MECHANICAL AND CORROSION BEHAVIOR OF IN-SITU PARTICULATES (Al_2O_3, TiB_2, TiC) REINFORCED Fe-Al INTERMETALLIC COMPOSITES

by

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LIST OF ABBREVIATION

MMCs	:	Metal matrix composites
PMCs	:	Polymer matrix composites
CMCs	:	Ceramic matrix composites
IMCs	:	Intermetallic matrix composites
LMMCs	:	Light metal matrix composites
FML	:	Fibre metal laminate
PCA	:	Process control agent
PDS	:	Pulse discharge sintering
HIP	:	Hot isostatic pressing
MA	:	Mechanical alloying
MM	:	Mechanical milling
ODS	:	Oxide dispersed strengthening
APB	:	Antiphase boundaries
rpm	:	Round per minute
SEM	:	Scanning electron microscope
XRD	:	X-ray diffraction
μm	:	Micrometer

KELAKUAN MEKANIKAL DAN KAKISAN KOMPOSIT INTERMETALIK Fe-Al DIPERKUAT OLEH PARTIKEL *IN SITU* (Al_2O_3, TiB_2, TiC).

ABSTRAK

Tujuan kajian ini adalah untuk menghasilkan komposit antara logam ferum aluminida dengan campuran partikel (Al_2O_3, TiB_2, TiC) secara *in situ*. Kaedah metalurgi serbuk telah digunakan untuk menghasilkan larutan pepejal ferum aluminida sebagai matriks dengan menggunakan kaedah pengaloian mekanik. Kajian ini menunjukkan pengaloian mekanikal memainkan peranan penting dalam pembentukan larutan pepejal dan seterusnya mempengaruhi sifat mekanik komposit. Keadaan optimum untuk menghasilkan komposit ferum aluminida adalah seperti berikut: (1) masa pengisaran ialah 10 jam pada kelajuan 300 rpm, (2) pemadatan dengan menggunakan tekanan hidraulik pada 350 MPa dan (3) suhu persinteran pada 1300 ^oC selama 1 jam. Ferum aluminida matriks membentuk Fe_3Al atau FeAl bergantung kepada kepekatan Al. Partikel (Al_2O_3, TiB_2, TiC) yang terbentuk secara *in situ* semasa persinteran adalah berbentuk partikel yang halus dengan saiz partikel 2 μm, 1μm dan 3μm masing-masing, dan bertabur secara seragam di dalam matriks. Ujian kekerasan micro-Vickers dan kekuatan tegangan secara mampatan diametral bagi sampel *in situ* menunjukkan peningkatan kekuatan berbanding dengan sampel *ex situ*. Ujian kekerasan dan kekuatan komposit ferum aluminida dengan campuran partikel TiB_2 *in situ* ialah 15.42 GPa dan 1253.5 MPa masing-masingnya, dan nilai-nilai ini lebih tinggi berbanding dengan besi alatan diperkuat, iaitu 8.9 GPa dan 309 MPa. Keputusan ini menunjukkan bahawa kepada komposit ferum aluminida diperkuat partikel TiB_2 *in situ* berpotensi untuk menggantikan besi alatan dengan kelebihan ia mempunyai ketumpatan yang lebih rendah dan seterusnya nisbah kekuatan kepada berat yang tinggi. Nilai kerintangan haus adalah berkadar langsung dengan nilai kekerasan, dan didapati nilai

kerintangan haus bagi komposit ferum aluminida diperkuat TiB2 adalah lebih baik dari besi alatan diperkuat. Berdasarkan keputusan kepolaran potentiodinamik, kadar kakisan komposit ferum aluminida bertambah dengan penambahan campuran partikel *in situ*. Secara keseluruhan, kadar kakisan komposit ferum aluminida adalah lebih tinggi daripada besi alatan diperkuat.

MECHANICAL AND CORROSION BEHAVIOR OF *IN SITU* PARTICULATE (Al_2O_3, TiB_2, TiC) REINFORCED Fe-Al INTERMETALLIC COMPOSITES

ABSTRACT

The purpose of this study is to synthesize iron aluminide intermetallic composite with *in situ* particulates (Al_2O_3, TiB_2, TiC). Powder metallurgy method was used in this work to produce iron aluminide solid solution as matrix by mechanical alloying. It was found that mechanical alloying process plays a significant role in solid solution formation and later on the mechanical properties. The optimum conditions to produce iron aluminide composite in this particular work are: (1) milling time is 10 h at speed of 300 rpm, (2) consolidation by hydraulic press at 350 MPa and (3) sintering at 1300 ^oC for 1 h. Matrix of iron aluminide can form Fe_3Al or FeAl depending on the Al concentration. The *in situ* particulate of Al_2O_3, TiB_2 and TiC were formed during sintering with average particles size of 2 μm, 1μm and 3μm, respectively, and they were uniformly distributed in the matrix. The micro Vickers hardness and tensile strength by diametral compression test showed that the strength improved compared to the *ex situ* samples. The hardness and ultimate tensile strength of iron aluminide composite with *in situ* TiB_2 were 15.42 GPa and 1253.5 MPa, respectively and showed higher value compared to tool steel of 8.9 GPa and 309 MPa, respectively. Hence, this can be a promising candidate to replace steel with the advantage of much lower density and this will translate to high strength to weight ratio. Wear resistance was proportional to hardness and, compared to tool steel hardened, composite with TiB_2 reinforcement has better wear resistance. Comparison to tool steel was made as this composite can be used in applications where hardness and wear resistance is of importance. Corrosion behavior revealed in terms of potentiodynamic polarization scan of iron aluminide composite

showed an increasing corrosion rate with increasing amount of *in situ* particulate and the rate is higher than tool steel.

CHAPTER 1

INTRODUCTION

1.1 Introduction

By the broadest definition, a composite material is one in which two or more materials that are different are combined to form a single structure with an identifiable interface. The possibility of combining various material systems (metal – ceramic – polymer) gives the opportunity for unlimited variation. The properties of these new materials are basically determined by the properties of each single component and the composition of the mixture using what is known as "Rule of Mixture".

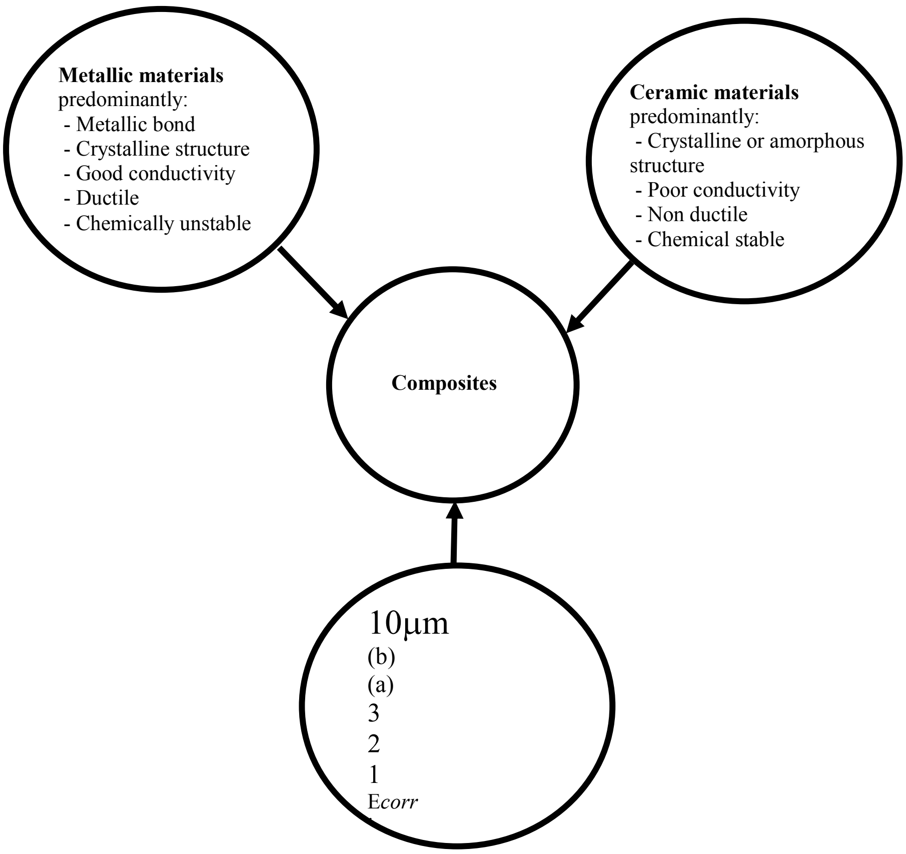

Figure 1.1 Classification of composites materials within the group of materials. (Daniel *et al.*, 2001).

Figure 1.1 shows the allocation of the composite materials into various groups of materials. The composite generally has characteristics better than or different from those of the elemental components. The matrix phase is the continuous phase, while the distributed phase, commonly called the reinforcement phase can be in the form of particles, whiskers or short fiber, continuous fiber or sheet. It is convenient to classify different types of composites as per the matrix materials characteristics: polymer matrix composites (PMCs), metal matrix composites (MMCs), and ceramic matrix composites (CMCs). The reinforcement in any matrix can be polymeric, metallic or ceramic. Polymeric matrix composites containing reinforcement fibers such as carbon, glass or aramid are quite commonly used as engineering materials. Metal containing ceramic particles, whiskers or fiber (short or long) are also gaining in importance. The ceramic matrix composites are the newest entrants in the composite field.

Metal matrix composites (MMCs) represent an alternative to conventional materials for the production of high performance materials. The materials can be manufactured in such a way as to exhibit a combination of the characteristics of the metallic matrix and the reinforcement phase. For many researchers the term metal matrix composites is often equated with the term light metal matrix composites. During the last number of years, confidence in using composite materials has increased dramatically, with great innovation in manufacturing, assembly, and repair method development. In transportation engineering, especially in the automotive and aerospace industry, MMCs have been used commercially in fibre reinforced pistons and aluminum crank cases with strengthened cylinder surfaces as well as particle-strengthened brake disks. The B-2 bomber, shown in Figure 1.2, is constructed using an even higher percentage of composites, as are current helicopters and vertical lift designs. Additionally, where metal alloys (steel, copper bronze, etc.) have isotropic characteristics (the same properties in all directions), composites can have very selective directional properties to meet specific application needs. Thus, composites are typically highly engineered materials targeted at specific applications.

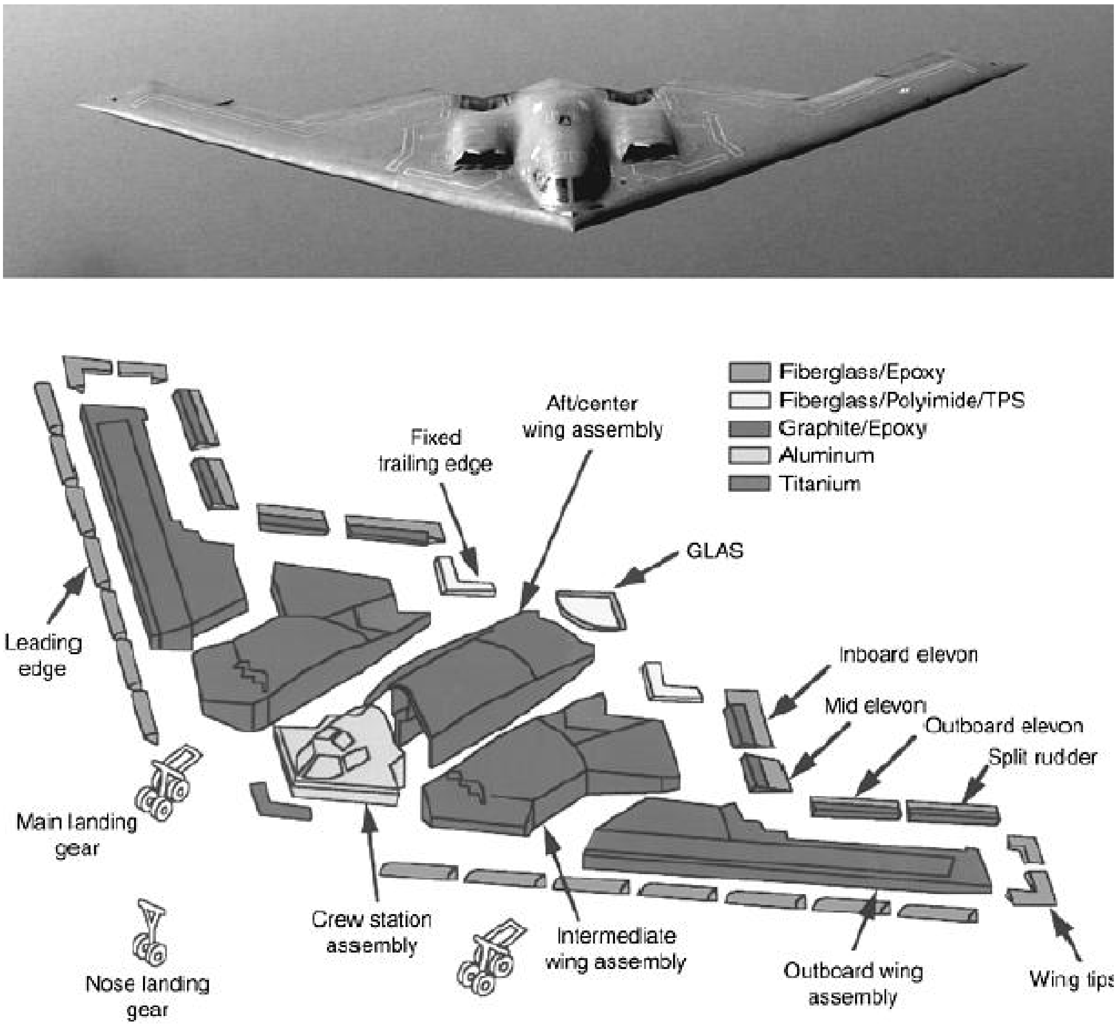

Figure 1.2 The US air force B-2 advanced “stealth” bomber (Daniel *et al.*, 2001).

In order to compete with other method, low production cost is important. If the manufacturer can control production routs and exploit the specific advantages of MMCs, new markets and opportunities will arise. A better understanding of MMCs, especially the iron aluminide based ones, will give advantages and feasibility to industrial side. Both time and money can be saved if the manufacturing processes of new components and ideas can be exploited carefully before.

1.2 Problem statement

Iron aluminide matrix composites based on Fe_3Al and FeAl are promising candidates for many structural applications due to the combination of low cost, low density, excellent oxidation and sulfidation resistance. In fact, since several decades ago ordered intermetallics based on aluminides of transition metals such as iron, nickel, niobium, titanium and cobalt have been evaluated for their potential as high temperature structural materials. The amount of aluminum in these alloys exceeds that of used in conventional alloys and can range from 10 to 30 %wt. This concentration of aluminum allows the formation of an impervious oxide layer which is responsible for the excellent oxidation, sulfidation and carburization resistance at temperatures of 1000 °C and higher (Subramanian *et al.*, 1998). Among these interesting candidates, iron aluminides are potentially less expensive as they contain no nickel or titanium and only minor amounts of other alloying elements.

In the present work, iron aluminide composites were synthesized by mechanical alloying process, characterized by repeated welding and fracturing of powder particles to form intermetallic phase, and then solid state sintering allows the reinforcement to be finely distributed which can increase the strength of composites.

Recent interest of processes developed to incorporate reinforcing particles into iron aluminide matrix includes an *in situ* technique in which a thermodynamically stable reinforcing phase is produced during processing. This technique can be the most economical on synthesis of composites besides offering an advantage of the ability to avoid contamination with oxidation films or other detrimental surface reactions on the surfaces of reinforcement (Ko *et at.*, 1999).

However, with increasing Al concentration, intrinsic grain boundary weakness further limits the tensile ductility of iron aluminides. Therefore in this work, three compositions are Fe-30 %at. Al, Fe-35 %at. Al and Fe-40 %at. Al were selected as matrix (hereafter referred as Fe30Al, Fe35Al and Fe40Al). The reinforcement of TiB_2, TiC and Al_2O_3 were added as hard ceramic particles. The mechanical properties and corrosion behavior will be evaluated in terms of different matrix compositions and amount of particulates.

In order for the composites to be used as structural materials, their mechanical properties have to be evaluated, especially strength. The composites are also expected to have high hardness and excellent wear resistance, hence suggesting their use in wear related applications. For both structural and wear applications, steel has been used for decades due to the low cost and excellent mechanical properties. Thus, in this work, results of hardness and wear behavior will be compared to that of tool steel hardened. Corrosion studies will also be conducted as the material has to be able to sustain environmental attack over the period of use.

1.3 Objectives of research

This study is focused on certain variables such as mechanical alloying time, sintering parameters (sintering temperature, sintering time and amount of reinforcements). Therefore, the objectives of this research are:

1. To investigate the optimum parameters (mechanical alloying time, sintering temperature and sintering time) to form iron aluminide composites.
2. To fabricate *in situ* TiB_2, Al_2O_3 and TiC particulate reinforced iron aluminide composites and study the mechanism of phase formation of Fe_3Al and FeAl upon the mechanical alloying and sintering.
3. To characterize the mechanical properties of iron aluminide composites Fe30Al, Fe35Al and Fe40Al with different amounts of reinforcement.
4. To study the corrosion behavior in 3.5 M NaCl of iron aluminide Fe30Al, Fe35Al and Fe40Al composite with different amounts of reinforcement.

1.4 Research scope

In general, the research is divided into two parts which will be described in more detail in Chapter 3. The first part is the synthesis of iron aluminide matrix Fe30Al, Fe35Al and Fe40Al using mechanical alloying process, subsequent consolidation and solid state sintering. The effects of parameters (mechanical alloying time, sintering time and sintering temperature) were studied. The characterization of the sample powders were carried out using XRD, SEM and the density were measured.

The second part is the characterization of iron aluminide composites with *in situ* reinforcements. In this part, micro Vickers hardness, compressive strength wear resistance and corrosion behaviors were explored to understand its mechanical properties. The full detail of methodology will be described in Chapter 3.

CHAPTER 2

LITERATURE REVIEW

2.1 Metal matrix composite

2.1.1 Introduction

Metal matrix composites (MMCs) materials have increasingly been used in many areas of daily life. Materials like cast iron with graphite or steel with a high carbide content, as well as tungsten carbide, consisting of carbide and metallic binder, also belong to this group of composite materials. For many researchers, the term metal matrix composites is often equated with the term light metal matrix composites (LMMCs). Substantial progress in the development of MMCs has been achieved in recent decades, so that they could be introduced into most important applications. In traffic engineering, especially in the automotive industry, MMCs have been used commercially in fiber reinforced piston and aluminum crank case with strengthened cylinder surface as well as particle strengthened brake disk.

These innovative materials open up unlimited possibilities for modern material science and development; the characteristics of MMCs can be designed into the material, custom-made, depending on the application. Thus, metal matrix composites potentially fulfill all the desired conceptions of the designer. This material group becomes interesting for use as constructional and functional materials. However, the technology of MMCs is in competition with other modern material technologies, for example powder metallurgy. The advantages of the composite materials are only realized when there is a reasonable cost–performance relationship in the component production. The use of a composite material is obligatory if a special properties profile can only be achieved by application of these materials.

The possibility of combining various material systems (metal – ceramic – non-metal) gives the opportunity for unlimited variation. The properties of these new materials are basically determined by the properties of their single components.

The reinforcement of metals can have many different objectives. The reinforcement of light metals opens up the possibility of using these materials in areas where weight reduction has first priority. The precondition here is that the properties of the component has to be improved. The objectives for developing light metal composite materials are:

- Increase in yield strength and tensile strength at room temperature and above while maintaining the minimum ductility or rather toughness,
- Increase in creep resistance at higher temperatures compared to that of conventional alloys,
- Increase in fatigue strength, especially at higher temperatures,
- Improvement of thermal shock resistance,
- Improvement of corrosion resistance,
- Increase in Young's modulus,
- Reduction of thermal elongation.

To summarize, an improvement in the weight specific properties can be obtained, offering the possibilities of extending the application area, substituting the common materials and optimization of component properties. With functional materials, the appropriate function of the material must be maintained.

Although increasing development activities have led to system solutions using metal composite materials, the use of especially innovative systems, particularly in the area of light metals, has not been realised. The reason for this is insufficient process stability and reliability, combined with production and processing problems and inadequate economic efficiency. Application areas, like traffic engineering, are very cost orientated and conservative, and the industry is not willing to pay additional costs for the use of such materials Krainer (2006). For all these reasons metal matrix composites are only at the beginning of the evolution curve of modern materials as showed in Figure 2.1.

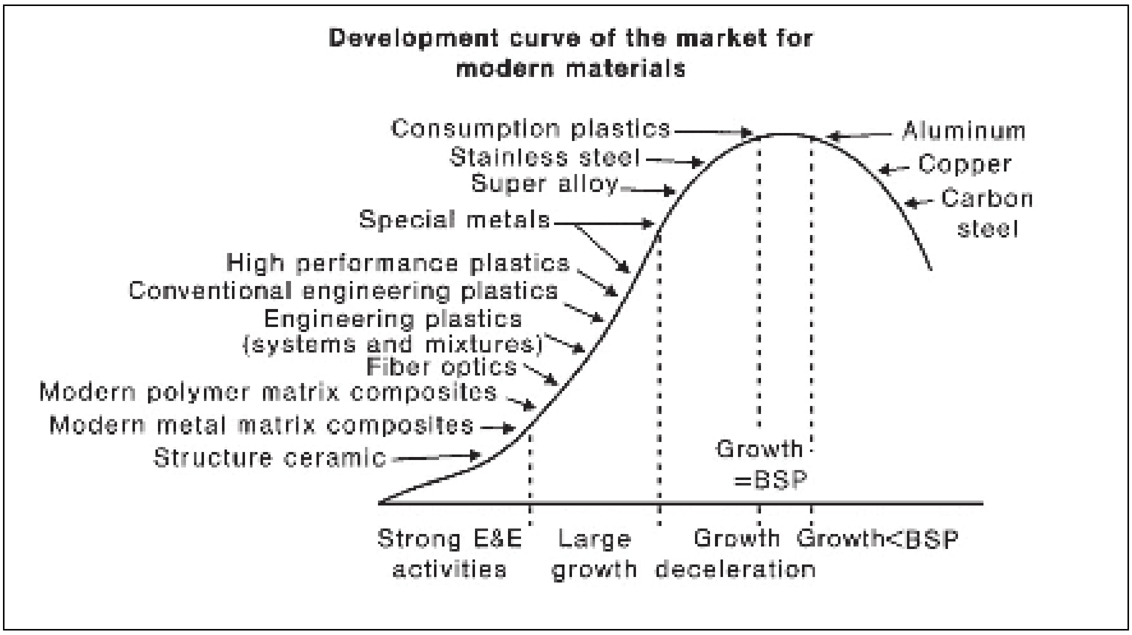

Figure 2.1 Development curve of the market for modern materials (Krainer, 2006)

2.1.2 Classification of metal matrix composites

Metal matrix composites can be classified in various ways. One classification is the consideration of type and contribution of reinforcement components in particle, fiber and structural reinforced as shown in Figure 2.2.

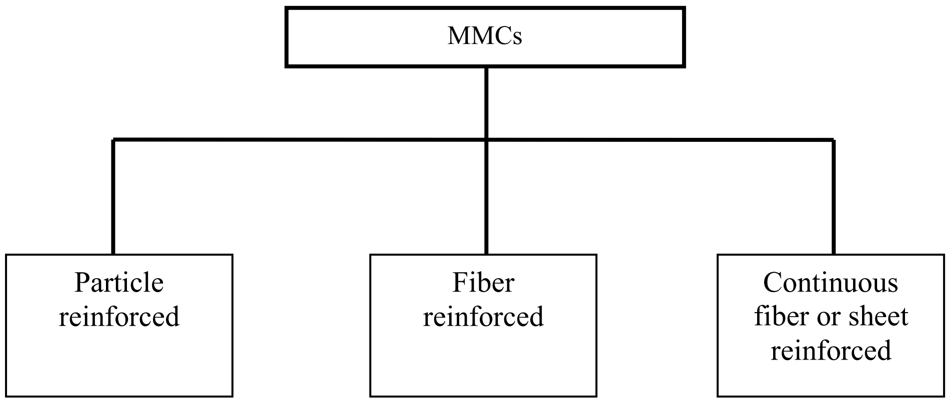

Figure 2.2 Different types of MMCs (Krainer, 2006)

2.1.2.1 Particle reinforced composite

The term particle or discontinuous naturally refers to the reinforcement that is in the shape of short fibers, whiskers or particles. Particulate MMCs are of special interest as their raw materials are inexpensive, and many fabrication methods can be used, making them suitable for application requiring relatively high volume production (Everett, 1991). Furthermore, to improve properties of particulate MMCs, addition of hard ceramic particles is required. Particle-matrix interactions occur at the atomic or molecular level for dispersion strengthened composites (Everett, 1991).

Usually, small particles size are used in the range of 10-1000 nm. Examples of particle reinforcement are SiC, Al_2O_3, TiC, TiN and WC. These reinforcing particles tend to restrain movement of the matrix phase in the vicinity of each particle, therefore, the matrix transfers some of the applied stress to the particles, which bear a fraction of the load. However, the matrix bears the major portion of an applied load while the small dispersed particles prevent the motion of dislocation. This resulted in restriction of plastic deformation, causing in an increment in the yield and tensile strengths, as well as hardness improved.

According to Tjong *et al.* (2008), the mechanical properties of discontinuously reinforced titanium matrix composite are mainly dependent upon the composition or microstructure of matrix, shape and volume content of reinforcement and matrix-reinforcement interface. The incorporation of discontinuous ceramic reinforcements to titanium and its alloys increase the tensile strength and stiffness of the materials at the expense of tensile ductility and fracture toughness at ambient temperature. TiC_p/Ti-6Al-4V (particulate) and TiB_w/Ti (whisker) composite have been used in their investigation as shown in Figure 2.3. The strengthening effect of the reinforcement is retained at elevated temperatures.

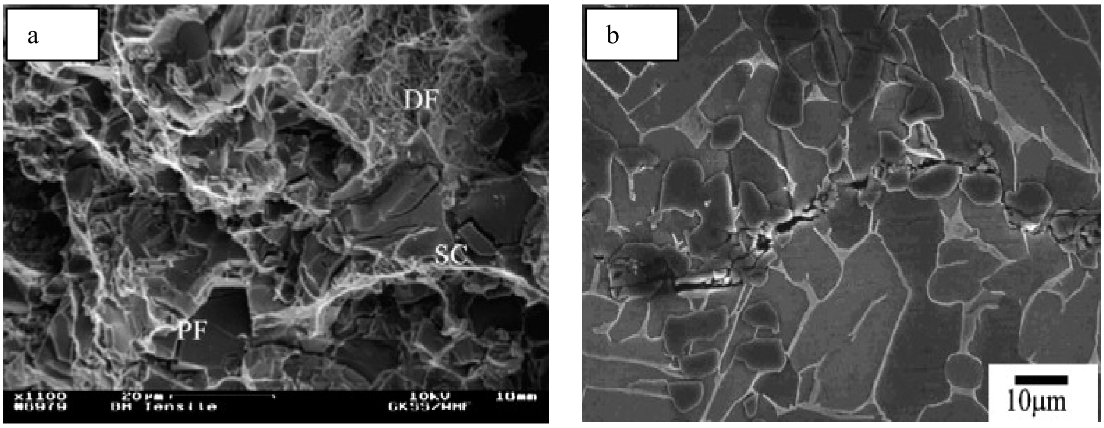

Figure 2.3 (a) SEM fractographs showing tensile fracture surface 10 %wt. TiC_p/Ti–6Al–4V composite at room temperature. DF: ductile fracture; PF: particle fracture; SC: secondary cracking, (b) SEM micrograph showing fracture of TiC particles on a polished surface of tensile specimen of *in situ* 10 %wt. TiC_p/Ti–6Al–4V composite. (Tjong *et al.*, 2008)

However, particulate MMCs have lower ductility, toughness and low-cycle fatigue properties in comparison to the unreinforced alloys, limiting their usefulness in practice.

2.1.2.2 Fiber reinforced composite

A fiber reinforcement can be described as an elongated material having more or less uniform diameter or thickness of less than 250 μm and an aspect ratio of more than 100 (Krainer, 2006). The basic requirements of fiber reinforcement are high strength and high modulus of elasticity in combination with low density, which determines their high specific values of strength and rigidity. In addition, the melting temperature of the fiber should be higher than that of the matrix alloy and the fiber have to maintain their properties at elevated temperature.

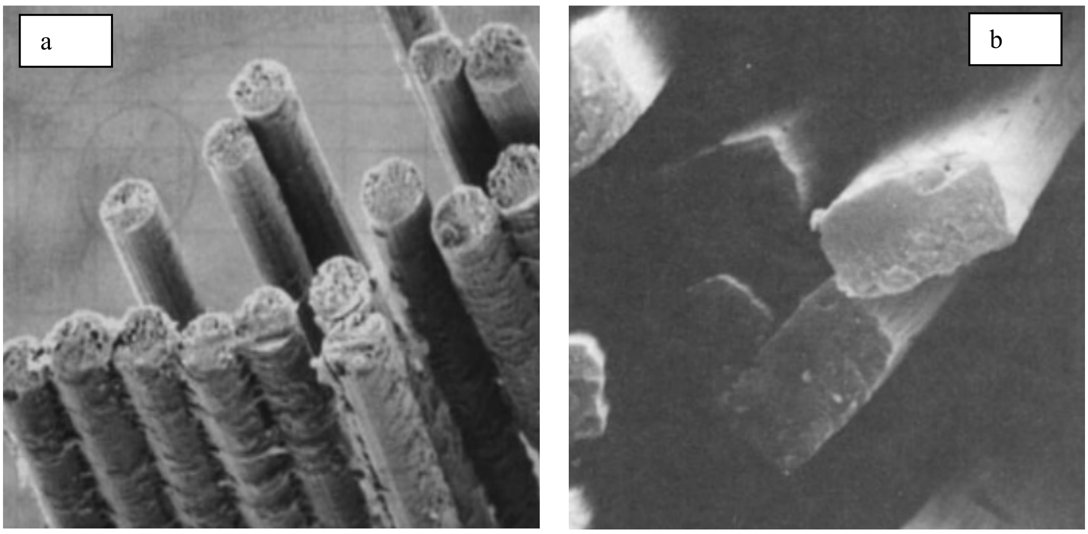

Figure 2.4 (a) Round tar-based fibers (b) Rectangular shape PAN-based fibers (Krainer, 2006)

At certain direction, the fiber are embedded into the matrix and resulted in anisotrophic behavior and affect the strength of composite directly. The most widely used at present are fibers of oxide, carbon, carbide, silicon or boron with various shapes as shown in Figure 2.4.

2.1.2.3 Structural reinforced composite

A structural composite is normally composed of both homogenous layers and cores, the properties of which depend not only on the properties of the constituent materials but also on the geometrical design of the various structural elements. The most common structural composite is aligned in laminar and sandwich like structure. Each of the layers is stacked up together where the orientation of high-strength direction varies for each successive layer.

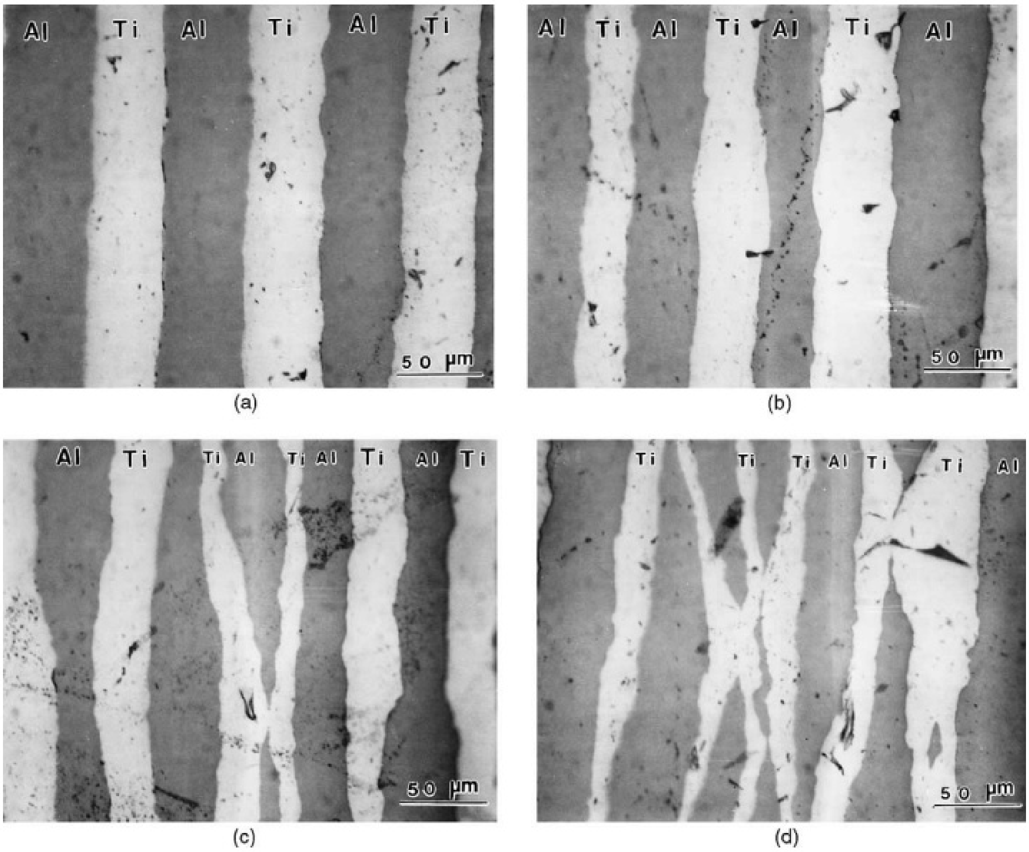

Figure 2.5 Light micrographs of the Ti/Al multi-layered composites in the as-cold roll bonded condition: (a) 40%, (b) 50%, (c) 72% and (d) 80% reduction (Chaudhari *et al.*, 2009).

Figure 2.5 showed morphologies of the multi-layered Ti/Al composites that were rolled to 40, 50, 72 and 80% reduction in thickness, which revealed that good bonding achieved. Alternating layers of titanium and aluminum foils were stacked one on top of the other to form a "sandwich" of titanium and aluminum.

When using continuous and oriented reinforcement fiber, the properties of material of the single layer is anisotropic. Nevertheless, if the stacking sequence and the orientation angles of all layers are well chosen, the resulting laminate can show quasi-isotropic material properties.

2.1.3 Metal matrix composites fabrication methods

There are varieties of fabrication methods that have been employed for MMCs over the years but most of these can be conveniently classified into the following categories:

- Solid state
- Liquid state
- Deposition
- *In situ*

2.1.3.1 Solid state processing

One of the solid state processing involves bringing the particles or foils into close contact with the reinforcement, with the application of a suitable combination of temperature and pressure. The method using foils is usually called diffusion bonding (Matthews and Rawlings, 1994).

The first stage in diffusion bonding is to sandwich a fibre mat, where the fibres are held in place by a polymer binder, between two sheets of foil to form a ply, as shown in Figure 2.6. In some cases, this is followed by consolidation of the ply. The plies are cut and stacked in the required sequence. After that, the stack is hot pressed in a die to form the component.

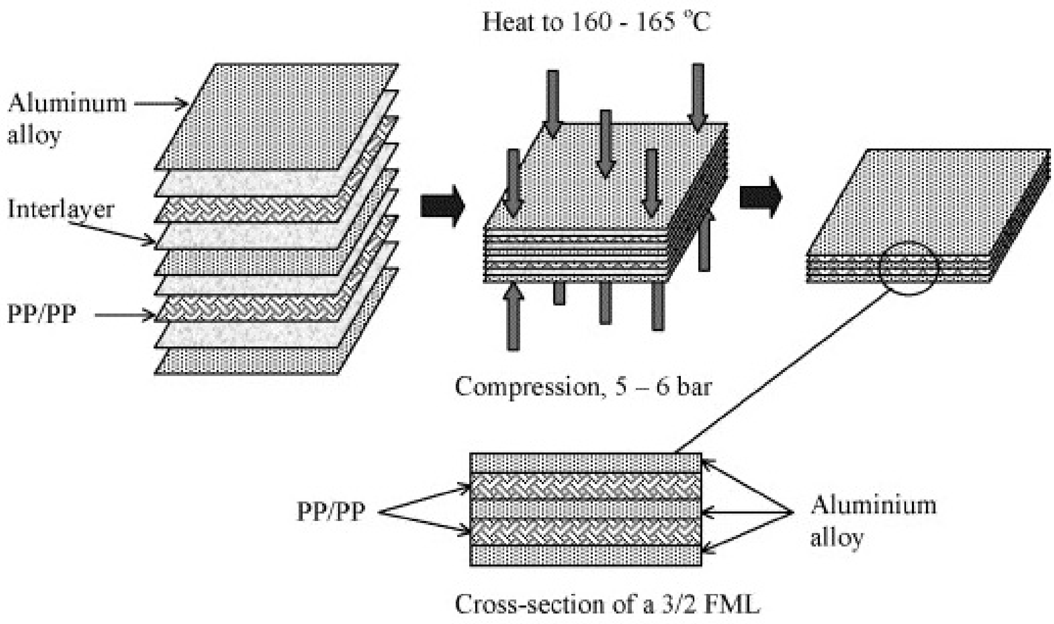

Figure 2.6 A metal matrix composite produced by the diffusion bonding method (Abdullah *et al.*, 2005).

Abdullah *et al.* (2005) produced polypropylene-based fibre–metal laminate (FML). The fibre–metal laminates were manufactured by stacking the appropriate number of composite plies, aluminium sheets and interlayer films in a picture-frame mould and placing the stack in a press. The laminates were then heated to 165 °C at a rate of approximately 10 °C/min before being cooled to room temperature at a rate of approximately 5 °C/min as shown in Figure 2.6.

Mechanical alloying (MA) is one of the solid state processes to produce metal matrix composites. MA is a solid state powder processing method which involves repeated cold welding and fracture of particles as a result of high energy ball-powders collisions. This process is normally carried out under inert atmosphere to prevent oxidation. This method, also known together with the *in situ* processing, which was used in the formation of Fe_3Al phase was introduced by Ko *et al.* (1999) and formation of particulate phases such as TiC, TiB_2, Al_2O_3, etc. (Subramanian *et al.*, 1998; Krasnowski *et al.*, 2002; Koch, 1998)

For instance, fabrication of TiC/Ti composite material using mechanical alloying was performed using starting reactant materials of pure Ti and carbon powders. A mixture of desired average composition was milled under argon atmosphere with ball to powder weight ratio of 10:1 (Sheriff, 2001). The milling process was carried out at room temperature using high energy ball mill with different milling time. After milling, the as-milled powder consists of two phases: unreacted metallic Ti and fully reacted TiC. After consolidation which took place at 1880^{o}C [just above the melting point of Ti (1870 ^{o}C) and far below the melting point of TiC (3100 ^{o}C)], the particles of TiC in the mixture powders are embedded in the molten Ti matrix to form composite Ti/TiC compact (Sherif, 2001).

In a research work carried out by Froyen *et al.*, 2003, TiB/Ti composite were fabricated by a self-propagating high temperature synthesis connected with hot isostatic pressing (HIP). Self-propagating high temperature synthesis (SHS) is a process which takes advantage of the extreme heat generated by the exothermic reaction of following reactions during processing:

$$(1 + x)Ti + B \rightarrow TiB + (x)\ Ti \qquad (2.1)$$

Several TiB/Ti reactant mixtures of different metal content (x=0, 20, 30, 40, 50, 60 %wt.) were tested. The reactants were milled in a stainless steel pot for 24 hours. The reactants were dried for 24 hours in a dryer. Then the reactants were cold pressed into square pellets by a uniaxial single acting. A heated coil is applied to heat the pellets. A heated coil at one end ignited the pellets and self-sustained combustion wave propagates from this heated end to the full sample due to the highly exothermic reaction. The compact was immediately pressed just after combustion waves passed and the products were still in the hot and plastic condition.

2.1.3.2 Liquid state processing

There are three major liquid infiltration methods to produce metal matrix composites. Casting adapted from the conventional technique was used to fabricate metal composite but problems of this technique are non-wetting of reinforcement and matrix reaction due to the high temperature involved. Various approaches are being pursued to overcome these problems such as adding an additive substance to improve wetting ability between the matrix and reinforcement and applying melt stirring method. In recent years, many researchers used external pressure during processing: spontaneous infiltration, squeeze casting, and gas pressure infiltration.

For example: carbon fiber composite base Al, the liquid state processing is the best way to fabricate these kinds of composites. The 2014 Al alloy was used as a matrix to fill in fibers preform Ni-coated continuous carbon fibre. The processing of the composite was carried out by gas pressure infiltration method. It was observed that the fibers were uniformly distributed in the aluminum alloy matrix and there is no sign of cluster fibre or residual porosity (Daoud, 2004). Schneibel *et al.* (1997)

also reported that $TiC/ZrB_2/TiB_2$ particles reinforced in iron aluminide FeAl matrix composite were produced by liquid phase processing.

Metal matrix composites reinforced by fibres also can be fabricated by low-pressure infiltration process (Mizumoto *et al.*, 2005). In their research Al-Cu alloy, Al-Si alloy and Mg-Al-Zn (AZ91) were used as matrix. The preform was made by SiC fiber, aluminum particles and organic binder formed in a cylindrical shape preform and was fired at 500 ^{o}C to remove binder. However, metal based composite can also be produced by high pressure infiltration method. According to Peng *et al.* (2004), high-volume-fraction of Si_3N_4–Al-based composites have been fabricated by high-pressure casting method. Ceramic–metal composites were fabricated by infiltrating the molten Al alloys into the porous preform using a vertical type, high-pressure casting machine.

The metal composite not only can be fabricated by low or high pressure infiltration but also can be fabricated by vacuum pressure infiltration method (Qin and Zhang, 2000). The SiC_p preform was filled up with molten 6061 Al alloy by vacuum pressure infiltration, as shown in Figure 2.7.

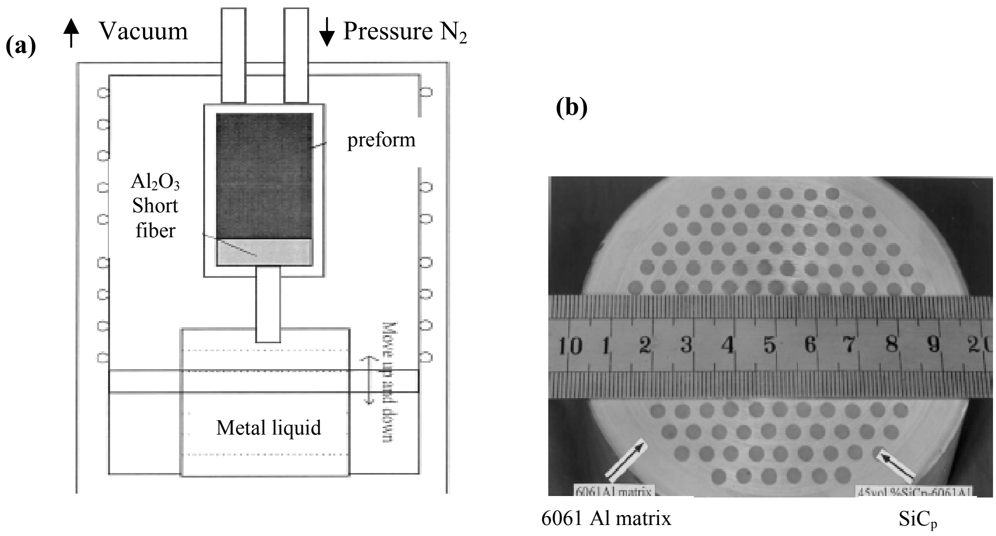

Figure 2.7 (a) The equipment of vacuum pressure infiltration, (b) The shape of the designed composite after infiltration (Chaudhury *et al.*, 2004)

2.1.3.3 Deposition

A deposition process which has considerable potential is spray co-deposition which is the modification of Osprey deposition process. It involves atomizing a melt and introduced the reinforcement particle into the spray of fine metal droplets. The metal and the reinforcement particles are then co-deposited on the substrate (Matthews and Rawlings, 1994). The atomized metal exists as discrete droplets for short time, of the order of a few milliseconds, and the rapid solidification leads to a matrix with a fine microstructure and reduces the possibility of extensive chemical reaction.

Many other deposition techniques have been tried to fabricate MMCs, such as chemical and physical deposition, sputtering and plasma spraying. An attractive factor for some of these techniques are that they operate at low temperature. Therefore, reactions at the reinforcement-fibre interface is minimized (Matthews and Rawlings, 1994).

A new spray forming technique that has been developed to synthesize aluminium based metal matrix composites was reported by Chaudhury *et al.* (2004). The schematic diagram of the new forming technique is shown in Figure 2.8. It consists of two metallic tubes being placed concentrically inside the confined type atomiser. Above the inner tube, a funnel was welded for feeding of ceramic particles. Mg was added in the melt to improve the wettability between the ceramic particles and aluminium melt. The melt and preheated ceramic particles were passed simultaneously through outer and inner tube, respectively. The melt was atomised by argon gas and then subsequently deposited on a rotating (10 rpm) copper substrate (200 mm diameter and 10mm thickness), placed vertically below the atomiser.

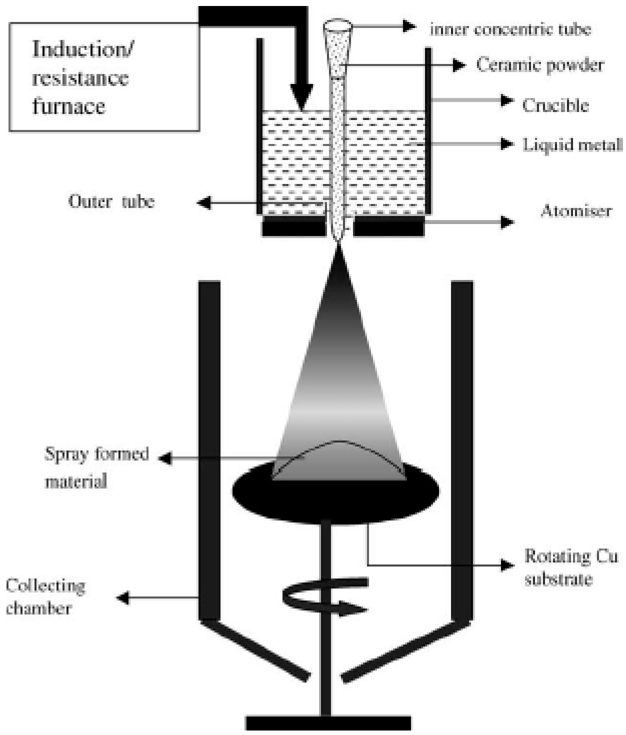

Figure 2.8 The schematic diagram of new forming spray technique (Chaudhury *et al.*, 2004).

2.1.3.4 *In situ* processing

In situ process offers interesting ways to produce intermetallics or metal matrix composite by powder metallurgy. The conventional way of producing MMCs is the so-called *ex situ* production where the reinforcing phase is prepared separately and then inserted into the matrix during a secondary process (typically via powder metallurgy or casting process). *In situ* MMCs, however, are composites where the reinforcing phases are synthesized in the metallic matrix during the composite fabrication itself. Most composites are particulate or dispersoid reinforced MMCs. *In situ* composites possess a number of advantages as compared to *ex situ* composites: thermodynamic stability of the reinforcing phases, clean particle-matrix interfaces with improved wetability, finer reinforcing particle size, more uniform distribution and no difficulties or safety risks in handling fine reinforcing particulates.

The various *in situ* routes can be classified according to the reaction mechanism via which the reinforcing phases are formed in the metallic matrix:

Liquid-Gas reaction: A typical example is the reactive gas injection, which involves an injection of a carbon or nitrogen bearing gas (e.g. CH_4 plus Ar) into molten alloy at a high temperature. The alloy contains reactive alloying elements which are strong carbide or nitride former leading to the formation of fine carbide or nitride particles in the metal matrix (Froyen *et al.*, 2003).

Liquid-Liquid reaction: A well known process is the salt-metal reaction involving addition of reactive salts to molten metal. It has been used to produce Al/TiB_2 MMCs where mixed Ti and B bearing salts, i.e. K_2TiF_6 + KBF_4 are added to molten aluminum and then dispersion of TiB_2 particles is formed in the Al matrix. The process is based on the well-established technology for producing Al-Ti-B master alloys (typically less than 3 %vol. borides) for grain refining (Froyen *et al.*, 2003).

Liquid-Solid reaction: Combustion synthesis, developed by Merzhanov and his co-workers (Merzhanov, 2009), is a process characterized by exothermic reactions, which are used to consolidate the solid product e.g. from compact powders. During this process, a combustion wave, which is self-sustained by the exothermic reactions, travel throughout the material. The process can be subdivided into thermal explosion mode process and self-propagating high-temperature process (SHS). In the thermal explosion mode, the powder mixture is heated up until a temperature where the reaction spontaneously occurs everywhere in the whole sample. In the SHS process, the combustion process is locally ignited and the combustion occurs locally and travels as a front throughout the whole sample. In both case, the process is characterized by high temperature conditions and front velocities from 1 to 250 mm/s. Anita *et al.* (2006) also reported that the dispersion of iron aluminide formed from iron powder in an aluminum matrix via a SHS synthesis with very fine particle size of iron aluminide obtained.

Solid-Solid reactions: Mechanical alloying and reactive milling are powder metallurgical routes for preparing composite powders from a mixture of starting powders by high energy ball-milling. *In situ* MMCs, intermetallics and IMCs can be successfully produced by MA plus a subsequent heat-treatment. Ko *et al.* (2002) showed results of the microstructure and synthesis path of *in situ* TiC reinforced Fe-28 %at. Al composite as a function of milling time and heat treatment temperature. Mechanical alloying of elemental powders promotes bcc solid solution formation, and the solid solution was completed at milling time of 400 h. More details of mechanical alloying will be described in the next part.

2.2 Mechanical alloying (MA)

In the present research work, mechanical alloying method was used to produce iron aluminide composite reinforced with TiB_2, TiC and Al_2O_3 from Fe-Al-Ti-B-C powder system. Therefore, a review on mechanical alloying provides a useful knowledge for this research. These reviews help to understand deeply about mechanical alloying process and what are the factors that affect the properties of the as-milled powders.

2.2.1 Introduction

Mechanical alloying (MA) is a powder metallurgy processing technique involving cold welding, fracturing, and rewelding of powder particles in a high-energy ball mill, and has now become an established commercial technique to produce oxide dispersion strengthened nickel- and iron-based materials. MA is also capable of synthesizing a variety of equilibrium and non-equilibrium alloy phases starting from blended elemental or prealloyed powders. The non-equilibrium phases synthesized include supersaturated solid solutions, metastable crystalline and quasicrystalline phases, nanostructures, and amorphous alloys. Recent advances in these areas and also on disordering of ordered intermetallics and mechanochemical synthesis of materials have been critically reviewed after discussing the process and process variables involved in MA. However, the "science" of MA is being investigated only during the past 10 years (Suryanarayana *et al.*, 2004).

Milling by mechanical collision using hard balls is a new approach to fabricating powder alloys from elemental powders. A jar mill of stainless steel filled