STUDY OF RELATION BETWEEN PIN TOOL GEOMETRY WITH FORMATION OF WORMHOLE IN FSW OF ALUMINUM AA5083

By

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15018

Dissertation is submitted in partial fulfillment of

the requirements for the Bachelor of Engineering (Hons)

(Mechanical)

MAY 2015

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CERTIFICATION OF APPROVAL

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In partial fulfillment of the requirement for
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(MECHANICAL)

Approved by,

____

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Department of Mechanical Engineering
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CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements and that the original work contained have not been undertaken of done by unspecified sources or persons.

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(AMIR HAKIM BIN SANIMAN)

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بسم الله الرحمن الرحيم

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ABSTRACT

Friction Stir Welding has developed as alternative machinery for joining metallic alloys that can be consider as almost impossible to join with conventional techniques. FSW process is employed for joining low melting point metal in several industries such as aerospace, rail, automotive and marine industries. Presence of cavity defect or wormhole is a common occurrence in Friction Stir Welding that been closely linked to weaken the joint mechanical properties. Formation of cavity at stirred area or nugget is a result of poor flow control of plasticize welding material, hence understanding movement of plasticize material is crucial to dictate the problem. Variables such as pin tool geometry, tilting angle and also direction of pin tool rotation are believed to impact the flow of melted workpiece. Nevertheless, frictional contact conditions at the tool–workpiece interface, the resulting microstructure, and the genesis of the cyclic variation in process responses are among the issues that need elucidation from critical experimentations. This project proposes pin tool design to reduce size of wormhole spawn from FSW process. Manipulative variables that were tested were pin tool geometry, and pin tool material. The experiment was conducted at room temperature on controlled test-piece dimension of Aluminum 5083. Parts of mechanical properties of the welded samples and also microstructure formation will be presented to conclude the study.

Keywords: Friction Stir Welding, wormhole size, pin tool geometry, pin tool material, aluminium 5083

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CHAPTER 1: INTRODUCTION

1.1 BACKGROUND STUDY

Friction Stir Welding is a joining technique that offers huge improvement to aerospace, shipbuilding, aircraft and automotive industries. Initially, it was invented by Wayne Thomas in December 1991 at The Welding Institute (TWI). Friction driven mechanism has made FSW operate at lower temperature than fusion welding hence provide joining to 2xxx – 7xxx series alloys which previously considered as unweldable. The FSW products are made of father-father materials which significantly has great mechanical properties. Among all joining technique, FSW has more advantages despite it produce the best properties of welding. However, small defects on work piece that affect mechanical properties might spawn from bad control of FSW parameters.

This project focuses on wormhole or tunnel defect that regularly occurs from poor flow of plasticized work piece. Besides, there are other suspects to contribution of wormhole such as poor heat control, unsuitable pin tool geometry profiles, clamping error, and also inefficient parameters. The formed cavity under the welded surface decreased the joint mechanical properties hence affect joint strength and safety. The project emphasizes on root cause of wormhole and pin tool design to reduce the size of wormhole as well as improved the joint effectiveness.

In general, FSW has been found to produce a low concentration of defects and is very tolerant of variations in parameters and materials.

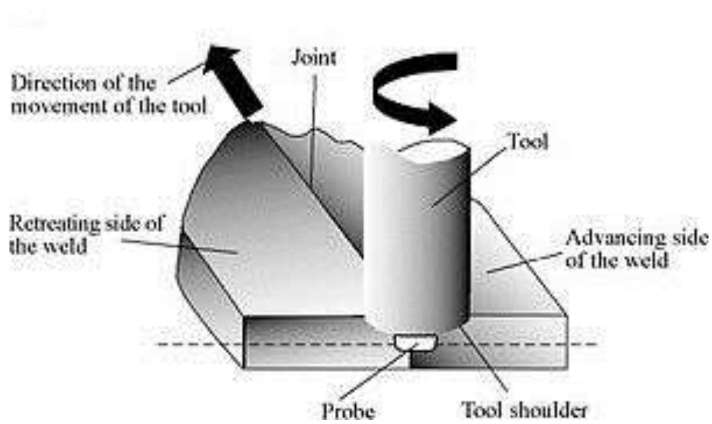


Figure 1 illustration of FSW process

1.2 PROBLEM STATEMENT

Friction stir welding (FSW) is a solid state welding process for joining aluminum and other metallic alloys and has been employed in aerospace, rail, automotive and marine industries. Since FSW joint superiority was better than conventional welding, its application takes us another step forward in structured system. However, FSW tends to produce defect that may affects aesthetic as well as strength in certain applications. One of the most occurs defect is wormhole.

This project aims to investigate relation between pin tool profiles with formation of wormhole. By varying pin tool profile, wormhole size reduction was investigated. As defect free weldment was desired, smaller wormhole size are preferred as it less likely to omit mechanical strength of the weldment, hence bring benefit to quality of metal joining.

Wormhole is voids coagulate along the welding line. Cavity formed between the joint created spaces for stress concentrator hence affects the weldment integrity. In terms of wormhole formation, there are a lot of aspects to look after. Generally, tunnel defect in the weldment occur while the welding process take action. Hence, variables regarding the welding process hugely impact the wormhole formation.

1.3 OBJECTIVE

The main objective of this research are :

i. To reveal relation between pin tool geometry and formation of wormhole.
ii. To identify preferable material between H13 Tool Steel and AISI D2 tool steel in reducing wormhole size in FSW of AA 5083
iii. To investigate relation between wormhole formation and mechanical properties of joint.
iv. To investigate relation between volume displace by the probe with formation of wormhole

1.4 SCOPE OF STUDY

The research on the topic will be based on the stated objectives. The scope of study in the objectives can be simplified as follow:

i. Friction Stir Welding Machine

   In order of preserving material and reducing cost, optimum parameters from previous research is applied in the study with addition of other new manipulative parameter.

ii. Process and procedure of Friction Stir Welding

   As to achieve the best result, detailed review on the welding procedures are made, hence applied in this research.

iii. Wormhole effect

   Effect of wormhole on welded joint is studied to differentiate and improve the impairment cause by the cavity.

iv. Manipulative variables that affect occurrence of wormhole

   Besides FSW machine’s basic parameter; rotation speed, travel speed, and down force; other criterion such as tilting angle, direction of rotation, type of material also impact presence of tunnel defect.

v. Pin tool impact

   Various design of pin tool geometry give different quality of welding and affect the flow of plasticize welding sample. Other than that, pin tool also hugely affect the superiority of the joint.

CHAPTER 2: LITERATURE REVIEW

2.1 FRICTION STIR WELDING (FSW)

FSW is a relatively new joining technique. Basically FSW utilizes frictional heating combined with forging pressure to produce high-strength bonds. Throughout the process, there is no melting condition involves, only solid-state. Frictional welding only use 70% - 90% of material's melting temperature which deals joining to the "unweldable". Among known advantages of FSW are the product has less porosity, less distortion, less shrinkage and also absence of filler metal. There are three main parameters in handling FSW:

- Rotation speed (ω)
- Travel speed (mm/min)
- Down force (F)

Good control of these parameters according to work material could fabricate a perfect joining. Other than that, pin tool suitability also crucial to achieve the best welding quality. In place to identify the way heat is generated and transferred to the joint area. A simplified model is described in the following equation: $Q = \mu\omega FK$

Where the heat (Q) is the result of friction (μ), tool rotation speed (ω) down force (F) and a tool geometry constant (K). The work piece temperature is precised by distributing heat conferring to material's heat capacity; varying the travel speed and rotational speed. The main objective of this project is to identify pin tool geometry that produce small wormhole size formed during FSW, thus knowing the machine's control is crucial.

As to investigate the most efficient parameters to minimize the defect occurrence, a study is conducted by S.K. Chionopoulos. The study used AA5083 aluminum alloy (Al–4.60Mg–0.73Mn–0.12Cr–0.03Fe–0.02Si) with dimensions of 300 mm x 150 mm x 5 mm rolled plates as welding material. Eight different sets of parameters were used to fabricate the joints, while two types of geometries concerning the pin tools were used. This study concluded that only the conical pin geometry

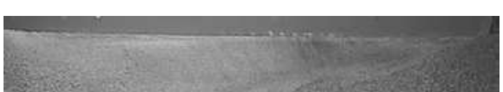


Figure 3 sample of perfect weldment of conical pin tool

Figure 2 sample of perfect weldment of tapered pin tool resulted in defect-free welds at specific welding parameters; 0.158 mm/rev (figure 1) and 0.179 mm/rev (figure 2) with both at 475 RPM (L. Fratini, 2010).

2.2 PIN TOOL

Pin tool is the interface between FSW machine and weld material. It is another core part of a FSW machine; the rotating pin tool that responsible for friction generation and intermixes of weldment. There are three main components in a single pin tool, probe, shoulder and also external features on the probe. According to Shude Ji, he opined how velocity flow of plasticize material being affected by three type of tool profile by using ANSYS FLUENT simulation; conventional tool (cylindrical probe), half-screw pin tool and a tool with a tapered-flute pin. He concluded that, the tool with half-screw pin and the tool with tapered-flute pin both obviously increase material flow velocity near the bottom of the work piece and both are beneficial in avoiding root flaws (Shude Ji, 2012). This shown that probe design is among the factor to control material flow.

Furthermore, a study experiment the rotation direction; clockwise or anti-clockwise effect on welding flow. It was concluded that the joint welded using a stir tool rotated in a counter clockwise direction exhibits better formation of microstructure than the joint welded in a clockwise direction (F.C. Zhang, 2012). Pin tool rotating direction relate with advancing side and retracting side of the weldment which further affect the microstructure arrangement.

Pin tool give many effect on welding quality despite other parameters. Pin tool design has to fit certain criteria in order to assure the success of friction stir welding. As to provide complete joining between two plates, the probe design has to penetrate $80\% - 90\%$ depth of both plates. Meanwhile, the shoulder design contributes to heat generation which require up to 70% of based metal melting temperature. Sufficient heat is needed to plasticize based metal and assure smoothness of pin tool transverse movement. Excessive heat will melt the based metal and formulate defect.

Furthermore, pin tool material selection is crucial based on based metal. Pin tool has to maintain great properties on elevated temperature. Throughout the welding, pin tool material has to remain rigid while colliding with based metal in rotating motion. Penetration from rotating pin tool thru sample will generate heat that dissipates at the welding line. Hence, it is important for pin tool material to be strong and hard throughout the process at any given temperature.

Other than that, external features on the probe are one the main factor that dictates the flow of the weldment. Different pin tool designs give different welding quality. As the probe penetrate into the work piece, it regulate stirring motion between both surface that cause the based metal to become rubber-like state by frictional heating. Both surface then intermix behind the pin tool as it moves transversely. Probe design affect the flow path of the weldment which poor flow control will result in defects such as tunnel defect.

2.3 WORMHOLE

Wormhole or tunnel defect is a cavity formed under welded surface where undetectable by naked eyes. The primary cause of wormhole is abnormal flow of plasticize material during welding. Due to the reduced joint area between the work pieces, wormholes severely weaken the mechanical properties of the weld bond (G. Huang, 2000). In Friction Stir weld cross-section, there are three main zones; the nugget (stirred zone), thermal mechanically affected zone (TMAZ), heat affected zone (HAZ). These three zones possess distinct mechanical properties accordingly while nugget and TMAZ is the weakest part of the joint (M. R. Uday, 2010).

The nugget is the region through which the tool piece pin passes, and thus experiences high deformation and high heat. It generally consists of fine equiaxed grains due to full recrystallisation. The TMAZ adjacent to the nugget is the region where the metal is plastically deformed as well as heated, but this is not sufficient to cause recrystallisation. The HAZ experiences only a heating effect, with no mechanical deformation (S. Rajakumar, 2011). The region above and under the nugget may represent the zone where tunnel defect might occur. Tunnelling defect occurs when the material flow around the pin tool is not adequate, result in irregular weld filling, one of the most critical types of the FSW defect is the tunnelling (S. Balos, 2013).

The effect of tunnelling defects on the joint strength efficiency is studied by L. Sidjinin. This study objective is to conclude that either the wormhole affect the mechanical properties of friction stir weld. Based from the findings, the tunnel obtained influenced the joint mechanical properties. In one of his sample, a combination of tunnel and a crack-shaped tunnel was found, hence proved to give more unfavourable effect on the mechanical properties. Sidjinin also conclude that the pin-to-concave-shoulder-volume ratio greatly influences the occurrence of the tunnelling defect (R. Nandan, 2008).

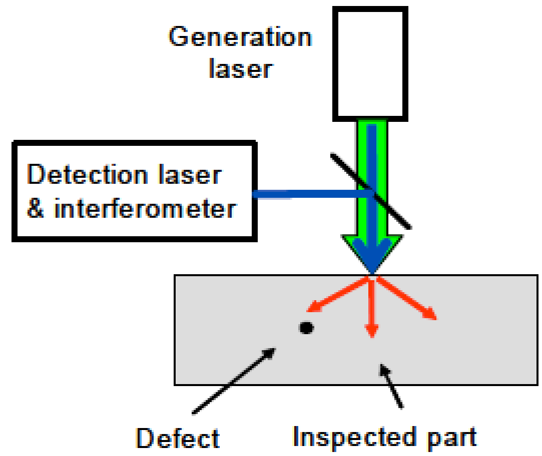


Figure 4 X-ray set up for weldment defect inspection

Wormhole occurs under the welding surface, hence undetectable by visual inspection. Based from previous research, wormholes can be identified by using x-ray, radiography, laser-ultrasonic technique and also infrared thermography. Other than that, destructive testing of sample give cross sectional of weldment will provide visual on wormhole. The microstructure and grain formation usually tested by the scanning electron microscope (SEM) equipment or optical microscopy (OM).

2.4 ALUMINUM 5083

Initially FSW was designed to encounter joining of Aluminum. Although, aluminium has advantage on its light weight, it could not form joint by fusion welding. Its’ low melting temperature (660.32℃) and boiling point (2470℃) do not suit fusion welding. However, FSW along with friction powered mechanism offers rapid and high quality weld to 2xxx – 7xxx series alloys who previously considered as unweldable. These aluminum alloys are generally classified as non-weldable because of the poor solidification microstructure and porosity in the fusion zone. Also, the loss in mechanical properties as compared to the base material is very significant. These factors make the joining of these alloys by conventional welding processes unattractive. Some aluminium alloys can be resistance welded, but the surface preparation is expensive, with surface oxide being a major problem (L. G. Zhang, 2012).

FSW offers improvement in many fields such as naval, aerospace, and aircraft with its’ innovation on dead-weight loading. By decreasing dead-weight loading value, it leads to less energy consumption while increasing load capacity. Besides, aluminium natural properties of highly corrosive resistance made its worthy for exploration. Further study might improve application of aluminium other properties such as excellent conductor of heat and electricity. Other than that, this project choose aluminium as main material as it easily saved resource because it can be recycled, and thus can be expected to be an environmentally friendly metallic material (S.K. Chionopoulos, 2008).

Among other aluminium series, 5xxx series are known as one of the strongest series. Shipbuilding, car body parts, pressure vessel, and aerospace are among the field most contributed by 5xxx series. This group of aluminium well suit with corrosive environment such as seawater and industrial chemical.

CHAPTER 3 : METHODOLOGY

3.1 PROJECT FLOWCHART

In the method in carrying this project, there are several steps to achieve the desired result.

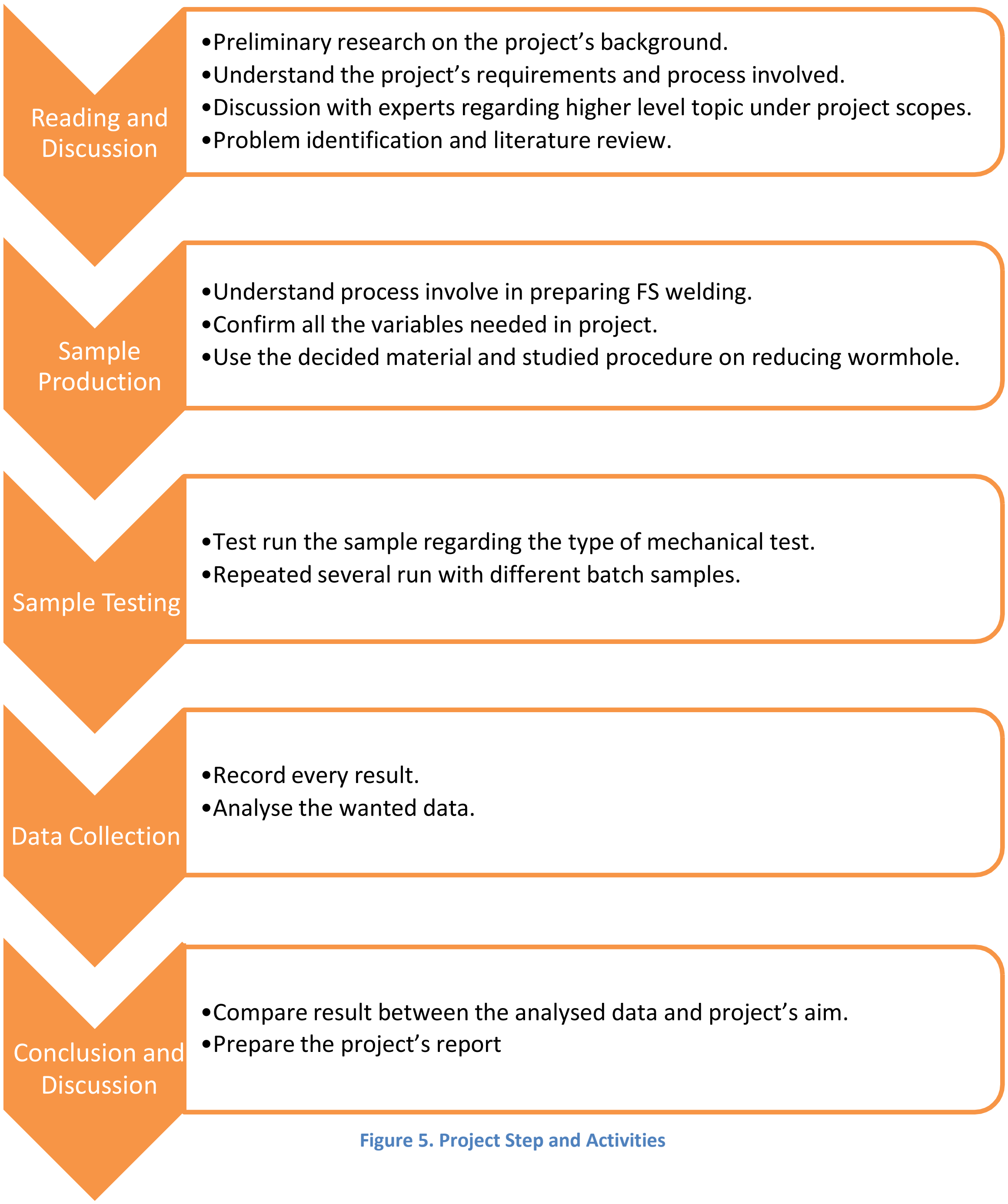


Figure 5. Project Step and Activities

3.2 PROJECT OVERVIEW

First of all, final year project was divided into two separate parts, FYP I and FYP II. Initially in FYP I, information were gathered by reviewing previous research, journal paper, technical handbook and other FSW related essays. Literature reviews was done by referring multiple available sources. In order to complete progress report, full procedure on conducting the research was crucial with detailed information. Besides, meeting with associated technicians and experienced student was arranged to share and collect more detailed data.

Final year project II (FYP II) requires execution of process in order to achieve objective. Based from provided methodology, investigation work was conducted. FYP II is the final part of the project where at the end of the period, full report of experiment was expected. The activities involved in FYP II for this project were pin tool fabrication, pin tool heat treatment, FSW of sample, and mechanical testing of joint. Most of the process requires approval and booking from respective technician before appointed machines and lab could be use. Any application on booking must be approved earlier by FYP supervisor.

3.3 PROJECT PHASE

3.3.1 MATERIAL SELECTION

There are a few materials that need to be supply in order to achieve the objective. Material selection defines the welding success and quality. Proper material selection is crucial to avoid process failure and loss. Currently the feasibility for FSW is limited. The project only uses CNC milling machine to imitate the FSW machine mechanism, hence the parameters set up was bounded to the milling machine. Since the milling machine was not equipped with default pin tool, it has to be fabricated. Another substantial that has to take into account was the based metal used for producing sample.

As a total, there are two parts that have to be prepared, two materials for pin tool and based metal. The main factors for choosing these materials are the strength and the hardness of material. Pin tool material has to be stronger and harder than based material and also endure great properties in elevated temperature. While the welding take place, the pin tool will penetrate the based metal in stirring motion. Friction mechanism will generate heat until based metal is plasticize and intermix in circular motion. Hence, it is important for the pin tool to have high wear resistance and remain rigid throughout the process.

Material selected as pin tool is Chromium Hot Work Tool Steel H13 (H13). Chromium hot-work tool steels are classified as group H steels by the AISI classification system. This series of steels start from H1 to H19.H13 was one of the commonly used metals because it has good properties as a tool. High hardenability, excellent wear resistance and hot toughness were among the properties H13 comprised. H13 usually used in hot work process as it has good thermal shock resistance, drastic temperature change barely affect H13 structure.

As H13 content, molybdenum and vanadium act as strengthening agents while chromium content aid the alloy to resist softening at high temperature. Aside from its magnificent properties, H13 have a high marketability around research area. Hence, H13 was selected as pin tool material.

Table 1 H13 Tool Steel Material Composition.

Element	Content (%)
Carbon , C	1.40 – 1.60
Manganese , Mn	0.60
Silicon , Si	0.60
Cobalt , Co	1.00
Chromium , Cr	11.00 – 13.00
Molybdenum , Mo	0.70 – 1.20
Vanadium , V	1.10
Phosphorus , P	0.03
Nickel , Ni	0.30
Copper , Cu	0.25
Sulphur , S	0.03

Another material chosen as pin tool material is AISI D2 cold-work tool steel. This type of steel has high carbon and high chromium content which classified in the group D steels. Group D steels have 1.50% – 2.35% of carbon and 12% of chromium, except for D3. One of the known criteria of group D steels are they have high wear resistance as a tool. Although group D steel are brittle, D2 have excellent hardness value. D2 tool steel was chosen as it suits the properties of pin tool, the most commonly used steel and also available nearby.

Aluminium 5083 was chosen as the based metal for friction stir welding. Initially FSW was developed for non-ferrous metal. Nonferrous alloys often light, strong and have good corrosion resistance, although they did not suit conventional welding and require costly preparation for the process. FSW which operates at $70\% - 90\%$ of based metal melting point offers joining to nonferrous metal.

Table 2 D2 Tool Steel composition

Element	Content (%)
Chromium , Cr	4.75 – 5.50
Molybdenum , Mo	1.10 – 1.75
Silicon , Si	0.80 – 1.20
Vanadium , V	0.80 – 1.20
Carbon , C	0.32 – 0.45
Nickel , Ni	0.30
Copper , Cu	0.25
Manganese , Mn	0.20 – 0.50
Phosphorus , P	0.03
Sulfur , S	0.03

Aluminium was known for its light weight, remarkable for its low density and also resistance towards corrosion as results of passivation. Aluminium was applicable almost in all fields while FSW improve its feasibility of joining. By using friction mechanism, nonferrous metal can be easily welded hence creating a new branch of structure engineering.

Among all aluminium series, Aluminium 5083 was considered as one of high strength series. 5083 grade was widely used in shipbuilding, pressure vessel, vehicle bodies and others. Aluminium 5083 stands firm in extreme environment with high resistant to seawater and also industrial chemical attack. Moreover, aluminium 5083 has excellent marketability around research area. Hence, studying aluminium 5083 wormhole formation will improve its effectivity on application.

Table 3 Aluminum AA 5083 composition

Element	Content (%)
Silicon , Si	0.40
Iron , Fe	0.40
Copper , Cu	0.10
Manganese , Mn	0.40 – 1.00
Magnesium , Mg	4.00 – 4.90
Zinc , Zn	0.25
Titanium , Ti	0.15
Chromium , Cr	0.05 – 0.25
Aluminum , Al	Balance

Based from supplier information, mechanical properties of H13 and AA 5083 were compared above. H13 was 4 times stronger and 3 times harder than AA 5083 material. Hence for friction stir welding of AA 5083, H13 suit the need as pin tool material.

Table 4 tensile strength and Vickers hardness comparison between materials

Properties	Material		
	H13 tool steel	D2 tool steel	AA 5083
Tensile strength (MPa)	1200	1650	300
Vickers hardness (HV)	230	748	70

Figure 5 H13 tool steel raw material

Figure 7 D2 tool steel raw material

3.3.2 PIN TOOL FABRICATION

In this investigation, two pin tool geometries are going to be tested. They are tapered pin tool (TP) and half-cone pin tool (HC). Both of the pin tools acquire same basic dimensions. The only difference they had are the probe geometry and also volume displace by the probe.

There are 2 main parts in pin tool design, the shoulder and the probe. Another part is the cylinder shape after the shoulder that used to socket the pin tool to the FSW machine. All other dimension is kept constant except the probe. For shoulder design, the diameter was fixed to 30 mm. The penetration depth was set based on the based material thickness. The thickness of AA 5083 plate that was used as based metal was $10\,mm$. Penetration of the probe only require $80\% - 90\%$ of the sample thickness. For this experiment, it was set to 85% penetration; hence the probe length was 8.5 mm for both geometries.

Difference in volume displace by the probe for both pin tool geometries was recorded. As for tapered pin tool, volume displace by the probe was calculated by using the following equation:

$$V = \frac{h\pi(R^2 + r^2 + Rr)}{3}$$

$$V = \frac{8.5\pi\left(8^2 + 7^2 + 8(7)\right)}{3}$$

$$V = 1504.30\ mm^3$$

Meanwhile volume displace by half-cone probe was calculated by using the following equation:

$$V = (\pi r^2 H) + (\pi r^2 \frac{h}{3})$$

$$V = \left(8^2\pi(4)\right) + \left(8^2\pi \frac{4.5}{3}\right)$$

$$V = 1105.84\ mm^3$$

Based on calculation, tapered probe has more volume than half-cone probe. Tapered pin tool covered 36% more volume than half-cone pin tool. Depth of penetration for both pin tool are the same, however tapered pin tool displace the based material more than half-cone pin tool. Relation between volume displace by the probe and wormhole formation was investigated.

Tapered pin tool can be considered as conventional pin tool that are commonly use. Meanwhile half-cone pin tool is rarely used and based from previous researches; pointed pin tool tested on FSW was only 5 percent of all pin tool geometries. Therefore, this experiment will compare both geometries efficiency for FSW of AA5083.

Each experimental pin tool were fabricated from raw H13 round bar with diameter of $30\,mm$ with length of $100\,mm$. By using CNC Lathe Machine, numerical coding was keyed in by respective technician based on geometry design. All the process was done by computer controlled data.

Figure 6 tapered pin tool made from H13 Tool Steel

Figure 9 half-cone pin tool made from AISI D2 Tool Steel

3.3.3 HEAT TREATMENT

As to increase FSW success rate, fabricated pin tool was treated with annealing to remove internal stress and further strengthen it. Annealing alter the samples microstructure by heating causing change in properties such as strength, hardness and ductility. Process temperature of annealing depends on the sample's glass transition temperature, T_g. Divided into three parts; annealing consist of preheat, maintaining suitable temperature above glass transition temperature, and cooling down.

Annealing of fabricated pin tool from H13 material utilized 850℃ temperature for 6 hours. increase, there are three reactions that occur on material. Firstly recovery, where it Glass transition temperature, T_g is always below the melting temperature, T_m. As the temperature soften the metal and remove primary linear defects (dislocation) and also internal stresses cause by them. Recrystallization occurs afterwards where new strain-free grains nucleate and grow to replace internal stresses. Once the crystallization completed and the annealing still going, grain growth will then occur.

Heat treatment for both pin tool was done by using Carbolite CWF 1100 DegC Laboratory Chamber Furnace. The furnace was located at heat treatment lab, block 17, UTP. Procedure for the heat treatment of H13 was mentioned as follows:

- Each samples were cleaned from impurities
- Samples were placed steadily inside the furnace. After that, the furnace was closed.
- The heater was set to increase temperature by $107\ ℃/15\ mins$ as preheat temperature.
- After two hours, the temperature was kept constant at 850℃ for 6 hours.
- After that, samples were let to cool to room temperature in the furnace.

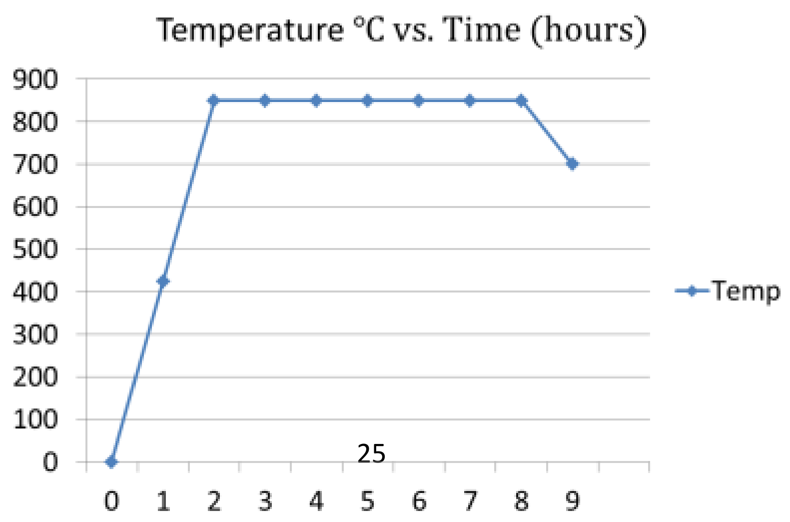


Figure 10 graph of temperature versus time for heat tratment of H13 Tool Steel

Heat treatment for both pin tool were done separately as it relate to different material, hence different temperature was applied. Heat treatment for pin tool made from AISI D2 Tool Steel was done as follows:

- Each samples were cleaned from impurities
- Samples were placed steadily inside the furnace. After that, the furnace was closed.
- The heater was set to heat at 400°C for 2 hours as preheat temperature.
- After two hours, the temperature was kept constant at 820°C for 6 hours.
- After that, samples were let to cool to room temperature in the furnace.

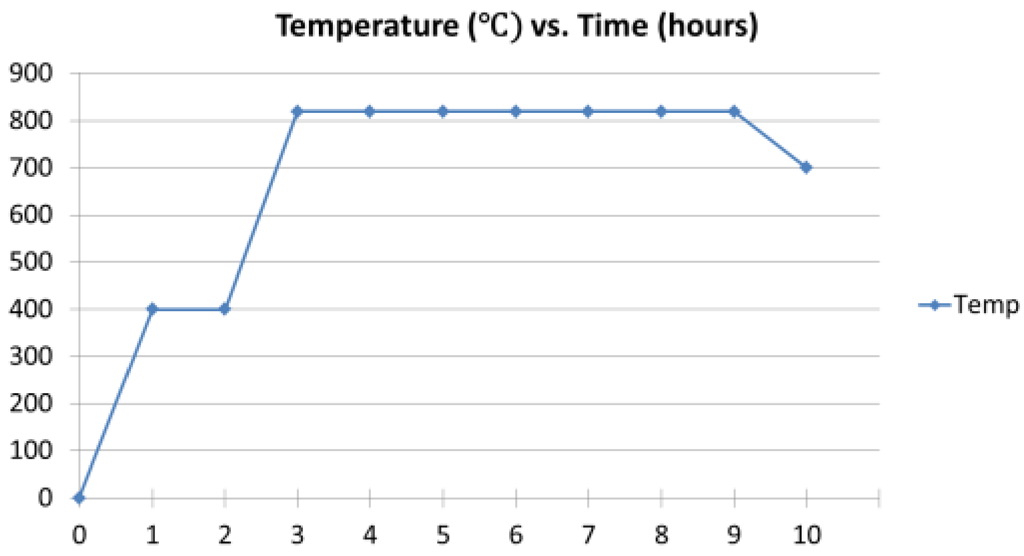


Figure 11 graph of temperature versus time for heat treatment of AISI D2 Tool Steel

Mechanical properties were altered by relieving internal stress and refine the microstructure creating homogenous region. As a result, pin tool properties were strengthen and harden than the pin tool before heat treatment.

Figure 7 pin tool made from AISI D2 Tool Steel after heat teratment

3.3.4 SAMPLE PRODUCTION

After the heat treatment, following schedule was producing joint sample from fabricated pin tool. Until recent April 2015, UTP has not acquired a Friction Stir Welding machine. Hence previously, all research on FSW was conducted by using CNC Milling Bridgeport. By that means, feasibility of the project depends on available machine and apparatus.

Before the welding take place, all tools and materials were inspected to follow the parameter set up. For each run, two AA 5083 plate with $100\,mm \times 100\,mm \times 10\,mm$ dimension will be joined together as butt joint by using experimental pin tool. Each tool (TP / HC) were used to produce three samples. After that, the joint structure and wormhole formation was analysed.

Initially, two AA 5083 plates were clamped and secured in a jig as butt joint arrangement. The jig functioned to hold the plates in place while the welding takes place. Each sides and top of the plates were clamped with tight in screw. It was crucial for the plates to remain rigid throughout the process as any vibration or movement from the plates will affect welding quality. Defect may occur as a result of clamping error. After the sample plates were secured, FSW was conducted by using experimental pin tool and selected parameters.

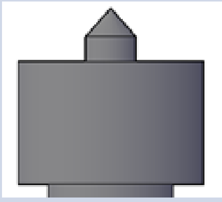
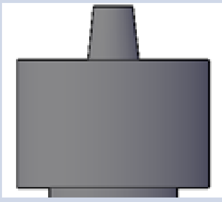
All other variables were decided based from previous research of successful FSW butt joint with thickness of $10\,mm$ with prior to AA 5083. Throughout the investigation, other specifications were kept constant which the only things that vary were pin tool geometry and volume displace by the probe. Parameters of Friction Stir Welding of Aluminum AA 5083 were set as mentioned in table below.

Table 5 parameter set up for FSW process

Parameter		Remark
Rotational speed	(rpm)	1600
Travel speed	$(^{mm}/_{min})$	15
Plunging speed	$(^{mm}/_{min})$	10
Dwell period	(s)	10
Tilting angle	(°)	0
Penetration depth	(mm)	-8.5

Manipulative controls for this experiment were the pin tool design and also pin tool material. While the different in pin tool design manipulate volume displace by the probe, pin tool with tapered design has more volume displaced rather than half-cone. The experiment were divided in to four independent samples.

Table 6 sample's pin tool detail

Sample No.	Probe Geometry	Material	Volume Displace (mm^2)
1		AISI D2 Tool Steel	1105.84
2		H13 Tool Steel	
3		AISI D2 Tool Steel	1504.30
4		H13 Tool Steel	

Each four samples were fabricated by using same FSW machine parameters as mention previously. After completion of all four samples, the results were then analysed.

Figure 8 CNC Bridgeport Milling Machine used to imitate FSW Machine

3.3.5 TESTING PROCESS

There were two testing procedure that have been conducted on the welding samples. They are Vickers hardness (HV), and optical microscopy (OM). Initially, the FSW samples were cross cut at $20mm$ from starting of weldment to reveal wormhole formation visually. Sizes of wormhole formed were compared from smallest to largest. Based on wormhole size order, pin tool material and probe design efficiency for this experiment was decided.

Other than that, mechanical properties of joint fabricated were tested with Vickers Hardness. Vickers hardness measures sample hardness by indentation principle which test the samples resistance to deformation due to constant compression load. The hardness values of the sample are then calculated from dimension of indentation left by the load. Hardness value measure the strength of the bond formed between microstructure where larger HV value indicates finer particle size. However, below critical grain-size, hardness decreases with decreasing grain size.

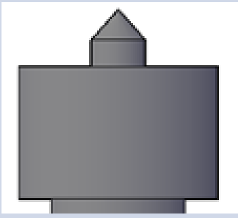
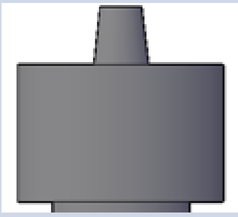
Besides that, microstructure formation of the samples was revealed by using optical microscopy (OM) magnification. Initially, samples were etched by using Krool’s Reagent. Samples surface were swabbed with etching solution for 30 seconds. OM utilizes light and magnifying lenses to magnify images of small sample. By using normal light, image were capture by using light sensitive camera. Micrograph was recorded and analysed.

CHAPTER 4 : RESULT AND DISCUSSION

4.1 FRICTION STIR WELDING RESULTS

In this study, four samples were produced in order to investigate relation between pin tool geometry with formation of wormhole. There are two pin tool geometries that were being tested. Each pin tool design was fabricated from two different materials. Hence, two samples were obtained from each geometry. Samples pin tool used for Friction Stir Welding of Aluminum AA5083 were shown below:

Table 7 sample's pin tool detail

Sample No.	Probe Geometry	Material	Volume Displace (mm^2)
1		AISI D2 Tool Steel	1105.84
2		H13 Tool Steel	
3		AISI D2 Tool Steel	1504.30
4		H13 Tool Steel	

In order to extract more knowledge, literature and understanding of FSW of Aluminum AA5083, the samples were analysed from two different variables. The samples are group into two investigations as follows:

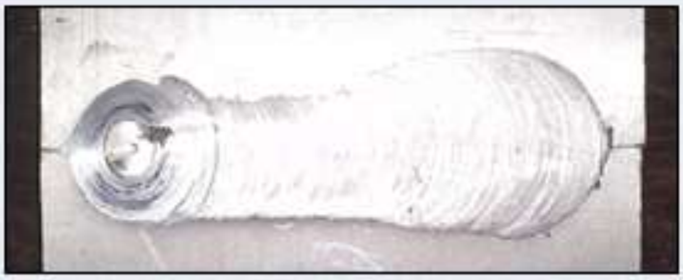
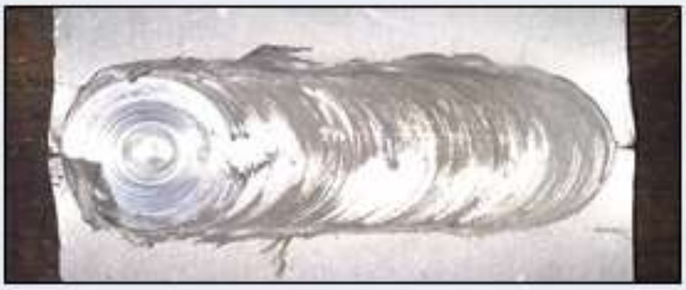
Table 8 samples set comparison based on variables

Variables	Samples Set	Type of investigation
Pin Tool Geometry	(1 & 2) vs (3 & 4)	To study effect of pin tool geometry on formation of wormhole in FSW of Aluminum AA 5083
Pin tool Material	(1 & 3) vs (2 & 4)	To investigate suitable pin tool material associated with FSW of Aluminum AA5083 in reducing wormhole formation

The results were analysed and compared based on formation of wormhole, hardness of joint and microstructure.

Results of FSW of Aluminum AA5083 were shown as follows.

Table 9 FSW results for all four samples

Samples No.	FSW Result
1	
2	
3	
4	

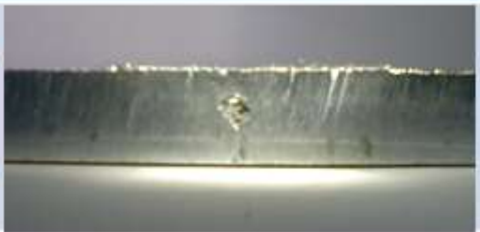
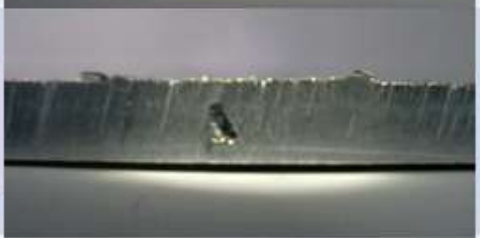
As observed, surface of all four weldment were not favourable as predicted from welding samples of aluminium from other series. Meanwhile other samples had waves defect and irregular welding surface. This was the result of excess heat generation which cause the based metal to melt and spilled outside relative to the stirring motion. Excess heat generation was due to large shoulder diameter of pin tool. As a conclusion, shoulder diameter of 30 mm was too big and caused excessive heat for FSW of AA 5083 with 10 mm thickness.

Only sample 2 has almost constant welding surface. Sample 2 has regular surface formation because the heat input was sufficient and the shoulder barely touch the plates while the welding take place, hence lower heat amount was generated. There was a clamping error while running sample 2 causing the based metal not align with the pin tool route. Hence, the penetration of pin tool was not constant.

4.2 WORMHOLE FORMATION

Each sample was then cut perpendicularly from welding line to view tunnel defect spawning. Samples were cut $40\,mm$ inside from the pin tool entrance. Image of wormhole spawn was as mention below

Table 10 wormhole formation for all four samples

Samples No.	Wormhole Formation		
1	RS		AS
2	RS		AS
3	RS		AS
4	RS		AS

Based on visual observation, all four samples produce wormhole at welding line approximately $40\,mm$ from pin tool penetration point. Out of all samples, there were no obvious different between advancing side (AS) weldment and retreating side (RS) weldment. For all four samples, wormholes spawn in the middle of nugget area where region above and below wormhole was joined completely.

However, there were apparent different in the size of wormholes formulate. Sample that produces the smallest wormhole was sample 2. Meanwhile, sample 3 produced largest cross sectional size of wormhole.

4.3 EFFECT ON PIN TOOL PROFILE

Pin tools with tapered geometry were compared between material they were fabricated. It was observe that pin tool made from H13 Tool Steel have more wear percentage then the pin tool made from AISI D2 Tool Steel. This was because AISI D2 Tool Steel perform better performance at elevated temperature with stronger and harder properties then H13 Tool Steel.

Figure 94 tapered pin tool, surface wear comparison

However, for half-cone pin tool, it was observed that H13 Tool Steel has no wear on its shoulder while AISI D2 tool steel has worn out shoulder surface. This was the result of clamping error of sample 2 where the pin tool penetration was not constant. Hence, the work piece barely touches the pin tool shoulder. On the pin tool probe, H13 tool steel was observe to have more wear percentage than AISI D2 tool steel.

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Figure 105 hal-cone pin tool, surface wear comparison

4.4 HARDNESS TESTING RESULTS

The samples were cut 40 mm from the pin tool entrance. Micro-hardness reading was taken at 5 different locations for each sample. The locations were at the weld nugget (0 mm from welding line), 2 points at both advancing and retreating side (4 mm and 8 mm from welding line). The results was tabulate below.

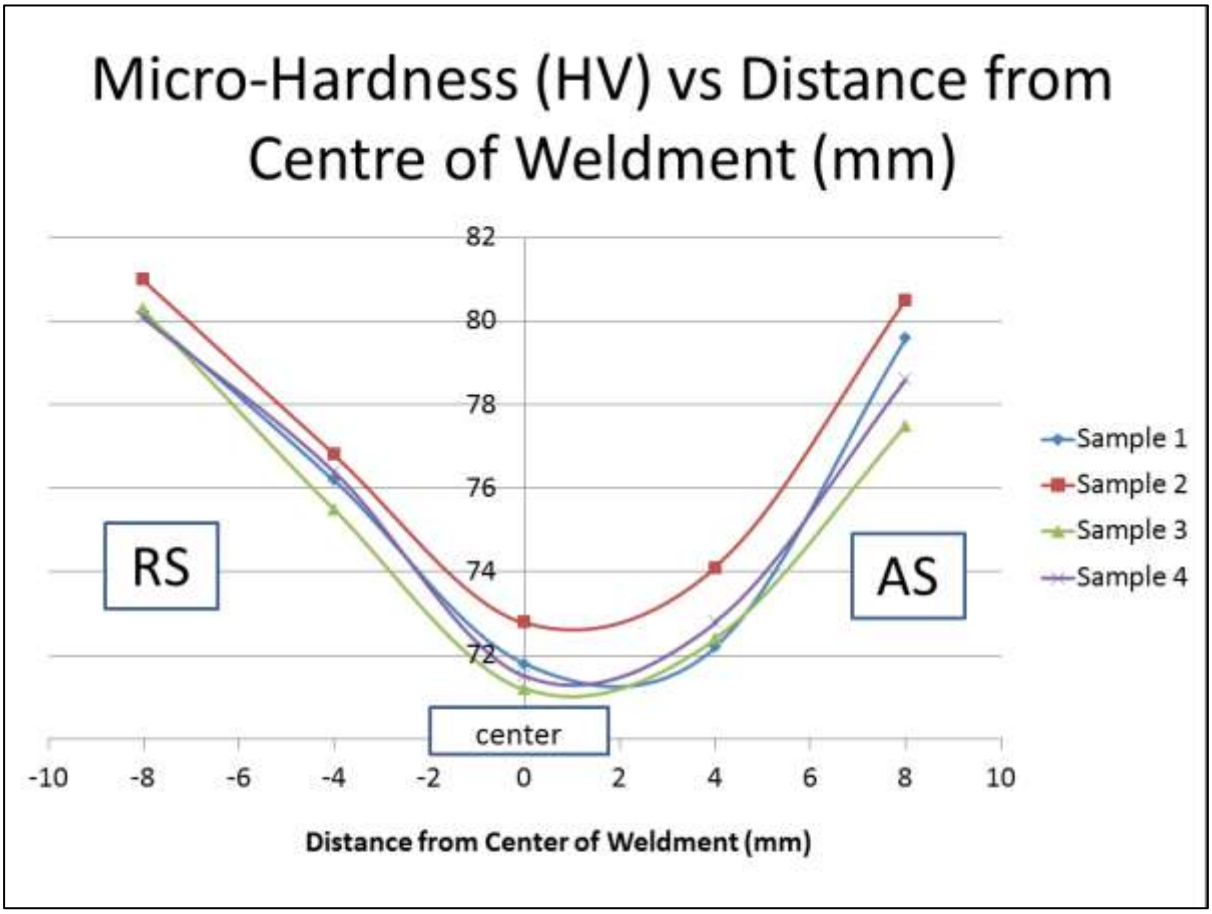

Figure 116 graph of micro-hardness versus distance from center of weldment for all samples

4.5 MICROGRAPH ANALYSIS

Image of the weld nugget for each samples were view in micro scale; $200\mu m$, $100\mu m$, $20\mu m$, and $10\mu m$ respectively. Based on the scales, the weld nugget of each sample was compared.

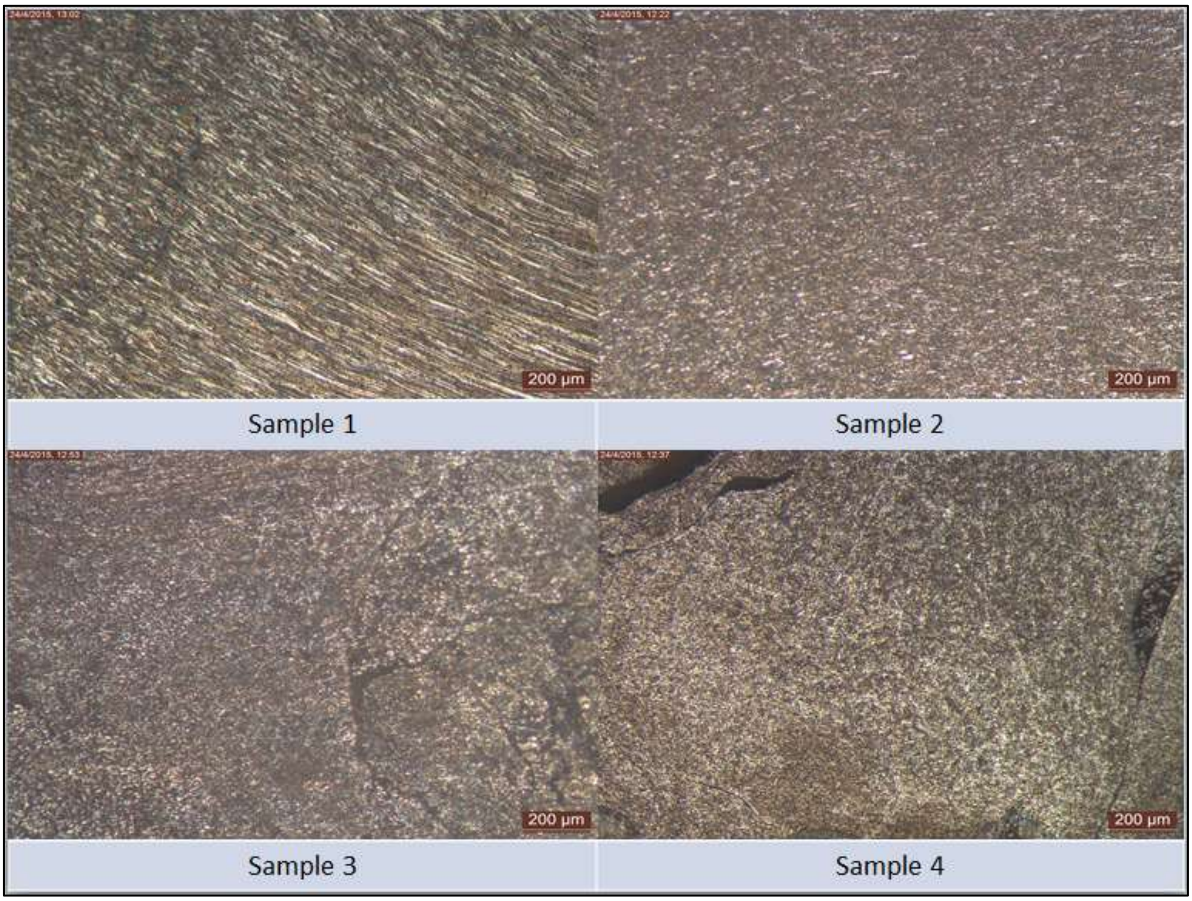

Figure 13 Sample micrograph comparison at $200\mu m$

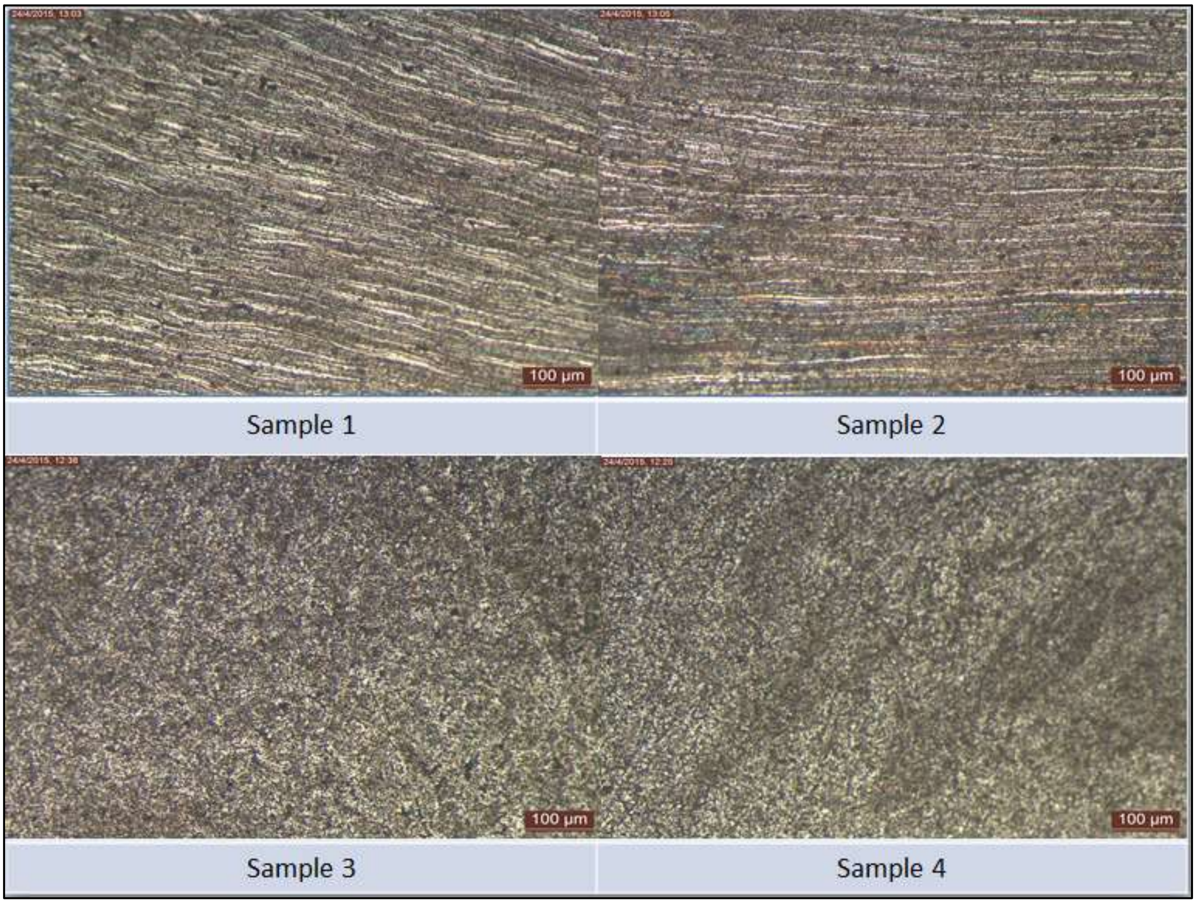

Figure 128 sample comparison at 100µm

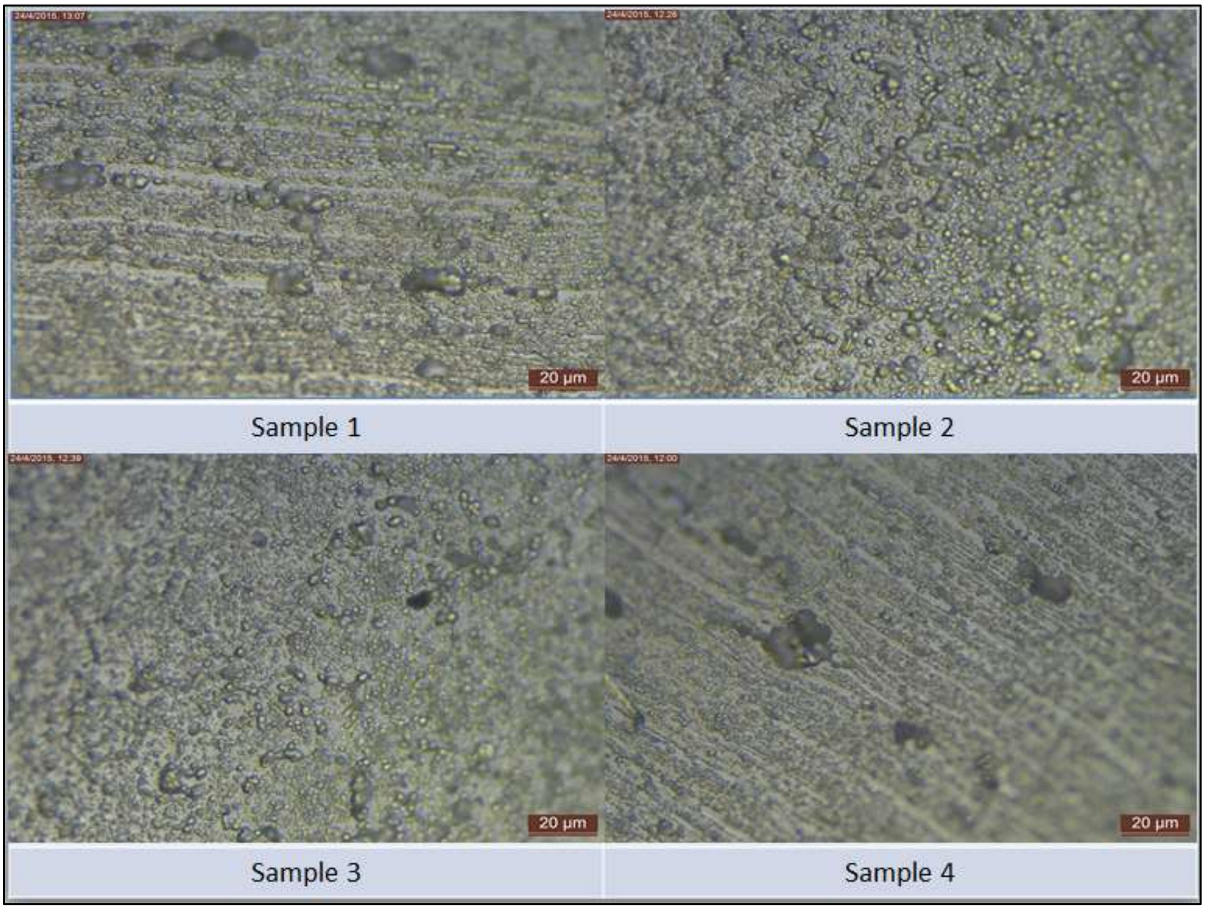

Figure 15 sample comparison at 20µm

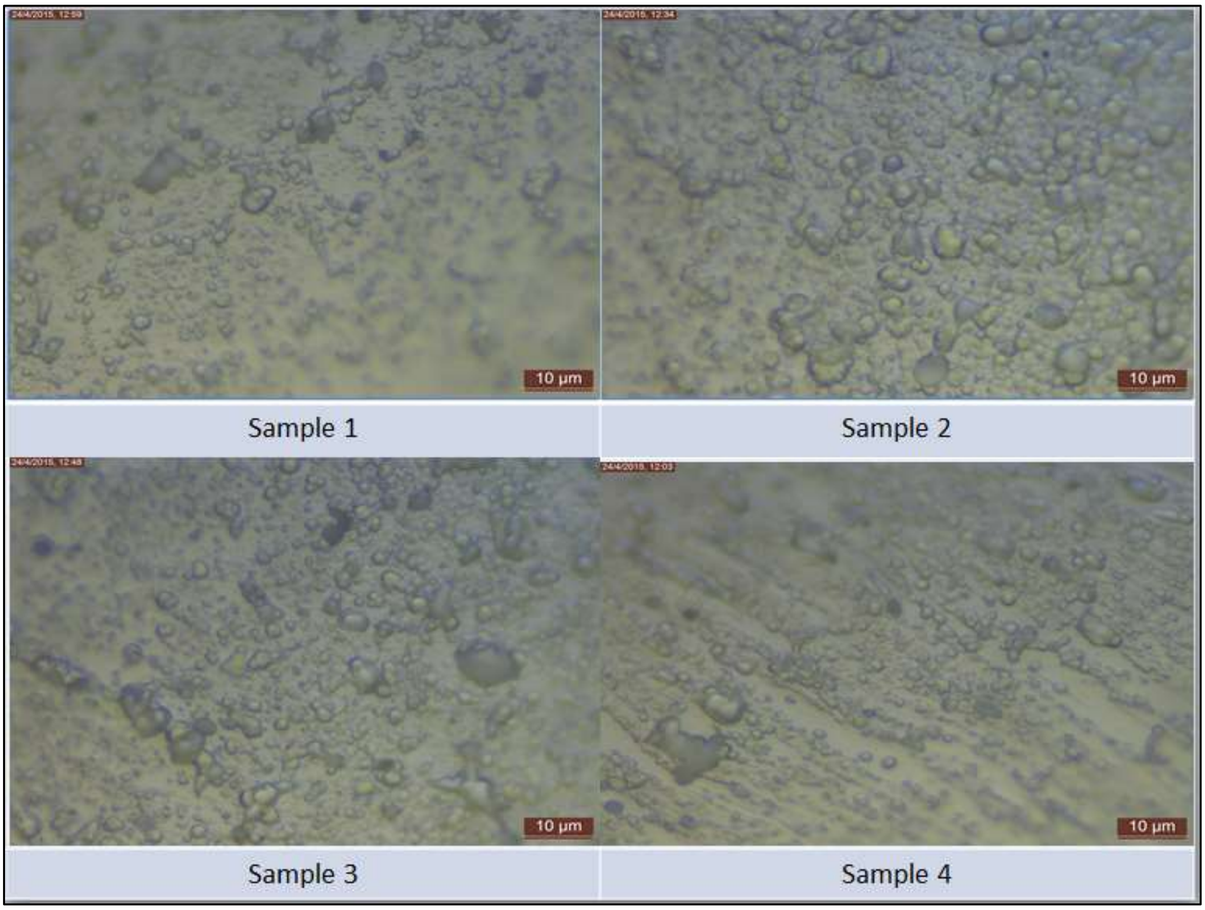

Figure 14 sample comparison at 10µm

CHAPTER 5: CONCLUSION AND RECOMMENDATION

5.1 ACHIEVED OBJECTIVES

The main objective of this study is to identify pin tool geometry relation with formation of wormhole in FSW of Aluminum AA5083. Based on 4 samples produced, the objective was achieved. Wormhole formation from each sample was reviewed and it was decided that half-cone pin tool produce less wormhole size compared to tapered geometry. Hence, for FSW of AA5083, it is recommended to utilize half-cone with a pointed probe as pin tool external features.

For each samples, there were five separate location of micro-hardness test conducted. It was observed that area around weld nugget has the lowest hardness among the sets, while area of retreating side has higher hardness than the advancing side. As the pin tool travel in stirring motion either clockwise or anti-clockwise direction, the retreating side commonly has high hardness than advancing side because of the feed of the motion. Plasticize metal at retreating side will be compacted while the pin tool travel in transverse direction hence improving microstructure properties. Area of weld nugget was the least hard because it was the most heat affected area among others.

Based on optical microscopy observation on the joint, where intermixes occur, all four samples shown bubble-like void at magnification of $10\ \mu m$. However, the void shown by sample 1 and 2 were smaller than sample 3 and 4. The bubble-like voids formed by trapped air during the FSW process. The air bubble was the result of excessive vibration of pin tool during the process, this was probably because of the CNC Milling machine was not rigid enough to hold the pin tool while carry out the process. Hence, conducting FSW by using a real FSW machine was recommended.

Furthermore, this study applied two different material used as pin tool. It was observe that pin tool made from H13 tool steel produce smaller wormhole than pin tool made from AISI D2 tool steel. Other than that, AISI D2 tool steel pin tool shows higher wear percentage on than H13 tool steel pin tool. From these two results it was recommended that H13 tool steel was more suitable for FSW of Aluminum AA5083.

Observation on the weld surface shows wave defect at the side of the weldment surface. This was the result of too much heat generation, where the based metal liquefied and splash outside as the pin tool stirring. Excessive heat was produce by the shoulder where in this case, pin tool shoulder was $30\ mm$ in diameter. Hence it was decided that $30\ mm$ shoulder diameter was too big for FSW of Aluminum AA5083 plates with $10\ mm$ thickness.

5.2 RECOMMENDATION

In order to understand pin tool geometry effect on flow of plasticize metal, it was recommended to simulate the process with ANSYS software. From the simulation, flow pattern from each geometry can be observed. Hence, factors of pin tool that dictate the flow of plasticize metal can be pointed out.

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APPENDIX

Standard Test Method for Knoop and Vickers Hardness of Materials[1]

This standard is issued under the fixed designation E384; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (´) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

´[1] NOTE—The title was editorially revised in March 2010.
´[2] NOTE—Section A1.5.2 and Table A1.1 and other editorial corrections were made throughout in April 2010.

1. Scope*

1.1 This test method covers determination of the Knoop and Vickers hardness of materials, the verification of Knoop and Vickers hardness testing machines, and the calibration of standardized Knoop and Vickers test blocks.

1.2 This test method covers Knoop and Vickers hardness tests made utilizing test forces in micro (9.807×10^{-3} to 9.807 N) (1 to 1000 gf) and macro (>9.807 to 1176.68 N) (>1 to 120 kgf) ranges.

NOTE 1—Previous versions of this standard limited test forces to 9.807 N (1 kgf).

1.3 This test method includes all of the requirements to perform macro Vickers hardness tests as previously defined in Test Method E92, Standard Test Method for Vickers Hardness Testing.

1.4 This test method includes an analysis of the possible sources of errors that can occur during Knoop and Vickers testing and how these factors affect the accuracy, repeatability, and reproducibility of test results.

NOTE 2—While Committee E04 is primarily concerned with metals, the test procedures described are applicable to other materials.

1.5 *Units*—When Knoop and Vickers hardness tests were developed, the force levels were specified in units of grams-force (gf) and kilograms-force (kgf). This standard specifies the units of force and length in the International System of Units (SI); that is, force in Newtons (N) and length in mm or µm. However, because of the historical precedent and continued common usage, force values in gf and kgf units are provided for information and much of the discussion in this standard as well as the method of reporting the test results refers to these units.

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C1326 Test Method for Knoop Indentation Hardness of Advanced Ceramics
C1327 Test Method for Vickers Indentation Hardness of Advanced Ceramics
E3 Guide for Preparation of Metallographic Specimens
E7 Terminology Relating to Metallography
E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications
E74 Practice of Calibration of Force-Measuring Instruments for Verifying the Force Indication of Testing Machines
E92 Test Method for Vickers Hardness of Metallic Materials
E122 Practice for Calculating Sample Size to Estimate, With Specified Precision, the Average for a Characteristic of a Lot or Process
E140 Hardness Conversion Tables for Metals Relationship Among Brinell Hardness, Vickers Hardness, Rockwell Hardness, Superficial Hardness, Knoop Hardness, and Scleroscope Hardness
E175 Terminology of Microscopy
E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

[1] This test method is under the jurisdiction of ASTM Committee E04 on Metallography and is the direct responsibility of Subcommittee E04.05 on Microindentation Hardness Testing.With this revision the test method was expanded to include the requirements previously defined in E28.92, Standard Test Method for Vickers Hardness Testing of Metallic Material that was under the jurisdiction of E28.06

Current edition approved Feb. 1, 2010. Published February 2010. Originally approved in 1969. Last previous edition approved in 2009 as E384 – 09. DOI: 10.1520/E0384-10.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

E766 Practice for Calibrating the Magnification of a Scanning Electron Microscope

2.2 *ISO Standards:*[3]

ISO 6507-1 Metallic Materials—Vickers hardness Test—Part 1: Test Method

ISO/IEC 17011 Conformity Assessment—General Requirements for Accreditation Bodies Accrediting Conformity Assessment Bodies.

ISO/IEC 17025 General Requirements for the Competence of Testing and Calibration Laboratories

3. Terminology

3.1 *Definitions*—For the standard definitions of terms used in this test method, see Terminology E7.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *calibrating*, *v*—determining the values of the significant parameters by comparison with values indicated by a reference instrument or by a set of reference standards.

3.2.2 *Knoop hardness number, HK*, *n*—an expression of hardness obtained by dividing the force applied to the Knoop indenter by the projected area of the permanent indentation made by the indenter.

3.2.3 *Knoop indenter*, *n*—a rhombic-based pyramidal-shaped diamond indenter with edge angles of ∠ A = 172° 30′ and ∠ B = 130° 0′ (see Fig. 2).

3.2.4 *microindentation hardness test*, *n*—a hardness test using a calibrated machine to force a diamond indenter of specific geometry into the surface of the material being evaluated, in which the test forces are 9.807×10^{-3} to 9.807 N (1 to 1000 gf) and the indentation diagonal, or diagonals are measured with a light microscope after load removal; for any test, it is assumed that the indentation does not undergo elastic recovery after force removal. The test results are normally in the Knoop or Vickers scales.

3.2.5 *macroindention hardness test*, *n*—a hardness test using a calibrated machine to force an indenter of specific geometry into the surface of the material being evaluated, in which the test forces are normally higher than 9.807 N (1 kgf). Macroindentation test scales include Vickers, Rockwell and Brinell.

NOTE 3—Use of the term microhardness should be avoided because it implies that the hardness, rather than the force or the indentation size, is very low.

3.2.6 *verifying*, *v*—checking or testing the instrument to assure conformance with the specification.

3.2.7 *Vickers hardness number, HV*, *n*—an expression of hardness obtained by dividing the force applied to a Vickers indenter by the surface area of the permanent indentation made by the indenter.

3.2.8 *Vickers indenter*, *n*—a square-based pyramidal-shaped diamond indenter with face angles of 136° (see Fig. 1).

3.2.9 *scale*, *n*—a specific combination of indenter (Knoop or Vickers) and the test force. For example, HV10 is a scale defined as using a Vickers indenter and a 10 kgf test force and HK 0.1 is a scale defined as using a Knoop indenter and a 100 gf test force. See 5.8 for the proper reporting of the hardness level and scale.

3.3 *Formulae*—The formulae presented in 5.5 and 5.6 for calculating Knoop and Vickers hardness are based upon an ideal tester. The measured value of the Knoop and Vickers hardness of a material is subject to several sources of errors. Based on Eq 1-9, variations in the applied force, geometrical variations between diamond indenters, and human errors in measuring indentation lengths can affect the calculated material hardness. The influence each of these parameters has on the

[3] Available from International Organization for Standardization (ISO), 1, ch. de la Voie-Creuse, Case postale 56, CH-1211, Geneva 20, Switzerland, http://www.iso.org.

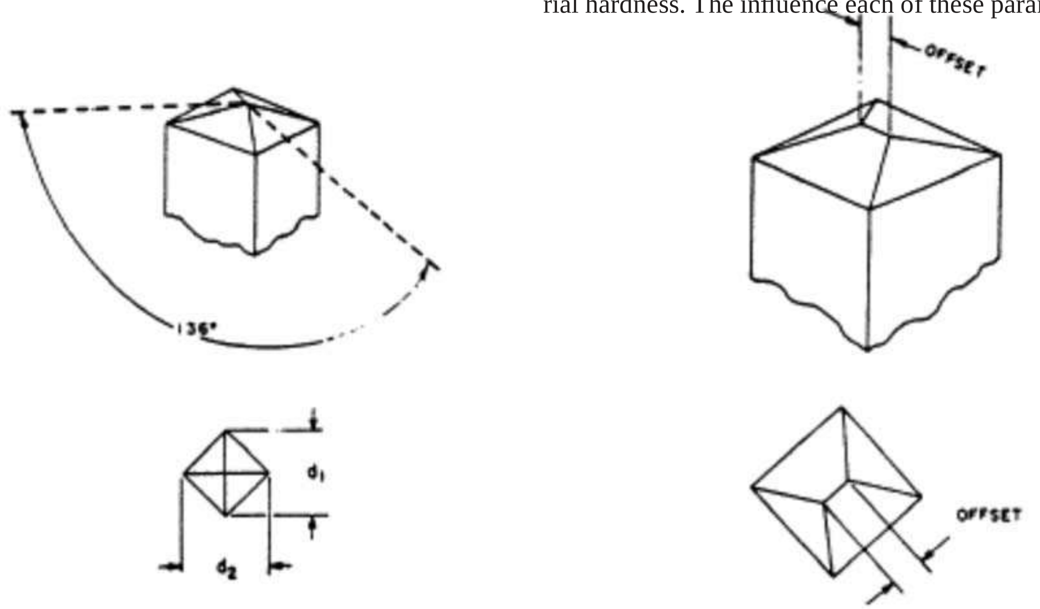

FIG. 1 Vickers Indenter

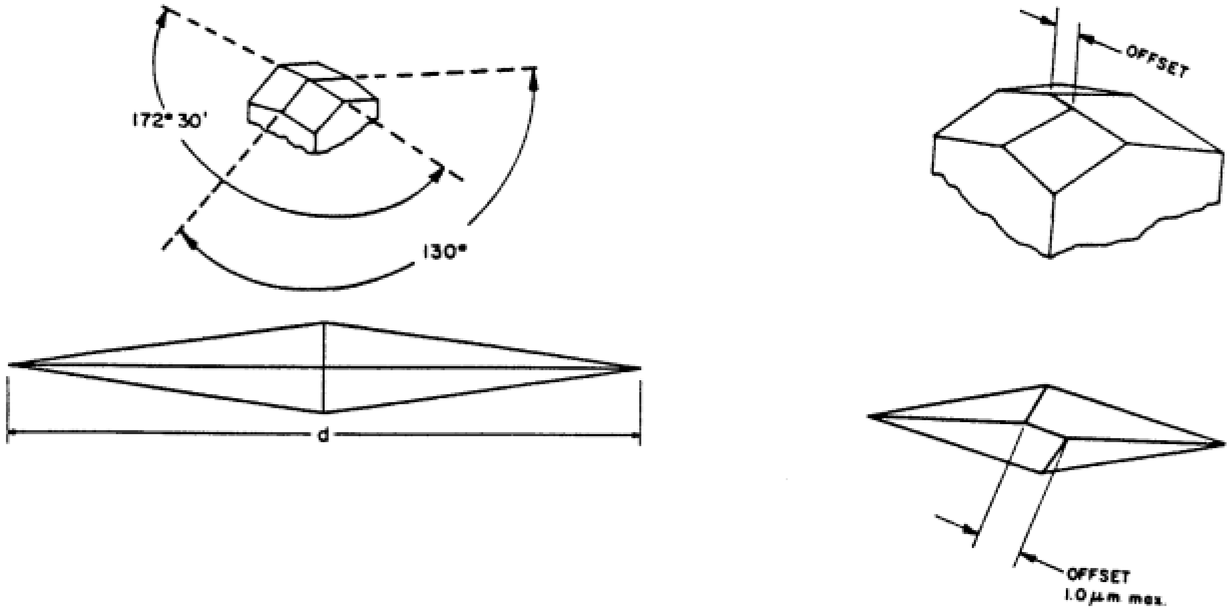

FIG. 2 Knoop Indenter

calculated value of a Knoop or Vickers measurement is discussed in Section 10.

4. Significance and Use

4.1 Hardness tests have been found to be very useful for materials evaluation, quality control of manufacturing processes and research and development efforts. Hardness, although empirical in nature, can be correlated to tensile strength for many metals, and is an indicator of wear resistance and ductility.

4.2 Microindentation hardness tests extend testing to materials that are too thin or too small for macroindentation hardness tests. Microindentation hardness tests also allow specific phases or constituents and regions or gradients too small for macroindentation hardness testing to be evaluated.

4.3 Because the Knoop and Vickers hardness will reveal hardness variations that may exist within a material, a single test value may not be representative of the bulk hardness.

4.4 The Vickers indenter usually produces a geometrically similar indentation at all test forces. Except for tests at very low forces that produce indentations with diagonals smaller than about 25 µm, the hardness number will be essentially the same as produced by Vickers machines with test forces greater than 1 kgf, as long as the material being tested is reasonably homogeneous. For isotropic materials, the two diagonals of a Vickers indentation are equal in size. Recommendations for low force microindentation testing can be found in Appendix X5.

4.5 The Knoop indenter does not produce a geometrically similar indentation as a function of test force. Consequently, the Knoop hardness will vary with test force. Due to its rhombic shape, the indentation depth is shallower for a Knoop indentation compared to a Vickers indentation under identical test conditions. The two diagonals of a Knoop indentation are markedly different. Ideally, the long diagonal is 7.114 times longer than the short diagonal, but this ratio is influenced by elastic recovery. Thus, the Knoop indenter is very useful for evaluating hardness gradients or thin coatings of sectioned samples.

5. Principle of Test

5.1 In this test method, a Knoop or Vickers hardness number is determined based on the formation of a relatively small indentation made in the test surface of samples being evaluated.

5.2 A Knoop or Vickers indenter, made from diamond of specific geometry, is pressed into the test specimen surface by an accurately controlled applied force using test machines specifically designed for such work.

5.3 Knoop and Vickers hardness testing is divided into micro and macro-test force ranges as defined:

Range	Test Force
Micro	9.807 3 10^{-3} to # 9.807 N (1 to # 1000 gf)
Macro	> 9.807 to # 1176.68 N (> 1 to # 120 kgf)

5.3.1 Knoop scale testing is normally performed using micro-range test forces (1kg and less) while the Vickers scale is used over both the micro and macro-ranges.

NOTE 4—The user should consult with the manufacturer before applying test forces in the macro-ranges (over 1 kg) with diamond indenters previously used for micro-range testing. The diamond mount may not be strong enough to support the higher test forces and the diamond may not be large enough to produce the larger indentation sizes.

5.4 The size of the indentation is measured using a light microscope equipped with a filar type eyepiece, or other type of measuring device (see Terminology E175). Micro-range indents are typically measured in µm (micrometers) and macro-range indents are measured in mm. The formulas for both units are given below.

(μm). The Knoop hardness number, in terms of gf and μm, is calculated using the following:

$$HK = 1.000 \times 10^3 \times (P/A_p) = 1.000 \times 10^3 \times P/(c_p \times d^2) \quad (1)$$

or

$$HK = 14229 \times P/d^2 \quad (2)$$

$$\text{Indenter constant} = c_p = \frac{\tan \frac{\angle B}{2}}{2 \tan \frac{\angle A}{2}} \quad (3)$$

where:

P = force, gf,
d = length of long diagonal, μm,
A_p = projected area of indentation, μm^2
$\angle A$ = included longitudinal edge angle, 172° 30'
$\angle B$ = included transverse edge angle, 130° 0' (see Fig. 2 and,
c_p = indenter constant relating projected area of the indentation to the square of the length of the long diagonal, ideally 0.07028.

NOTE 5—HK values for a 1gf (9.807×10^{-3} N) test force are contained in Appendix X6. To obtain HK values when other test forces are employed, multiply the HK value from Table X6.1 for the d value by the actual test force, gf.

5.5.2 The Knoop hardness, in terms of kgf and mm, is determined as follows:

$$HK = 14.229 \times P_1/d_1^2 \quad (4)$$

where:

P_1 = force, kgf, and
d_1 = length of long diagonal, mm.

5.5.3 The Knoop hardness reported with units of GPa is determined as follows:

$$HK = 0.014229 \times P_2/d_2^2 \quad (5)$$

where:

P_2 = force, N, and
d_2 = length of the long diagonal of the indentation, mm.

5.6 The Vickers hardness number is based upon the force divided by the surface area of the indentation.

5.6.1 For the micro-range Vickers hardness test loads are typically in grams-force (gf) and indentation diagonals are in micrometers (μm). The Vickers hardness number, in terms of gf and μm, is calculated as follows:

$$HV = 1.000 \times 10^3 \times P/A_s = 2.000 \times 10^3 \times P \sin(\alpha/2)/d^2 \quad (6)$$

or

$$HV = 1854.4 \times P/d^2 \quad (7)$$

where:

P = force, gf,
A_s = surface area of the indentation, μm^2,
d = mean diagonal length of the indentation, μm, and
α = face angle of the indenter, 136° 0' (see Fig. 1).

NOTE 6—HV numbers for a 1 gf (9.807×10^{-3} N) test load are contained in Appendix X6. To obtain HV values when other test forces are employed, multiply the HV value from Table X6.2 for the d value by the actual test force, gf.

5.6.2 Macro range Vickers hardness is typically determined using kgf and mm and is calculated as follows:

$$HV = 1.8544 \times P_1/d_1^2 \quad (8)$$

where:

P_1 = force, kgf, and
d_1 = mean diagonal length of the indentations, mm.

5.6.3 The Vickers hardness reported with units of GPa is determined as follows:

$$HV = 0.0018544 \times P_2/d_2^2 \quad (9)$$

where:

P_2 = force, N, and
d_2 = mean diagonal length of the indentations, mm.

5.7 It is assumed that elastic recovery does not occur when the indenter is removed after the loading cycle. That is, it is assumed that the indentation retains the shape of the indenter after the force is removed. In Knoop testing, it is assumed that the ratio of the long diagonal to the short diagonal of the indentation is the same as for the indenter.

5.8 The symbols HK for Knoop hardness, and HV for Vickers hardness shall be used with the reported numerical values.

5.8.1 For this standard, the hardness test results can be reported in several different ways. For example, if the Knoop hardness was found to be 400, and the test force was 100 gf, the test results may be reported as follows:

5.8.1.1 In the kilogram force system: 400 HK 0.1.

5.8.1.2 In the gram force system: 400 HK 100 gf.

5.8.1.3 In the SI system: 3.92 GPa.

5.8.1.4 For nonstandard dwell times, other than 10 to 15 s, the hardness would be reported as 400 HK 0.1 /22. In this case, 22 would be the actual time of full load dwell time in seconds.

5.9 The reported Knoop and Vickers hardness number shall be reported rounded to three significant digits in accordance with Practice E29 (for example, 725 HV 0.1, 99.2 HK 1).

6. Apparatus

6.1 *Test Machine*—The test machine shall support the test specimen and control the movement of the indenter into the specimen under a preselected test force, and should have a light optical microscope to select the desired test location and to measure the size of the indentation produced by the test. The plane of the surface of the test specimen should be perpendicular to the axis of the indenter and the direction of the force application.

6.1.1 *Vibration Control*—During the entire test cycle, the test machine should be protected from shock or vibration. To minimize vibrations, the operator should avoid contacting the machine in any manner during the entire test cycle.

6.2 *Vickers Indenter*—The ideal Vickers indenter (see Fig. 1) is a highly polished, pointed, square-based pyramidal diamond with face angles of 136° 0'. The effect that geometripoint. The line of junction (offset) between opposite faces shall not exceed the limits defined in A1.3.5.1.

6.3 *Knoop Indenter*—The ideal Knoop (see Fig. 2) indenter is a highly polished, pointed, rhombic-based, pyramidal diamond. The included longitudinal edge angles are 172° 30' and 130° 0'. The ideal indenter constant, c_p, is 0.07028. The effect that geometrical variations of these angles have on the measured values of Knoop hardness are discussed in Section 10.

6.3.1 The four faces of the Knoop indenter shall be equally inclined to the axis of the indenter and shall meet at a sharp point. The line of junction (offset) between opposite faces shall not exceed the limits defined in A1.3.5.2.

6.4 *Measuring Equipment*—The measuring device shall be capable of reporting the diagonal lengths to within 0.5 µm or 0.5% whichever is larger. For microindention hardness testing the measuring device should be able to report the diagonal lengths in 0.1 µm increments.

NOTE 7—This is the reported length and not the resolution of the system used for performing the measurements. As an example, if a length of 200 µm corresponds to 300 filar units or pixels, the corresponding calibration constant would be 200/300 = 0.667. This value would be used to compute diagonal lengths, but the reported length would only be reported to the nearest 0.5 or 0.1 µm.

6.4.1 The measuring device may be an integral part of the tester or a stand alone instrument.

6.4.2 The optical portion of the measuring device should have Köhler illumination (see Appendix X1).

6.4.3 To obtain maximum resolution, the measuring microscope should have adjustable illumination intensity, adjustable alignment, aperture, and field diaphragms.

6.4.4 Magnifications should be provided so that the diagonal can be enlarged to greater than 25 % but less than 75 % of the field width.

6.5 *Verifications*—All testers and indenters used to perform Knoop and Vickers hardness tests shall meet the requirements defined in Annex A1 prior to performing hardness tests.

7. Test Specimen

7.1 There is no standard shape or size for a Knoop or Vickers test specimen. The specimen on which the indentation is made should conform to the following:

7.1.1 *Preparation*—For optimum accuracy of measurement, the test should be performed on a flat specimen with a polished or otherwise suitably prepared surface. The quality of the required surface finish can vary with the forces and magnifications used. The lower the test force and the smaller the indentation size, the more critical is the surface preparation. Specimen preparation should be performed in accordance with applicable section of Guide E3. In all tests, the preparation should be such that the indentation perimeter and the indentation tips in particular, can be clearly defined when observed by the measuring system.

7.1.1.1 The test surface shall be free of any defects that could affect the indentation or the subsequent measurement of the diagonals. It is well known that improper grinding and polishing methods can alter test results either due to excessive heating or cold work. Some materials are more sensitive to preparation-induced damage than others; therefore special precautions must be taken during specimen preparation. Specimen preparation must remove any damage introduced during these steps.

7.1.1.2 The specimen surface should not be etched before making an indentation. Etched surfaces can obscure the edge of the indentation, making an accurate measurement of the size of the indentation difficult. However, when determining the microindentation hardness of an isolated phase or constituent, a light etch can be used to delineate the object of interest.

7.1.2 *Alignment*—To obtain usable information from the test, the specimen should be prepared or mounted so that the test surface is perpendicular to the axis of the indenter. This can readily be accomplished by surface grinding (or otherwise machining) the opposite side of the specimen parallel with the side to be tested. Non-parallel samples can be tested using clamping and leveling fixtures designed to align the test surface properly to the indenter.

7.1.3 *Mounted Samples*—In many instances, it is necessary to mount the specimen for convenience in preparation and to maintain a sharp edge when surface gradient tests are to be performed on the sample. When mounting is required, the specimen must be adequately supported by the mounting medium so that the specimen does not move during force application, that is, avoid the use of polymeric mounting compounds that creep under the indenter force.

7.1.4 *Thickness*—the thickness of the specimen tested shall be such that no bulge or other marking showing the effect of the test force appears on the side of the piece opposite the indentation. The thickness of the material under test should be at least ten times the depth of the indentation. This is also to be used as a guideline for the minimum depth of a coating on a material.

7.1.5 *Radius of Curvature* —due caution should be used in interpreting or accepting the results of tests made on spherical or cylindrical surfaces. Results will be affected even in the case of the Knoop test where the radius of curvature is in the direction of the short diagonal. Table 1 provides correction factors that shall be applied when performing Vickers test on spherical surfaces.

NOTE 8—A method for correcting Vickers hardness readings taken on spherical or cylindrical surfaces can be found in the International Organization for Standardization (ISO) Vickers Hardness Standard (ISO 6507-1).

8. Procedure

8.1 *Test temperature*—Knoop and Vickers hardness tests should be carried out at a temperature within the limits of 10 to 35°C (50 to 95°F). Because variations within this temperature range may affect results, users may choose to control temperature within a tighter range.

8.2 *Indenter*—Select the desired indenter, either Knoop or Vickers, to suit the desired test scale to be performed. Refer to the manufacturer's instruction manual for the proper procedure if it is necessary to change indenters.

TABLE 1 Correction Factors for Use in Vickers Hardness Tests Made on Spherical Surfaces

Convex Surface		Concave Surface	
d/D[A]	Correction Factor	d/D[A]	Correction Factor
0.004	0.995	0.004	1.005
0.009	0.990	0.008	1.010
0.013	0.985	0.012	1.015
0.018	0.980	0.016	1.020
0.023	0.975	0.020	1.025
0.028	0.970	0.024	1.030
0.033	0.965	0.028	1.035
0.038	0.960	0.031	1.040
0.043	0.955	0.035	1.045
0.049	0.950	0.038	1.050
0.055	0.945	0.041	1.055
0.061	0.940	0.045	1.060
0.067	0.935	0.048	1.065
0.073	0.930	0.051	1.070
0.079	0.925	0.054	1.075
0.086	0.920	0.057	1.080
0.093	0.915	0.060	1.085
0.100	0.910	0.063	1.090
0.107	0.905	0.066	1.095
0.114	0.900	0.069	1.100
0.122	0.895	0.071	1.105
0.130	0.890	0.074	1.110
0.139	0.885	0.077	1.115
0.147	0.880	0.079	1.200
0.156	0.875	0.082	1.125
0.165	0.870	0.084	1.130
0.175	0.865	0.087	1.135
0.185	0.860	0.089	1.140
0.195	0.855	0.091	1.145
0.206	0.850	0.094	1.150

[A] *D* = diameter of sphere in millimeters.
d = mean diagonal of indentation in millimeters.

8.2.1 After each change, or removal and replacement, of the indenter it is recommended that a daily verification be performed as defined in A1.5. At least two preliminary indentations should be made to ensure that the indenter is seated properly. The results of the preliminary indentations shall be disregarded.

8.2.2 Occasionally clean the indenter with a cotton swab and alcohol. Avoid creating static charges during cleaning. Indenting a piece of paper will often remove oil from the indenter

8.2.3 Indenters should be examined periodically and replaced if they become worn, dulled, chipped, cracked or separated from the mounting material. Checks of the indenter by the user may be performed by visual inspection of the resulting indentation; it is sufficient to verify the absence of defects from the shape of indentations performed on test blocks

8.3 *Magnitude of Test Force*—Select the desired test force on the tester by following the manufacturers instructions.

8.3.1 After each change of a test force, it is recommended that the operation of the machine be checked by performing a daily verification as defined in A1.5.

8.4 *Mount the specimen to the tester*—Mount the specimen on the tester stage or place it in the top-surface indexed mounting fixture on the stage so that the test surface is perpendicular to the indenter axis.

8.5 *Locate the test point*—Focus the measuring microscope with a low power objective so that the specimen surface can be observed. Adjust the light intensity and adjust the diaphragms for optimum resolution and contrast. Adjust the position of the sample so that the indentation will be made in the desired location on the test surface. Before applying the force, make a final focus using the measuring objective or the highest magnification objective available.

8.6 *Force Application*—Apply the selected test force as follows without shock or vibration:

8.6.1 For micro test force range testing, the indenter shall contact the specimen at a velocity between 15 and 70 µm/s. For macro test force ranges the contact velocity should not exceed 0.2 mm/s.

8.6.2 The time from the initial application of the force until the full test force is reached shall not be more than 10 s.

8.6.3 The full test force shall be applied for 10 to 15 s unless otherwise specified.

8.6.3.1 For some applications it may be necessary to apply the test force for longer times. In these instances the tolerance for the time of the applied force shall be ± 2 s. The application time shall be defined in the report

8.6.4 Remove the test force without shock or vibration.

8.7 *Test location*—After the force is removed, switch to the measuring mode, and select the proper objective lens. Focus the image, adjust the light intensity if necessary, and adjust the diaphragms for maximum resolution and contrast.

8.7.1 Examine the indentation for its position relative to the desired location and for its symmetry.

8.7.2 If the indentation did not occur at the desired spot, the tester is out of alignment. Consult the manufacturer's instruction manual for the proper procedure to produce alignment. Make another indentation and recheck the indentation location. Readjust and repeat as necessary.

8.8 *Indentation examination*:

8.8.1 For a Knoop indentation, if one half of the long diagonal is greater than 10 % longer than the other, or if both ends of the indentation are not in sharp focus, the test specimen surface may not be perpendicular to the indenter axis. Check the specimen alignment and make another test. Indents that exceed the 10% limit should be noted in the test report.

8.8.2 For a Vickers indentation, if one half of either diagonal is more than 5 % longer than the other half of that diagonal, or if the four corners of the indentation are not in sharp focus, the test surface may not be perpendicular to the indenter axis. Check the specimen alignment and make another test. Indents that exceed the 5% limit should be noted in the test report.

8.8.3 If the diagonal legs are unequal as described in 8.8.1 or 8.8.2 rotate the specimen 90° and make another indentation in an untested region. If the nonsymmetrical aspect of the

8.8.4 Some materials may have nonsymmetrical indentations even if the indenter and the specimen surface are perfectly aligned. Tests on single crystals or on textured materials may produce such results. When this occurs, check the alignment using a test specimen, such as a standardized test block, known to produce uniformly shaped indentations. Testers that do not perform symmetrical indents on those specimens shall not be used until they meet the requirements of sections 8.8.1 and 8.8.2.

8.8.5 Brittle materials such as ceramics may crack as a result of being indented. Specific details for testing ceramics are contained in Test Methods C1326 and C1327.

8.9 *indentation Measurement*:

8.9.1 Measure the long diagonal of a Knoop indentation, or both diagonals of a Vickers indentation, by operating the measuring device in accordance with the manufacturer's instruction manual.

8.9.2 Determine the length of the diagonals to 0.5 µm or less (see 6.4).

8.9.3 For the Vickers indentations, average the two diagonal length measurements.

8.10 *Knoop or Vickers hardness calculation*:

8.10.1 Compute the Knoop or Vickers hardness number using the appropriate equation in 5.5 or 5.6 or Table X6.1 or Table X6.2, respectively. Table X6.1 and Table X6.2 show the Knoop or Vickers hardness for indentations with diagonal lengths from 1 to 200.9 µm using 1 gf. If the force was not 1 gf, multiply the value from Table X6.1 or Table X6.2 by the actual gram-force value to obtain the correct hardness number.

8.11 *Spacing of Indentations*—Generally more than one indentation is made on a test specimen. It is necessary to ensure that the spacing between indentations is large enough so that adjacent tests do not interfere with each other.

8.11.1 For most testing purposes, the minimum recommended spacing between separate tests, and minimum distance between an indentation and the edge of the specimen are illustrated in Fig. 3.

8.11.2 For some applications, closer spacing of indentations than those shown in Fig. 3 may be desired. If closer indentation spacing is used, it shall be the responsibility of the testing laboratory to verify the accuracy of the testing procedure.

9. Report

9.1 Report the following information:

9.1.1 The results (see 5.8), the number of tests, and, where appropriate, the mean and standard deviation of the results,

9.1.2 Test force,

9.1.3 The total force application time if outside the limits of 10 to 15 s as defined in 8.6.3.

9.1.4 Any unusual conditions encountered during the test, and

9.1.5 The test temperature, when the outside the recommended allowable range of 10°C to 35°C (50°F to 95°F).

10. Precision and Bias

10.1 The precision and bias of Knoop and Vickers hardness measurements depend on strict adherence to the stated test procedure and are influenced by instrumental and material factors and indentation measurement errors.

10.2 The consistency of agreement for repeated tests on the same material is dependent on the homogeneity of the material, reproducibility of the hardness tester, and consistent, careful measurement of the indents by a competent operator.

10.3 Instrumental factors that can affect test results include: accuracy of loading; inertia effects; speed of loading; vibrations; the angle of indentation; lateral movement of the indenter or specimen; indentation and indenter shape deviations.

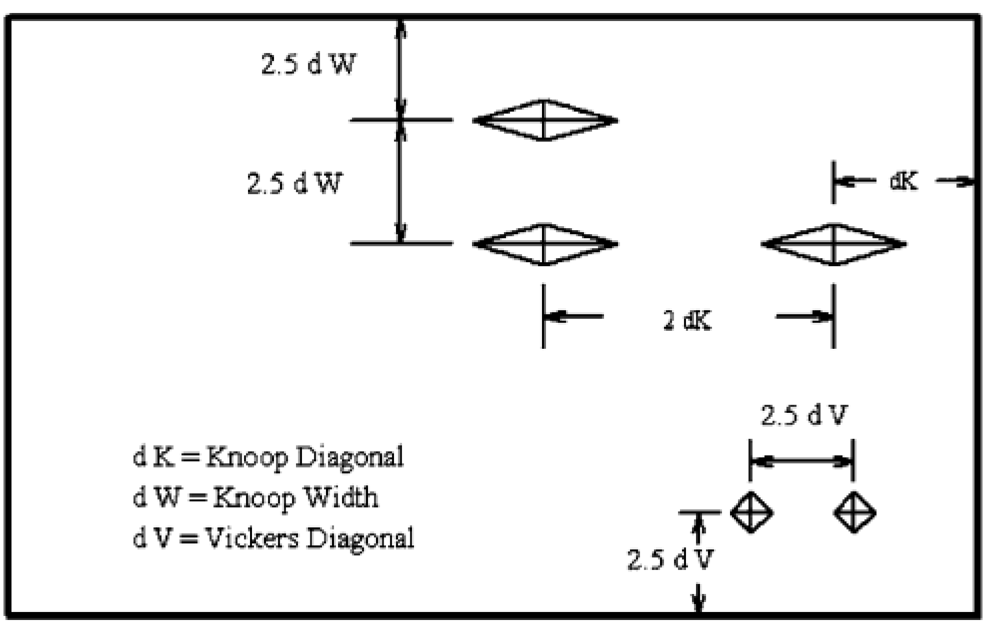

FIG. 3 Minimum Recommended Spacing for Knoop and Vickers Indentations

10.3.1 Vibrations during indenting will produce larger indentations with the influence of vibrations becoming larger as the force decreases (**1**, **2**).[4]

10.3.2 The angle between the indenter axis and specimen surface should be within 2° of perpendicular. Greater amounts of tilting produce nonuniform indentations and invalid test results.

10.4 Material factors that can affect test results include: specimen homogeneity, orientation or texture effects; improper specimen preparation; low specimen surface reflectivity; transparency of the specimen.

10.4.1 Residual deformation from mechanical polishing must be removed, particularly for low-force testing.

10.4.2 Distortion of the indentation shape due to either crystallographic or microstructural texture influences diagonal lengths and the validity of the calculated hardness.

10.4.3 Plastic deformation during indenting can produce ridging around the indentation periphery that will affect diagonal measurement accuracy.

10.4.4 Testing of etched surfaces, depending on the extent of etching, can produce results that are different from those obtained on unetched surfaces (**1**).

10.5 Measurement errors that can affect test results include: inaccurate calibration of the measuring device; inadequate resolving power of the objective; insufficient magnification; operator bias in sizing the indentations; poor image quality; nonuniform illumination, improper zeroing of the measuring device.

10.5.1 The accuracy of Knoop and Vickers hardness testing is strongly influenced by the accuracy to which the indentations can be measured.

10.5.2 The error in measuring the diagonals increases as the numerical aperture of the measuring objective decreases (**3**, **4**).

10.5.3 Bias is introduced if the operator consistently undersizes or oversizes the indentations.

10.6 Some of the factors that affect test results produce systematic errors that influence all test results while others primarily influence low-force test results (**5**). Some of these problems occur continually, others may occur in an undefined, sporadic manner. Low force hardness tests are influenced by these factors to a greater extent than high force tests.

10.7 For both the Vickers and Knoop hardness tests, the calculated hardness is a function of three variables: force, indenter geometry and diagonal measurement. Total differentials of the equations used to calculate the hardness can be used to evaluate the effect variations in these parameters can cause.

10.7.1 *Vickers*—using Eq 6, the total differential for the Vickers hardness number is:

$$dV = \left(\frac{\partial V}{\partial P}\right) dP + \left(\frac{\partial V}{\partial d}\right) dd + \left(\frac{\partial V}{\partial \alpha}\right) d\alpha \tag{10}$$

and

$$\left(\frac{\partial V}{\partial P}\right) = 2 \times 10^{3} \times d^{-2} \times \sin\left(\frac{\alpha}{2}\right) \tag{11}$$

$$\left(\frac{\partial V}{\partial d}\right) = -4 \times 10^{3} \times P \times d^{-3} \times \sin\left(\frac{\alpha}{2}\right) \tag{12}$$

$$\left(\frac{\partial V}{\partial \alpha}\right) = 10^{3} \times P \times d^{-2} \cos\left(\frac{\alpha}{2}\right) \tag{13}$$

For a material having a hardness of 500 HV when tested with a 500 gf force, d = 43.06 µm, α = 136°, and $\sin\left(\frac{\alpha}{2}\right)$ = 0.927184.

10.7.1.1 Consider introducing a 1 % error into the hardness of the material through an error in either the applied force, the indenter constant or the measured diagonal length. In this case, the hardness would be HV = 505 or dV = 5. Using Eq 11-13, the corresponding errors in the various parameters are as shown in Table 2. Thus a 1 % change in P or a 2.836 % error in α creates a 1 % error in the Vickers hardness number. However, only a 0.5 % error in the measured diagonal is needed to create a 1 % error in Vickers hardness. Furthermore, this analysis indicates that the calculated Vickers hardness number is not strongly influenced by errors in the angle of the indenter.

10.7.2 *Knoop*—Similarly, using Eq 1, it follows that:

$$dK = \left(\frac{\partial K}{\partial P}\right) dP + \left(\frac{\partial K}{\partial c_p}\right) dc_p + \left(\frac{\partial K}{\partial d}\right) dd \tag{14}$$

$$\frac{10^{3}}{c_p\, d^{2}} dP + \frac{10^{3}\, P}{c_p^{2}\, d^{2}} dc_p + \frac{-2 \times 10^{3}\, P}{c_p\, d^{3}} dd \tag{15}$$

and since the indenter has two different angles, A and B,

$$dc_p = \left(\frac{\partial c_p}{\partial A}\right) dA + \left(\frac{\partial c_p}{\partial B}\right) dB \tag{16}$$

$$\left(\frac{\partial c_p}{\partial A}\right) = \frac{-\tan\left(\frac{B}{2}\right)}{4 \sin^{2}\left(\frac{A}{2}\right)} \tag{17}$$

and

$$\left(\frac{\partial c_p}{\partial B}\right) = \frac{\cot\left(\frac{A}{2}\right)}{4 \cos^{2}\left(\frac{B}{2}\right)} \tag{18}$$

10.7.2.1 Using the differentials cited in 10.7.2, for the Knoop test at various forces, for a 1 % error in hardness that is, HK = 505 or dK = 5, the corresponding errors in the force, diagonal measurement and indenter angle are as shown in Table 3. From this analysis it follows that 1 % error in P creates

TABLE 2 Vickers Hardness Analysis—1 % Error

		1 % Error		
Force, gf	Diagonal, µm	Δ P, gf	Δ Diagonal, µm	Δ Angle, °
10	6.090	0.100	–0.030	2.836
20	8.612	0.200	–0.043	2.836
50	13.617	0.499	–0.068	2.836
100	19.258	0.999	–0.096	2.836
200	27.235	1.998	–0.136	2.836
500	43.062	4.994	–0.215	2.836

[4] The boldface numbers in parentheses refer to the list of references at the end of this standard.

TABLE 3 Knoop Hardness Analysis—1 % Error

		1 % Error			
Force, gm	Diagonal, µm	Δ P gf	Δ diagonal, µm	Δ A, °	Δ B, °
10	16.87	0.10	–0.08	0.075	0.439
20	23.86	0.20	–0.12	0.075	0.439
50	37.72	0.50	–0.19	0.075	0.439
100	53.35	1.00	–0.27	0.075	0.439
200	75.45	2.00	–0.38	0.075	0.439
500	119.29	5.00	–0.60	0.075	0.439
1000	168.71	10.00	–0.84	0.075	0.439
				4′ 30″	26′ 20″

a 1 % error in HK, 0.5 % error in the measured diagonal creates a 1 % error in HK, and 1 % error in c creates a 1 % error in HK.

10.7.2.2 Since the indenter constant is composed of terms from two different angles, either a 4′ 3″ error in ∠A, or a 26′ 20″ error in ∠B produces a 1 % error in HK. Unlike the Vickers indenter, the calculated Knoop hardness number is very strongly influenced by small errors in the two angles of the indenter. The A angle, 172° 30′ 00″, is the most sensitive of these parameters. The actual value of c_p for each indenter can be calculated using the certified A and B angles provided by the indenter manufacturer. This will enhance the accuracy of the test measurements.

10.8 Over a period of several years, four separate interlaboratory studies have been conducted in accordance with Practice E691 to determine the precision, repeatability, and reproducibility of this test method. The four studies are defined as follows:

a) Knoop and Vickers tests, six test forces in the micro range, twelve laboratories, manual measurements, seven different hardness level samples. See 10.8.1 and Appendix X3.

b) Knoop and Vickers tests, two test forces in the micro range, seven laboratories, Image Analysis and manual measurements, four different hardness level samples. See 10.8.2 and Appendix X4.

c) Knoop and Vickers tests, six test forces in the micro range, twenty-five laboratories, manual measurements, six different hardness level samples. See 10.8.3.

d) Vickers tests, four test forces in the macro range, seven laboratories, manual measurements, three different hardness level samples. See 10.8.4.

10.8.1 An interlaboratory test program was conducted in accordance with Practice E691 to develop information regarding the precision, repeatability, and reproducibility of the measurement of Knoop and Vickers indentations in the micro ranges[5]. The test forces were 25, 50, 100, 200, 500, and 1000 gf on three ferrous and four nonferrous specimens **(6, 7)**. Twelve laboratories measured the indentations, five of each type at each force on each sample. Additional details of this study are given in Appendix X3.

10.8.1.1 Tests of the three ferrous specimens revealed that nine laboratories produced similar measurements while two laboratories consistently undersized the indentations and one laboratory consistently oversized the indentations. These latter results were most pronounced as the force decreased and specimen hardness increased (that is, as the diagonal size decreased) and were observed for both Vickers and Knoop indentations. Results for the lower hardness nonferrous indentations produced better agreement. However, none of the laboratories that obtained higher or lower results on the ferrous specimens measured the nonferrous indentations.

10.8.1.2 *Repeatability Interval*—The difference due to test error between two test results in the same laboratory on the same material increases with increasing specimen hardness and with decreasing test force (see X3.4.4).

10.8.1.3 *Reproducibility Interval*—The difference in test results on the same material tested in different laboratories increased with increasing specimen hardness and with decreasing test force (see X3.4.5).

10.8.1.4 The within-laboratory and between-laboratory precision values improved as specimen hardness decreased and test force increased. The repeatability interval and reproducibility interval were generally larger than the precision estimate, particularly at low test forces and high specimen hardnesses.

10.8.2 *Image Analysis Measurements*—An interlaboratory test program was conducted in accordance with Practice E691 to develop information regarding the repeatability and reproducibility of Knoop and Vickers measurements made with automated Image Analysis systems and manual procedures. Four ferrous specimens were used in the round robin. The test were conducted at 100 gf and 300 gf. The participants in the test program measured the same indentations on the four specimens. Seven labs measured the specimens using both procedures. The Knoop indentations on specimen C1 were too long for accurate measurements to be made by one lab; hence, only six sets of measurements were made on this specimen. Near the end of the test program, specimen B1 was lost in shipping; thus only six sets of measurements were made on this specimen. Additional details of the study are contained in Appendix X4.

10.8.2.1 Repeatability concerns the variability between individual test results obtained within a single laboratory by a single operator with a specific set of test apparatus. For both the manual and automated measurements, the repeatability interval increased with specimen hardness and decreasing test force, Appendix X4. For equivalent testing conditions, the repeatability interval for automated measurements was slightly larger than for manual measurements.

10.8.2.2 Reproducibility deals with the variability between single test results obtained by different laboratories applying the same test methods to the same or similar test specimens. For both the manual and automated measurements, the reproducibility interval increased with specimen hardness and decreasing test force, Appendix X4. For equivalent testing conditions, the reproducibility interval for automated measure-

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:E04-1004.

the manual and automated procedures. However, this information is graphically represented for comparative purposes, X4.6.

10.8.3 The precision of this test method is based on an interlaboratory study of E384-07, Standard Test Method for Microindentation Hardness of Materials, conducted in 2007. Twenty-five laboratories tested a total of six ferrous materials for Vickers Hardness and thirteen laboratories submitted Knoop Hardness results. Every "test result" was recorded, and the laboratory means represent an average of five individual determinations (for Knoop) or five separate measurements, each the average of two readings (for Vickers). Practice E691 was followed for the design and analysis of the data; the details are given in ASTM Research Report No. E04-1006.[6]

10.8.3.1 *Repeatability limit (r)*—Two test results obtained within one laboratory shall be judged not equivalent if they differ by more than the "r" value for that material; "r" is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment on the same day in the same laboratory.

10.8.3.2 Repeatability limits in diagonal lengths (µm) are listed Table 4 and Table 5 and in hardness units (HK, HV) in Table 6 and Table 7.

10.8.3.3 *Reproducibility limit (R)*— Two test results shall be judged not equivalent if they differ by more than the "R" value for that material; "R" is the interval representing the critical difference between two test results for the same material, obtained by different operators using different equipment in different laboratories.

10.8.3.4 Reproducibility limits in diagonal lengths (µm) are listed in Table 4 and Table 5 and Fig. 4 and Fig. 5 and in hardness units (HK, HV) in Table 6 and Table 7 and Fig. 6 and Fig. 7.

10.8.3.5 The above terms (repeatability limit and reproducibility limit) are used as specified in Practice E177.

10.8.3.6 Any judgment in accordance with statements 10.8.3.1 and 10.8.3.3 would have an approximate 95% probability of being correct.

10.8.3.7 The precision statement was determined through statistical examination of results from twenty-five laboratories, on six ferrous materials. These six ferrous materials were described as:

Specimen A: H13, mill annealed, hardness less than 20 HRC

[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:E04-1006.

TABLE 4 Precision Statistics for an Interlaboratory Study of the Knoop Microindentation Hardness Test for Ferrous Specimens in Diagonal Units (µm)

Specimen	Test Force (gf)	500 100 0 –	Average Diagonal (µm) *d*	88.27 126.96	Standard Deviation (µm) S_x 1.40 1.33	Repeatability Standard Deviation (µm) S_r	0.66 0.75 Reproducibility Standard Deviation (µm) S_R
A	25		35.61		1.65 2.63	0.72	1.54
	50		51.77		2.07 1.72	1.11	1.66
	100		74.84		0.95 0.94	1.77	2.28
	300		132.28		1.12 1.39	2.57	3.50
	500		171.51		1.68 1.65	2.46	3.02
	1000		243.11		1.33 1.14	2.96	3.16
B	25		23.66		1.05 1.25	0.48	1.04
	50		34.33		1.50 1.79	0.56	1.07
	100		49.61		1.04 0.85	0.65	1.26
	300		88.64		1.08 0.94	0.88	1.59
	500		115.48		1.16 2.03	1.11	1.95
	1000		164.38		0.72 1.00	1.52	2.14
C	25		27.62		1.15 1.00	0.49	1.41
	50		39.47		1.06 1.27	0.50	1.22
	100		56.66		0.88 1.03	0.64	1.20
	300		100.14		1.45 1.39	0.81	1.44
	500		130.19		1.11 1.47	0.83	1.68
	1000		184.84			1.19	2.08
D	25		31.04			0.46	1.11
	50		44.64			0.46	0.95
	100		64.22			0.67	1.24
	300		113.94			0.82	1.19
	500		148.16			0.74	1.33
	1000		210.10			1.64	2.50
E	25		20.02			0.48	0.84
	50		29.03			0.48	1.09
	100		42.21			0.52	1.24
	300		76.03			0.53	1.11
	500		99.25			0.49	1.15
	1000		141.67			0.85	1.48
T	25		17.14			0.48	0.98
	50		25.59			0.47	1.12
	100		37.20			0.52	1.52
	300		67.43			0.65	

TABLE 5 Precision statistics for an Interlaboratory Study of the Vickers Microindentation Hardness Test for Ferrous Specimens in Diagonal Units (µm)

Specimen	Test Force (gf)	Average Diagonal (µm) $\bar{d}$	Standard Deviation (µm) S_x	Repeatability Standard Deviation (µm) S_r	Reproducibility Standard Deviation (µm) S_R	Repeatability Limit (µm) r	Reproducibility Limit (µm) R
A	25	13.89	0.75	0.30	0.80	0.85	2.24
	50	19.81	0.61	0.34	0.68	0.95	1.91
	100	28.10	0.57	0.45	0.70	1.26	1.96
	300	49.19	0.75	0.72	0.99	2.02	2.77
	500	63.65	0.81	0.88	3.16	2.47	1.13
	1000	90.48	0.98	1.31	1.53	3.66	4.28
B	25	9.35	0.40	0.25	0.46	0.69	1.28
	50	13.06	0.37	0.23	0.42	0.63	1.18
	100	18.51	0.39	0.39	0.52	1.09	1.47
	300	32.11	0.43	0.30	0.50	0.84	1.41
	500	41.68	0.51	0.36	0.60	1.00	1.69
	1000	59.21	0.55	0.52	0.72	1.46	2.03
C	25	10.81	0.53	0.19	0.56	0.54	1.56
	50	15.13	0.42	0.20	0.46	0.57	1.29
	100	21.34	0.40	0.22	0.45	0.62	1.25
	300	36.85	0.38	0.21	0.43	0.59	1.20
	500	47.68	0.55	0.24	0.59	0.67	1.64
	1000	67.60	0.58	0.33	0.65	0.93	1.83
D	100	24.50	0.43	0.29	0.50	0.82	1.40
	300	42.52	0.41	0.28	0.48	0.80	1.35
	500	55.02	0.50	0.25	0.55	0.70	1.54
	1000	78.14	0.70	0.34	0.77	0.97	2.15
E	100	15.61	0.40	0.18	0.43	0.52	1.20
	300	27.25	0.41	0.25	0.46	0.70	1.30
	500	35.26	0.43	0.20	0.46	0.55	1.30
	1000	50.06	0.41	0.24	0.46	0.67	1.29
T	300	23.94	0.47	0.17	0.49	0.49	1.38
	500	31.00	0.51	0.21	0.55	0.59	1.53
	1000	44.12	0.50	0.25	0.55	0.69	1.53

Specimen B: H13, austenitized, quenched, and tempered ~ 50 HRC
Specimen C: H13, austenitized, quenched, and tempered ~ 40 HRC
Specimen D: H13, austenitized, quenched, and tempered ~ 30 HRC
Specimen E: O1, austenitized, quenched and tempered O1 steel, ~ 60 HRC
Specimen T: T15 P/M, austenitized, quenched and tempered ~ 67 HRC
To judge the equivalency of two test results, it is recommended to choose the material closest in characteristics to the test material.

10.8.4 The macro Vickers precision statement is based on an interlaboratory study of E92, Standard Test Method for Vickers Hardness of Metallic Materials, conducted in 2001. (With this revision Test Method E92 is now part of E384) Seven laboratories tested three different standard hardness test blocks using macro range test forces of 1kg, 5kg, 10kg, and 20kg. Only four laboratories were also able to provide results at 50kg test force. Every "test result" represents an individual determination of the Vickers hardness of the material. Each laboratory was asked to report triplicate test results in order to permit the estimation of Intralaboratory precision. Practice E691 was followed for the design and analysis of the data; the details are given in ASTM Research Report No. RR:E04-1007.[7]

10.8.4.1 *Repeatability limit (r)*—Two test results obtained within one laboratory shall be judged not equivalent if they differ by more than the "r" value for that material; "r" is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment on the same day in the same laboratory. Repeatability limits are listed in Tables 8-12 below.

10.8.4.2 *Reproducibility limit (R)*—Two test results shall be judged not equivalent if they differ by more than the "R" value for that material; "R" is the interval representing the critical difference between two test results for the same material, obtained by different operators using different equipment in different laboratories. Reproducibility limits are listed Tables 8-12 in below.

10.8.4.3 The above terms (repeatability limit and reproducibility limit) are used as specified in Practice E177.

10.8.4.4 Any judgment in accordance with statements 10.8.4.1 and 10.8.4.2 would have an approximate 95% probability of being correct.

10.8.4.5 *Bias*—There is no recognized standard by which to estimate the bias of this test method.

10.8.4.6 The precision statement was determined through statistical examination of 288 results, from seven laboratories,

TABLE 6 Precision statistics for an Interlaboratory Study of the Knoop Microindentation Hardness Test for Ferrous Specimens in Hardness units (HK)

Specimen	Test Force	Average Diagonal (µm)	Standard Deviation (HK)	Repeatability Standard Deviation (HK)	Reproducibility Standard Deviation (HK)	Repeatability Limit (HK)	Reproducibility Limit (HK)
	(gf)	*d*	S_x	S_r	S_R	r	R
A	25	35.61	22.07	11.35	24.29	31.56	68.41
	50	51.77	13.64	11.39	17.03	32.05	47.98
	100	74.84	11.20	12.02	15.49	33.68	43.61
	300	132.28	9.70	9.48	12.91	26.60	36.21
	500	171.51	5.84	6.94	8.52	19.45	23.86
	1000	243.11	3.41	5.86	6.26	16.43	17.52
B	25	23.66	51.07	25.79	55.92	72.09	157.50
	50	34.33	33.07	19.70	37.65	55.27	105.55
	100	49.61	26.11	15.15	29.38	42.45	82.72
	300	88.64	17.04	10.79	19.49	30.04	54.74
	500	115.48	15.52	10.26	18.02	28.75	50.50
	1000	164.38	10.57	9.74	13.71	27.24	38.34
C	25	27.62	44.96	16.55	47.67	46.65	134.05
	50	39.47	26.39	11.57	28.24	32.19	79.67
	100	56.66	16.43	10.01	18.78	28.02	52.50
	300	100.14	10.63	6.89	12.24	19.22	34.29
	500	130.19	9.67	5.35	10.83	15.03	30.26
	1000	184.84	8.07	5.36	9.37	15.01	26.24
D	25	31.04	24.75	10.94	26.42	30.48	74.60
	50	44.64	13.60	7.36	15.20	20.80	42.46
	100	64.22	11.61	7.20	13.33	20.32	37.34
	300	113.94	5.43	4.73	6.87	13.22	19.23
	500	148.16	5.08	3.24	5.82	9.01	16.32
	1000	210.10	6.23	5.03	7.67	14.06	21.49
E	25	20.02	63.88	42.57	74.54	120.86	208.90
	50	29.03	58.20	27.92	63.44	78.02	178.37
	100	42.21	43.53	19.68	46.94	55.28	131.37
	300	76.03	19.43	10.30	21.56	28.76	60.27
	500	99.25	15.43	7.13	16.74	19.94	46.74
	1000	141.67	12.71	8.51	14.81	23.92	41.55
T	25	17.14	124.50	67.85	138.69	191.33	395.07
	50	25.59	87.53	39.91	95.19	112.23	266.90
	100	37.20	80.22	28.75	84.10	80.77	237.05
	300	67.43	38.71	18.10	42.06	50.70	117.74
	500	88.27	22.97	13.65	26.07	38.28	73.09
	1000	126.96	20.44	10.43	22.39	29.07	62.90

on three test blocks. The materials were described as the following:

Material 1: 200 HV
Material 2: 400 HV
Material 3: 800 HV

11. Conversion to Other Hardness Scales or Tensile Strength Values

11.1 There is no generally accepted method for accurate conversion of Knoop or Vickers hardness numbers to other hardness scales or tensile strength values. Such conversions are limited in scope and should be used with caution, except for special cases where a reliable basis for the conversion has been obtained by comparison tests. For loads ≥ 100 gf microindentation Vickers hardness numbers are in reasonable agreement with macroindention Vickers hardness numbers. Refer to Test Method E140 for hardness conversion tables for metals.

12. Keywords

12.1 hardness; indentation; Knoop; microindentation; macroindentation; Vickers

TABLE 7 Precision statistics for an Interlaboratory Study of the Vickers Microindentation Hardness Test for Ferrous Specimens in Hardness units (HV)

Specimen	Test Force (gf)	Average Diagonal (µm) *d*	Standard Deviation (HV) S_x	Repeatability Standard Deviation (HV) S_r	Reproducibility Standard Deviation (HV) S_R	Repeatability Limit (HV) r	Reproducibility Limit (HV) R
A	25	13.89	25.99	10.38	27.73	29.46	78.52
	50	19.81	14.56	8.11	16.23	22.69	45.77
	100	28.10	9.53	7.52	11.70	21.08	32.84
	300	49.19	7.01	6.73	9.26	18.90	25.94
	500	63.65	5.83	6.33	22.75	17.78	8.13
	1000	90.48	4.91	6.56	7.66	18.34	21.45
B	25	9.35	45.41	28.37	52.24	78.48	146.56
	50	13.06	30.81	19.15	34.98	52.51	98.63
	100	18.51	22.81	22.81	30.42	63.85	86.24
	300	32.11	14.45	10.08	16.81	28.24	47.43
	500	41.68	13.06	9.22	15.37	25.62	43.32
	1000	59.21	9.83	9.29	12.87	26.09	36.29
C	25	10.81	38.95	13.95	41.16	39.69	115.71
	50	15.13	22.50	10.71	24.64	30.54	69.32
	100	21.34	15.27	8.40	17.18	23.67	47.79
	300	36.85	8.45	4.67	9.56	13.12	26.70
	500	47.68	9.41	4.11	10.09	11.46	28.07
	1000	67.60	6.96	3.96	7.80	11.17	21.98
D	100	24.50	10.85	7.31	12.61	20.69	35.36
	300	42.52	5.93	4.05	6.95	11.58	19.55
	500	55.02	5.57	2.78	6.12	7.79	17.15
	1000	78.14	5.44	2.64	5.99	7.54	16.72
E	100	15.61	39.01	17.55	41.94	50.73	117.35
	300	27.25	22.55	13.75	25.30	38.50	71.56
	500	35.26	18.19	8.46	19.46	23.27	55.03
	1000	50.06	12.12	7.10	13.60	19.81	38.15
T	300	23.94	38.12	13.79	39.74	39.74	112.09
	500	31.00	31.75	13.07	34.24	36.73	95.35
	1000	44.12	21.59	10.80	23.75	29.80	66.11

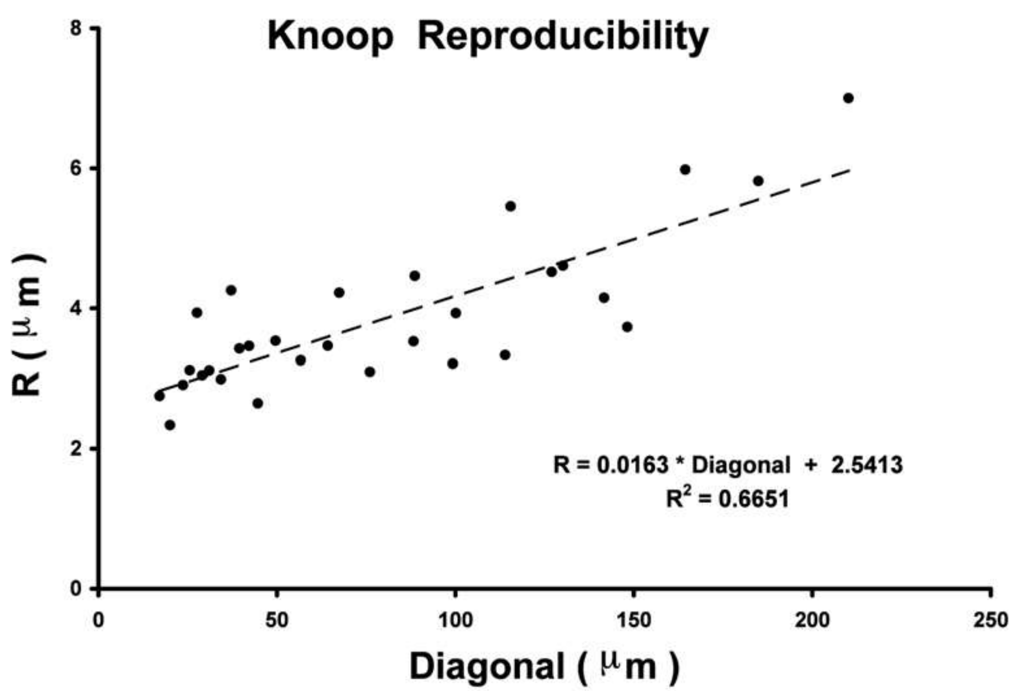

FIG. 4 The Relationship between Reproducibility (R) and Diagonal length (*d*) from Table 4 in µm units, for the Knoop Hardness Tests for Specimens B, C, D, E and T

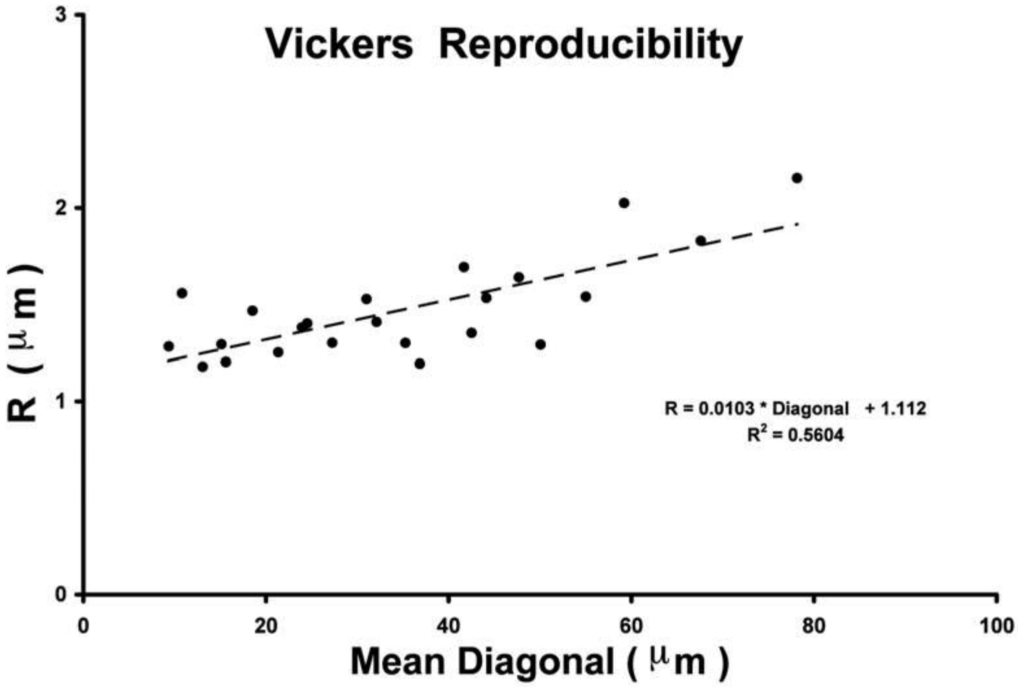

FIG. 5 The Relationship between Reproducibility and Diagonal length (*d*) from Table 5 in µm units, for the Vickers Hardness Tests for Specimens B, C, D, E and T

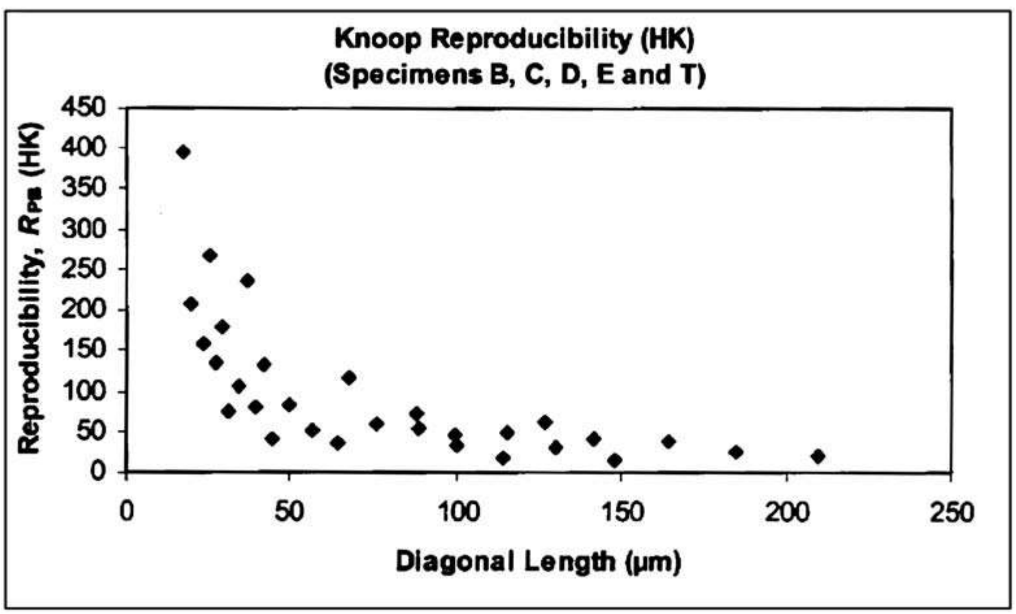

FIG. 6 The Relationship between Reproducibility (R) and Diagonal length (*d*) from Table 6 in HK units, for the Knoop Hardness Tests for Specimens B, C, D, E and T

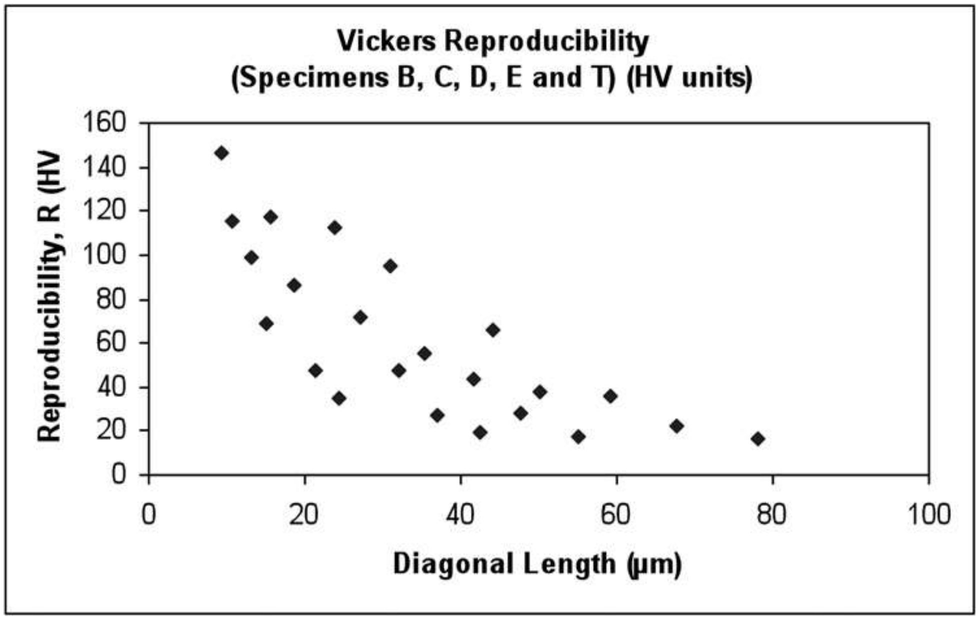

FIG. 7 The Relationship between Reproducibility (R) and Diagonal length (*d*) from Table 7 in HV units, for the Vickers Hardness Tests for Specimens B, C, D, E and T

TABLE 8 Vickers hardness at 1 kgf Test Force (HV)

Test Block Nominal Hardness (HV)	Average (HV)	Bias	Repeatability Standard Deviation (HV)	Reproducibility Standard Deviation (HV)	Repeatability Limit (HV)	Reproducibility Limit (HV)
	$\bar{X}$	%	s_r	s_R	r	R
200	209.2	N/A	4.1	7.1	11.5	19.9
400	413.8	N/A	8.1	15.6	22.8	43.7
800	812.9	N/A	21.8	21.8	61.1	61.1

TABLE 9 Vickers hardness at 5 kgf Test Force (HV)

Test Block Nominal Hardness (HV)	Average (HV)	Bias	Repeatability Standard Deviation (HV)	Reproducibility Standard Deviation (HV)	Repeatability Limit (HV)	Reproducibility Limit (HV)
	$\bar{X}$	%	s_r	s_R	r	R
200	199.0	N/A	1.7	5.2	4.7	14.5
400	421.8	N/A	4.8	7.3	13.3	20.5
800	828.0	N/A	8.9	19.5	25.0	54.6

TABLE 10 Vickers hardness at 10 kgf Test Force (HV)

Test Block Nominal Hardness (HV)	Average (HV)	Bias	Repeatability Standard Deviation (HV)	Reproducibility Standard Deviation (HV)	Repeatability Limit (HV)	Reproducibility Limit (HV)
	$\bar{X}$	%	s_r	s_R	r	R
200	198.1	N/A	2.1	3.0	6.0	8.5
400	398.5	N/A	2.9	9.1	8.2	25.4
800	800.2	N/A	2.3	11.7	6.6	32.7

TABLE 11 Vickers hardness at 20 kgf Test Force (HV)

Test Block Nominal Hardness (HV)	Average (HV)	Bias	Repeatability Standard Deviation (HV)	Reproducibility Standard Deviation (HV)	Repeatability Limit (HV)	Reproducibility Limit (HV)
	$\bar{X}$	%	s_r	s_R	r	R
200	197.2	N/A	1.8	3.5	4.9	9.9
400	415.7	N/A	2.5	5.1	7.0	14.2
800	811.5	N/A	8.3	16.6	23.3	46.6

TABLE 12 Vickers hardness at 50 kgf Test Force (HV)

Test Block Nominal Hardness (HV)	Average (HV)	Bias	Repeatability Standard Deviation (HV)	Reproducibility Standard Deviation (HV)	Repeatability Limit (HV)	Reproducibility Limit (HV)
	$\bar{X}$	%	s_r	s_R	r	R
200	191.2	N/A	0.5	1.5	1.4	4.3
400	399.9	N/A	1.1	2.0	3.1	5.7
800	814.4	N/A	2.8	12.0	7.7	33.6

ANNEXES

(Mandatory Information)

A1. VERIFICATION OF KNOOP AND VICKERS HARDNESS TESTING MACHINES AND INDENTERS

A1.1 Scope

A1.1.1 Annex A1 specifies three types of procedures for verifying Knoop and Vickers hardness testing machines: direct verification, indirect verification, and daily verification. This annex also contains geometric specifications for the indenter.

A1.1.2 Direct verification is a process for verifying that critical components of the hardness testing machine are within allowable tolerances by directly measuring the test forces, indentation measuring system, and testing cycle.

A1.1.3 Indirect verification is a process for periodically verifying the performance of the testing machine by means of standardized test blocks.

A1.1.4 The daily verification is a process for monitoring the performance of the testing machine between indirect verifications by means of standardized test blocks.

A1.2 General Requirements

A1.2.1 The testing machine shall be verified at specific instances and at periodic intervals as specified in Table A1.1, and when circumstances occur that may affect the performance of the testing machine.

A1.2.2 All instruments used to make measurements required by this Annex shall be calibrated traceable to national standards when a system of traceability exists, except as noted otherwise.

A1.2.3 Indirect verification of the testing machine shall be performed at the location where it will be used.

A1.2.4 Direct verification of newly manufactured or rebuilt testing machines may be performed at the place of manufacture, rebuild or the location of use.

TABLE A1.1 Verification Schedule for a Knoop and Vickers Hardness Testing Machine

Verification Procedure	Schedule
Direct Verification	When a testing machine is new, or when adjustments, modifications or repairs are made that could affect the application of the test forces or the measuring system. When a testing machine fails an indirect verification.
Indirect Verification	Shall be preformed following a direct verification before placing the tester in service. Shall be no longer than every 18 months. Recommended every 12 months. Recommended when a test machine is installed or moved.
Daily Verification	Required each day that the machine is used. Required whenever the machine is moved. Recommended whenever the indenter or test force is changed.

NOTE A1.1—It is recommended that the calibration agency that is used to conduct the verifications of Knoop or Vickers, hardness testing machines and indenters be accredited to the requirements of ISO/IEC 17025 (or an equivalent) by an accrediting an body recognized by the International Laboratory Accreditation Cooperation (ILAC) as operating to the requirements of ISO/IEC 17011.

A1.3 Direct Verification

A1.3.1 A direct verification of the testing machine shall be performed at specific instances in accordance with Table A1.1. The test forces, indentation measuring system, testing cycle, and indenters shall be verified as follows.

NOTE A1.2—Direct verification is a useful tool for determining the being offset by errors in another component.

A1.3.2 *Verification of the Test Forces*—For each Knoop and Vickers hardness scale, or both, that will be used, the corresponding test force shall be measured. The test forces shall be measured by means of a Class A elastic force measuring instrument having an accuracy of at least 0.25 %, as described in Practice E74.

A1.3.2.1 Make three measurements of each force. The forces shall be measured as they are applied during testing; however, longer dwell times are allowed when necessary to enable the measuring device to obtain accurate measurements.

A1.3.2.2 Each test force *P* shall meet the requirements specified in Table A1.2.

A1.3.3 *Verification of the Indentation Measuring System*—Each magnification of the measuring device used to determine the diagonal of the indentation shall be verified at five evenly spaced intervals over the working range by comparison with an accurate scale such as a stage micrometer. The accuracy of the certified line interval of the stage micrometer shall be 0.1 µm or 0.05 % of any interval, which ever is greater. Throughout the range covered, the difference between the reading of the device and of the stage shall not exceed 0.4 µm or 0.5 % , which ever is greater.

A1.3.4 *Verification of the Testing Cycle*—The testing machine shall be verified to be capable of meeting the testing cycle tolerances specified in 8.6. Direct verification of the testing cycle is to be verified by the testing machine manufacturer at the time of manufacture, or when the testing machine is returned to the manufacturer for repair, or when a problem with the testing cycle is suspected. Verification of the testing cycle is recommended but not required as part of the direct verification at other times.

NOTE A1.3—Instruments that have timing controlled by software or other nonadjustable components do not have to be verified providing that the design has been proven to produce the correct time cycles.

A1.3.5 *Verification of Indenters*—The geometry of each indenter shall be directly verified when new before placing into service. The device used to verify the indenter angles shall have a maximum uncertainty of 6 40 min. The indenter geometry tolerances are specified as follows:

A1.3.5.1 *Vickers Indenter*:

(1) The Vickers diamond indenter, see Fig. 1, used for standard testing and indirect verifications shall have face angles of 136° 0' 6 30'. As an alternate, the 136° face angles may be verified by measuring the angles between the opposite edges rather than the faces. When measured, the edge angles shall be 148° 6' 36" 6 45'. The edge angles shall be equally inclined to the axis of the indenter within 6 30'.

(2) The offset shall not exceed 1 µm when testing with test forces of 1 kgf and greater. When testing with forces less than 1 kgf the offset shall not exceed 0.5 µm.

NOTE A1.4—It is permissible to verify the offset by using a microscope with at least 5003 magnification to view an indentation created by the indenter and compare the offset length to a known dimension.

(3) The four faces of the diamond shall be equally inclined to the axis of the indenter to within 6 308

A1.3.5.2 *Knoop Indenter*:

(1) The Knoop diamond indenters (see Fig. 2, used for standard testing and indirect verifications shall have included longitudinal edge angle A of 172° 308 60.10° (6')

(2) The corresponding angle B = 130° must be contained within the dimensions listed in Table A1.3 and graphically as described by Fig. A1.1.

(3) The indenter constant (c_p) shall be 0.07028 within 6 1 % (0.06958 # c_p# 0.07098).

(4) The offset shall not be more than 1 µm in length for indentations greater than 15 µm in length, as shown in Fig. 2. For shorter indentations the offset should be proportionally less. (See Note A1.4.)

(5) The four faces of the diamond shall be equally inclined to the axis of the indenter to within 6 308.

A1.3.6 *Direct Verification Failure*—If any of the direct verifications fail the specified requirements, the testing machine shall not be used until it is adjusted or repaired. If the test forces, indentation measuring system or testing cycle may have been affected by an adjustment or repair, the affected components shall be verified again by a direct verification.

A1.3.7 *Indirect Verification*—Following a successful direct verification, an indirect verification according to A1.4 shall be performed.

A1.4 Indirect Verification

A1.4.1 An indirect verification of the testing machine shall be performed in accordance with the schedule given in Table A1.1. Indirect verifications may be required more frequently than stated in Table A1.1 and should be based on the usage of the testing machine.

A1.4.2 The testing machine shall be verified for each test force and for each indenter that will be used prior to the next indirect verification. Hardness tests made using Knoop or Vickers hardness scales that have not been verified within the schedule given in Table A1.1 do not meet this standard.

A1.4.3 Standardized test blocks used for the indirect verification shall meet the requirements of Annex A2.

NOTE A1.5—It is recognized that appropriate standardized test blocks are not available for all geometric shapes, materials, or hardness ranges.

A1.4.4 The indenter(s) to be used for the indirect verification shall meet the requirements of A1.3.5.

A1.4.5 *As-found Condition*—It is recommended that the as-found condition of the testing machine be assessed as part of an indirect verification. This is important for documenting the historical performance of the machine. This procedure should

TABLE A1.2 Accuracy of Applied Forces

TABLE A1.3 Angular Tolerances for Knoop Indenters

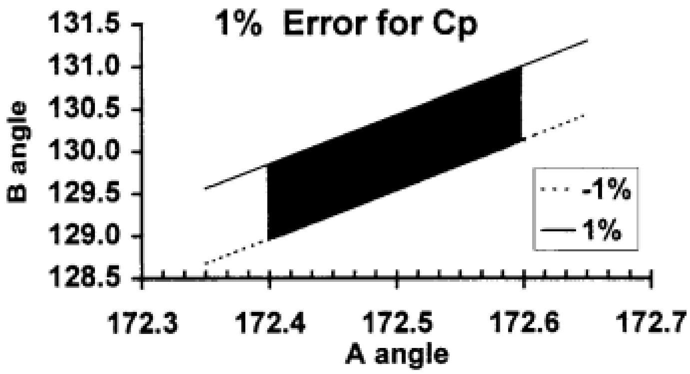

FIG. A1.1 Schematic Representing the Acceptable Regions of Knoop Indenter Angles

be conducted by the verification agency prior to any cleaning, maintenance, adjustments, or repairs.

A1.4.5.1 The as-found condition of the testing machine shall be determined with the user's indenter that is normally used with the testing machine. One or more standardized test blocks in the range of normal testing should be used for each Knoop or Vickers hardness scale that will undergo indirect verification.

A1.4.5.2 On each standardized test block, make at least three measurements distributed uniformly over the test surface. Let d_1, d_2, ..., d_n be the indentation diagonal measurement values, and $\bar{d}$ be the average of the measurements.

NOTE A1.6—When testing at low forces it may be necessary to increase the number of tests in order to obtain more consistent results.

A1.4.5.3 Determine the repeatability R and the error E in the performance of the testing machine for each standardized test block that is measured using Eq A1.1 and Eq A1.3 in section A1.7.

A1.4.5.4 The repeatability R and the error E should be within the tolerances of Table A1.5 or Table A1.6.

A1.4.5.5 If the calculated values of the repeatability R or the error E fall outside the specified tolerances, this is an indication that the hardness tests made since the last indirect verification may be suspect.

A1.4.6 *Cleaning and Maintenance*—Perform cleaning and routine maintenance of the testing machine when required in accordance with the manufacturer's specifications and instructions.

A1.4.7 *Indirect Verification Procedure*—The indirect verification procedure is designed to verify that for all of the Knoop and Vickers hardness scales to be used, each test force is being accurately applied, each indenter is correct, and the measuring device is calibrated correctly for the range of indentation sizes that these scales produce. This is accomplished by making hardness measurements on test blocks that have been calibrated for appropriate Knoop and Vickers hardness scales that employ each of the corresponding test forces.

TABLE A1.4 Hardness Ranges Used for Indirect Verification

Range	Knoop	Vickers
Low	< 250	< 240
Mid	250–650	240–600
High	> 650	> 600

A1.4.7.1 The testing machine shall be verified with the user's indenter(s) normally used for testing.

A1.4.7.2 A minimum of two standardized test blocks shall be used for the verification of the testing machine. The hardness values and hardness scales of the test blocks shall be chosen such that the following criteria are met:

A1.4.7.3 Each test force will be used.

A1.4.7.4 At least one hardness test block calibrated according to Annex A2, shall be used for each scale to be verified.

A1.4.7.5 At least two of the blocks shall be from different hardness ranges, low, mid or high hardness as specified in Table A1.4. The hardness difference between the two blocks used for verification shall be a minimum of 100 points. For example, if only one scale is to be verified, and one block having a hardness of 220 is used to verify the low range, then a block having a minimum hardness of 320 shall be used to verify the mid hardness range. See more examples below of the test blocks needed when performing multi-scale verifications.

A1.4.7.6 The highest test force shall be verified on a block from the lower of the chosen hardness ranges to produce the largest indentation size, and the lowest test force shall be used on the block from the higher of the chosen hardness ranges to produce the smallest indentation size. The two extremes of indentation size will verify the capability of the measuring device.

Example 1—A testing machine is to be verified for the HV 0.5 and HK 1 scales. Two test blocks are chosen for the verification: 450 HV 0.5 (mid-range) and 200 HK 1 (low-range). In this case, both of the test forces are verified by using only two blocks. The highest test force (1000 gf) is used on a low-range hardness block, and the lowest test force (500 gf) is used on a mid-range test block, which is the higher of the two hardness ranges.

Example 2—A testing machine is to be verified for the HK 0.1, HV 0.3 and HV 1 scales. Three test blocks are chosen for the verification: 720 HK 0.1 (high-range), 480 HV 0.3 (mid-range) and 180 HV 1 (low-range). In this case, there are three test forces that must be verified. The highest test force (1000 gf) is used on a low-range hardness block, and the lowest test force (100 gf) scale is used on the high-range test block. The middle test force (300 gf) scale could be used on either a low-range or mid-range test block.

Example 3– A testing machine is to be verified for the HV 0.5 and HV 1 scales. Two test blocks are chosen for the verification: 150 HV (low-range) and 450 HV (mid-range). In this case, both of the test forces are verified by using only two blocks. The highest test force (1000 gf) is used on a low-range hardness block, and the lowest test force (500 gf) is used on a mid-range test block, which is the higher of the two hardness ranges

Example 4– A testing machine is to be verified for the HV

TABLE A1.5 Repeatability and Error of Test Machines—Indirect Verification by Standardized Test Blocks Based on Measured Diagonal Lengths Using Test Forces 1000 gf and Less[A]

Hardness Range of Standardized Test Blocks		Force, gf	*R* Maximum Repeatability (%)	*E* Maximum Error (%)
Knoop	Vickers			
HK > 0	HV > 0	1 # P <100	13	3
HK < 100	HV < 100	100 # P # 1000	13	3
100 # HK # 250	100 # HV # 240	100 # P < 500	13	2
250 < HK # 650	240 < HV # 600		5	2
HK > 650	HV > 600		4	2
100 # HK # 250	100 # HV # 240	500 # P # 1000	8	2
250 < HK # 650	240 < HV # 600		4	2
HK > 650	HV > 600		3	2

[A] In all cases, the repeatability is the greater of the percentage given or 1 µm. The maximum error is the greater of the percentage given, or 0.5 µm.

TABLE A1.6 Repeatability and Error of Test Machines—Indirect Verification by Standardized Test Blocks Based on Measured Diagonal Lengths Using Test Forces greater than 1000 gf[A]

Hardness Range of Standardized Test Blocks	Force, gf	*R* Maximum Repeatability (%)	*E* Maximum Error (%)
# 100 to # 240	>1000	4	2
> 240 to # 600	>1000	3	2
>600	>1000	2	2

[A] In all cases, the repeatability is the greater of the percentage given or 1 µm. The maximum error is the greater of the percentage given, or 0.5 µm.

(5000 gf) is used on a low-range hardness block, and the lowest test force (1000 gf) scale is used on the high-range test block. The middle test force (3000 gf) scale could be used on either a low-range or mid-range test block.

A1.4.7.7 On each standardized test block, make five measurements distributed uniformly over the test surface. Let d_1, d_2, ..., d_5 be the five indentation diagonal measurement values, and $\bar{d}$ be the average of the five measurements. Determine the repeatability *R* and the error *E* in the performance of the testing machine using Eq A1.1 and Eq A1.3 in section A1.7, for each hardness level of each Knoop and Vickers hardness scale to be verified. The repeatability *R* and the error *E* shall be within the tolerances of Table A1.5 or Table A1.6.

A1.4.7.8 If the measurements of error *E* or repeatability *R* using the user's indenter fall outside of the specified tolerances, the indirect verification measurements may be repeated using a different indenter.

A1.4.7.9 The indirect verification shall be approved only when the testing machine measurements of repeatability and error meet the specified tolerances with the user's indenter.

A1.4.8 In cases where it is necessary to replace the indenter during the period between indirect verifications, the new indenter must be verified for use with the specific testing machine. The user shall perform the verification by following the as-found procedures given in A1.4.5. If the repeatability, *R*, and error, *E*, values fall within the tolerances in Table A1.5 or Table A1.6 the indenter can be used.

A1.4.9 When the combination of block hardness and test force produces indentations with diagonals less than 20 µm long, indirect verification using standardized test blocks is not recommended. In these situations, the indentation measurement error represents a significant proportion of the diagonal length. This can lead to substantial deviations in hardness from the stated value. Examples of these errors are contained in Section 10 and Tables 2 and 3. Also see Appendix X5, Recommendations for Light Force Microindentation Hardness Testing.

A1.5 Daily Verification

A1.5.1 The daily verification is intended as a tool for the user to monitor the performance of the testing machine between indirect verifications. At a minimum, the daily verification shall be performed in accordance with the schedule given in Table A1.1 for each Knoop and Vickers hardness scale that will be used. The daily procedure shall be preformed whenever the testing machine is moved.

A1.5.2 It is recommended that the daily verification procedures be performed whenever the indenter or test force is changed.

A1.5.3 *Daily Verification Procedures*—The procedures to use when performing a daily verification are as follows.

A1.5.3.1 At least one standardized test block that meets the requirements of Annex A2 shall be used for each hardness scale to be used. When test blocks are commercially available, the

A1.5.3.3 Before performing the daily verification tests, ensure that the testing machine is working freely, the stage and test block are clean, and the measuring device is properly adjusted and zeroed.

A1.5.3.4 Make at least three hardness measurements on each of the verification test blocks. The tests shall be distributed uniformly over the surface of the test blocks.

A1.5.3.5 Let $\bar{d}$ be the average of the measurements. Determine the error E in the performance of the testing machine using Eq A1.3 for each standardized test block that is measured.

A1.5.3.6 If the error E calculated for each test block is within the tolerances given in Table A1.5 or Table A1.6, the testing machine with the indenter may be regarded as performing satisfactorily.

A1.5.3.7 If the error E calculated for any of the test blocks is outside the tolerances, follow the manufacturers trouble shooting recommendations and repeat the test. If the average of the hardness measurements again falls outside of tolerances for any of the test blocks, an indirect verification shall be performed.

A1.5.3.8 Whenever a testing machine fails a daily verification, the hardness tests made since the last valid daily verification may be suspect.

NOTE A1.7—It is highly recommended that the results obtained from the daily verification testing be recorded using accepted Statistical Process Control techniques, such as, but not limited to, *X*-bar (measurement averages) and *R*-charts (measurement ranges), and histograms.

A1.6 Verification Report

A1.6.1 A verification report is required for direct and indirect verifications. A verification report is not required for a daily verification.

A1.6.2 The verification report shall be produced by the person performing the verification and include the following information when available as a result of the verification performed.

A1.6.2.1 Reference to this ASTM test method.

A1.6.2.2 Method of verification.

A1.6.2.3 Identification of the hardness testing machine and the indenters used.

A1.6.2.4 Means of verification (test blocks, elastic proving devices,etc.) with statements defining traceability to a national standard.

A1.6.2.5 The Knoop and Vickers hardness scale(s) verified.

A1.6.2.6 The individual or calculated results used to determine whether the testing machine meets the requirements of the verification performed. Measurements made to determine the as-found condition of the testing machine shall be included whenever they are made.

A1.6.2.7 Description of adjustments or maintenance done to the testing machine.

A1.6.2.8 Date of verification and reference to the verifying agency or department.

A1.6.2.9 Signature of the person performing the verification.

A1.7 Example Calculations of Repeatability and Error

A1.7.1 *Repeatability of Knoop and Vickers Hardness Testers*:

A1.7.1.1 Repeatability, *R*, of the tester (%) is calculated by the following equation:

$$R = 100 \left(\frac{d_{max} - d_{min}}{\bar{d}} \right) \tag{A1.1}$$

where

d_{max} = is the longest of the five diagonals (or mean diagonals),
d_{min} = is the shortest of the five diagonals, and
$\bar{d}$ = is the mean diagonal length.

The repeatability is acceptable if it meets the requirements given in Table A1.5 or Table A1.6.

A1.7.1.2 The following is an example of a repeatability calculation. Assume that five Knoop indentations were made on a test block with a nominal hardness of 420 HK at the certified block test force of 300 gf and that the five readings are d_1 = 103.9, d_2 = 104.8, d_3 = 102.3, d_4 = 102.8 and d_5 = 100.2 µm, respectively. Therefore, $d_{max} - d_{min}$ = 104.8 – 100.2 = 4.6 µm and R = 100(4.6)/102.8 = 4.47 %. According to Table A1.5, the repeatability for a test block with a hardness >250 to 650 HK should be $\leq$5 %. In this example, the tester met the repeatability requirement for this hardness test block and force. However, if these diagonals had been obtained using a test block with a nominal hardness of 700 HK and a certified test force of 300 gf, then the repeatability would be inadequate as Table A1.5 requires $R \leq$ 4 % for a hardness >650 HK.

A1.7.2 *Error of Knoop and Vickers Hardness Testers*:

A1.7.2.1 The error, *E*, of the machine is:

$$E = \bar{d} - d_s \tag{A1.2}$$

The percent error, %*E*, is calculated by the following equation:

$$\%E = 100 \left(\frac{\bar{d} - d_s}{d_s} \right) \tag{A1.3}$$

Where:

$\bar{d}$ = is the measured mean diagonal length in µm, and
d_s = is the reported certified mean diagonal length, µm.

A1.7.2.2 The error between the certified mean diagonal and the measured mean diagonal shall not exceed the tolerances in Table A1.5, or ± 0.5 µm, whichever is greater.

A1.7.2.3 The following is an example of an error calculation based on the data given in A1.7.1.2, and a certified mean diagonal length for the test block, d_s, of 100.8 µm (420 HK 300gf). Since $\bar{d}$ = 102.8 µm, ($\bar{d} - d_s$) = 102.8 – 100.8 = 2.0 µm. Thus, $E \cong$ 1.98 %. In this case, the percent error meets the maximum of ± 2 %, which is greater than ± 0.5 µm