800 µm (0.800 mm) (i.e. in the extreme region of cohesive mode of failure), which is out of the scope of this study. Kendall (1971) has proved the negligible effect of the diameter of the discs on the tensile bond strength based on gelatine of thickness between 1000 µm (1.000 mm) and 4800 µm (4.800 mm) as adhesive materials and Perspex as substrates. Figure 6.14 shows the plot of the test results. It can be concluded that the adhesive bond strength is mainly influenced by the thickness of adhesive materials rather than the aspect ratio of specimens, which in this case was correlated with the diameter of the discs.



Notes: 1. Substrates: Perspex
2. Adhesive Materials: Gelatine
3. Conditioning Procedures: Dry condition at room temperature
4. Testing Conditions: Deformation rate of 0.6 mm/minute at room temperature

Figure 6.14 Relationship between maximum tensile bond strength and diameter of discs (Source: Kendall 1971)

Figure 6.15 shows the relationship between average total percentage area of adhesive failure and diameter of discs. Based on Figure 6.15, for thickness of less than 250 µm (0.250 mm), the total percentage area of adhesive failure was found to be constant regardless of the diameter of the discs. However, for thickness of more than 500 µm (0.500 mm), the total percentage area of adhesive failure was found to decrease with increasing diameter of the discs, which indicated the dependency of the specimens on the aspect ratio. Based on the previous section, for total percentage area of adhesive failure of less than 50%, cohesive mode of failure and also the occurrence of cobwebbing and cavitations are expected. Hence, it can be concluded that the adhesive bond strength and failure characteristics behave differently with respect to the aspect ratio of specimens, depending on the mode of failure and thus thickness of adhesive layer of bitumen.

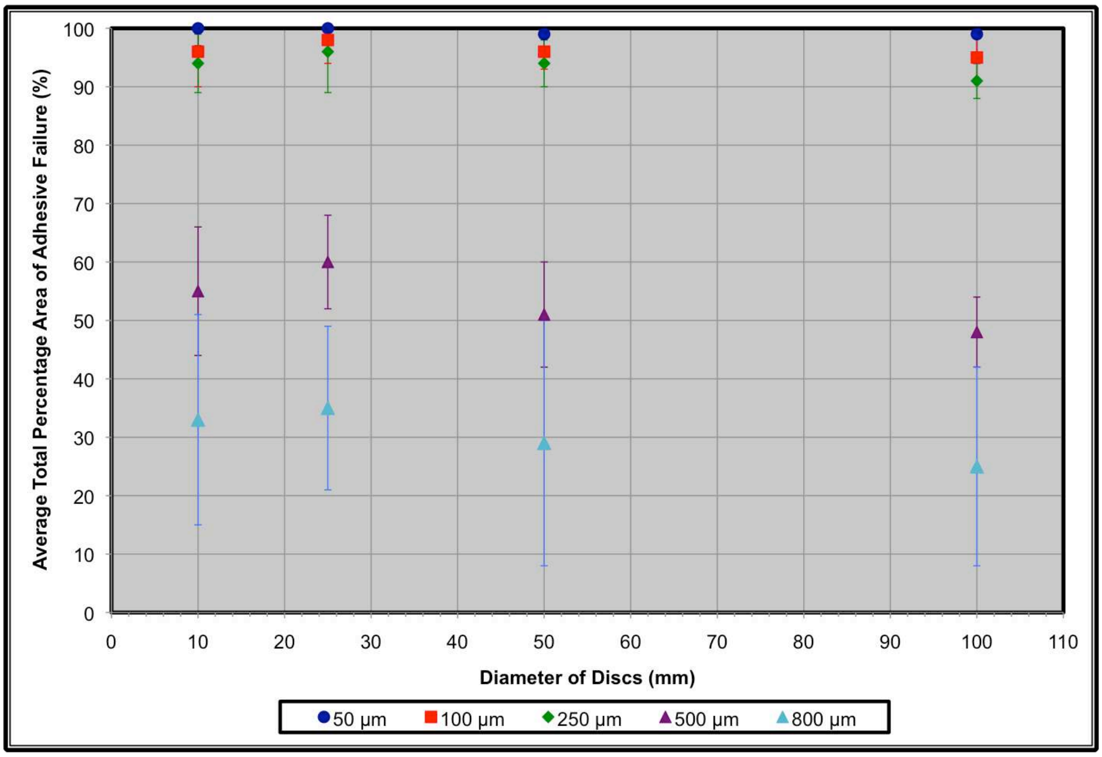


Figure 6.15 Relationship between average total percentage area of adhesive failure and diameter of discs

6.4 Analysis to Determine the Effect of Various Testing Conditions on the Test Results

This section is a continuation of part of the analysis from the previous chapter (i.e. Chapter 5) in which limited number of specimens had been subjected to various testing conditions in terms of deformation rates and test temperatures, in order to generally observe the effect on the test results and also to propose suitable testing conditions. Based on the data of the previous chapter, the ranges of suitable testing conditions in terms of deformation rate and test temperature were suggested to be 10 mm/minute and 20 mm/minute, and 15°C and 25°C respectively. In this section, further evaluation was conducted in order to verify the ranges of suitable testing conditions and thus to validate the reliability and efficiency of the proposed adhesion test method. A total of 72 specimens were prepared and tested based on the following combinations of asphalt mixture materials and testing conditions.

1. Conventional 70/100 penetration grade of bitumen was used as adhesive materials.

2. Aluminium alloy and Ivonbrook Limestone were used as substrates.

3. Specimens were subjected to either dry or wet conditionings at specified test temperature for 24 hours prior to testing (Note: For wet conditioning, specimens were immersed in container filled with water as shown in Figure 6.16 before being placed in the conditioning cabinet).

4. Testing conditions in terms of deformation rate and test temperature were 10 mm/minute or 20 mm/minute, and 15°C, 20°C or 25°C respectively.

Table 6.10 shows the summarised data of the test results. Figures 6.17 and 6.18 show the relationship between average maximum tensile bond strength and test temperature over wide ranges of deformation rates and conditioning procedures, based on aluminium alloy and Ivonbrook Limestone as substrates, respectively.

Figure 6.16 Specimen immersed in container filled with water

Table 6.10 Summarised data of the test results based on various combinations of asphalt mixture materials and testing conditions

Substrates	Conditioning Procedures	Average Thickness of Adhesive Layer of Bitumen (μm)	Deformation Rate (mm/minute)	Test Temperature (°C)	Average Total Percentage Area of Adhesive Failure (%)	Average Maximum Tensile Bond Strength (kPa)
Aluminium Alloy	Dry Conditioning at Specified Test Temperature for 24 Hours	50	10	15	100	1361
				20	100	1255
				25	100	1193
			20	15	100	1462
				20	100	1397
				25	100	1314
	Wet Conditioning at Specified Test Temperature for 24 Hours	50	10	15	100	1281
				20	100	1187
				25	100	1096
			20	15	100	1391
				20	100	1316
				25	100	1225
Ivonbrook Limestone	Dry Conditioning at Specified Test Temperature for 24 Hours	50	10	15	100	1244
				20	100	1173
				25	100	1061
			20	15	100	1398
				20	100	1312
				25	100	1238
	Wet Conditioning at Specified Test Temperature for 24 Hours	50	10	15	100	1106
				20	100	1011
				25	100	942
			20	15	100	1194
				20	100	1107
				25	100	1016

Notes: 1. Adhesive Materials: Conventional 70/100 penetration grade of bitumen

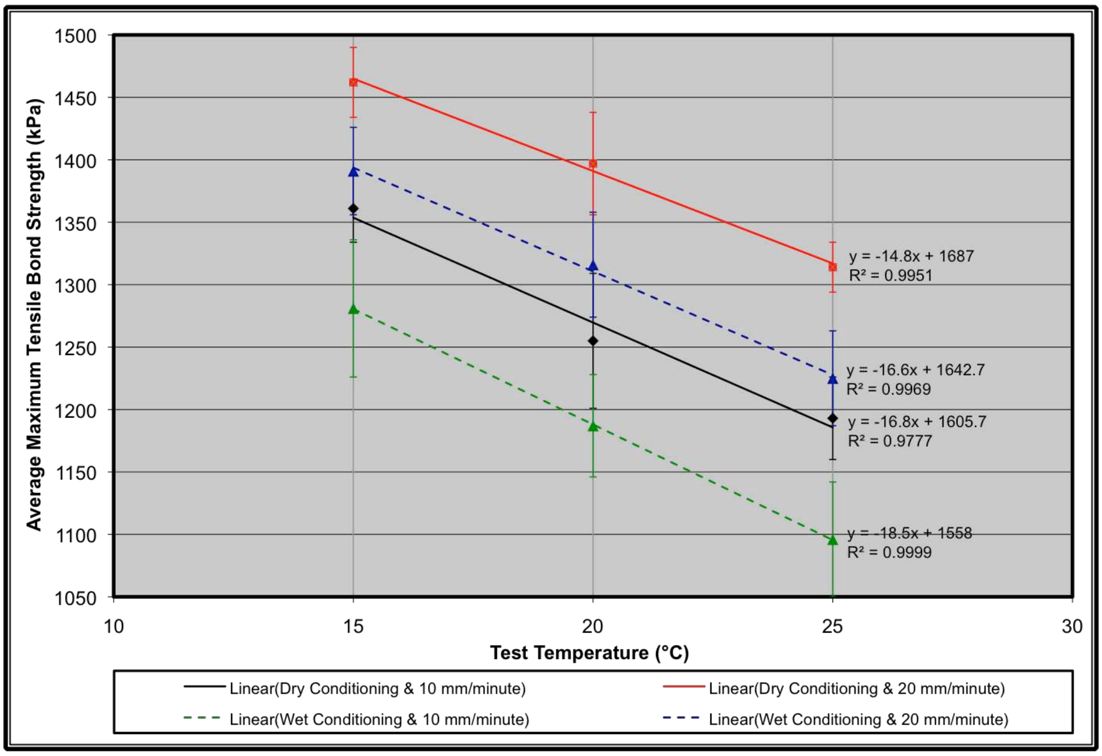


Figure 6.17 Relationship between average maximum tensile bond strength and test temperature over wide range of deformation rates and conditioning procedures (Aluminium alloy as substrates)

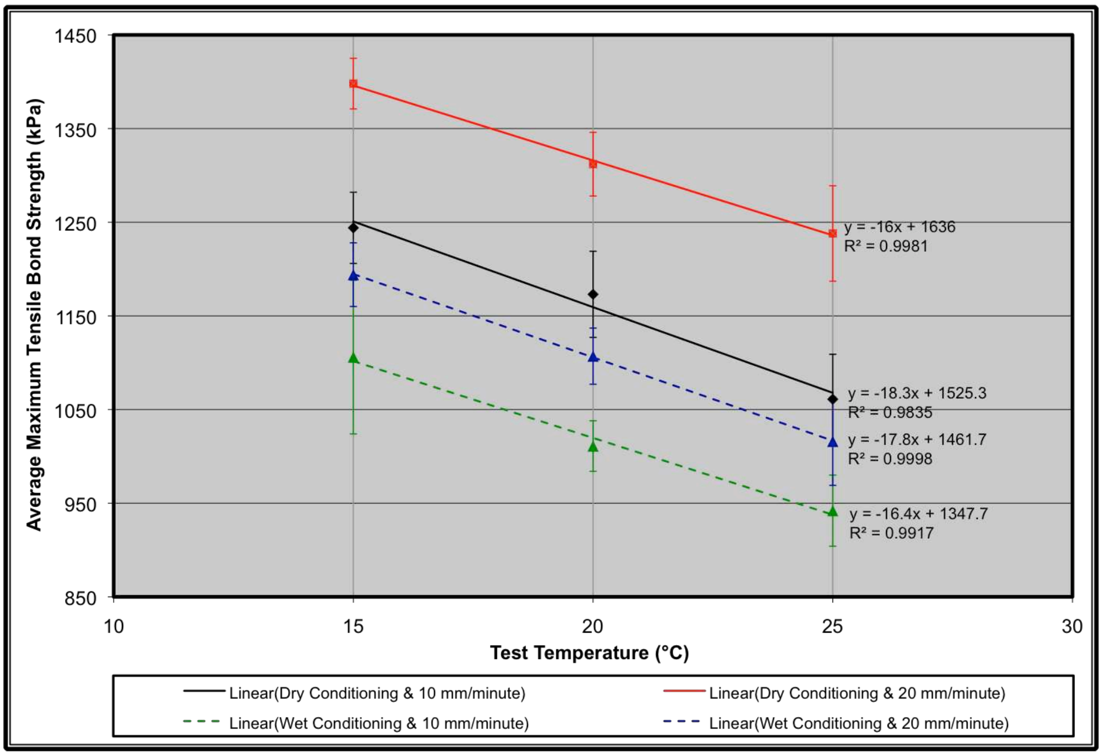


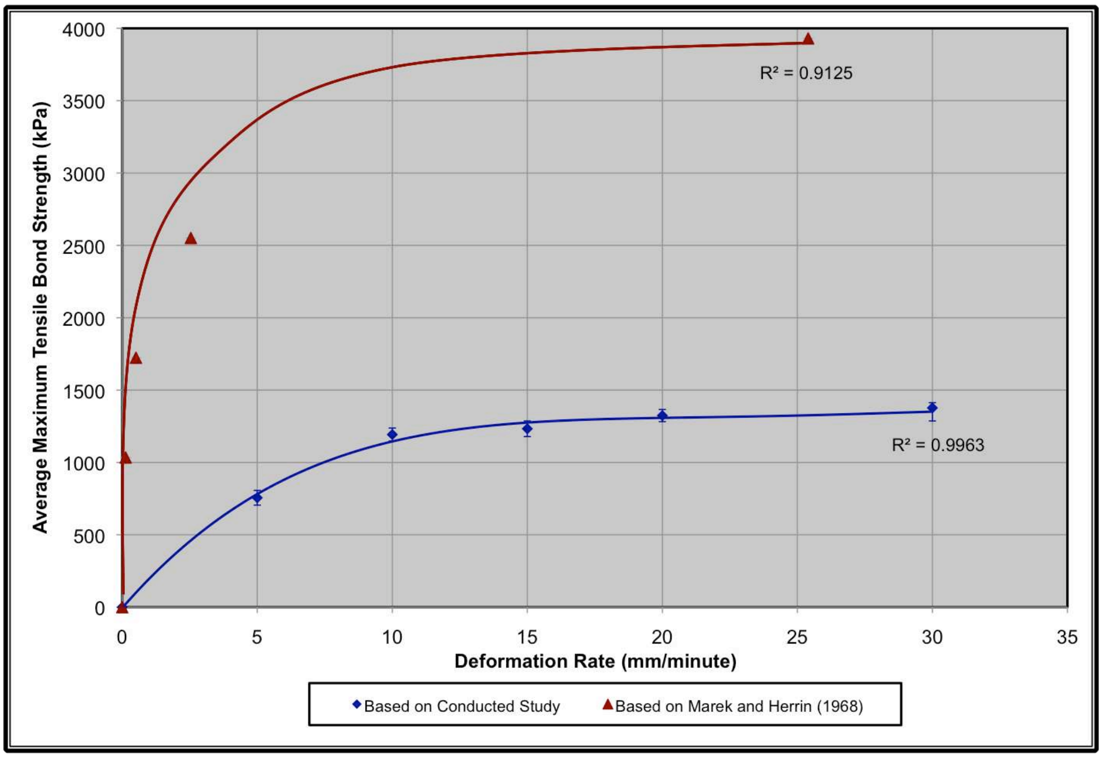
Figure 6.18 Relationship between average maximum tensile bond strength and test temperature over wide range of deformation rates and conditioning procedures (Ivonbrook Limestone as substrates)

Based on the Figures 6.17 and 6.18, the ranges of suitable testing conditions in terms of deformation rate and test temperature were verified as within 10 mm/minute and 20 mm/minute, and 15°C and 25°C respectively, regardless of the substrates and conditioning procedures. An inversely proportional relationship exists between maximum tensile bond strength and test temperature with constant shift due to the variation of deformation rate and conditioning procedures (i.e. dry and wet conditionings). Based on the study conducted in the previous chapter (i.e. Chapter 5), beyond the test temperature of 25°C, the maximum tensile bond strength was expected to converge to a constant value, regardless of the testing conditions (i.e. deformation rate and test temperature). It can be concluded that within the ranges of suitable testing conditions, the value of the maximum tensile bond strength is expected to increase with the increasing deformation rate and decreasing test temperature, regardless of the substrates and conditioning procedures.

Conditioning procedures (i.e. dry and wet conditionings) were found to have a profound influence on the test results, especially when using aggregates as substrates. Maximum tensile bond strength of specimens subjected to dry conditioning was higher compared to the specimens subjected to wet conditionings. Aluminium alloy, which is a non porous material and provides low ability for water to penetrate into, has the least effect of conditioning procedures (i.e. dry and wet conditionings) on the maximum tensile bond strength as compared to the Ivonbrook Limestone. Differences in terms of the average maximum tensile bond strength between dry and wet conditionings over wide ranges of deformation rates and test temperatures based on the aluminium alloy and Ivonbrook Limestone as substrates were 81 kPa and 175 kPa respectively.

A simple study was conducted in order to analyse and compare the variation of the maximum tensile bond strength over wide ranges of deformation rates with the data of the study conducted by Marek and Herrin (1968). Aluminium alloy, conventional 70/100 penetration grade of bitumen, dry conditioning for 24 hours prior to testing and 25°C were the selected asphalt mixture materials (i.e. substrates and adhesive materials), conditioning procedures and test temperature respectively. A total of 15 specimens of thickness of 50 μm (0.050 mm) were tested at the following deformation rate; 5 mm/minute, 10 mm/minute, 15 mm/minute, 20 mm/minute and 30 mm/minute.

Figure 6.19 shows the summarised data of the test results and also the data of the study conducted by Marek and Herrin (1968). The error bar for the data of the test results represents the one-standard deviation above and below the average maximum tensile bond strength. Data of the study conducted by Marek and Herrin (1968) has been plotted based on the estimated average values of the maximum tensile bond strength, according to Figure 2.48. It should be noted that the asphalt cement K used by Marek and Herrin (1968) has lower penetration grade of bitumen (i.e. 52) and thus justify the high value of the average maximum tensile bond strength. Based on Figure 6.19, it can be concluded that as the deformation rate is increased beyond 20 mm/minute, the maximum tensile bond strength is expected to reach steady state conditions. Further increment of the deformation rate beyond this point is wasteful.



Notes:

Based on Conducted Study

1. Substrates: Aluminium alloy
2. Adhesive Materials: Conventional 70/100 penetration grade of bitumen (Penetration at 25°C is 68)
3. Conditioning Procedures: Dry conditioning at 25°C for 24 hours prior to testing
4. Testing Conditions: Test temperature of 25°C

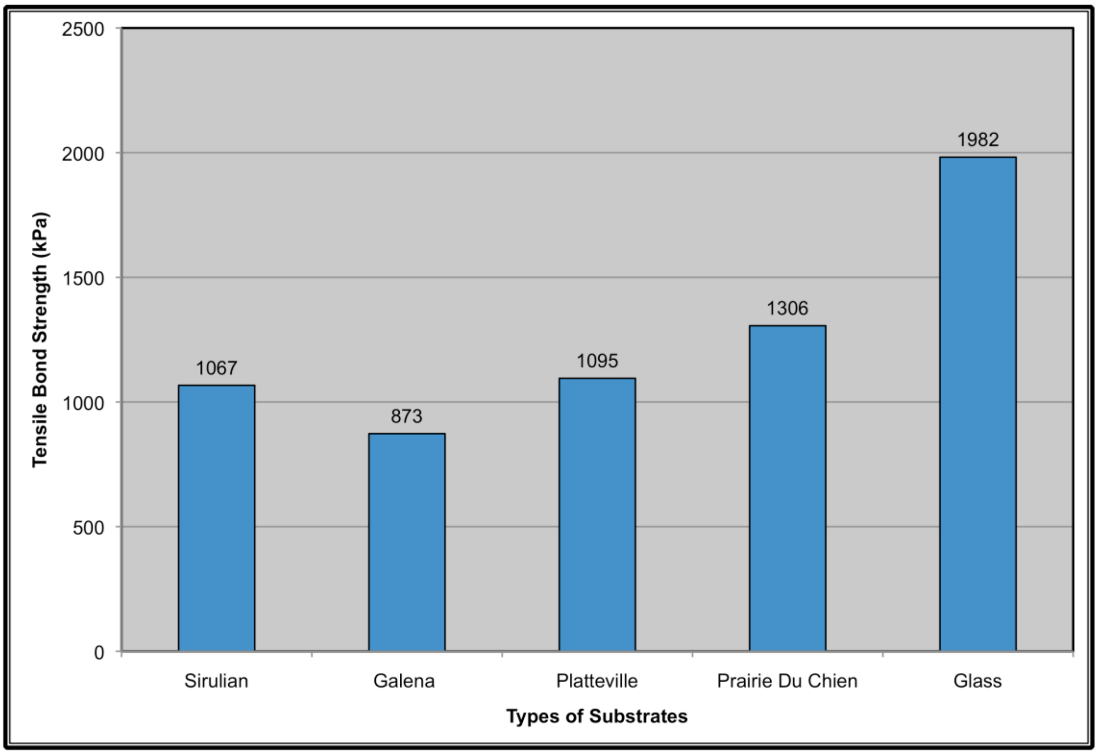
Based on Marek and Herrin (1968)

1. Substrates: Aluminium alloy
2. Adhesive Materials: Asphalt cement K (Penetration at 25°C is 52)
3. Conditioning Procedures: Dry conditioning at room temperature for 3 hours prior to testing
4. Testing Conditions: Test temperature of 25°C

Figure 6.19 Average maximum tensile bond strength over wide ranges of deformation rates

6.5 Analysis to Determine the Effect of Substrates and Conditioning Procedures on the Test Results

In this section, data analysis was conducted in order to determine the effect of different types of substrates and conditioning procedures (i.e. dry and wet conditionings) on the test results. Based on the study conducted by Poulikakos and Partl (2010), the effect of different types of substrates was minimum in the absence of water or moisture in the conditioning procedures (i.e. dry conditioning). Thus, regardless of the substrates, specimens subjected to dry conditioning were expected to exhibit approximately the same value of the test results, provided other testing variables and parameters are held constant. This is also supported by the study conducted by Kanitpong and Bahia (2003), as illustrated in Figure 6.20. Based on Figure 6.20, the tensile bond strength of different types of substrates (i.e. Sirulian, Galena, Platteville and Prairie Du Chien) was in the range of 873 kPa and 1306 kPa, except for glass of 1982 kPa.



Notes:
1. Thickness of Adhesive Materials: 200 μm
2. Adhesive Materials: PG 58-28
3. Conditioning Procedures: Dry conditioning at room temperature for 24 hours prior to testing
4. Testing Conditions: Deformation rate of 65.7 kPa/second at room temperature

Figure 6.20 Tensile bond strength of different types of substrates (Source: Kanitpong & Bahia 2003)

In the presence of water or moisture, significant difference in the test results between dry and wet conditionings, and also among the different types of substrates was expected. As stated in the literature review, adhesive bond strength between adhesive layer of bitumen and substrates can be influenced by physical properties of the substrates such as roughness of the surface and porosity, and also classification of the aggregates as either hydrophilic (i.e.

attract water) or hydrophobic (i.e. repulse water). Based on the properties of the aggregates discs listed in Table 6.5, Mount Sorrel Granite, which has high porosity (i.e. percentage of water absorption of 0.30%) and is classified as acidic (i.e. hydrophilic), was expected to result in significant difference in the test results between dry and wet conditionings. Also, based on Table 6.5, tensile bond strength of different types of substrates subjected to wet conditioning was expected to be ranked from high to low as follows; aluminium alloy, Dene Limestone, Ivonbrook Limestone and Mount Sorrel Granite.

A total of 80 specimens of various combinations of substrates and conditioning procedures were prepared and tested at fixed deformation rate and test temperature of 20 mm/minute and 25°C respectively. Substrates were prepared into discs of 25 mm diameter (i.e. 490.87 mm^2 area of contact), and thickness of 50 µm (0.050 mm) was selected for the adhesive layer of bitumen. Table 6.11 and Figure 6.21 present the data of the test results.

Table 6.11 Results based on combinations of different types of substrates and conditioning procedures

Substrates	Conditioning Procedures	Data Sets	Average Thickness of Adhesive Layer of Bitumen (μm)	Total Percentage Area of Adhesive Failure (%)	Maximum Tensile Bond Strength (kPa)	Tensile Energy Required to Produce Failure Per Unit Volume (kJ/m^3)
Aluminium Alloy	Dry Conditioning at 25°C for 24 Hours Prior to Testing	1	50	100	1300	2599
		2		100	1450	2079
		3		100	1260	2471
		4		100	1270	1399
		5		95	1530	2682
		6		100	1500	2481
		7		100	1270	2038
		8		90	1150	2115
		9		100	1220	1824
		10		100	1370	2289
Aluminium Alloy	Wet Conditioning at 25°C for 24 Hours Prior to Testing	1	50	100	1350	1717
		2		100	1210	1569
		3		95	1160	2420
		4		100	1420	1975
		5		100	1240	1701
		6		100	1020	1908
		7		100	1270	2436
		8		100	1200	1048
		9		100	1240	1771
		10		100	1330	2415
Dene Limestone	Dry Conditioning at 25°C for 24 Hours Prior to Testing	1	50	95	1390	2144
		2		95	1490	2805
		3		100	1260	1513
		4		100	1350	1814
		5		100	1210	2358
		6		100	1190	2702
		7		100	1270	2663
		8		90	1200	1356
		9		100	1320	3125
		10		100	1250	2933
Dene Limestone	Wet Conditioning at 25°C for 24 Hours Prior to Testing	1	50	100	1120	1243
		2		100	1080	1436
		3		100	1140	1446
		4		90	950	1567
		5		100	920	1080
		6		100	1210	2072
		7		90	1070	1255
		8		100	1000	988
		9		100	1030	1106
		10		100	940	1047

Table 6.11 Results based on combinations of different types of substrates and conditioning procedures (continued)

Substrates	Conditioning Procedures	Data Sets	Average Thickness of Adhesive Layer of Bitumen (μm)	Total Percentage Area of Adhesive Failure (%)	Maximum Tensile Bond Strength (kPa)	Tensile Energy Required to Produce Failure Per Unit Volume (kJ/m^3)
Ivonbrook Limestone	Dry Conditioning at 25°C for 24 Hours Prior to Testing	1	50	100	1210	2151
		2		100	1160	1702
		3		100	1280	1146
		4		95	1170	3952
		5		100	1320	1910
		6		90	1100	2330
		7		100	1250	3057
		8		100	1410	2954
		9		100	1090	2360
		10		100	1330	2065
Ivonbrook Limestone	Wet Conditioning at 25°C for 24 Hours Prior to Testing	1	50	100	1140	1105
		2		100	980	978
		3		100	1030	2087
		4		100	1020	1142
		5		100	880	1264
		6		100	910	1210
		7		95	990	1175
		8		100	950	929
		9		100	1020	1597
		10		100	950	1153
Mount Sorrel Granite	Dry Conditioning at 25°C for 24 Hours Prior to Testing	1	50	100	1300	2009
		2		90	1380	1964
		3		100	1200	2305
		4		95	1160	1886
		5		100	1270	2309
		6		100	1320	1370
		7		100	1400	3067
		8		100	1290	2931
		9		95	1430	2289
		10		100	1210	2782
Mount Sorrel Granite	Wet Conditioning at 25°C for 24 Hours Prior to Testing	1	50	100	860	770
		2		95	610	1093
		3		100	650	1110
		4		100	1140	828
		5		90	770	787
		6		100	970	900
		7		100	1230	1546
		8		90	610	889
		9		100	560	758
		10		100	910	1037

Notes: 1. Adhesive Materials: Conventional 70/100 penetration grade of bitumen
2. Testing Conditions: Deformation rate and test temperature of 20 mm/minute and 25°C respectively

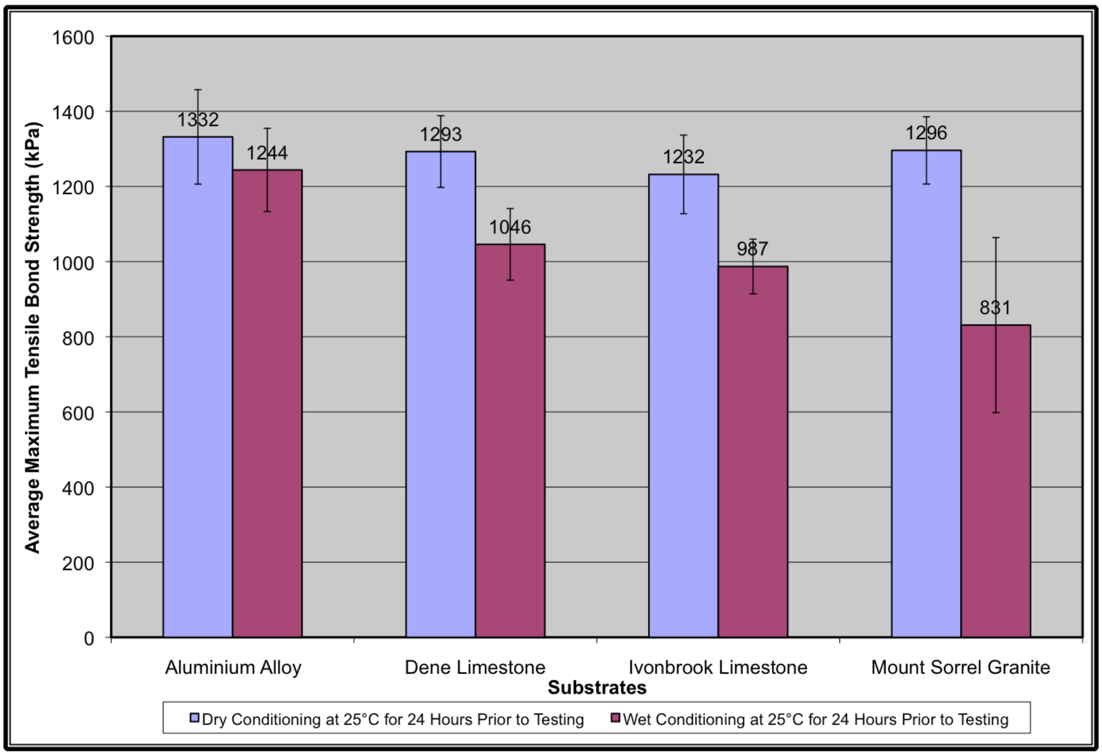


Figure 6.21 Average maximum tensile bond strength over wide ranges of substrates and conditioning procedures

Based on Table 6.11, the mode of failure for all specimens can be classified as adhesive due to the large value of the total percentage area of adhesive failure of more than 90%, regardless of the conditioning procedures (i.e. dry and wet conditionings). As has been stated in the previous part, the occurrence of the adhesive mode of failure can still be achieved in the absence of water or moisture in the conditioning procedures (i.e. dry conditioning), provided that the thickness of adhesive layer of bitumen is thin enough.

Based on Figure 6.21, the average maximum tensile bond strength for all substrates subjected to dry conditioning was in the range of 1232 kPa and 1332 kPa. The error bar represents the one-standard deviation above and below the average maximum tensile bond strength. As expected, the maximum tensile bond strength of aluminium alloy was highest due to uniform surface characteristics and strong structural properties as compared to the aggregates. Chipping due to cutting and brittleness, and also dusty surface are the common problems of the aggregates, which affect the tensile bond strength. Ivonbrook Limestone has the lowest value of the average tensile bond strength in the dry conditioning due to the aforementioned problems. For specimens subjected to wet conditioning, the average maximum tensile bond strength can be ranked from high to low as follows; aluminium alloy, Dene Limestone, Ivonbrook Limestone and Mount Sorrel Granite. Data of the test results has thus validated the theoretically expected performance of the different types of substrates.

Unpaired (i.e. Independent) Two-Samples t-Test procedure, at level of significance, α of 0.05, was conducted in order to determine the existence of significant statistical difference in the average maximum tensile bond strength among different types of substrates. Theoretically, the effect of different types of substrates was minimum in the absence of water or moisture in the conditioning procedures (i.e. dry conditioning), and vice versa. Table 6.12 summarises the output of the analysis of the Unpaired (i.e. Independent) Two-Samples t-Test procedure. The decision rule in determining the existence of significant statistical difference in the pair of data sets is as follows.

If two-tailed p-value is smaller than level of significance, α, significant statistical difference exists in the pair of data sets.

Otherwise, there is no significant statistical difference in the pair of data sets.

Table 6.12 Statistical analysis of Unpaired (i.e. Independent) Two-Samples t-Test procedure (Effect of different types of substrates)

	Pair of Data Sets for Average Maximum Tensile Bond Strength					
	Aluminium Alloy & Dene Limestone		Aluminium Alloy & Ivonbrook Limestone		Aluminium Alloy & Mount Sorrel Granite	
	Dry	Wet	Dry	Wet	Dry	Wet
95.0% CI	(-65.85, 143.85)	(100.89, 295.11)	(-8.74, 208.74)	(168.90, 345.10)	(-66.59, 138.59)	(241.56, 584.44)
t-statistic, t	0.7814	4.2837	1.9320	6.1289	0.7372	5.0610
Two-tailed p-value	0.4447	0.0004	0.0693	0.0001	0.4705	0.0001
Significant Difference	NO	YES	NO	YES	NO	YES

	Pair of Data Sets for Average Maximum Tensile Bond Strength					
	Dene Limestone & Ivonbrook Limestone		Dene Limestone & Mount Sorrel Granite		Ivonbrook Limestone & Mount Sorrel Granite	
	Dry	Wet	Dry	Wet	Dry	Wet
95.0% CI	(-33.10, 155.10)	(-20.62, 138.62)	(-89.92, 83.92)	(47.76, 382.24)	(-155.58, 27.58)	(-6.18, 318.18)
t-statistic, t	1.3618	1.5569	0.0725	2.7008	1.4683	2.0209
Two-tailed p-value	0.1900	0.1369	0.9430	0.0146	0.1593	0.0584
Significant Difference	NO	NO	NO	YES	NO	NO

Based on Table 6.12, it can be concluded that there is sufficient evidence of no significant difference in the average maximum tensile bond strength among different types of substrates, in the absence of water or moisture in the conditioning procedures (i.e. dry conditioning). Data of the tensile bond strength of specimens subjected to dry conditioning was found to be approximately the same, regardless of the substrates. In the presence of water or moisture (i.e. wet conditioning), significant difference in the test results was found in all pair of data sets except for Dene Limestone and Ivonbrook Limestone, and Ivonbrook Limestone and Mount Sorrel Granite. Hence, data of the statistical analysis has justified the theoretically predicted performance of average maximum tensile bond strength among different types of substrates due to the effect of water or moisture in the conditioning procedures. Further analysis to observe the effect of different types of substrates on the test results was then conducted, as shown in Table 6.13. Aluminium alloy was selected as control substrates.

Table 6.13 Effect of different types of substrates on the test results

Properties	Dry Conditioning at 25°C for 24 Hours			Wet Conditioning at 25°C for 24 Hours		
	DL / AA	IL / AA	MSG / AA	DL / AA	IL / AA	MSG / AA
Maximum Tensile Bond Strength (kPa)	0.97	0.92	0.97	0.88	0.79	0.67
Tensile Energy Required to Produce Failure Per Unit Volume (kJ/m^3)	1.07	1.08	1.04	0.70	0.67	0.51

Notes: 1. AA: Aluminium Alloy; DL: Dene Limestone; IL: Ivonbrook Limestone; MSG: Mount Sorrel Granite
2. Adhesive Materials: Conventional 70/100 penetration grade of bitumen
3. Testing Conditions: Deformation rate and test temperature of 20 mm/minute and 25°C respectively

As has been stated before, in the dry conditioning, the effect of different types of substrates on the test results of maximum tensile bond strength and tensile energy required to produce failure per unit volume was minimum. Data of the test results of specimens subjected to dry conditioning can be concluded to be approximately the same, regardless of the substrates. However, for specimens subjected to wet conditioning, data of the test results was found to decrease in comparison to the control substrates of aluminium alloy. Ratio of the maximum tensile bond strength and tensile energy required to produce failure per unit volume between different types of aggregates (i.e. Dene Limestone, Ivonbrook Limestone and Mount Sorrel Granite) and aluminium alloy were 0.88, 0.79 and 0.67; and 0.70, 0.67 and 0.51 respectively. Mount Sorrel Granite has the lowest values of the maximum tensile bond strength and tensile energy required to produce failure per unit volume in the wet conditioning, in comparison to the control substrates of aluminium alloy. The reason for this can be attributed to the properties of the aggregates (i.e. high porosity and acidic). The effect of water or moisture in the conditioning procedures was more noticeable in the presence of mineral aggregates as substrates than aluminium alloy as substrates, as shown in Tables 6.14 and 6.15.

Table 6.14 Statistical analysis of Unpaired (i.e. Independent) Two-Samples t-Test procedure (Effect of conditioning procedures)

	Pair of Data Sets of Dry Conditioning & Wet Conditioning for Average Maximum Tensile Bond Strength			
	Aluminium Alloy	Dene Limestone	Ivonbrook Limestone	Mount Sorrel Granite
95.0% CI	(-23.39, 199.39)	(157.47, 336.53)	(160.27, 329.73)	(299.14, 630.86)
t-statistic, t	1.6598	5.7960	6.0752	5.8901
Two-tailed p-value	0.1143	0.0001	0.0001	0.0001
Significant Difference	NO	YES	YES	YES

Table 6.15 Effect of conditioning procedures on the test results

Properties	$\frac{AA_{WET}}{AA_{DRY}}$	$\frac{DL_{WET}}{DL_{DRY}}$	$\frac{IL_{WET}}{IL_{DRY}}$	$\frac{MSG_{WET}}{MSG_{DRY}}$
Maximum Tensile Bond Strength (kPa)	0.93	0.81	0.80	0.64
Tensile Energy Required to Produce Failure Per Unit Volume (kJ/m^3)	0.86	0.57	0.53	0.42

Notes: 1. AA: Aluminium Alloy; DL: Dene Limestone; IL: Ivonbrook Limestone; MSG: Mount Sorrel Granite
2. Adhesive Materials: Conventional 70/100 penetration grade of bitumen
3. Testing Conditions: Deformation rate and test temperature of 20 mm/minute and 25°C respectively

Based on Table 6.14, as expected, aluminium alloy has shown no significant statistical difference in the average maximum tensile bond strength between dry and wet conditionings. Aluminium alloy has the lowest difference of approximately 7% for maximum tensile bond strength and 14% for tensile energy required to produce failure per unit volume, as shown in Table 6.15, which can be attributed to the small percentage of water absorption of 0.05%. Low ability for water to penetrate into (i.e. non porous material) was found to result in minimum effect of water or moisture on the test results.

By comparing with the properties of the aggregates discs as listed in Table 6.5, it can be concluded that porosity and classification of the aggregates as either hydrophilic or hydrophobic have a profound influence on the test results, especially in the presence of water or moisture in the conditioning procedures (i.e. wet conditioning). Mount Sorrel Granite, which has high porosity and is classified as acidic was found to result in the most significant difference in the average maximum tensile bond strength between dry and wet conditionings, of approximately 36%. As shown in Table 6.15, reduction of the values of the test results between dry and wet conditionings of Dene Limestone and Ivonbrook Limestone was found to be approximately the same, which can be attributed to the same classification of the aggregates (i.e. basic).

6.6 Analysis to Determine the Effect of Adhesive Materials and Conditioning Procedures on the Test Results

In this section, data analysis was conducted in order to determine the effect of different types of bitumen (bitumen-filler mastic) of different penetration grade, and conditioning procedures (i.e. dry and wet conditionings) on the test results. Based on the study conducted by Marek and Herrin (1968), penetration grade of bitumen was found to have a profound influence on the test results, especially in terms of the types of failure and tensile bond strength. The values of the percentage area of adhesive failure and tensile bond strength were found to decrease with the increasing value of the penetration grade of bitumen (i.e. from hard to soft bitumen grade). Hence, based on the properties of the bitumen (bitumen-filler mastic) listed in Table 6.6, the values of the percentage area of adhesive failure and tensile bond strength of conventional 70/100 penetration grade of bitumen with different types of filler were expected to be ranked from high to low as follows; 20% by volume of Hydrated Lime filler, 20% by volume of Limestone filler, 20% by volume of Gritstone filler and without filler.

A total of 80 specimens of various combinations of asphalt mixture materials and conditioning procedures were prepared and tested at fixed deformation rate and test temperature of 20 mm/minute and 25°C respectively. Aluminium alloy and Ivonbrook Limestone were selected as substrates in order to study the behaviour of different types of bitumen (bitumen-filler mastic) without and with the influence of the mineral aggregates, respectively. Specimens of average thickness of adhesive layer of bitumen of 50 μm (0.050 mm) were subjected to either dry or wet conditionings at 25°C for 24 hours prior to testing. Table 6.16 and Figure 6.22 present the data of the test results.

Table 6.16 Results based on various combinations of asphalt mixture materials and conditioning procedures

Adhesive Materials	Substrates	Conditioning Procedures	Data Sets	Total Percentage Area of Adhesive Failure (%)	Maximum Tensile Bond Strength (kPa)	Tensile Energy Required to Produce Failure Per Unit Volume (kJ/m^3)
Conventional 70/100 Penetration Grade of Bitumen	Aluminium Alloy	Dry Conditioning at 25°C for 24 Hours Prior to Testing	1	100	1300	2599
			2	100	1350	2079
			3	100	1330	2471
			4	100	1270	1399
			5	95	1430	2682
		Wet Conditioning at 25°C for 24 Hours Prior to Testing	1	100	1250	1717
			2	100	1210	1569
			3	95	1220	2420
			4	100	1320	1975
			5	100	1240	1701
	Ivonbrook Limestone	Dry Conditioning at 25°C for 24 Hours Prior to Testing	1	100	1210	2151
			2	100	1260	1702
			3	100	1280	1146
			4	95	1170	3952
			5	100	1220	1910
		Wet Conditioning at 25°C for 24 Hours Prior to Testing	1	100	1040	1105
			2	100	980	978
			3	100	1030	2087
			4	100	1020	1142
			5	100	980	1264
Conventional 70/100 Penetration Grade of Bitumen and 20% by Volume of Hydrated Lime Filler	Aluminium Alloy	Dry Conditioning at 25°C for 24 Hours Prior to Testing	1	100	1620	3241
			2	100	1530	2788
			3	100	1490	2945
			4	100	1410	2674
			5	95	1540	2803
		Wet Conditioning at 25°C for 24 Hours Prior to Testing	1	100	1480	2314
			2	100	1430	2687
			3	95	1480	2796
			4	100	1400	1985
			5	100	1510	2964
	Ivonbrook Limestone	Dry Conditioning at 25°C for 24 Hours Prior to Testing	1	100	1370	3042
			2	100	1290	2498
			3	95	1350	2677
			4	100	1330	2303
			5	100	1380	2609
		Wet Conditioning at 25°C for 24 Hours Prior to Testing	1	100	1180	1986
			2	100	1190	2063
			3	100	1220	2012
			4	100	1250	2771
			5	100	1270	2154

Table 6.16 Results based on various combinations of asphalt mixture materials and conditioning procedures (continued)

Adhesive Materials	Substrates	Conditioning Procedures	Data Sets	Total Percentage Area of Adhesive Failure (%)	Maximum Tensile Bond Strength (kPa)	Tensile Energy Required to Produce Failure Per Unit Volume (kJ/m^3)
Conventional 70/100 Penetration Grade of Bitumen and 20% by Volume of Limestone Filler	Aluminium Alloy	Dry Conditioning at 25°C for 24 Hours Prior to Testing	1	100	1380	2642
			2	95	1480	2849
			3	100	1460	3105
			4	100	1530	2457
			5	100	1320	2055
		Wet Conditioning at 25°C for 24 Hours Prior to Testing	1	100	1380	1887
			2	100	1360	2546
			3	100	1310	2251
			4	100	1290	2409
			5	100	1370	2311
	Ivonbrook Limestone	Dry Conditioning at 25°C for 24 Hours Prior to Testing	1	100	1280	2973
			2	90	1290	2610
			3	100	1330	2294
			4	100	1350	2101
			5	100	1340	1976
		Wet Conditioning at 25°C for 24 Hours Prior to Testing	1	100	1110	2154
			2	100	1130	1965
			3	100	1190	1841
			4	100	1080	1569
			5	100	1200	2001
Conventional 70/100 Penetration Grade of Bitumen and 20% by Volume of Gritstone Filler	Aluminium Alloy	Dry Conditioning at 25°C for 24 Hours Prior to Testing	1	100	1410	2882
			2	100	1500	2495
			3	100	1330	1980
			4	100	1370	2537
			5	95	1420	2687
		Wet Conditioning at 25°C for 24 Hours Prior to Testing	1	100	1310	1836
			2	100	1250	1944
			3	95	1310	1673
			4	100	1260	2030
			5	100	1320	2174
	Ivonbrook Limestone	Dry Conditioning at 25°C for 24 Hours Prior to Testing	1	100	1330	2348
			2	100	1250	2219
			3	100	1320	1952
			4	100	1260	1801
			5	100	1270	2649
		Wet Conditioning at 25°C for 24 Hours Prior to Testing	1	100	1110	1811
			2	100	920	1154
			3	100	1060	1349
			4	100	1010	1788
			5	100	1060	1551

Notes: 1. Testing Conditions: Deformation rate and test temperature of 20 mm/minute and 25°C respectively
2. Average Thickness of Adhesive Layer of Bitumen: 50 μm

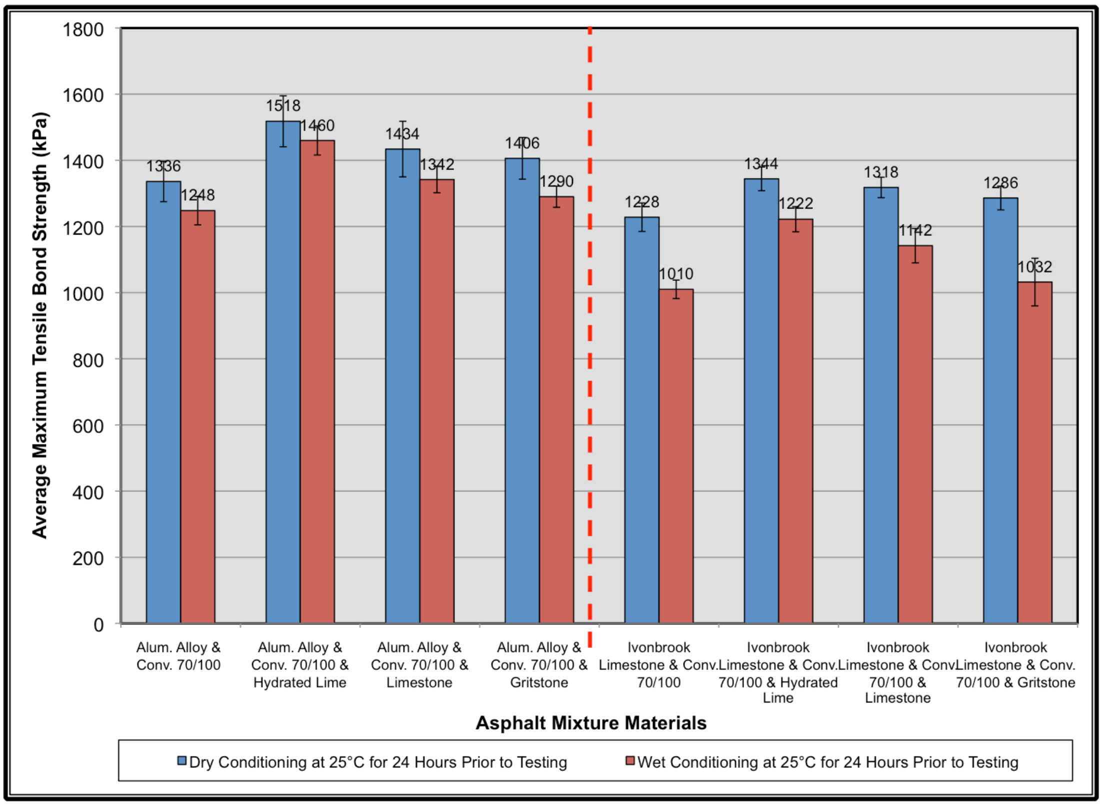
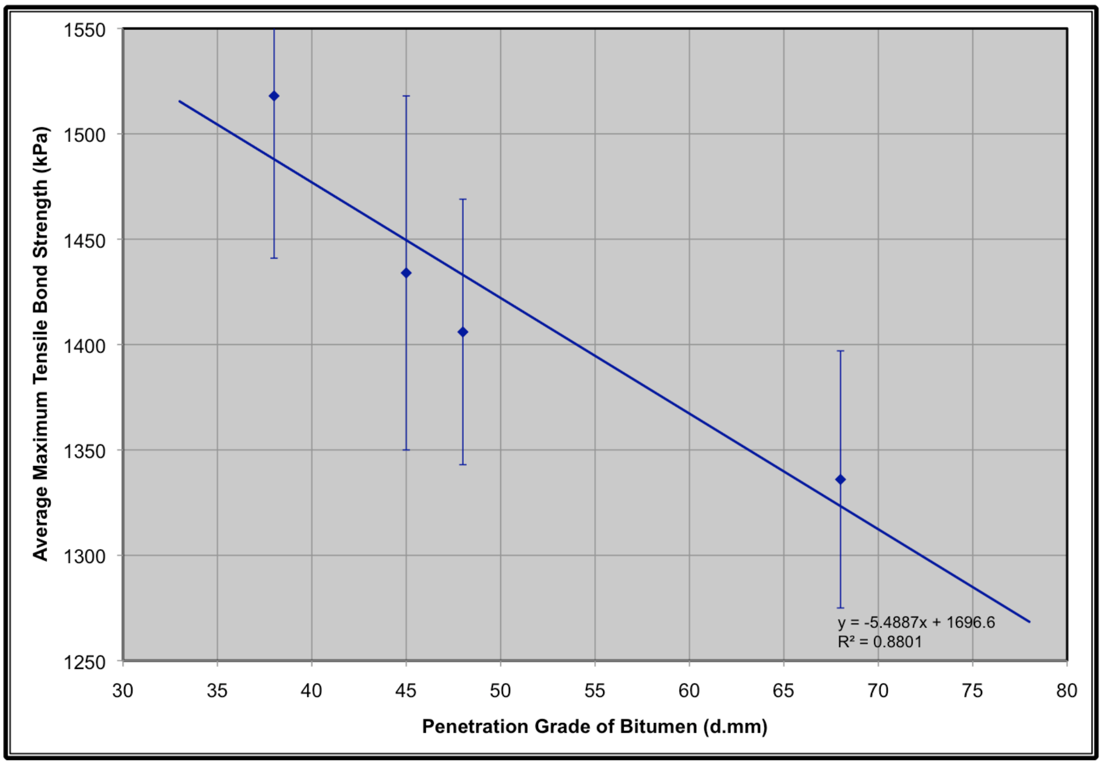


Figure 6.22 Average maximum tensile bond strength over wide ranges of asphalt mixture materials and conditioning procedures

Based on Table 6.16, the mode of failure for all specimens can be classified as adhesive due to the large value of the total percentage area of adhesive failure of more than 90%. The occurrence of the adhesive mode of failure can be correlated with the thickness of adhesive layer of bitumen. Based on Figure 6.22, addition of basic filler (i.e. Hydrated Lime and Limestone) to bitumen was found to increase the value of the maximum tensile bond strength, regardless of the substrates and conditioning procedures. Addition of Hydrated Lime and Limestone has resulted in increase in the value of the maximum tensile bond strength of approximately 11.54% and 7.35% for specimens subjected to dry conditioning, and 18.99% and 10.30% for specimens subjected to wet conditioning respectively, regardless of the substrates. While addition of Gritstone as mineral filler only increased the maximum tensile bond strength by 4.98% and 2.78% for specimens subjected to dry and wet conditionings respectively. Mineral filler, which is classified as basic was found to significantly improve the value of the tensile bond strength in the presence of water or moisture. In Figure 6.22, the error bar represents the one-standard deviation above and below the average maximum tensile bond strength.

Based on Table 6.16 and Figure 6.22, the average maximum tensile bond strength of conventional 70/100 penetration grade of bitumen with different types of filler can be ranked as follows; 20% by volume of Hydrated Lime filler, 20% by volume of Limestone filler, 20% by volume of Gritstone filler and without filler. As expected, data of the test results has justified the theoretically predicted performance of the different types of bitumen (bitumen-filler mastic), which was based on the properties listed in Table 6.6. The values of the maximum tensile bond strength were found to decrease with the increasing value of the penetration grade of bitumen (bitumen-filler mastic). Figure 6.23 shows the relationship between average maximum tensile bond strength and penetration grade of bitumen (bitumen-filler mastic), based on the data of the test results of specimens consisting of aluminium alloy as substrates and subjected to dry conditioning.



Notes:
1. Substrates: Aluminium alloy
2. Conditioning Procedures: Dry conditioning at 25°C for 24 hours prior to testing
3. Testing Conditions: Deformation rate and test temperature of 20 mm/minute and 25°C respectively
4. Thickness of Adhesive Layer of Bitumen: 50 μm

Figure 6.23 Relationship between average maximum tensile bond strength and penetration grade of bitumen

Unpaired (i.e. Independent) Two-Samples t-Test procedure, at level of significance, α of 0.05, was conducted in order to determine the existence of significant statistical difference in the average maximum tensile bond strength of conventional 70/100 penetration grade of bitumen due to the addition of different types of mineral filler. Table 6.17 summarises the output of the analysis. Again, the same decision rule in determining the existence of significant statistical difference in the pair of data sets was applied.

If two-tailed p-value is smaller than level of significance, α, significant statistical difference exists in the pair of data sets.

Otherwise, there is no significant statistical difference in the pair of data sets.

Table 6.17 Statistical analysis of Unpaired (i.e. Independent) Two-Samples t-Test procedure (Effect of addition of mineral filler)

	Pair of Data Sets for Average Maximum Tensile Bond Strength					
	Aluminium Alloy as Substrates					
	Addition of 20% by Volume of Hydrated Lime Filler		Addition of 20% by Volume of Limestone Filler		Addition of 20% by Volume of Gritstone Filler	
	Dry	Wet	Dry	Wet	Dry	Wet
95.0% CI	(-282.78, -81.22)	(-275.74, -148.26)	(-204.48, 8.48)	(-154.49, -33.51)	(-160.55, 20.55)	(-97.73, 13.73)
t-statistic, t	4.1644	7.6699	2.1224	3.5837	1.7826	1.7380
Two-tailed p-value	0.0031	0.0001	0.0666	0.0071	0.1125	0.1204
Significant Difference	YES	YES	NO	YES	NO	NO

	Pair of Data Sets for Average Maximum Tensile Bond Strength					
	Ivonbrook Limestone as Substrates					
	Addition of 20% by Volume of Hydrated Lime Filler		Addition of 20% by Volume of Limestone Filler		Addition of 20% by Volume of Gritstone Filler	
	Dry	Wet	Dry	Wet	Dry	Wet
95.0% CI	(-173.88, -58.12)	(-261.13, -162.87)	(-144.96, -35.04)	(-192.75, -71.25)	(-116.34, 0.34)	(-101.68, 57.68)
t-statistic, t	4.6216	9.9497	3.7763	5.0107	2.2927	0.6367
Two-tailed p-value	0.0017	0.0001	0.0054	0.0010	0.0511	0.5421
Significant Difference	YES	YES	YES	YES	NO	NO

Note: Conventional 70/100 penetration grade of bitumen without any addition of mineral filler was selected as control adhesive materials.

Based on Table 6.17, it can be concluded that there is sufficient evidence of significant statistical difference in the average maximum tensile bond strength of conventional 70/100 penetration grade of bitumen due to the addition of basic filler (i.e. Hydrated Lime and Limestone), regardless of the substrates and conditioning procedures, except for combination of aluminium alloy as substrates, Limestone filler and dry conditioning. Hence, a conclusion can be made that mineral filler, which is classified as basic could significantly improve the tensile bond strength. Addition of Gritstone as mineral filler to bitumen was found to result in minimum effect on the test results due to the classification as acidic (i.e. hydrophilic) (i.e. no significant difference in the average maximum tensile bond strength of conventional 70/100 penetration grade of bitumen due to the addition of Gritstone).

Additional analysis to observe the effect on the test results due to the addition of different types of mineral filler to bitumen was conducted, as shown in Table 6.18. Based on Table 6.18, in terms of the data of the test results of maximum tensile bond strength and tensile energy required to produce failure per unit volume, addition of Hydrated Lime as mineral filler was found to result in the highest increase of the properties, followed by Limestone and Gritstone, and thus justified the theoretically expected performance.

Table 6.18 Effect of different types of adhesive materials on the test results

Properties	Dry Conditioning at 25°C for 24 Hours						Wet Conditioning at 25°C for 24 Hours					
	Alum. Alloy			Ivonbrook Lime.			Alum. Alloy			Ivonbrook Lime.		
	HF/NF	LF/NF	GF/NF	HF/NF	LF/NF	GF/NF	HF/NF	LF/NF	GF/NF	HF/NF	LF/NF	GF/NF
Maximum Tensile Bond Strength (kPa)	1.14	1.07	1.05	1.09	1.07	1.05	1.17	1.08	1.03	1.21	1.13	1.02
Tensile Energy Required to Produce Failure Per Unit Volume (kJ/m^3)	1.29	1.17	1.12	1.21	1.10	1.01	1.36	1.22	1.03	1.67	1.45	1.16

Notes: 1. NF: Conventional 70/100; HF: Conventional 70/100 & Hydrated Lime Filler; LF: Conventional 70/100 & Limestone Filler; GF: Conventional 70/100 & Gritstone Filler
2. Testing Conditions: Deformation rate and test temperature of 20 mm/minute and 25°C respectively

The effect of water or moisture in the conditioning procedures was more noticeable in the presence of mineral aggregates as substrates (i.e. Ivonbrook Limestone), regardless of the adhesive materials, as shown in Tables 6.19 and 6.20. Specimens consist of mineral aggregates as substrates are expected to result in differences in the test results between dry and wet conditionings, which can be attributed to the porosity and classification of the aggregates as either hydrophilic (i.e. attract water) or hydrophobic (i.e. repulse water). For aluminium alloy as substrates, theoretically, no significant statistical difference in the test results between dry and wet conditionings should be observed due to the properties as non porous material, which provides low ability for water to penetrate into. However, based on Table 6.19, conventional 70/100 penetration grade of bitumen without filler and with addition of 20% by volume of Gritstone filler have shown significant statistical difference in the average maximum tensile bond strength between dry and wet conditionings. The reason for this can be attributed to the small sample size of 5, in which a small error and differences could affect the test results. Based on the previous data of the test results shown in Table 6.14 using a sample size of 10, aluminium alloy has shown no significant statistical difference in the average maximum tensile bond strength between dry and wet conditionings.

Table 6.19 Statistical analysis of Unpaired (i.e. Independent) Two-Samples t-Test procedure (Effect of conditioning procedures)

	Pair of Data Sets of Dry Conditioning & Wet Conditioning for Average Maximum Tensile Bond Strength			
	Aluminium Alloy as Substrates			
	Without Filler	20% by Volume of Hydrated Lime Filler	20% by Volume of Limestone Filler	20% by Volume of Gritstone Filler
95.0% CI	(11.17, 164.83)	(-33.20, 149.20)	(-3.36, 187.36)	(42.50, 189.50)
t-statistic, t	2.6413	1.4666	2.2248	3.6392
Two-tailed p-value	0.0297	0.1807	0.0568	0.0066
Significant Difference	YES	NO	NO	YES

	Pair of Data Sets of Dry Conditioning & Wet Conditioning for Average Maximum Tensile Bond Strength			
	Ivonbrook Limestone as Substrates			
	Without Filler	20% by Volume of Hydrated Lime Filler	20% by Volume of Limestone Filler	20% by Volume of Gritstone Filler
95.0% CI	(164.71, 271.29)	(67.92, 176.08)	(113.78, 238.22)	(170.86, 337.14)
t-statistic, t	9.4338	5.2021	6.5230	7.0447
Two-tailed p-value	0.0001	0.0008	0.0002	0.0001
Significant Difference	YES	YES	YES	YES

Table 6.20 Effect of conditioning procedures on the test results

Properties	Aluminium Alloy				Ivonbrook Limestone			
	$\frac{NF_{WET}}{NF_{DRY}}$	$\frac{HF_{WET}}{HF_{DRY}}$	$\frac{LF_{WET}}{LF_{DRY}}$	$\frac{GF_{WET}}{GF_{DRY}}$	$\frac{NF_{WET}}{NF_{DRY}}$	$\frac{HF_{WET}}{HF_{DRY}}$	$\frac{LF_{WET}}{LF_{DRY}}$	$\frac{GF_{WET}}{GF_{DRY}}$
Maximum Tensile Bond Strength (kPa)	0.93	0.96	0.94	0.92	0.82	0.91	0.87	0.80
Tensile Energy Required to Produce Failure Per Unit Volume (kJ/m^3)	0.84	0.88	0.87	0.77	0.61	0.84	0.80	0.70

Notes: 1. NF: Conventional 70/100; HF: Conventional 70/100 & Hydrated Lime Filler; LF: Conventional 70/100 & Limestone Filler; GF: Conventional 70/100 & Gritstone Filler
2. Testing Conditions: Deformation rate and test temperature of 20 mm/minute and 25°C respectively

Also, based on Table 6.20, in terms of the tensile energy required to produce failure per unit volume, for aluminium alloy as substrates, the ratio between dry and wet conditionings has shown a reduction as follows; 16%, 12%, 13% and 23% for conventional 70/100 penetration grade of bitumen without filler and with addition of 20% by volume of Hydrated Lime, Limestone and Gritstone respectively. While for Ivonbrook Limestone as substrates, the ratio between dry and wet conditionings for conventional 70/100 penetration grade of bitumen without filler and with addition of 20% by volume of Hydrated Lime, Limestone and Gritstone were 39%, 16%, 20% and 30% respectively.

Generally, it can be concluded that penetration grade of bitumen, types of substrates and mineral filler, and conditioning procedures have a profound influence on the test results. Hard bitumen grade (i.e. low penetration grade of bitumen), low porosity of substrates and classification of mineral filler as basic were expected to improve the performance of the asphalt mixtures, especially in terms of the adhesive bond strength.

6.7 Conclusions

In this chapter, the established criteria and procedures for the proposed adhesion test method were subjected to detailed evaluation in order to quantify the adhesive bond strength and failure characteristics of various combinations of asphalt mixture materials over wide ranges of testing conditions. A simple study was conducted in order to determine the effect of the amount of poured bitumen (i.e. under filling and overfilling) on the test results. Data of the test results has shown that the amount of poured bitumen has no or little effect, based on assumptions that a reasonable amount of bitumen has been poured (i.e. under filling and overfilling are not too extreme relative to the sufficiently filled).

Thickness of adhesive layer of bitumen was found to have a profound influence on the test results. The types of failure of specimens of either adhesive or cohesive can be modelled as a S-shaped curve due to the increasing or decreasing value of the thickness of adhesive layer of bitumen. Based on the following assumption; adhesive mode of failure is characterised by the value of the total percentage area of adhesive failure of more than 90%, thickness of 200 μm (0.200 mm) was deduced as point of transition of the mode of failure of either adhesive or mixed cohesive and adhesive. The value of the tensile bond strength within the region of thickness of less than 200 μm (0.200 mm) can be used to represent the adhesive bond strength between adhesive layer of bitumen and substrates. However, in order to generalise the conclusion over wide ranges of asphalt mixture materials and testing conditions, and also to take into account the worst-case scenario that might possibly occur, factor of safety of 2.0 has been applied. Hence, thickness of 100 μm (0.100 mm) has been deduced as the final thickness for the occurrence of the adhesive mode of failure in this study. Based on the data of the test results and also data of Marek and Herrin (1968), suitable thickness of adhesive layer of bitumen was suggested to be within 40 μm (0.040 mm) and 100 μm (0.100 mm), in order to allow for the occurrence of adhesive mode of failure and at the same time maintaining the uniformity and accuracy of the test results. Also, the mode of failure of entirely cohesive, mixed cohesive and adhesive and entirely adhesive can be characterised by the value of the total percentage area of adhesive failure as follows; less than 50%, between 50% and 90% and more than 90% respectively.

For thickness less than 500 μm (0.500 mm), aspect ratio of specimens was expected to result in negligible effect on the test results as long as the thickness of adhesive layer of bitumen remained constant. Hence, for the purpose of comparative analysis, it is important to prepare the specimen to uniform thickness of adhesive layer of bitumen despite the various area of contact between bitumen and substrates. However, beyond the thickness of 800 μm (0.800 mm) (i.e. in the extreme region of cohesive mode of failure), further study is suggested.

Based on the data of the previous chapter and also the continuation of the study in this part, the ranges of suitable testing conditions in terms of deformation rate and test temperature were verified as within 10 mm/minute and 20 mm/minute, and 15°C and 25°C respectively, regardless of the substrates and conditioning procedures. Also, based on the conducted study and comparison with the data of Marek and Herrin (1968), tensile bond strength was found to reach steady state conditions as deformation rate is increased beyond 20 mm/minute. Hence, application of higher deformation rate of more than 20 mm/minute for the laboratory adhesion test method seems to be wasteful.

Regardless of the substrates, specimens subjected to dry conditioning were found to exhibit approximately the same adhesive bond strength and failure characteristics, provided other testing variables and parameters were held constant. In the presence of water or moisture in the conditioning procedures (i.e. wet conditioning), significant difference in the test results among different types of substrates and also between dry and wet conditionings was expected, which mainly influenced by the physical properties of the substrates such as roughness of the surface and porosity, and also classification of the substrates as either hydrophilic (i.e. attract water) or hydrophobic (i.e. repulse water). Also, in the presence of water or moisture in the conditioning procedures (i.e. wet conditioning), different types of bitumen (bitumen-filler mastic) of different penetration grade were found to have a profound influence on the test results. Hard bitumen grade (i.e. low penetration grade of bitumen) and addition of mineral filler, especially basic (i.e. hydrophobic) filler was expected to improve the adhesive bond strength and failure characteristics of the asphalt mixtures.

Within the ranges of suitable testing conditions, tensile bond strength of specimens subjected to dry conditioning was higher compared to the specimens subjected to wet conditioning. However, the types of failure of specimens of either adhesive or cohesive are mainly influenced by the thickness of adhesive layer of bitumen rather than the presence of water or moisture. Data of the test results has shown that the occurrence of the adhesive mode of failure can still be achieved in the absence of water or moisture provided that the thickness of adhesive layer of bitumen is thin enough.

Generally, the values of the tensile bond strength and tensile energy required to produce failure per unit volume were expected to increase with the increasing deformation rate, and decrease with the increasing test temperature and the increasing value of the penetration grade of bitumen (bitumen-filler mastic) (i.e. from hard to soft bitumen grade). Based on the data of the test results of the whole study, the established criteria and procedures for the proposed adhesion test method were verified as capable in quantifying the adhesive bond strength and failure characteristics of various combinations of asphalt mixture materials (i.e. bitumen (bitumen-filler mastic) and aggregates) over wide ranges of thicknesses of adhesive layer of bitumen, aspect ratio of specimens, testing conditions (i.e. deformation rates and test temperatures) and conditioning procedures (i.e. dry and wet conditionings). Limitations of the testing variables and parameters were described in detail in this chapter.

CHAPTER 7

CONCLUSIONS AND RECOMMENDATIONS FOR FUTURE RESEARCH DEVELOPMENT

The overall objective of this study was to develop and establish a simple, practical and reliable monotonically-loaded laboratory adhesion test method for direct measurement of the adhesive bond strength of bitumen (bitumen-filler mastic) and aggregates. The established criteria and procedures for the laboratory adhesion test method were then subjected to detailed evaluation in order to quantify the adhesive bond strength and failure characteristics of various combinations of asphalt mixture materials over wide ranges of testing conditions, to propose suitable testing conditions and to validate the reliability and efficiency. Adhesion between bitumen (bitumen-filler mastic) and aggregates is considered as one of the main fundamental properties of the asphalt mixtures, which can be correlated with the quality, performance and serviceability. However, there are no established testing techniques and procedures that can be used to quantify the adhesive bond strength of bitumen (bitumen-filler mastic) and aggregates, and therefore research in this area is crucial and evidently needed. There have been some efforts in developing the testing techniques and procedures such as published by Copeland (2007), Kanitpong and Bahia (2003), Kanitpong and Bahia (2004) and Kanitpong and Bahia (2005). However, the developed testing techniques and procedures have not enjoyed universal success and acceptance, and not yet established. Also, there is no published research in the pavement related areas that had determined the effect of different types of filler (i.e. bitumen-filler mastic) on the adhesive bond strength and failure characteristics of asphalt mixtures.

7.1 Conclusions

Based upon the data of the test results and analysis of the three parts of the study, the following conclusions were made.

1. Part 1: Selection and Justification of the Proposed Adhesion Test Method

   Based on the comprehensive literature review on various testing techniques and procedures used to measure the adhesive bond strength, adhesion test method based on the pull off (tension) mode was concluded as the most suitable and realistic approach for development of laboratory adhesion test method for asphalt mixtures. Pull off (tension) mode was found to be the best approach to describe the adhesive bond strength and failure characteristics of asphalt mixtures, and this was also supported by the study conducted by Harvey (2000), Kanitpong and Bahia (2003) and Marek and Herrin (1968). Also, several factors such as ease of specimen preparation, cost effectiveness of test setup and apparatus, availability of suitable testing equipment (i.e. INSTRON servo hydraulic frame and Ductilometer testing apparatus) and compatibility with asphalt mixtures were taken into account in making the selection.

2. Part 2: Development of Criteria and Procedures for the Proposed Adhesion Test Method

   A general concept based on the pull off (tension) mode was subjected to trial and error experimental approach, in order to establish the criteria and procedures for the laboratory adhesion test method. Throughout this part, the development of criteria and procedures in terms of test setup and apparatus, specimen preparation, testing and data analysis was conducted based on the consideration of the INSTRON servo hydraulic frame and Ductilometer testing apparatus as testing equipments. Both of the testing equipments were subjected to preliminary and subsequent study.

Based on the preliminary and subsequent study, the INSTRON servo hydraulic frame was found to be the most suitable and practical testing equipment compared to the Ductilometer testing apparatus. Draft of standard criteria and procedures for the laboratory adhesion test method based on the INSTRON servo hydraulic frame as testing equipment was developed and presented in Appendix C. Based on the conducted analysis, the established criteria and procedures were found capable to measure the adhesive bond strength with excellent uniformity and repeatability. Total conditioning time of 24 hours has been considered as the standard conditioning procedures for both dry and wet conditionings, regardless of the substrates. Temperature for the conditioning procedures was dependent on the test temperature. Also, a standard procedure in determining the types of failure of specimens as either adhesive or cohesive was established, as presented in Appendix B.

3. Part 3: Detailed Evaluation and Validation of the Proposed Adhesion Test Method

In this part, the established criteria and procedures for the laboratory adhesion test method were subjected to further evaluation in quantifying the adhesive bond strength and failure characteristics of various combinations of asphalt mixture materials (i.e. bitumen (bitumen-filler mastic) and aggregates) over wide ranges of thicknesses of adhesive layer of bitumen, aspect ratio of specimens, testing conditions (i.e. deformation rates and test temperatures) and conditioning procedures (i.e. dry and wet conditionings). Results of the study were then used for comparative analysis in order to determine the effect of various variables and parameters on the test results, to propose suitable testing conditions and to validate the reliability and efficiency of the laboratory adhesion test method. In order to consider wide ranges of asphalt mixture materials, various types of substrates and adhesive materials of distinct properties were utilised, and details of the asphalt mixture materials were summarised in Tables 6.5 and 6.6.

Initial study was conducted in order to determine the effect of the amount of poured bitumen (i.e. under filling, sufficiently filled or overfilling) on the test results. Results of the study have shown that the amount of poured bitumen has no or little effect on the test results, as long as a reasonable amount of bitumen is poured (i.e. excessive or insufficient amount is not too extreme). Also, in this part, thickness of adhesive layer of bitumen was found to have a profound influence on the types of failure of specimens, regardless of conditioning procedures (i.e. dry and wet conditionings).

Based on the data of the test results and data of Marek and Herrin (1968), suitable thickness of adhesive layer of bitumen for the laboratory adhesion test method was suggested to be within 40 μm (0.040 mm) and 100 μm (0.100 mm), in order to allow for the occurrence of adhesive mode of failure and at the same time maintaining the uniformity and accuracy of the test results. Aspect ratio of specimens was expected to result in negligible effect on the test results as long as the thickness of adhesive layer of bitumen remained constant. Ranges of suitable testing conditions in terms of deformation rate and test temperature for the laboratory adhesion test method were suggested as within 10 mm/minute and 20 mm/minute, and 15°C and 25°C respectively, regardless of the substrates and conditioning procedures. It can be concluded that within the ranges of suitable testing conditions, the value of the tensile bond strength and tensile energy required to produce failure per unit volume were expected to increase with the increasing deformation rate, and decrease with the increasing test temperature and the increasing value of the penetration grade of bitumen (bitumen-filler mastic) (i.e. from hard to soft bitumen grade).

Based on the data of the test results of the whole study, the established criteria and procedures for the laboratory adhesion test method were verified as capable in quantifying the adhesive bond strength and failure characteristics of various combinations of asphalt mixture materials (i.e. bitumen (bitumen-filler mastic) and aggregates) over wide ranges of testing conditions. In terms of the uniformity and repeatability of the test results, except for the tensile energy required to produce failure per unit volume, all parameters (i.e. thickness of adhesive layer of bitumen, total percentage area of adhesive failure and maximum tensile bond strength) have resulted in coefficient of variation of less than 7%. High variability of the tensile energy required to produce failure per unit volume can be correlated with the curve fitting procedures and the various parameters that governed the values of the tensile energy required to produce failure per unit volume such as tensile load, pull off displacement and thickness of adhesive layer of bitumen. However, this can be considered as the first step to gain basic knowledge and understanding of the tensile energy required to produce failure per unit volume of asphalt mixture materials. Draft of standard criteria and procedures for the laboratory adhesion test method that can be used as guiding principles in conducting the test can be referred in Appendix C.

7.2 Recommendations for Future Research Development

Considering the overall work that have been done in this study and based on the data of the test results and analysis, the following recommendations are suggested for future research development.

1. Round robin test is suggested in order to validate the reproducibility of the developed laboratory adhesion test method. Reproducibility can be defined as the ability of the developed laboratory adhesion test method to be used independently by different operators at different laboratories in obtaining consistent data of the test results as compared to the replicate specimens under the same testing conditions. In order to perform the round robin test, different operators at different laboratories are suggested to conduct the laboratory adhesion test method using INSTRON servo hydraulic frame on the same asphalt mixture materials under the same testing conditions, based on the developed draft of standard criteria and procedures, as presented in Appendix C. Hence, the degree of agreements of the data of the test results between different operators at different laboratories can be observed and verification on how well the operators dealing with the criteria and procedures of the laboratory adhesion test method based on the instructions given in Appendix C can be determined.

2. Round robin test is also recommended in order to evaluate the consistency and accuracy of the procedures in determining the types of failure of specimens, which were based on visual observations, as presented in Appendix B. Several different operators can be used to evaluate the types of failure of the same specimens subjected to laboratory adhesion test method, and the variability of the data of the test results in terms of the percentage area of failure based on different operators can be used to verify the degree of reliability of the procedures. Alternatively, image analysis that is capable to differentiate and measure the types of failure of specimens as either adhesive or cohesive is suggested.

3. Draft of standard criteria and procedures for the laboratory adhesion test method in terms of the test setup and apparatus, specimen preparation, testing and data analysis are presented in Appendix C. However, for the laboratory adhesion test method to become a standardised method, improved setup of the moulds (i.e. pair of plates) is suggested, as illustrated in Figure 7.1. Details of the suggested improved test setup and apparatus are given in Appendix E. Based on the improved setup of the moulds (i.e. pair of plates), better precision and accuracy for the thickness of adhesive layer of bitumen can be achieved without the need of spacers. Also, limitation in the measurement of the thickness of adhesive layer of bitumen can be improved by implementing a more precise measurement tool.

4. Based on the analysis of the effect of the amount of poured bitumen (i.e. under filling, sufficiently filled or overfilling) on the test results, which had been conducted in Section 6.1.1, improved procedures for specimen

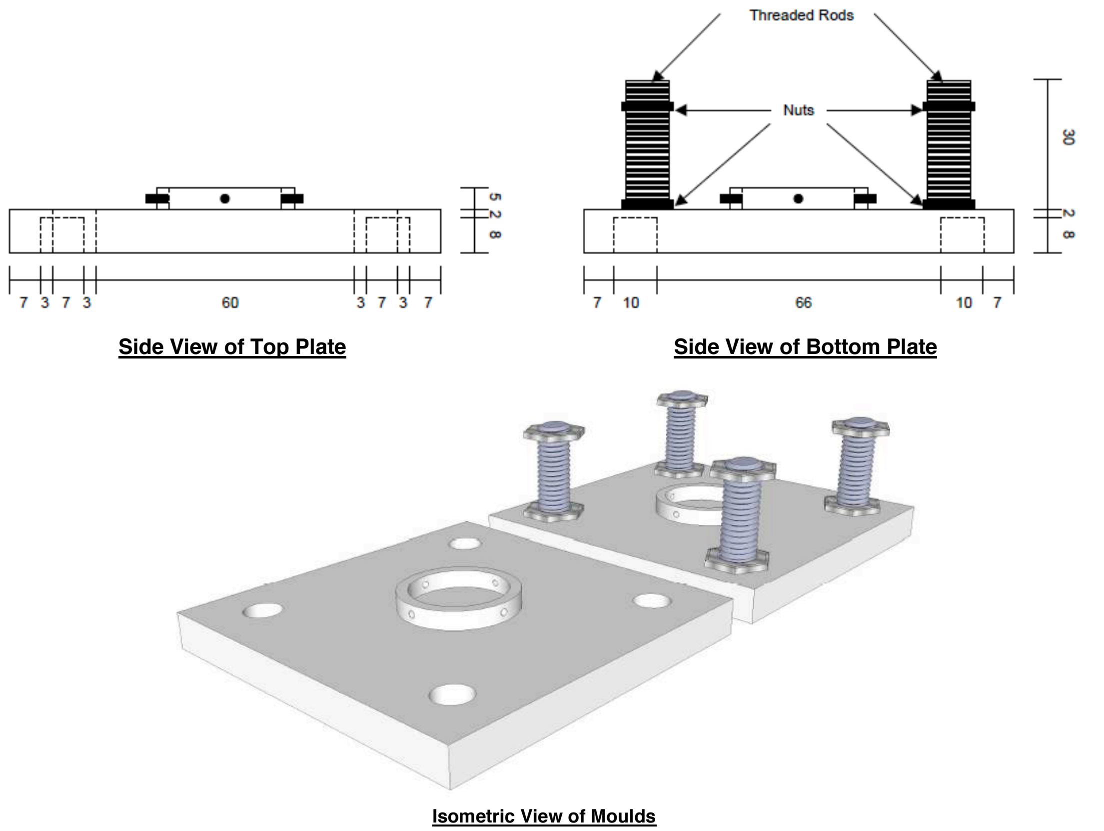
preparation are required in order to ensure the uniformity and consistency of the amount of poured bitumen. It is suggested that a reasonable amount of bitumen be poured until fully coated surface of the bottom plate is achieved in order to result in overfilling, followed by the placement of the top plate, and the required thickness of adhesive layer of bitumen is then achieved via compression. The amount of poured bitumen is suggested to be within the range of sufficiently filled and overfilling in order to minimise the discrepancy and inaccuracy of the test results.

5. Laboratory adhesion test method has been shown to be an effective method for quantifying the adhesive bond strength and failure characteristics of various combinations of asphalt mixture materials over wide ranges of testing conditions, which include deformation rates, test temperatures and conditioning procedures (i.e. dry and wet conditionings). Currently, there is limitation in terms of the applicability in conducting test in a temperature controlled water bath (i.e. conducting test under water), as per Ductilometer testing apparatus. However, measurement of the adhesive bond strength via test conducted in a temperature controlled water bath (i.e. conducting test under water) is assumed to be of little significance due to the relatively short duration of the test.

6. Based on Bhasin et al. (2006), Cheng et al. (2002) and Masad et al. (2006), thermodynamic surface free energy characteristics of aggregates, bitumen and water (moisture) can be used in calculating the intrinsic (theoretical) adhesion and thus the adhesive bond strength of asphalt mixtures. This approach has allowed the adhesive bond energy

related parameters to be calculated for various combinations of bitumen and aggregates. Adhesive bond energy related parameters consist of adhesive bond energy of aggregates and bitumen in dry conditioning and reduction of surface free energy when bitumen debonds from aggregates surface in the presence of water or moisture. Hence, data of the test result from the established laboratory adhesion test method in this study which is based on the direct measurement of the adhesive bond strength can be used to correlate with the adhesive bond energy related parameters based on the thermodynamic surface free energy concept. Combination of these two approaches will thus pave the way for further development and refinement of the assessment of the adhesive bond strength of asphalt mixtures.

7. Based on the relationship between tensile bond strength and thickness of adhesive layer of bitumen analysed in Chapter 6, beyond the thickness of more than 600 μm (0.600 mm), constant value of tensile bond strength was observed. These constant values of tensile bond strength can be directly correlated with the cohesive bond strength of bitumen (bitumen-filler mastic) (i.e. intermolecular force developed within adhesive layer of bitumen). Cohesive bond strength of bitumen (bitumen-filler mastic) is mainly influence by viscosity, and hence Dynamic Shear Rheometer (DSR) and ductility test are suggested for validation of the assumptions. Kanitpong and Bahia (2003) has developed a method known as Tackiness Test of Asphalt using Dynamic Shear Rheometer (DSR) in order to measure the cohesive bond strength of bitumen. Details of the developed method are presented in Chapter 2 (Section 2.4.1).



Note: All units are in millimeter (mm)

Figure 7.1 Improved setup of the moulds (i.e. pair of plates)

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APPENDIX A

LOP 9.28 Tensile Bond Testing (TBT) Laboratory Operation Procedures - Test Methods/Testing

9.28 Tensile Bond Testing (TBT)

Laboratory Operation Procedures – Test Methods/Testing

Facility: Pavement Research Building NTEC
Last Modified by Mick Winfield on 20/08/2008

Status: Approved by Jon Watson

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Summary: This procedure explains how to conduct tensile bond testing of asphalt samples using the INSTRON servo hydraulic loading frame for in-house test PT 0127, following guidance provided in DMRB HD 47/99 Appendix B.

Changes to previous version: Includes section on data retrieval 9.28.5

9.28.1 General

This document outlines the procedure, which will be followed by the laboratory staff for testing multi-layer specimens to measure the adhesion strength between layers in tension, according to in-house procedure PT 0127. The tensile bond test (TBT) uses worksheet WS-PT0127.

Samples for this test are normally provided with 100 mm x 100 mm cross-section and are located on the central line of a 305 mm x 305 mm. The samples generally consist of a number of layers of materials e.g. asphalt/asphalt or asphalt/concrete. Steel end plates of 100 mm x 100 mm cross-section with a threaded hole are glued to the top of the specimen. Once bonded, threaded "eye" hooks are then screwed into the end plates and other fixtures are then used to connect to the INSTRON servo-hydraulic frame. The test is conducted within a temperature-controlled cabinet and the sample is loaded at a controlled rate until bond failure is observed and maximum bond strength recorded.

9.28.2 Health and Safety

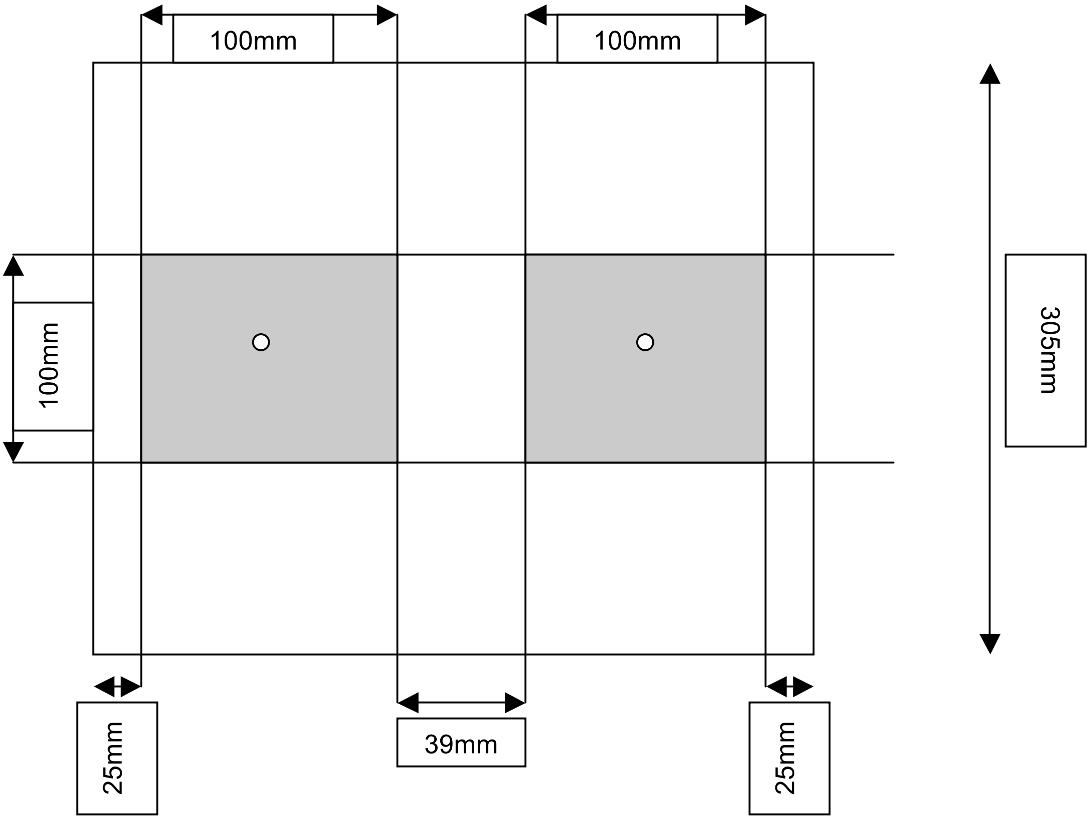
Read Risk Assessment and COSHH assessment information associated with the instruments and adhesives used.

When using the hydraulic load frames ensure that items of clothing and fingers etc. do not get trapped between the loading rams.

9.28.3 Sample Preparation

The 305 mm x 305 mm slabs need to be trimmed as per the diagram below (see LOP for safe use of the clipper saw). Once the slab has dried, the two steel plates can be glued with araldite to the two test specimens (shaded area on the drawing). The steel plates can be held in place with two steel box sections (located in A10). Weights can then be added to the top of the steel plates. Silver foil will be needed to close up the saw cuts, to avoid glue dripping down the sides of the specimen.

When the glue has cured, place the specimens in a conditioning cabinet at the required temperature for a minimum of 4 hours and a maximum of 24 hours.



Plan View of a 305mm x 305mm slab

9.28.4 Procedure for "TBT" testing using the Instron

1) Set the test cabinet to the test temperature, where able (cabinets range -5 to +60°C). Also place the testing apparatus into the cabinet and record the time. Standard test temperature is 23 ± 2°C.

2) Samples must be in the conditioning cabinet for at least 4 hours.

3) Turn on the hydraulic pump as stated in LOP 11.13.

4) Turn on the computer situated next to the INSTRON.

5) Click on the Rubicon Icon on the computer screen.

6) Click Run.

7) Click start manual. Check stroke and click OK.

8) Press **Δ** on the controller attached to the INSTRON. This gives manual control and allows the ram to be raised or lowered, to enable the TBT block to be fitted into place using the up and down arrows on the controller.

(Using the up and down arrows allows for small movement of the hydraulic ramp. For greater travel the knobs on the front of the machine can be used. First by using the unlocking knob, and then using the up and down knobs)

9) Check that the hydraulic ramp (when the block is in position) has plenty of travel (at least 10 mm) to allow the test to be conducted.

10) Click Command on the computer

11) Select Ramps 1. Input the following:

- Control mode : Stroke
- End point : +10mm (1st sample) there on based on 1st sample.
- Relative or Absolute : Relative
- Rate/Time : 20mm/minutes
- Next : Manual
- Profile : Linear
- Update : Check Maxima and Minima
- Targets 1: If REL STROKE >= +10.0 (as required) then FREEZE
  2: If ABS LOAD >= +20Kn (as required) then FREEZE

Depending on the material being used and the test temperature, a higher load than 20 kN may be required. If this is the case go into the global trips and adjust the max load accordingly!!!

- Check overview (to ensure all parameters are set correctly)

Select Ramp (check box at bottom right hand side of the overview screen)

- Check trips
- Click Run (TAB)
- Pre-release x-y plot (ICON)
- Finish Logging : Yes
- Select axis : Y= Load X= Stroke
- Then OK
- Save data into a file: OK **(Save into G:NCPE DATA\SHATS-TATS\Tensile adhesion\Job number\Test order number)**
- Write in file name: OK **(Ensure sample numbers have an underscore instead of a dash or the data will not save)**
- Start new test
- Threshold v change to load (50 N or as required)
  v other sensor: change to stroke (50 um or as required)
  (This is the interval at which the computer saves the information)
- Click on GO
- Are you ready to start new test: Yes
- Once the sample has failed click the Finish Logging button
- Convert to ASCII FORMAT
- Re-write file name
- Click on the bottom right corner arrow on the screen
- Regain control of the RAM by selecting the Stroke mode.

The testing is now complete. Repeat the above steps to carry out any subsequent tests.

9.28.5 Procedure Data retrieval

- Open the .asc version of the file needed e.g. **G:\Ncpe data\ITS data\jn950\07-3243.asc**
- If the file doesn't open in Notepad, right click, click on Open With and select Notepad

07_3243 - Notepad

File Edit Format Help

Control interface cycle number at the beginning of the test: 0

Point	Time	Cycle	Stroke	Load	Extension		Strain-4	
#	Sec	#	m	N	mm	mm	mm	
0	0.452	0	1.2785E-2	-4.3333E+0	1.6833E-2	1.2767E-1	1.1667E-6	4000
1	0.457	1	1.2782E-2	-3.3333E+0	1.6167E-2	1.3033E-1	0.0000E+0	4005
2	0.465	1	1.2787E-2	-1.6667E+0	1.6167E-2	1.2833E-1	5.0000E-7	4005
3	0.490	1	1.2773E-2	-1.6667E+0	1.6167E-2	1.3033E-1	0.0000E+0	4005
4	0.515	1	1.2767E-2	-3.3333E+0	1.6167E-2	1.2800E-1	-1.6667E-7	4005
5	0.541	1	1.2747E-2	-4.3333E+0	1.5667E-2	1.2900E-1	1.6667E-7	4005
6	0.566	1	1.2735E-2	-3.3333E+0	1.6167E-2	1.2933E-1	6.6667E-7	4005
7	0.591	1	1.2713E-2	1.0000E+0	1.5667E-2	1.2800E-1	0.0000E+0	4005
8	0.641	1	1.2673E-2	-1.5267E+2	1.5667E-2	1.2767E-1	5.0000E-7	4005
9	0.667	1	1.2647E-2	-3.2767E+2	1.5833E-2	1.2900E-1	6.6667E-7	4005
10	0.692	1	1.2632E-2	-5.1667E+2	1.6167E-2	1.2833E-1	-6.6667E-7	4005
11	0.715	1	1.2607E-2	-7.2733E+2	1.5000E-2	1.2933E-1	6.6667E-7	4005
12	0.740	1	1.2587E-2	-9.2500E+2	1.5667E-2	1.2800E-1	5.0000E-7	4005
13	0.765	1	1.2575E-2	-1.1420E+3	1.6167E-2	1.2767E-1	-1.6667E-7	4005
14	0.791	1	1.2552E-2	-1.3433E+3	1.5667E-2	1.2933E-1	5.0000E-7	4005
15	0.816	1	1.2528E-2	-1.5470E+3	1.5667E-2	1.2833E-1	5.0000E-7	4005
16	0.841	1	1.2513E-2	-1.7353E+3	1.4333E-2	1.2900E-1	8.3333E-7	4005
17	0.866	1	1.2480E-2	-1.9540E+3	1.5000E-2	1.2900E-1	0.0000E+0	4005
18	0.891	1	1.2462E-2	-2.1550E+3	1.5000E-2	1.2767E-1	-6.6667E-7	4005
19	0.917	1	1.2447E-2	-2.3407E+3	1.5667E-2	1.2833E-1	5.0000E-7	4005
20	0.942	1	1.2420E-2	-2.4717E+3	1.5667E-2	1.2767E-1	6.6667E-7	4005
21	0.965	1	1.2400E-2	-2.6220E+3	1.5000E-2	1.2933E-1	5.0000E-7	4005
22	0.990	1	1.2380E-2	-2.8020E+3	1.5500E-2	1.2800E-1	6.6667E-7	4005
23	1.015	1	1.2367E-2	-2.9580E+3	1.5167E-2	1.2900E-1	5.0000E-7	4005
24	1.041	1	1.2347E-2	-3.1020E+3	1.5167E-2	1.2767E-1	6.6667E-7	4005
25	1.066	1	1.2320E-2	-3.1873E+3	1.4500E-2	1.2933E-1	5.0000E-7	4005
26	1.091	1	1.2312E-2	-3.3117E+3	1.5167E-2	1.2900E-1	0.0000E+0	4005
27	1.116	1	1.2282E-2	-3.4620E+3	1.5000E-2	1.2767E-1	5.0000E-7	4005
28	1.141	1	1.2253E-2	-3.5500E+3	1.5000E-2	1.3067E-1	5.0000E-7	4005
29	1.167	1	1.2240E-2	-3.6593E+3	1.5000E-2	1.2800E-1	-5.0000E-7	4005
30	1.192	1	1.2220E-2	-3.7513E+3	1.4333E-2	1.2933E-1	5.0000E-7	4005
31	1.215	1	1.2202E-2	-3.8340E+3	1.5167E-2	1.3067E-1	6.6667E-7	4005
32	1.240	1	1.2173E-2	-3.9313E+3	1.5000E-2	1.2767E-1	5.0000E-7	4005
33	1.265	1	1.2167E-2	-4.0140E+3	1.5667E-2	1.2800E-1	6.6667E-7	4005
34	1.291	1	1.2142E-2	-4.1207E+3	1.5000E-2	1.2800E-1	0.0000E+0	4005
35	1.316	1	1.2113E-2	-4.2243E+3	1.4333E-2	1.2800E-1	5.0000E-7	4005
36	1.341	1	1.2100E-2	-4.2873E+3	1.5000E-2	1.2800E-1	5.0000E-7	4005
37	1.366	1	1.2085E-2	-4.3783E+3	1.5167E-2	1.2900E-1	6.6667E-7	4005
38	1.391	1	1.2060E-2	-4.4727E+3	1.4333E-2	1.3033E-1	-6.6667E-7	4005
39	1.442	1	1.2018E-2	-4.5927E+3	1.4333E-2	1.2800E-1	5.0000E-7	4005
40	1.465	1	1.1988E-2	-4.6620E+3	1.5000E-2	1.2767E-1	5.0000E-7	4005
41	1.490	1	1.1980E-2	-4.7283E+3	1.4333E-2	1.3067E-1	-1.6667E-7	4005
42	1.515	1	1.1953E-2	-4.7967E+3	1.5000E-2	1.2767E-1	-1.6667E-7	4005
43	1.566	1	1.1913E-2	-4.9050E+3	1.5500E-2	1.2900E-1	6.6667E-7	4005
44	1.616	1	1.1868E-2	-4.9900E+3	1.4333E-2	1.2767E-1	-1.6667E-7	4005
45	1.667	1	1.1827E-2	-5.0833E+3	1.4833E-2	1.2900E-1	5.0000E-7	4005
46	1.715	1	1.1780E-2	-5.1657E+3	1.5000E-2	1.2833E-1	0.0000E+0	4005

- To find the Peak Load, scroll down the **Load** column until you get to the largest number. In the case of ITS and SAT the load is recorded as negative and Roofing adhesion, TAT and TBT the load is positive.
- The letter E means x 10 and the positive or negative to the right is the power the 10 is timed by. So E+3 = $x10^3$ = x1000, E+2 = $x10^2$ = x100, E+1 = 10^1 = x10
- From the above example the Peak Load is 5165.7 N (-5.1657E+3) or 5.1657 kN.

9.28.6 Report

Report according to HD 47/99 Appendix B, or raw data, as requested by client.

<<End Document>>>

APPENDIX B

Procedures for Determination of Types of Failure of Specimens

APPENDIX B

PROCEDURES FOR DETERMINATION OF TYPES OF FAILURE OF SPECIMENS

B.1 General Background

Procedures in determining the types of failure of specimens subjected to the laboratory adhesion test method were developed based on the BS EN ISO 4624:2003 Paints and Varnishes-Pull-Off Test for Adhesion, which had used the simplest, easiest and commonly used method; visual observation. Example of the determination of the types of failure of specimens as either adhesive or cohesive is presented in the following section.

Types of failure (adhesive or cohesive failure) were determined via visual observation of the top and bottom of each pair of plates, and then calculated based on the percentage area of adhesive failure. Figure B.1 shows the top and bottom of a pair of plates after being subjected to testing.

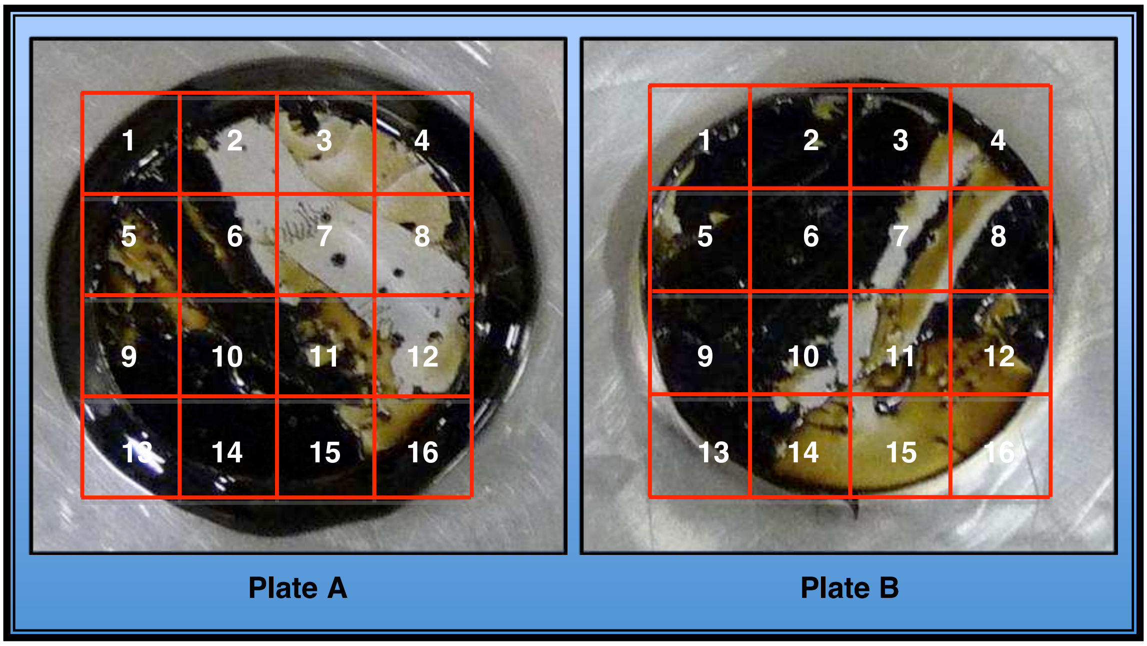


Figure B.1 Area of contact of adhesive materials and substrates of Plates A and B

B.2 Procedures

1. Divide the area of contact of adhesive materials and substrates for each plate (i.e. Plates A and B) into 16 squares of equal area as shown in the Figure B.1.
2. Analyse each square via visual observation and estimate to the nearest 10% the value of percentage area of adhesive failure. (Note: Approximate estimation is made in determining the percentage area of adhesive failure for square that is not fully covered by the area of contact of adhesive materials and substrates (i.e. Square 1, 2, 3, 4, 13, 14, 15 and 16)).
3. Tabulate the value of the percentage area of adhesive failure for each square as in Table B.1.
4. Calculate the average of percentage area of adhesive failure for each plate (i.e. Plates A and B) and then calculate the combined total percentage area of adhesive failure for pair of plates, respectively.

Round the value of combined total percentage area of adhesive failure for pair of plates to the highest 5%.

5. A value of 100% has been set as the maximum total percentage area of adhesive failure for pair of plates.

Table B.1 Example of determination of types of failure via visual observation

Square	Percentage Area of Adhesive Failure (%)[1]	
	Plate A	Plate B
(1 & 2)[2]	60	10
(3 & 4)[2]	90	20
5	50	30
6	40	0
7	100	70
8	90	30
9	10	0
10	10	50
11	40	80
12	90	70
(13 & 14)[2]	0	50
(15 & 16)[2]	10	90
Average of Percentage Area of Adhesive Failure (%)	49.20	41.70
Combined Total Percentage Area of Adhesive Failure for Pair of Plates (%)[3]	49.20 + 41.70 = 90.90 (i.e. 95% Adhesive Failure)[4]	

Notes:
[1]Percentage area of adhesive failure is estimated to the nearest 10%;
[2]Squares are combined in order to allow for a more accurate estimation;
[3]Combined total percentage area of adhesive failure for pair of plates is rounded to the highest 5%;
[4]If combined total percentage area of adhesive failure for pair of plates is more than 80%, considered as adhesive failure, otherwise cohesive failure.

APPENDIX C

Draft of Standard Criteria and Procedures for the Laboratory Adhesion Test Method Using INSTRON Servo Hydraulic Frame

APPENDIX C

DRAFT OF STANDARD CRITERIA AND PROCEDURES FOR THE LABORATORY ADHESION TEST METHOD USING INSTRON SERVO HYDRAULIC FRAME

C.1 Scope

This draft sets out recommended criteria and procedures in terms of test setup and apparatus, specimen preparation, testing and data analysis for conducting laboratory adhesion test method using INSTRON servo hydraulic frame.

Laboratory adhesion test method using INSTRON servo hydraulic frame is intended to quantify the adhesive bond strength and failure characteristics of various combinations of asphalt mixture materials (i.e. bitumen (bitumen-filler mastic) and aggregates) over wide ranges of testing conditions (i.e. deformation rates and test temperatures) and conditioning procedures (i.e. dry and wet conditionings). Results in terms of the percentage area of failure, tensile bond strength and tensile energy required to produce failure can be assessed upon completion of the test.

Parts of the criteria and procedures for the laboratory adhesion test method are developed in accordance with the referenced documents as listed below. However, several contents are changed for adaptation purposes.

(Note: Ranges of suitable testing conditions for the laboratory adhesion test method in terms of deformation rate and test temperature are 10 mm/minute and 20 mm/minute, and 15°C and 25°C, respectively)

C.2 Referenced Documents

1. BS EN ISO 4624:2003 Paints and Varnishes-Pull-Off Test for Adhesion.
2. LOP 9.28 Tensile Bond Testing (TBT) Laboratory Operation Procedures - Test Methods/Testing.
3. LOP 11.24 Blending Bitumen and Filler Laboratory Operation Procedures – Equipment Usage.

C.3 Test Setup and Apparatus

1. Oven
   - For heating bitumen (bitumen-filler mastic) and aggregates discs.
   - Capable of maintaining temperature ranges up to 200°C ± 5°C.
2. Pair of plates (i.e. top and bottom plates) (Figure C.1)
   - Measuring an area of 100 mm by 100 mm and thickness of 10 mm for each plate.
   - Capable for the insertion of various types of aggregates as substrates in the forms of 25 mm diameter discs.
   - Four rods to be inserted at each corner of the pair of plates in order to prevent any lateral movement.
3. Compression device (Figure C.2)
   - Consists of micrometer of accuracy up to 10 μm.
   - Capable of distributing compressive load onto pair of plates up to the required thickness of adhesive layer of bitumen.
4. Conditioning cabinet
   - Capable of maintaining temperature ranges between 0°C and 60°C.

5. Rigid testing rig (Figure C.3)
    - Consists of two parts; top plate with two vertical hollow rods and base plate with two vertical solid rods, which can be slide into each other.
    - Capable of distributing axial tensile load onto pair of plates.
    - Capable of resulting failure based on the pull off (tension) mode only (i.e. excludes the effects of peel and shear mode).
6. Universal joint attachment (Figure C.4)
    - Capable of attaching and thus transferring tensile load in perpendicular direction from upper part of the INSTRON servo hydraulic frame to the rigid testing rig.
7. Two carver clamps (Figure C.5)
    - Capable of securing rigid testing rig to hydraulic ramp (i.e. lower part) of the INSTRON servo hydraulic frame.
8. Linear Variable Differential Transducer (LVDT)
    - Capable of measuring vertical pull off displacement of tested pair of plates. Captured data of the vertical pull of displacement is then analysed via built-in software of the INSTRON servo hydraulic frame.
9. INSTRON servo hydraulic frame
    - Capable in applying loads in tension and compression.
    - Capable in conducting test over wide ranges of deformation rates and test temperatures within the ranges of 10 mm/minute and 20 mm/minute, and 15°C and 25°C respectively.

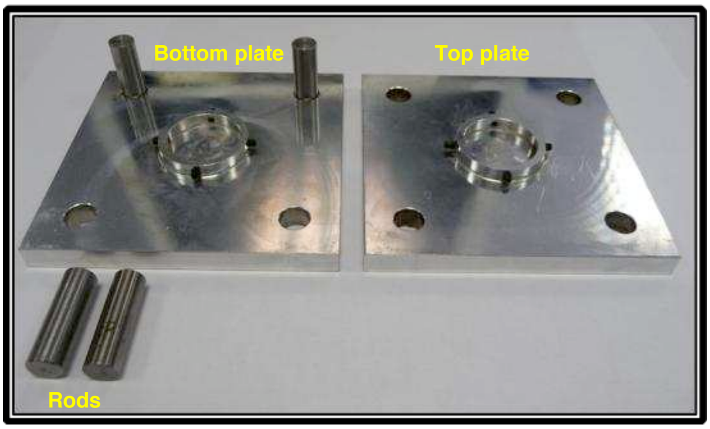


Figure C.1 Pair of plates (i.e. top and bottom plates) and four rods

Figure C.2 Compression device

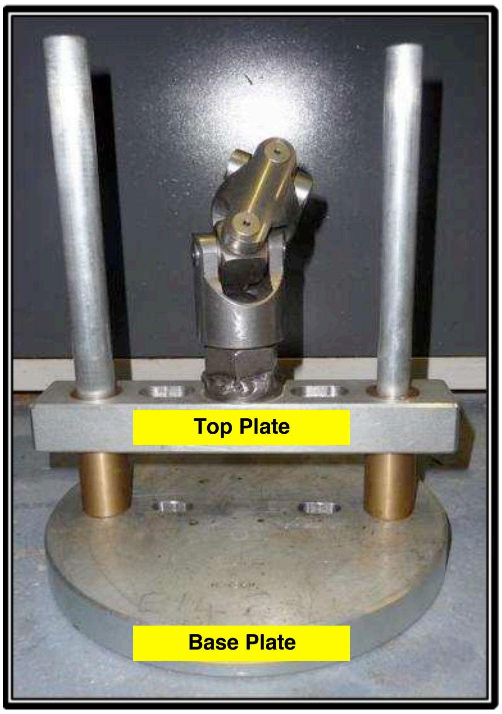


Figure C.3 Rigid testing rig and bolts to secure pair of plates

Figure C.4 Universal joint attachment

Figure C.5 Carver clamp

C.4 Materials

1. Preparation of aggregates discs

    a. Aggregates are prepared into 25 mm diameter discs (i.e. 490.87 mm^2 area of contact) and 8 mm thickness via the cutting of 25 mm diameter cylindrical core specimens extracted from boulder. (Figure C.6)

    b. Prior to specimen preparation, aggregates discs are dried at 80ºC for 30 minutes in oven, and surface of area of contact must be clean, dry and free from dust.

2. Preparation of bitumen (bitumen-filler mastic)

    a. For bitumen-filler mastic, mineral filler should be satisfactory to the requirement of 70% to 100% of particles passing a 63-µm sieve.

    b. Also, in order to achieve the thickness of adhesive layer of bitumen (bitumen-filler mastic) of 50 µm, mineral filler is further sieved through a 45-µm sieve (British Standard Sieve Series Mesh No. 350).

    c. Procedures for mixing bitumen-filler mastic as presented in Appendix D (i.e. LOP 11.24 Blending Bitumen and Filler Laboratory Operation Procedures – Equipment Usage) can be used as guidelines.

    d. Bitumen (bitumen-filler mastic) is then filled separately in small containers prior to storage for ease of pouring during specimen preparation.

    e. Prior to specimen preparation, bitumen (bitumen-filler mastic) is heated to approximately 160°C for at least two hours in oven, and is stirred thoroughly to ensure uniformity and homogeneity.

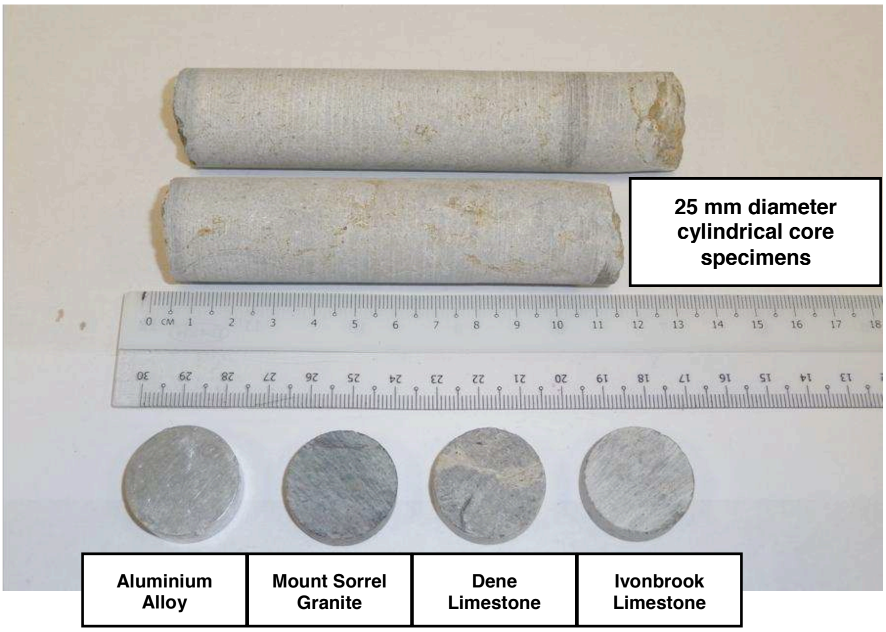


Figure C.6 Aggregates discs and cylindrical core specimens

C.5 Procedures for Specimen Preparation

1. Clean pair of plates using chemical solution (i.e. white spirit solvent) in order to ensure cleanliness and then followed by acetone (ethyl acetate) in order to remove the remaining chemical solution of the white spirit solvent.
2. Insert aggregates discs, which have been dried at 80ºC for 30 minutes into top and bottom plates respectively, and secure the position of the aggregates discs by tightening four screws (at 90º to each other) using Hex (Allen) key. (Note: Ensure surface of area of contact of the aggregates discs of top and bottom plates are levelled to each other).
3. Measure initial thickness of pair of plates (i.e. without adhesive layer of bitumen (bitumen-filler mastic)) using micrometer attached to the compression device and record the reading as T_0.
4. Pour bitumen (bitumen-filler mastic) which has been heated to approximately 160°C for at least two hours onto aggregates discs of the

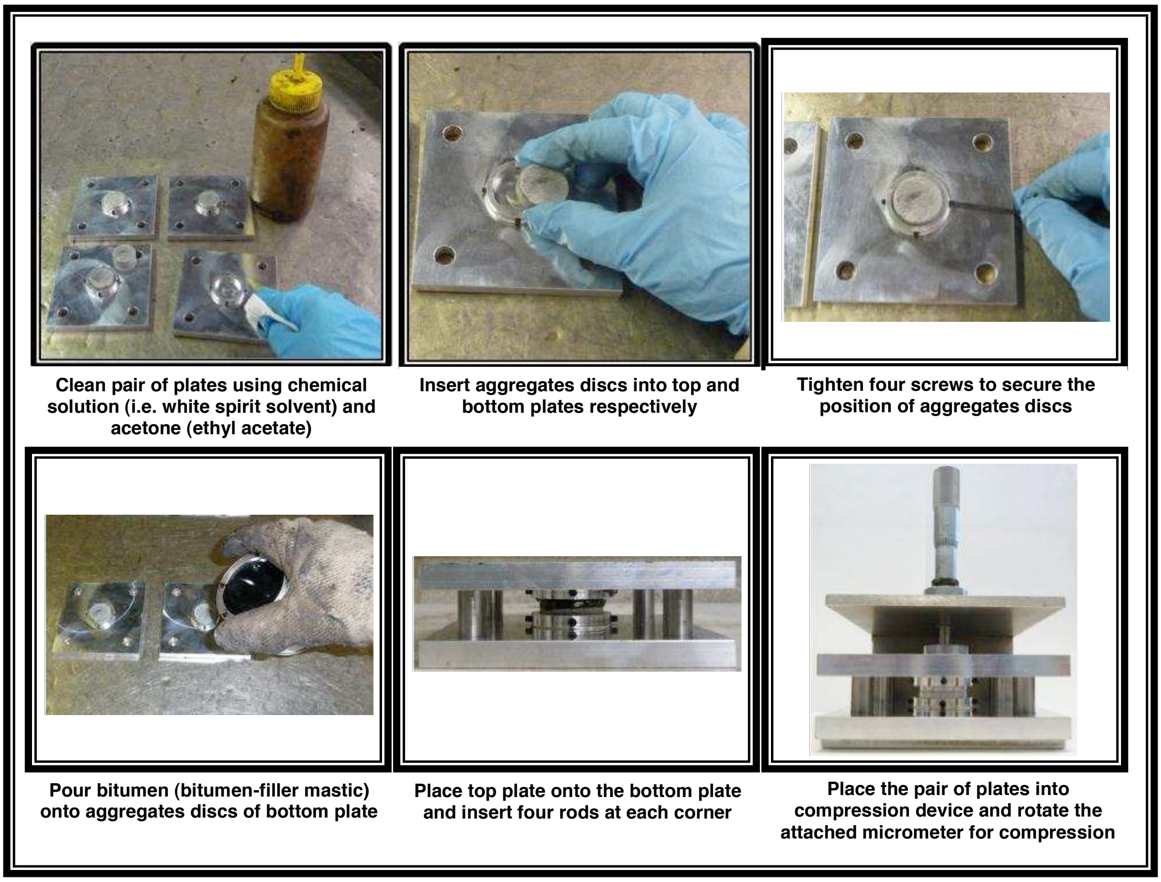
bottom plate, starting from centre of the aggregates discs up to about two-third filled in order to minimise the amount of excess bitumen (bitumen-filler mastic).

5. Place the top plate onto the bottom plate and insert four rods at each corner of the pair of plates in order to prevent any lateral movement.
6. **Optional Procedures**: Insert spacers in between the pair of plates in order to control the thickness of adhesive layer of bitumen.
7. Place the pair of plates into compression device and rotate the attached micrometer in order to compress up to the required thickness of adhesive layer of bitumen. (Note: Use the value of the initial thickness of pair of plates (i.e. without adhesive layer of bitumen (bitumen-filler mastic)) and the required thickness of adhesive layer of bitumen to determine the amount of rotation required).

Equation C.1

$$
\begin{aligned}
&(\text{Final Thickness of Pair of Plates, } T_1) \\
&\quad = (\text{Initial Thickness of Pair of Plates, } T_0) \\
&\quad - (\text{Required Thickness of Adhesive Layer of Bitumen})
\end{aligned}
$$

8. Leave the pair of plates in the compressed position for about 15 minutes prior to conditioning procedures.



Clean pair of plates using chemical solution (i.e. white spirit solvent) and acetone (ethyl acetate)

Insert aggregates discs into top and bottom plates respectively

Tighten four screws to secure the position of aggregates discs

Pour bitumen (bitumen-filler mastic) onto aggregates discs of bottom plate

Place top plate onto the bottom plate and insert four rods at each corner

Place the pair of plates into compression device and rotate the attached micrometer for compression

Figure C.7 Summary of the procedures for specimen preparation

C.6 Conditioning Procedures

Pair of plates is subjected to either dry or wet conditionings at specified test temperature for 24 hours prior to testing in the conditioning cabinet. (Note: For wet conditioning, immerse the pair of plates in container filled with water and place the container in the conditioning cabinet).

C.7 Procedures for Testing

1. Take out the pair of plates from conditioning cabinet and carefully remove the four rods at each corner of the pair of plates.
2. Place the pair of plates in between the top and base plate of the rigid testing rig and secure the test setup by inserting and tightening the bolts.
3. Attach the universal joint attachment to the test setup by fastening to the top plate of the rigid testing rig.
4. Turn on the hydraulic pump and INSTRON servo hydraulic frame.
5. Set the test cabinet of the INSTRON servo hydraulic frame to the required test temperature.
6. Place the test setup (i.e. pair of plates, rigid testing rig and universal joint attachment) on hydraulic ramp (i.e. lower part) of the INSTRON servo hydraulic frame and secure the position using two carver clamps.
7. Follow the following procedures for operating the built-in software of the INSTRON servo hydraulic frame. (Note: To be used as guidelines only and might vary depending on the version and types of the built-in software).
   a. Select Icon Rubicon.
   b. Click Run > Click Start Manual > Select Stroke > Click OK.
   c. Press Icon ∆ on the controller attached to the INSTRON servo hydraulic frame (Note: Enable manual control for the movement of up and down of hydraulic ramp (i.e. lower part) of the INSTRON servo hydraulic frame).
   d. Adjust the position of the hydraulic ramp to enable the universal joint attachment to be fastened to the upper part of the INSTRON servo hydraulic frame.

e. Ensure that hydraulic ramp (i.e. lower part) of the INSTRON servo hydraulic frame has sufficient space (i.e. at least 10 mm) to allow movement during testing.

(Note: Condition the test setup (i.e. pair of plates, rigid testing rig and universal joint attachment) in the test cabinet of the INSTRON servo hydraulic frame for at least 15 minutes prior to testing)

Figure C.8 Test setup (i.e. pair of plates, rigid testing rig and universal joint attachment) is positioned in the INSTRON servo hydraulic frame

f. Click Command > Click Ramp > Click 1

g. Input as follows:

- Control Mode: Stroke
- End point: +10.000 mm
- Checkbox Relative
- Rate/Time: **(Insert the required deformation rate)**
- Next: Manual

- Profile: Linear
- Checkbox Maxima > Checkbox Minima
- Targets 1: If REL STROKE >= +10.000 then FREEZE
- Targets 2: If ABS LOAD >= +20.000 kN then FREEZE

h. Click Overview (Note: Ensure that all parameters are correctly inserted) > Checkbox Select Ramps (i.e. at the right hand bottom)

i. Click Run > Click X-Y Plot

- Click Finish Logging > YES
- Click Select Axis > (Select Y= Load) > (Select X=Stroke) > OK
- Click Save Data Into File > OK **(Select destination to save the file)**

j. Click Start New Test > Click Test Setup > Checkbox Threshold

- Checkbox Load > Load = 500 N
- Checkbox Other Sensor > Stroke = 50 μm
- Click GO > (Are You Ready to Start New Test: YES)

k. Once completed, click Finish Logging

l. Convert file to ASCII Format

m. Re-write file name

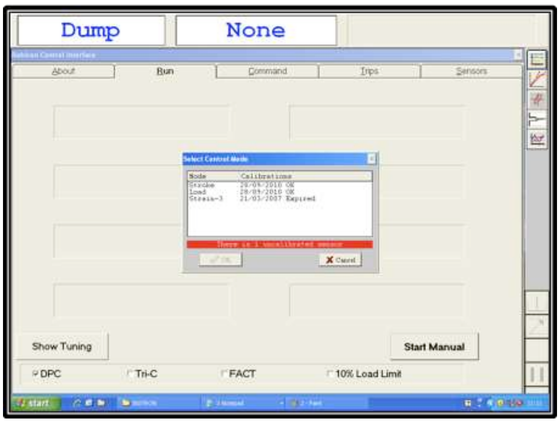
n. Exit X-Y Plot

o. Regain control of the hydraulic ramp

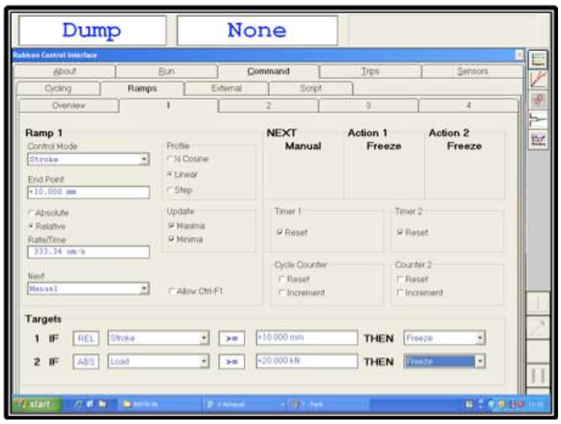
Click Start Manual > Click Stroke

8. Testing is now completed. Carefully remove the test setup (i.e. pair of plates, rigid testing rig and universal joint attachment) from the INSTRON servo hydraulic frame.
9. Carefully remove the separated pair of plates from the top and base plate of the rigid testing rig.

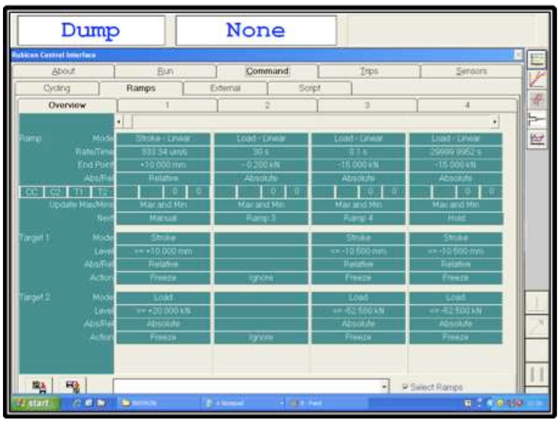
Select Icon Rubicon



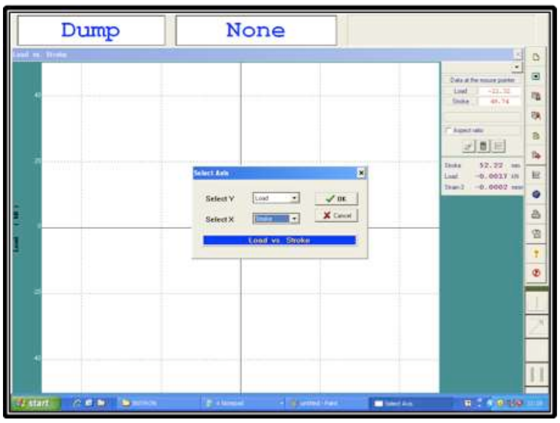
Click Run > Click Start Manual > Select Stroke > Click OK



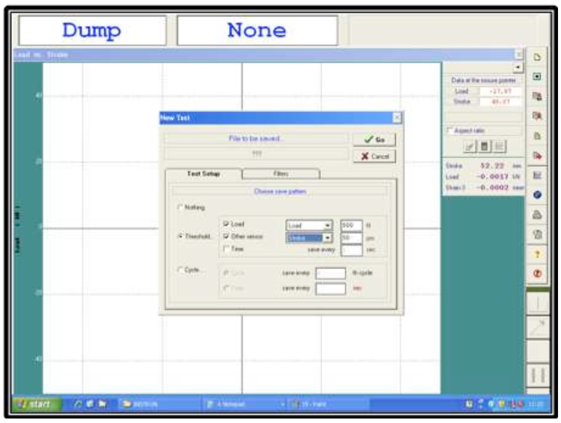
Click Command > Click Ramp > Click 1
Input as follows



Click Overview > Checkbox Select Ramps (i.e. at the right hand bottom)



Click Select Axis > (Select Y= Load) > (Select X=Stroke) > OK



Click Start New Test > Click Test Setup >
Checkbox Threshold
Input as follows

Figure C.9 Summary of the procedures for testing

C.8 Data Analysis

1. Percentage area of failure

   Observe and record the percentage area of failure via visual observation. Refer Appendix B for detailed procedures in determining the types of failure of specimens subjected to laboratory adhesion test method using INSTRON servo hydraulic frame.

2. Tensile bond strength

   Built-in software of the INSTRON servo hydraulic frame records the value of tensile load and pull off displacement at specified intervals. Determine the maximum tensile load from data of the test results. In order to determine the maximum tensile bond strength, divide the value of the maximum tensile load with the area of contact of adhesive materials and substrates (i.e. area of circle of 25 mm diameter = 490.87 mm^2).

3. Tensile energy required to produce failure

   Use CurveExpert 1.4 or any graphical software to calculate the area under the curve for graph of tensile load versus pull off displacement. Figure C.10 shows the example of the graphs of tensile load versus pull off displacement of the original and corrected curve for the tested specimen. The curvature of initial part of the original curve is attributed to the initial seating and adjustment of the apparatus and also testing equipments. Hence, corrected curve is required in order to eliminate these effects. For the corrected curve, correction is determined by projecting the linear portion of the curve to the pull off displacement axis and horizontal shift based on the distance between the intersection and the origin is then applied.

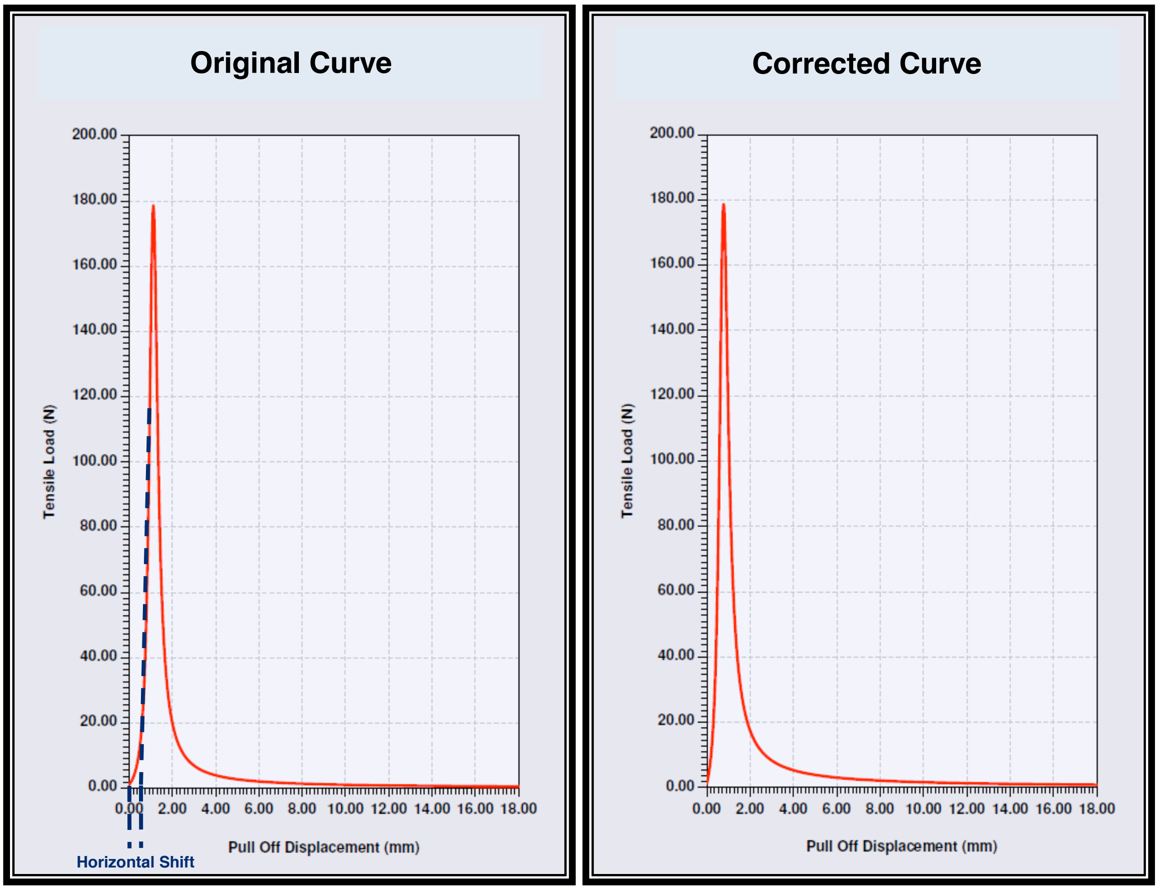


Figure C.10 Plots of tensile load versus pull off displacement

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APPENDIX D

LOP 11.24 Blending Bitumen and Filler Laboratory Operation Procedures – Equipment Usage

11.24 Blending Bitumen and Filler

Laboratory Operation Procedures – Equipment Usage

Facility: Pavement Research Building NTEC

Last Modified by Mick Winfield on 31/03/2009

Status: Approved by Mick Winfield

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Summary: This document outlines the procedures that shall be followed by laboratory staff when blending filler into base bitumen.

Changes to previous version: Alter fonts to bring LOP in line with template.

11.24.1 Scope

Filler is blended into base bitumen to produce a product with new properties.

11.24.2 Health & Safety

The appropriate risk assessment located in the laboratory shall be read and adhered to by staff when carrying out the procedures detailed below.

11.24.3 Equipment

1) Oven
2) Hot plate
3) Digital thermometer and dedicated probe
4) Balance with a resolution of at least 1 gram
5) Large pallet knife
6) E1520 variable speed stirrer and paddle

11.24.4 Procedures

Before proceeding with the procedures detailed below, the technician shall ensure that the client's approval of the blend has been obtained and recorded on the worksheet.

1) Place the bulk sample of bitumen in an oven until it is molten enough to pour.
2) Whilst the bitumen is heating up, weigh out the required amount of filler into a clean glass beaker and record the mass on the appropriate worksheet and also switch on a hot plate to warm up before blending.
3) Weigh out the required mass of bitumen into a clean tin and record the mass.
4) When the required mass of bitumen has been weighed, remove the tin from the balance and place on the hot plate in the fume cupboard.

If the blend is relatively small and/or the filler content is low or the filler content is very high (i.e. above 75%): Use a large pallet knife (strongly heated for very high filler contents) and slowly add the filler stirring continuously until all the filler is added to the binder. Continue stirring until no filler can be seen and the sample appears totally homogenous.

If the blend is relatively large and/or the filler content is high but not very high: Lower the (pre heated) stirrer paddle down into the bitumen and switch on. Gradually build the speed up to minimise splattering and slowly add the filler stirring continuously until all the filler is added to the binder. Continue stirring until no filler can be seen and the sample appears totally homogenous.

5) If during blending the bitumen cools too much or the filler starts to coagulate stop adding the filler and return the tin into the oven allowing it to get back to temperature, before proceeding with blending.

6) When the blend is complete, re-label the tin with its new NTEC sample number and ensure the stirrer paddles and/or pallet knife are cleaned ready for the next user.

7) It is usual practice to sub-sample a small amount of the blend whilst it is still hot in order to determine the new binder's initial properties.

8) Place the freshly made binder into the oven and keep warm until the new properties have been established.

9) Once the properties are established and deemed to be correct by the client, the binder can now either be re-canned or used in further blending.

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APPENDIX E

Details of Suggested Improved Test Setup and Apparatus for Laboratory Adhesion Test Method

APPENDIX E

DETAILS OF SUGGESTED IMPROVED TEST SETUP AND APPARATUS FOR LABORATORY ADHESION TEST METHOD

Design and fabrication of pair of plates (i.e. top and bottom plates) for laboratory adhesion test method are suggested as follows.

1. Top plate (Note: Top plate remains the same as in Appendix C)
   - Measuring an area of 100 mm by 100 mm and thickness of 10 mm.
   - Capable for the insertion of various types of aggregates in the forms of 25 mm diameter discs.
   - Four screws at 90˚ to each other are used to secure the position of aggregates discs.
   - Consists of four holes of 10 mm diameter at each corner.

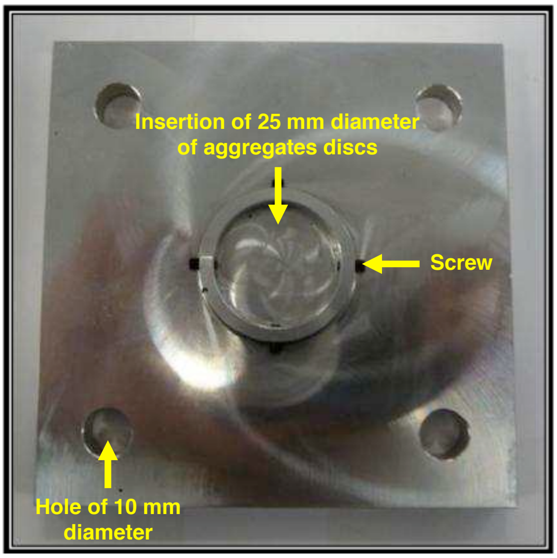
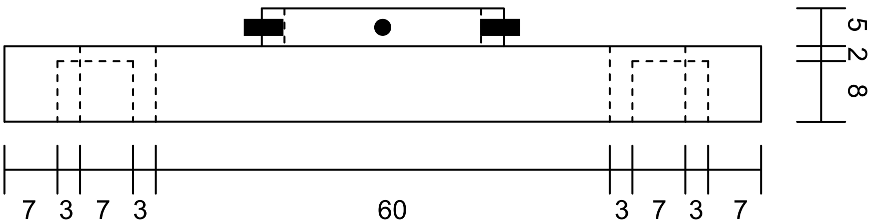


Figure E.1 Top plate

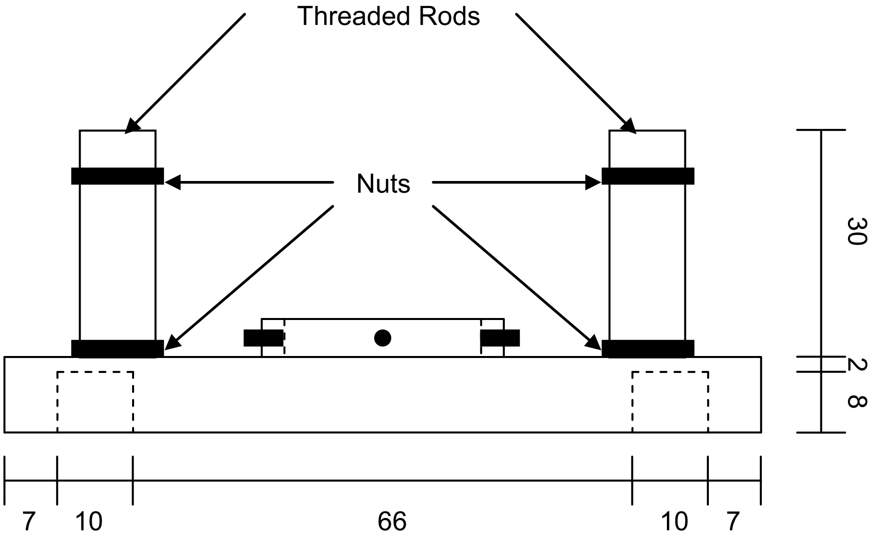
2. Bottom plate

- Measuring an area of 100 mm by 100 mm and thickness of 10 mm.
- Capable for the insertion of various types of aggregates in the forms of 25 mm diameter discs.
- Four screws at 90˚ to each other are used to secure the position of aggregates discs.
- Instead of using four removable rods as before, four threaded rods of approximately 10 mm diameter and 30 mm height are fixed at each corner of the bottom plate in order to prevent any lateral movement between the pair of plates. Also, by implementing the fixed threaded rods, nuts can be used to cater for various thicknesses of adhesive layer of bitumen. Hence, spacers are no longer required. (Note: Diameter of the fixed threaded rods is suggested to be slightly less than the diameter of the hole of the top plate (i.e. 10 mm) in order to prevent any friction due to sliding between the pair of plates during testing).



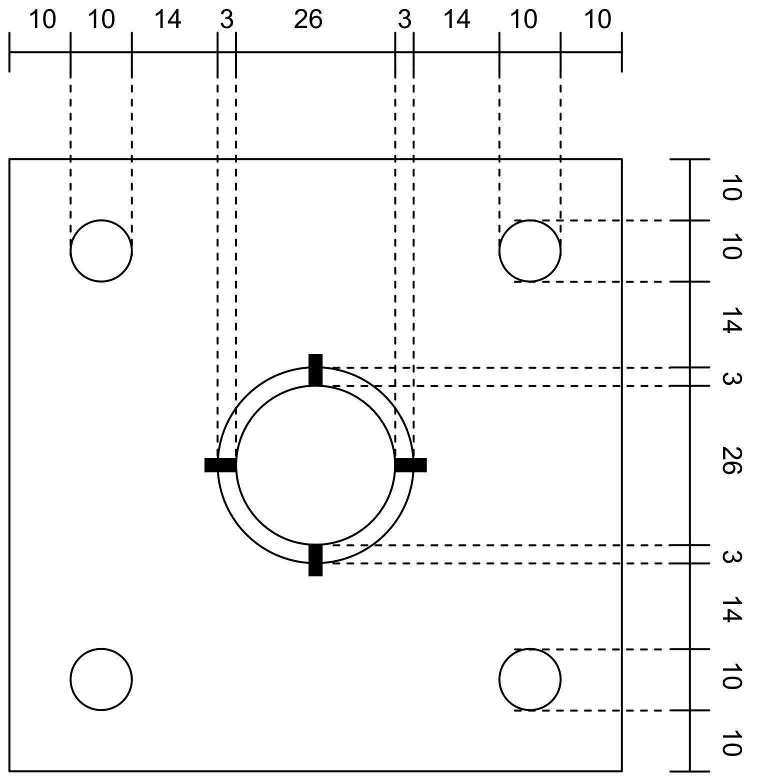
Note: All units are in millimeter (mm)

Figure E.2 Side view of top plate



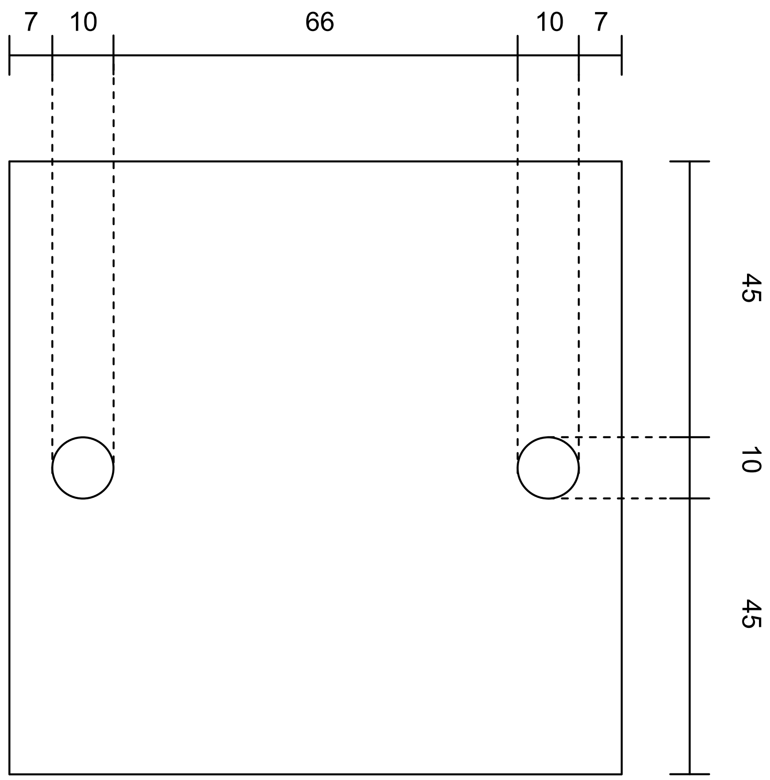
Note: All units are in millimeter (mm)

Figure E.3 Side view of bottom plate



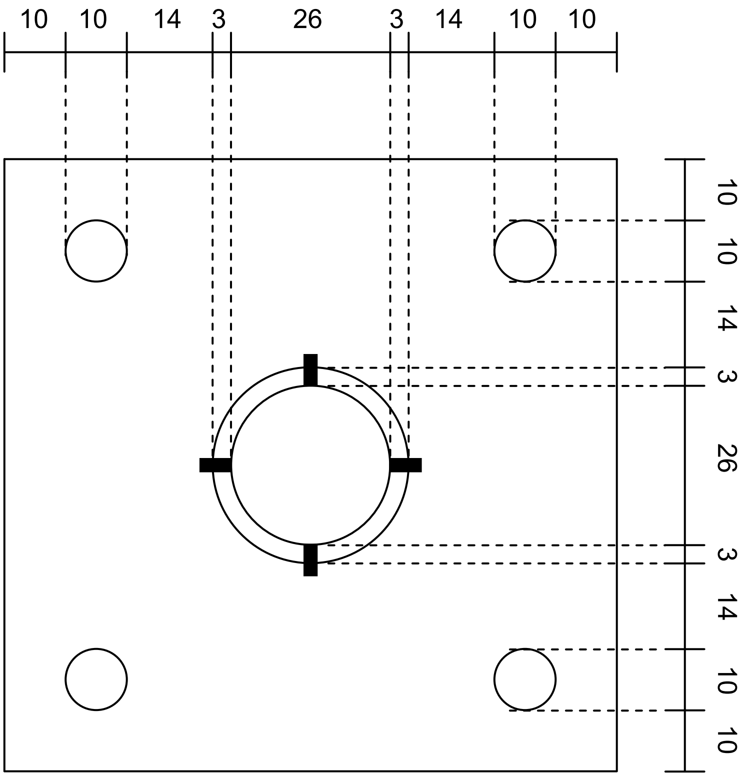
Note: All units are in millimeter (mm)

Figure E.4 Top view of top plate



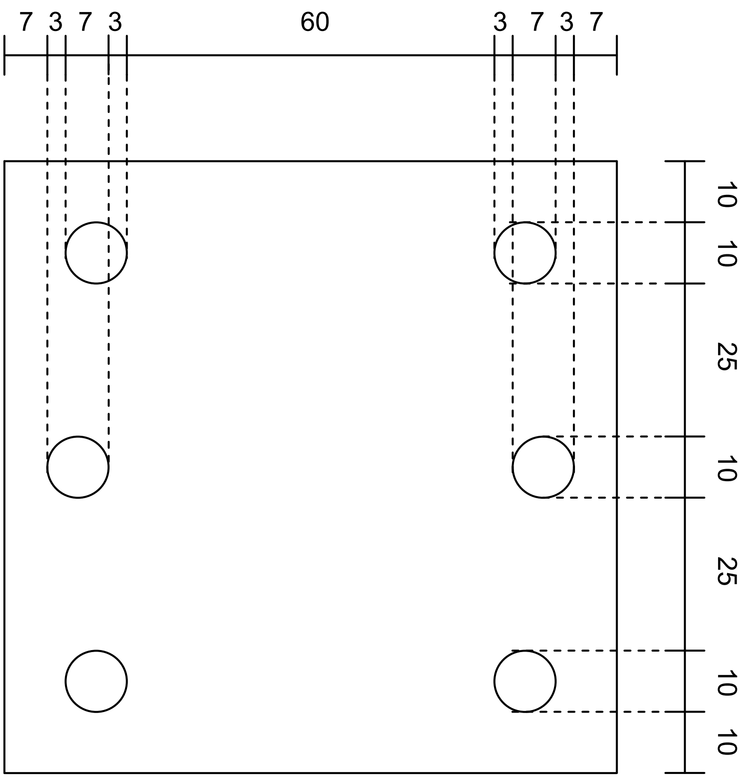
Note: All units are in millimeter (mm)

Figure E.5 Bottom view of top plate



Note: All units are in millimeter (mm)

Figure E.6 Top view of bottom plate



Note: All units are in millimeter (mm)

Figure E.7 Bottom view of bottom plate

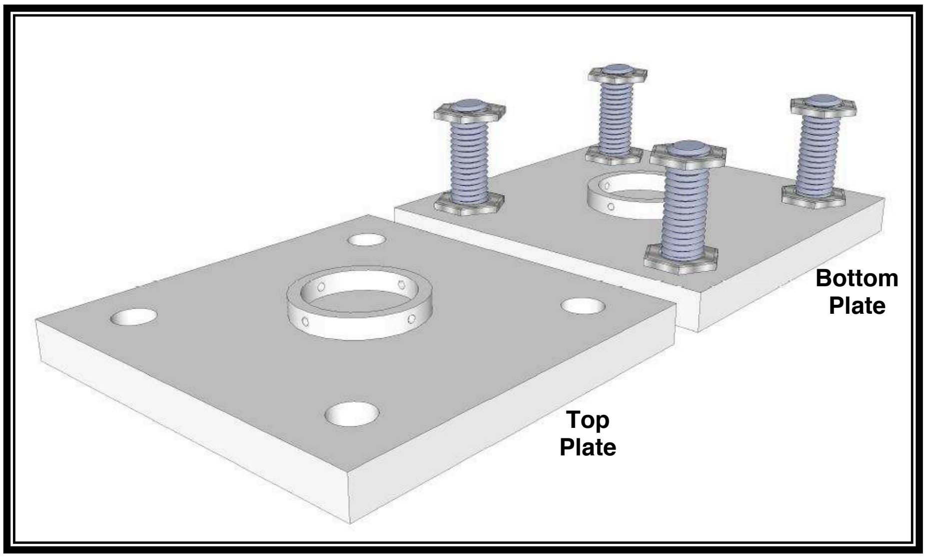


Figure E.8 Improved pair of plates (i.e. Top and bottom plates)

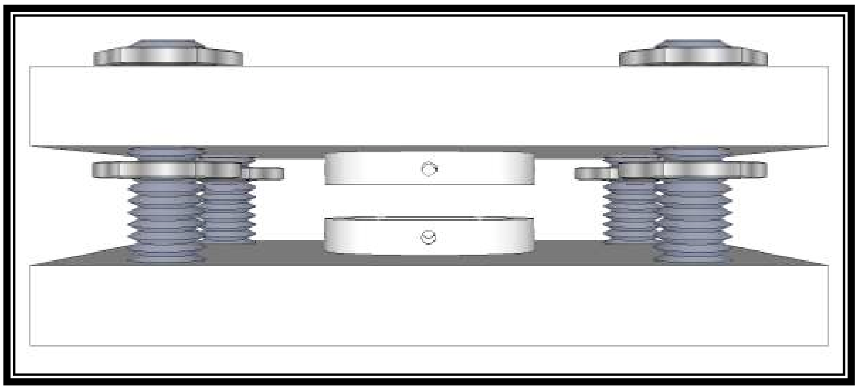


Figure E.9 Improved pair of plates

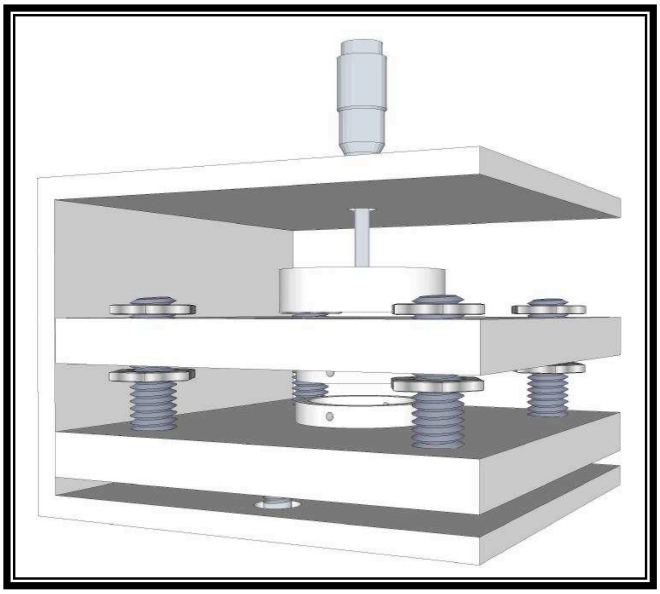


Figure E.10 Pair of plates assembled in compression device