CHARACTERIZATION OF TITANIUM ALUMINIDE ALLOYS SYNTHESIZED BY MECHANICAL ALLOYING TECHNIQUE

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ABSTRACT

One of the method in powder metallurgy namely as mechanical alloying is not only a simple and inexpensive process, but also a promising technique in improving material's properties. However, the final product is highly depending on the milling parameters and conditions and very sensitive to environmental factors. In this present study, processing of Ti-Al powders by mechanical alloying technique and subsequent heat treatment was performed with the purpose to gain better understanding of their effect on structural and phase formation of the titanium aluminide alloys, and its relationships to physical, thermal and mechanical properties. It was found that, dry milling at an intermediate speed of 300 rpm is the most optimum condition to produce Ti-Al with crystallite size in nano range. Solid state reaction during mechanical alloying resulted in the formation of Ti(Al) solid solution and remarkable reduction of crystallite size as low as 20 nm. In the initial stage of milling, the thermal stability was influenced by the crystallite size refinement, but after prolonged milling, the stability was regulated by alloying degree of powder constituent. The density and micro-hardness value tag along with the crystallite refinement with gradual decreased of densities from 4.1 g/cm^3 down to 3.51 g/cm^3, while the hardness increased systematically from 43 to 117 HV by milling duration. After the secondary processing step by subsequent heat treatment up to 850 ^{o}C, the powder constituent were transform into a new inter-metallic phase of a dominant $TiAl_3$. When the heating temperature was raised to 1000 ^{o}C, it transformed into an inter-metallic mixtures varying from $TiAl_3$, γ-TiAl and α2-Ti_3Al. The dual phase γ-TiAl + α2-Ti_3Al were obtained in powder milled for 80 and 100 h. A remarkable increased in hardness as high as 622 HV were obtained, while the density values were around 3.7 – 3.9 g/cm^3. Mechanical alloying technique was proven as an effective method for producing nano-structured Ti-Al powder, where the parameter need to be selected carefully and thoroughly to obtain the optimum crystallite size. The results conclude that physical, thermal and mechanical properties of powder product has high dependency on crystallite size and alloying degree of the powder constituent produce from mechanical alloying technique. However, after heat treatment, the properties of the alloys were influenced by new phases of the powder constituent in turn governs their performance. In order to get a broader picture on the effect of mechanical alloying process on the formation of Ti-Al alloys, optimal heat-treatment conditions to produce a desired microstructure, and the influence of grain growth need to be further studied. In addition, selection of consolidation process should be reconsidered in other to performed additional mechanical test and analysis.

ABSTRAK

Salah satu kaedah yang dalam metalurgi serbuk yang dikenali sebagai pengaloian mekanikal bukan sahaja proses yang mudah dan murah, tetapi juga sebagai satu teknik berkesan dalam meningkatkan sifat bahan. Walau bagaimanapun, produk akhir hasil daripada proses ini adalah sangat bergantung kepada parameter pengaloian mekanikal dan sangat sensitif kepada faktor-faktor persekitaran. Dalam kajian ini, pemprosesan serbuk Ti-Al menggunakan teknik pengaloian mekanikal diikuti rawatan haba di lakukan bagi mendapatkan pemahaman yang lebih jelas mengenai kesannya terhadap struktur dan perubahan fasa aloi titanium alumida, dan hubungkaitnya kepada sifat fizikal, termal dan mekanikal. Hasil kajian mendapati bahawa pemprosesan kering pada kelajuan pertengahan 300 rpm adalah keadaan yang paling optimum untuk menghasilkan Ti-Al dengan saiz kumin hablur dalam skala nano meter. Tindak balas pepejal-pepejal semasa pengaloian mekanikal menyebabkan pembentukan larutan pepejal Ti(Al) dan pengurangan saiz kumin hablur yang sangat ketara serendah 20 nm. Pada peringkat awal pemprosesan, kestabilan terma serbuk logam Ti-Al adalah dipengaruhi oleh pengurangan saiz kumin, tetapi selepas proses dipanjangkan, kestabilannya adalah dipengaruhi oleh tahap pengaloian dan juzuk serbuk Ti-Al. Nilai ketumpatan pula berkadar terus dengan pengurangan saiz kumin dengan beransur-ansur menurun daripada 4.1 g/cm^3 kepada 3.51 g/cm^3, manakala nilai kekerasan mikro Vickers berkadar songsang dengan pengurangan saiz kumin dengan peningkatan secara sistematik daripada 43 HV kepada 117 HV. Selepas pemprosesan sekunder melalui rawatan haba pada suhu sehingga 850 ^{o}C dilakukan, juzuk serbuk Ti-Al telah berubah menjadi fasa logam baru di mana $TiAl_3$ terbentuk sebagai fasa dominan. Apabila suhu pemanasan dinaikkan kepada 1000 ^{o}C diikuti oleh penuaan, juzuk serbuk Ti-Al berubah menjadi fasa baru dengan campuran fasa yang berbeza-beza dari $TiAl_3$, γ-TiAl dan $\alpha 2$-Ti_3Al. Fasa dwi γ-TiAl + $\alpha 2$-Ti_3Al telah diperolehi pada serbuk yang diproses pengaloian mekanikal selama 80 dan 100 jam. Nilai kekerasan mikro didapati meningkat dengan sangat ketara setinggi 622 HV, manakala nilai ketumpatan yang diperolehi adalah sekitar 3.7 – 3.9 g/cm^3. Melalui kajian ini, teknik pengaloian mekanikal terbukti sebagai kaedah yang berkesan untuk menghasilkan serbuk logam Ti -Al berstruktur nano, dimana parameter pengaloian perlu dipilih dengan berhati-hati dan teliti untuk mendapatkan saiz kumin hablur yang optimum. Kajian ini menyimpulkan bahawa sifat fizikal, termal dan mekanikal produk serbuk mempunyai kebergantungan yang tinggi terhadap saiz kumin hablur dan kadar pengaloian juzuk serbuk Ti-Al yang dihasilkan melalui teknik pengaloaian mekanikal. Selepas proses rawatan haba pula, sifat-sifat aloi ditentukan oleh fasa baru juzuk aloi yang seterusnya mengawal prestasi aloi yang terbentuk. Walau bagaimanapun, untuk mendapatkan gambaran yang lebih jelas tentang kesan proses pengaloian mekanikal pada pembentukan Ti -Al aloi, keadaan haba rawatan yang optimum untuk menghasilkan mikrostruktur yang dikehendaki, dan pengaruh pertumbuhan kumin hablur memerlukan kajian lanjut. Di samping itu, pemilihan proses penyatuan juga perlu dipertimbangkan semula bagi membolehkan ujian mekanikal dan analisis tambahan dijalankan.

TABLE OF CONTENTS

CHAPTER 1 INTRODUCTION

CHAPTER 2 LITERATURE REVIEW

CHAPTER 3 EXPERIMENTAL PROCEDURES

CHAPTER 4 RESULTS AND DISCUSSION

LIST OF TABLES

LIST OF FIGURES

LIST OF SYMBOLS

at.%	atomic percent
°C	degrees Celsius, unit measure of temperature
β	full width at half maximum (FWHM) of the XRD peaks
β_M	full width at half maximum (FWHM) obtained
β_I	correction factor for instrumental broadening
°F	degrees Fahrenheit, unit measure of temperature
σ_y	yield strength
σ_o	lattice friction stress
K_y	Hall-Petch constant
d	crystallite size
D	average crystallite size
K	shape factor of the XRD
λ	CuK_α radiation of the XRD
θ	diffraction angles of the XRD

LIST OF ABBREVIATIONS

Al	Aluminium
ASTM	American Society for Testing and Materials
cm	centi meters, unit measure of length -10^{-2} meters
CTE	coefficient of thermal expansion
DP	duplex microstructure
EDX	electron dispersive x-ray
FESEM	field emission scanning electron microscope
g	gram, unit measure of mass
GPa	giga pascal, unit measure of stress – 10^{-9} Pascals
h	hour, unit measure of time – 3600 second
HRTEM	high resolution transmission electron microscope
HV	Vickers hardness, unit measure of micro hardness - kg/mm^2
HIP	hot isostatic pressing
kg	kilogram, unit measure of mass – 10^3 grams
LVDT	linear variable differential transformer
MA	mechanical alloying
MPa	mega pascal, unit measure of stress – 10^6 Pascals
N	Newton, unit measure of force
NASA	National Aeronautics and Space Administration
NL	near lamellar microstructure
nm	nano meter, unit measure of length – 10^{-12} meters
PM	powder metallurgy
Psi	pound per square inch - unit measure of stress
SPS	spark plasma sintering
SEM	scanning electron microscope
Ti	Titanium
TiAl	Titanium aluminide
XRD	X-ray diffraction
YS	yield strength

CHAPTER 1

INTRODUCTION

1.1 BACKGROUND

Advanced technology such as aerospace, chemical, nuclear, medical and even weaponry sectors has high dependency towards great quality of alloys in ensuring its reliability and durability in hostile environment and extreme operational conditions. Hasty development in the industrial sectors and advancement in the technology application demanded for materials that are lighter, stronger, stiffer, and useful at higher temperatures. Reducing structural weight is one of the major ways to reduce carbon emissions and improve aircraft performance. Lighter and stronger materials allow greater range and speed, and may also contribute to reducing operational costs. Tri-aluminide, especially titanium aluminide base alloys, are regarded as one of the most promising candidates for high temperature structural materials in the 21st century. This is due to their low density combined with good mechanical properties and corrosion resistance at medium high temperature range. Titanium aluminide is a group of intermetallics created by alloying of titanium and aluminum along with minor addition ternary elements. The combination of their low density (3.7 - 4.1g/cm^3), high specific strength and relatively good properties at an elevated temperature, good creep characteristics at temperatures up to 900 °C (Xiao et al., 2009) and, excellent oxidation and corrosion resistance makes them very attractive as high-temperature structural materials for aerospace, automotive and other applications (Lagos and Agote, 2013).

Titanium aluminide base alloys have been prototyped and tested for use in aircraft turbines engine, automotive and hypersonic airplanes. In particular, titanium aluminide base alloys offer a great reduction in weight and acceptable mechanical performance characteristics compared to nickel base super-alloys, which can help attain weight-savings and cost reduction goals. Hence, replacement of nickel base super-alloys parts and component with titanium aluminide base alloys in high performance aircraft's gas turbine engines is expected to reduce the structural weight by 20–30% and will produce leaner and efficient structural systems (Voice et al., 2005) which also contribute to fuel savings and the reduction of CO_2 emissions. In automotive industries, the high stability of titanium aluminide exhaust valve in hot environment has resulted in a significant improvement in engine performance from the reduced inertia and friction loss in normal engines (Froes and Suryanarayana, 1996). Eventually, this will increase the fuel efficiency and contribute in reduction of the pollutant emissions, along with noise reduction during operation.

Nevertheless, the attractive properties of titanium aluminide-based alloys were outweighed by poor ductility at ambient temperature below 700^{o}C, low strength at elevated temperature, and insufficient oxidation resistance above 850^{o}C (Al-Dabbagh et al., 2013). The low ductility of titanium aluminide further caused difficulties in processing and machining at room temperature, and resulted in high manufacturing cost. In order to overcome this problem, many efforts have been devoted to fabricate nano-structured titanium aluminide alloys, which have an additional advantage of giving rise to a good combination of strength and ductility (Farhang et al., 2010). The initial processing route for fabrication of titanium aluminide base alloys components was investment casting and wrought processing of ingot metallurgy materials. This method have received the most attention because of their potential for low cost and high volume production. However, Clemens and Kestler (2000) reported that this processing route can lead to fluctuations in the Al content of more than ± 2 at.%, leading to a non-uniform microstructure. These microstructures have a significant variation in the mechanical properties leading to the deterioration of the properties of the alloys. On the other hand, powder metallurgy (PM) technologies offers for more precise control of composition and microstructure, as well as reducing the fabrication costs of producing complex, near net shape components (Moll and McTiernan, 2000).

An innovative development of PM method has enable a viable commercial practice for cost effective materials to be produce with controlled, uniformly refined structures and optimum mechanical properties (Zhao et al., 1997). Amongst the wide options of PM method, the MA technique has been considered the most powerful tool for the fabrication of nano-structured materials because of its simplicity and relatively inexpensive equipment (Koch, 1993). MA has attracted the attention of a large numbers of researchers because of it ability for the production of fine dispersion of second phase (usually oxide) particles, extension of solid solubility limits, refinement of grain sizes down to nano meter range, synthesis of novel crystalline and quasi-crystalline phases, development of amorphous (glassy) phases and disordering of ordered intermetallics (Cahn et al., 1991). Courtney and Maurice (1989), praised MA technique as the "Aladdin's lamp of powder metallurgy" because of the capability of this technique to produce alloys and compounds that are difficult or impossible to be obtained by conventional melting and casting techniques. In addition, this method has opened the possibility for producing materials with unique physical and mechanical properties at room temperature, which can be scaled up in large quantities up to several tons (Koch, 1997).

1.2 PROBLEM STATEMENT

The properties of titanium aluminide alloys are depend on their phases and strongly influenced by their microstructures, which is controlled by processing methods and composition modifications. Mechanical alloying (MA) is one of the techniques in PM route which employs a reputation that offers not only a refinement of crystallite size to nano-structure, but also for obtaining materials of high structural homogeneity (Farhang et al., 2010). Unlike ingot metallurgy and investment cast, materials produced from MA have a fine and homogeneous starting microstructure which can be an important advantage to achieve desired microstructure. However, the properties of specific materials obtained by MA are very sensitive to experimental conditions such as; milling type, rotation speed, type and amount of process control agents (PCA) and atmosphere (Suryanarayana, 2001). Recent publication by El-Eskandarany (2013) imply that the selection of these parameters, are vital in obtaining alloys in nano range crystallite size with specific phase and desired properties. Even then, all these process variables are not completely independent.

The possibility of producing Ti-Al intermetallics phase by using MA technique has only been explored recently in powder metallurgy (PM) materials. Many researchers have devoted their work to understand the formation of titanium aluminide through MA from elemental Ti-Al powders. However, there is still no general consensus on the precise mechanism behind the phase formation of these alloys by mechanical alloying technique. In addition, most of the results and findings implicate a significant difference and are in contradiction with the existing understanding and viewpoint regarding the mechanism of titanium aluminide phase formation (Guo et al., 1990, Suryanarayana et al., 1992, Oehring et al., 1993, Itsukaichi et al., 1993, Bhattacharya et al., 2004 and Forouzanmehr et al., 2009, Gonzalez et al., 2009 and Farhang et al., 2010). The main reason is because of the parameters selection for MA involves a large degree of uncertainty in obtaining desired phases and microstructures. For example, the optimum milling time depends on the type of mill, size of the grinding medium, temperature of milling, ball-to-powder ratio, etc. Such inter dependent effect of these variables make this simple process, become more complicated to obtained desirable microstructure and properties for specific materials (El-Eskandarany, 2013).

In this research project, the production factor of MA processes were investigate thoroughly under specific and controlled parameters in order to synthesis nano-structured Ti-Al alloys. Elemental titanium and aluminium powder were used which has been proven useful than pre-alloyed intermetallic powder because they are cheaper and due to their ability to generate exothermic heat from their reaction. In turn, the reaction taking place for the synthesis of the Ti-Al intermetallic compound often results in temperatures lower than the melting point of aluminium. Combined with increased of inter-diffusion reaction between Ti and Al by mechanical activation during ball milling, the reaction energy is utilized, making it more energy efficient. Furthermore, the optimum composition of Al were used as starting materials for the synthesis of dual phase γ-TiAl + α2-Ti_3Al alloys making it more economic.

1.3 OBJECTIVES OF THE STUDY

This work is intended to gain a better understanding on the effect MA processes and subsequent heat treatment on the phase formation and its relationships to physical, thermal and mechanical properties of the titanium aluminide alloys. The specific goals of this work include;

i. To investigate the effect of MA parameters on the production of nano-structured Ti-Al powders.

ii. To study the effect of MA and heat treatment parameters on the phase formation of titanium aluminide alloys.

iii. To assess the relationship of titanium aluminide alloys characteristics on the densities and mechanical properties.

1.4 SCOPE OF THE STUDY

The scope of this work is to produce nano crystalline titanium aluminide alloys from blended elemental Ti and Al powders with a composition of 50% of Ti and 50% of Al. It is aimed to explore the feasibility of MA processes by planetary ball mill and subsequent heat treatment (annealing) with interest to produce nanostructured dual phase γ-TiAl + $\alpha 2$-Ti_3Al alloys. The evaluations on the phase transformation, structure (crystallite size), physical (density), mechanical (Vickers micro hardness) and thermal (DTA) properties were essentials in order to determine whether MA can alter the phase formation of Ti-Al intermetallics and improve the critical properties of titanium aluminide alloys.

1.5 OVERVIEW OF THE THESIS

The layout of the thesis report is divided into five chapters. Chapter 1 is an introduction of the research. Chapter 2 provides a literature review covering relevant background research conducted in the area. Chapter 3 describes the experimental procedures. Results and discussions are presented in Chapter 4 and finalized with a conclusion and recommendations for future work in Chapter 5. This research work can be validate by numbers of papers published in international journals and presented at international conferences. Appendix A provides a list of all publications and proceedings related to this research work.

CHAPTER 2

LITERATURE REVIEW

2.1 INTRODUCTION

This chapter provides a literature review covering relevant background research conducted in the area including the introduction to nano-crystalline materials, titanium aluminide base alloys and the processing route to be explored in this course of research. The review on nano-crystalline materials discussed on the theoretical background, classification, processing route and it's potential over conventional materials. The review on titanium aluminide discussed on the development and current status of titanium aluminide base alloys with emphasis on its properties with comparison to superalloys. Emphasized were also given to both mechanical alloying processes and heat treatment in the production of titanium aluminide base alloys.

2.2 NANO-CRYSTALLINE MATERIALS

Materials scientists have been continuously research towards improving the properties and performance of materials for significant enhancements in mechanical and physical properties. Advancement in nanotechnology has enable for the creation of systematic synthesis techniques to control the structure of the materials in order to provide a precisely tailored set of properties for designated applications. One of the promising strategies in improving material's properties is by reducing the crystallite size. The driving force behind this effort was the possibility of synthesizing materials with strengths approaching the theoretical value by reducing the crystallite size to nano scale (Meyers et al., 2006). Furthermore, nano-crystalline materials may exhibit increased strength and hardness, improved toughness, reduced elastic modulus and ductility, enhanced diffusivity, higher specific heat, enhanced thermal expansion coefficient (CTE), and superior soft magnetic properties in comparison with conventional polycrystalline materials (Kadkhodapour et al., 2009). Nano-crystalline materials is a distinctive class of advanced engineering materials have now become a major identifiable activity in materials science and engineering with vast prospect for applications in a wide range of fields. These materials are expected to play a key role in the next generation of human civilization because of their superior properties when compared with their coarse grained counterparts (Meyers et al., 2006). Nano-crystalline materials are single or multi-phase polycrystalline solids with a crystallite size of a few nano meters and typically less than 100 nm. Attention in nano-crystalline materials arises from the realization that by controlling the sizes of the crystallite size, one begins to alter a variety of the physical, mechanical, and chemical properties of bulk materials (Roco, 2011). There has been shown that, by decreasing the crystallite size by 2 - 3 orders of magnitude, nanostructured materials are at least 4 - 5 times stronger than their conventional coarse-grained counterparts.

The properties of nano-crystalline materials could be tailored or engineered through the modification of micro-structural features, more precisely by controlling the crystallite size, morphology, and composition specifically. All of those are made possible by controlling the process parameters by a numbers of different techniques including inert gas condensation, rapid solidification from the liquid state, mechanical alloying, electro-deposition, crystallization from amorphous materials, severe plastic deformation, cryo-milling, plasma synthesis, chemical vapour deposition, pulse electron deposition, sputtering, physical vapour deposition and spark erosion (Meyers et al., 2006). Amongst many such processes which are in commercial use, rapid solidification from the liquid state, mechanical alloying, plasma processing, and vapour electro-deposition have been receiving serious attention from researchers. It has been shown that materials processed this way possess improved physical and mechanical characteristics in comparison with materials processed by conventional ingot solidification (Murty et al., 2003). Gleiter (1989) proposed the basic idea of nano-crystalline materials in which 50% or more of the atoms are situated in the grain boundaries. Since the grain sizes are too small, a significant volume of the microstructure in nano-crystalline materials is composed of interfaces, mainly grain boundaries. The large volume fraction of the atoms resides in grain boundaries consequently yielded in nano-crystalline materials exhibit properties that are significantly different from, and often improved over their conventional coarse-grained counterparts (Suryanarayana, 1995). In addition, comparison to the coarse-grained materials, nano-crystalline materials are also notable in enhanced diffusivity, and superior soft and hard magnetic properties. Even a cooperative phenomenon such as melting point and melting thermodynamic functions such as enthalpy and entropy of melting point could be affected in nano-crystalline materials.

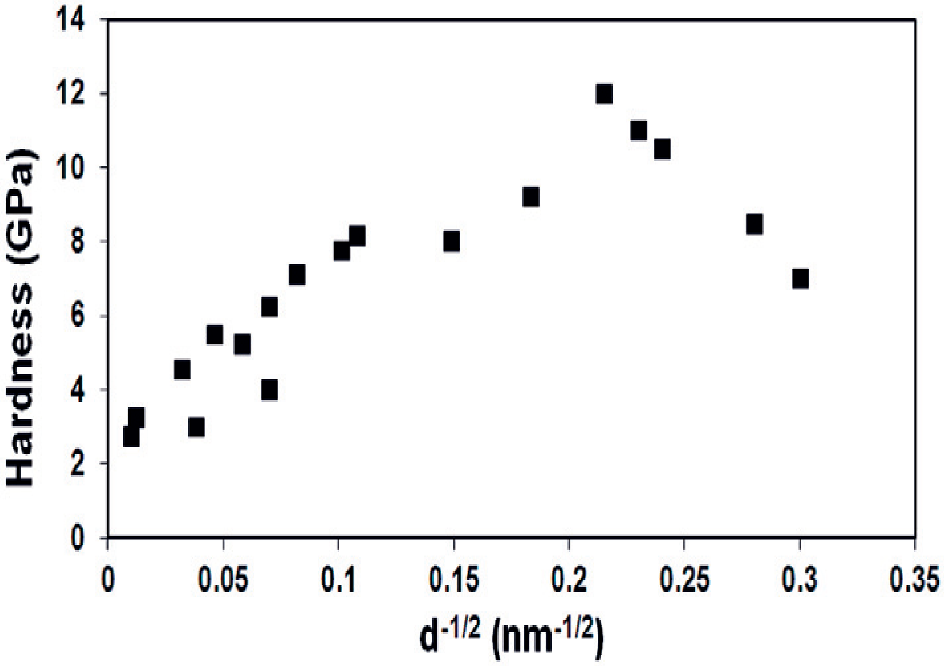

Figure 2.1: Variation of hardness with reciprocal of the square root of crystallite size in γ-TiAl alloys produced by different methods.

Hall (1951) and Petch (1953) proposed that, nano-crystalline materials are harder and stronger than one that is coarse grained material because of it has greater total grain boundary area to impede dislocation motion. For most materials, the yield strength increases with a decrease in crystallite size according to the Hall-Petch (Hall, 1951 and Petch 1953) equation:

$$\sigma_y = \sigma_o + K_y d^{-1/2} \tag{2.1}$$

where, σ_y is the yield strength, σ_o is the lattice friction stress, K_y is the Hall-Petch constant, and d is the crystallite size. However, experiments on many nano-crystalline materials demonstrated that when the crystallite reached the critical size which is typically less than about 30 nm, the strength has been found to decrease gradually with decreasing in crystallite size as shown in Figure 2.1 (Suryanarayana, 2012). This phenomenon has been referred as the inverse Hall–Petch effect. Figure 2.1 shows the hardness of γ-TiAl alloys plotted as a function of crystallite size from materials produced by conventional casting methods to novel mechanical alloying and deposition methods which demonstrate the inverse Hall–Petch effect when the crystallite size reached criticality below a size of 30 nm.

2.3 TITANIUM ALUMINIDE BASE ALLOYS

2.3.1 Development of Titanium Aluminide Base Alloys

The earliest major work on titanium aluminide base alloys was initiated by the U.S. Air Force Materials Laboratory between periods of 1975 to 1983 (Kim, 1994). This collaboration with Pratt and Whitney recommended "first generation" Ti-48Al-1V-(0.1C) as the best alloy composition based on ductility and creep resistance. The second major development program between the Air Force and General Electric (GE) during a periods of 1986-1991, identified a "second generation" Ti-48Al2(Cr or Mn)-2Nb as the best alloy composition (Kim, 1994). Improved and relatively high ductility, modest strength, but combined with good environmental resistance and manufacturability, this alloys make suitable for low pressure turbine (LPT) blade applications (Kim, 1995a). GE conducted successful engine tests on a full-set wheel of gamma blades, which improved overall confidence in the material in 1993. But it is more than a decade later GE announced its commercialization of titanium aluminide-based alloy for its low-pressure turbine (LPT) blades to be utilized in GEnx type engines (Figure 2.2) to power the Boeing 787 Dreamliner passenger craft. This is the first mass commercial production of titanium aluminide base alloys (Norris, 2006). In addition, titanium aluminide were also extensively studied for use in High Speed Civil Transport (HSCT) aircraft which is designed to fly at Mach 2.4. This programme is designated to meet environmental goals of reduced exhaust and noise pollutions while take-off and landing at conventional airports (Bartolotta and Krause, 1999). In oeder to meet these stringent requirements, several critical components of the large exhaust nozzle of HSCT propulsion system are fabricated from γ-titanium aluminide. This is including the divergent flap and hybrid type of nozzle sidewall made from a fabrication of both wrought gamma and cast gamma substructure, proposed for noise attenuation and exhaust reduction (Kim, 1995a). In automotive industries, the first commercial use of titanium aluminide base alloys is for turbochargers and exhaust valves (Figure 2.3) for Formula One and sports cars to replace the existing conventional Ti-base alloys (Beschliesser et al., 2003). Parts and component made out of titanium aluminide have also been prototyped and tested to replace Ni-based super-alloys but the limiting factor is to match the mechanical performance and significantly in production costs of titanium metal and it's processing (Tetsui, 1999). Through deeper understanding of titanium aluminide's microstructure, deformation mechanisms, advances in micro-alloying, and development in advance processing technologies initiated by NASA Glenn Research Center (USA), Plansee (Austria), and GKSS Research Center (Germany) has led to the production of titanium aluminide sheet material namely, Gamma Met PX (GMPX) which is commercially available (Appel et al., 2000). This sheet alloys are manufactured by using Advanced Sheet Rolling Process (ASRP) is homogenous thin titanium aluminide sheet with high Nb-content based on the TNB alloys developed by GKSS has resulted in cost reduction to \$150/lb (NASA, 2003).

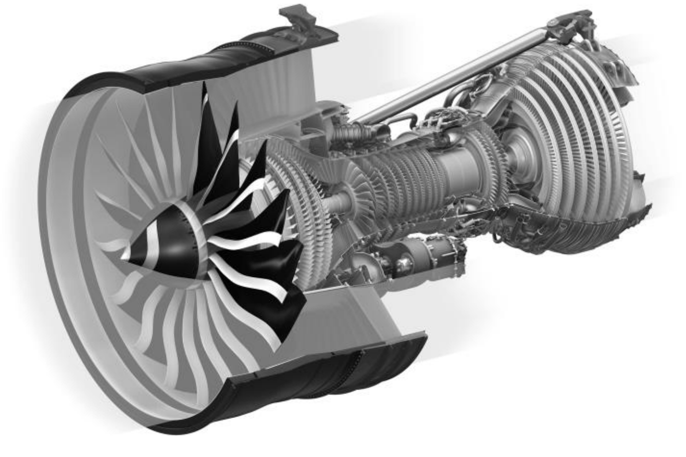

Figure 2.2: General Electric GEnx-1B engine for Boeing 787 Dreamliner with low pressure turbine blade made from cast TiAl (Appel et al., 2011).