MICROSTRUCTURAL CHARACTERISATION OF AS-CAST BINARY NEAR GAMMA TITANIUM ALUMINIDES

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Abstract. Intermetallics based on Ti-Al compounds exhibits promising properties for high temperature applications. However, only two phase near gamma alloys are able to some extent fulfill the crucial high temperature applications. Therefore during the past decade, a number of research efforts were directed to optimise the application of this recently emerging class of alloy. Since the high temperature property which mainly concerns creep is strongly microstructure dependent, a comprehensive understanding on the microstructure is needed. In this paper, the current knowledge on the microstructures of as-cast binary near gamma titanium aluminides will be addressed.

Keyword: Near gamma titanium aluminides, microstructure, lamellae, nearly lamellae, duplex

Abstrak. Sebatian antara logam berasaskan sebatian Ti-Al mempamerkan sifat yang menyakinkan untuk aplikasi suhu tinggi. Walau bagaimanapun, hanya aloi dua fasa hampir gamma mampu sedikit sebanyak memenuhi aplikasi suhu tinggi yang penting. Oleh itu, pada dekad yang lepas, banyak kajian telah ditumpukan kepada mengoptimumkan aplikasi aloi baru ini. Oleh kerana sifat suhu tinggi terutama berkaitan dengan rayapan yang sangat bergantung kepada mikrostruktur, kefahaman menyeluruh mengenai mikrostruktur ini diperlukan. Dalam kertas kerja ini, pengetahuan semasa mengenai mikrostruktur aloi tuangan binari hampir gamma titanium aluminaid akan dibincangkan.

Kata kunci: Hampir gamma titanium aluminaid, mikrostruktur, saiz bijian, pecahan isipadu, morphologi sempadan bijian

1.0   INTRODUCTION

Intermetallic compounds based on titanium and aluminium are light (low density), relatively stiff (high modulus) and have attractive high temperature mechanical properties (tensile and creep properties) [1].

Examination of the Ti-Al phase diagram shows that there are three compounds in this system; Ti_3Al (super alpha, α_2), TiAl (gamma, γ) and $TiAl_3$. Among these, only Ti_3Al and TiAl have been extensively investigated. Ti_3Al has a tetragonal DO_{19} structure and TiAl has the hexagonal LI_0 structure. In both cases, the compounds

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exhibit limited ductility at room temperature, particularly in binary state [1]. However, there is another phase; near gamma ($\alpha_2 + \gamma$) which are more reliable for structural applications than super alpha and gamma titanium aluminides.

γ-TiAl alloys have no significant engineering applications because of their poor ductility and fracture toughness, although they exhibit better performance in hostile environment [2]. The low ductility of single phase TiAl (more than 52%at Al) is not even improved by alloying addition of, e.g. V, Nb, Cr, W or Mn [3]. Super alpha, α_2-Ti$_3$Al are not preferred due to their high density, high susceptibility to stress corrosion cracking, reduced tensile ductility, creep failure at high temperature, low oxidation limit temperature and low specific strength and modulus [4].

Near gamma titanium aluminides are classified into single and two phase alloys. Near gamma alloys which were mentioned earlier for structural applications are referred to the two phase alloys; Ti-(46-52)at% Al [5]. The single phase near gamma alloys have no significant engineering properties. Figure 1 shows the composition range for two phase near gamma alloys [5].

The single phase near gamma alloys exhibit a lamellae microstructure. On the other hand, the significant two phase near gamma alloy exhibits two different structure as to the aluminum content which is nearly lamellae, Ti-(46-48)at% Al and duplex,
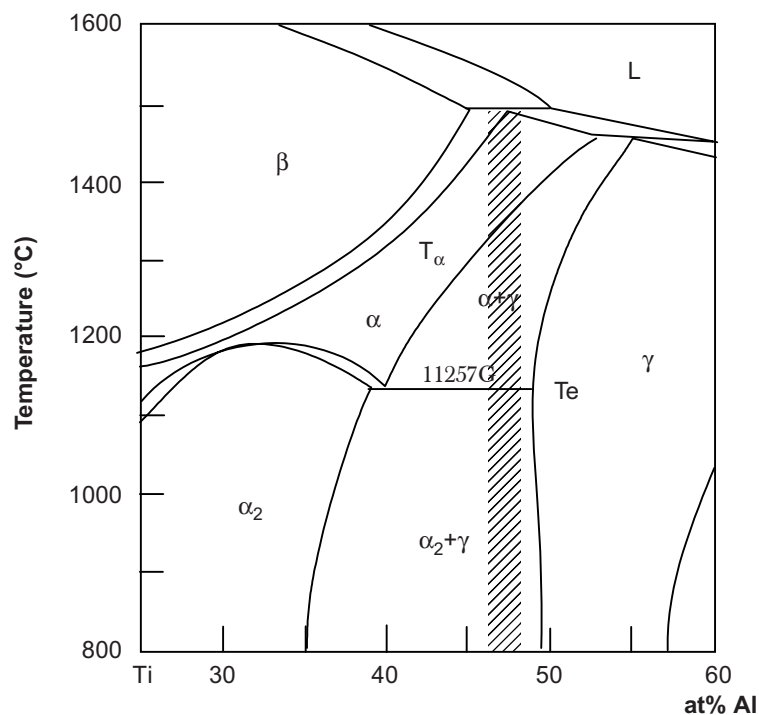

Figure 1 Central portion of binary Ti-Al phase diagram showing the composition range for two phase near gamma alloys [1]

Ti-(49-52)at% Al. Researchers had defined both microstructures to distinguish them. Nearly lamellae are defined as coarse lamellae grains with minor amounts of fine γ grains [3] or when gamma grains can be seen in the lamellae grain boundaries [6]. Whereas for duplex microstructure, it is a combination of equiaxed gamma grains and lamellae grains [3, 6].

The lamellae structure consists of alternating plates of the γ-TiAl and α_2-Ti$_3$Al phases. Such a lamellae structure results from the solid state phase transformation of the primary disordered α dendrites. The γ regions surrounding the lamellae grains result from the transformation of the aluminium rich interdendritic melt [7].

Even if the microstructures are well defined and distinguished, there is still confusion regarding this because there is no any complete comprehensive paper which describes this individually. Therefore, beginners have to go through a lot of literature to understand well about the microstructure, which is the basis for the studies. In this paper, all the three microstructures of near gamma alloys will be characterized and discussed

2.0   MATERIALS AND METHOD

Ti-45at.%Al, Ti-48at.%Al and Ti-50at.%Al were produced by IRC, University of Birmingham, UK by plasma melting casting technique to produce buttons weighing 2 kg each. The chemical composition of the as-cast samples is shown in Table 1. The samples will be referred in atomic percentage (at.%) throughout the manuscript.

Table 1   Chemical composition of the as-cast samples in at.%

| Sample	Ti	Al
Ti-45at.%Al	55	45
Ti-48at.%Al	52	48
Ti-50at.%Al	50	50

The samples for metallography analysis were cut into cubic shapes by using Electro Discharge Wire Cut Machine with dimensions of $10 \times 10 \times 10$ mm. The samples were then grounded and polished in accordance to the standard procedures. The polished surface was etched by using Kroll's reagent (94 ml H$_2$O + 4 ml HNO$_3$ + 2 ml HF).

Optical and scanning electron microscopes (back scattered electron mode) were used to characterise the microstructures, whereas x-ray diffraction (XRD) measurements was performed on the samples to identify the phases and its crystal structure. The grain size and volume fraction were measured using the intercept and point counting method respectively. The grain boundary morphology was determined by observing several micrographs which shows the grain boundaries.

3.0   RESULTS AND DISCUSSION

3.1   Microstructures

Figures 2(a), 2(b) and 2(c) show the microstructures of as-cast Ti-45Al, Ti-48Al and Ti-50Al which was observed under optical microscope at magnification of 50x whereas Figures 3(a), 3(b) and 3(c) show the microstructures of as-cast Ti-45Al, Ti-48Al and Ti-50Al which was observed under scanning electron microscope with magnification of 2000x. Ti-45Al, Ti-48Al and Ti-50Al exhibited lamellae, nearly lamellae and duplex microstructure respectively. The as-cast sample Ti-48Al and Ti-50Al exhibited a microstructure with strong microseggregations, being composed of primary α dendrites, which transformed into lamellae $\alpha_2 + \gamma$ during solid state cooling and
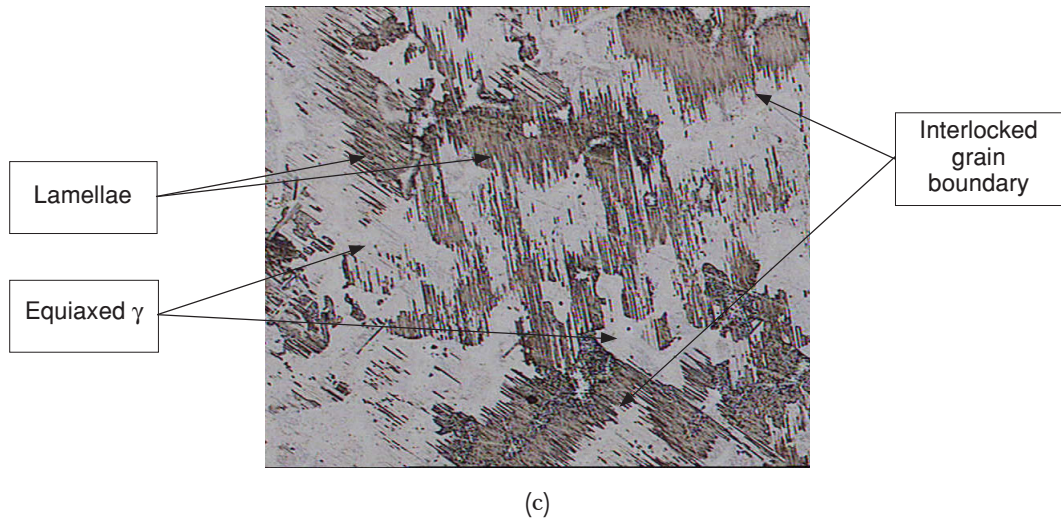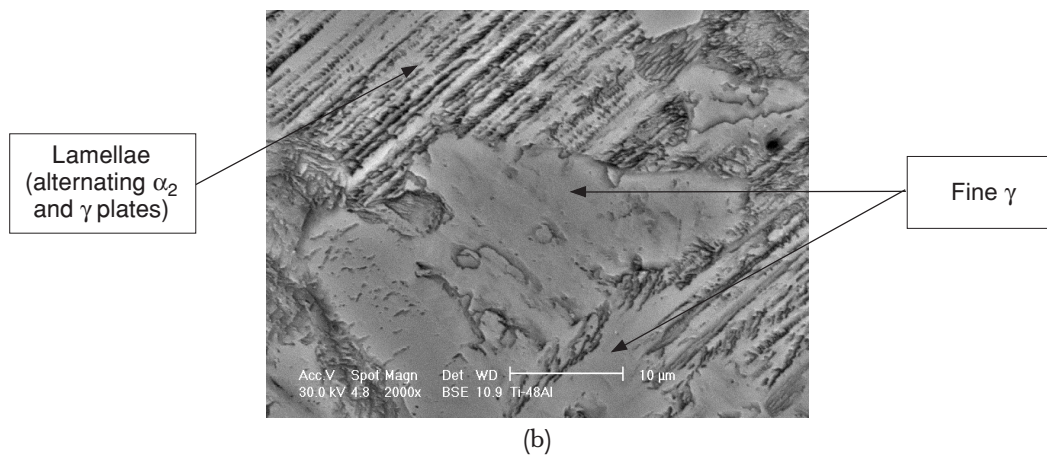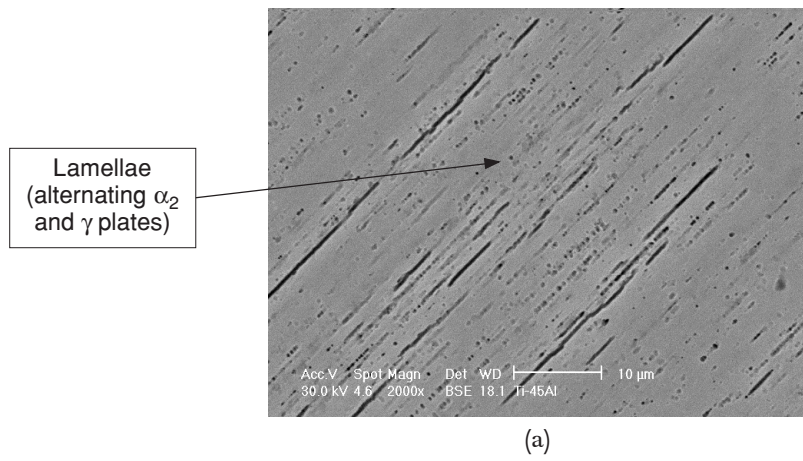

(a)

(b)

(c)
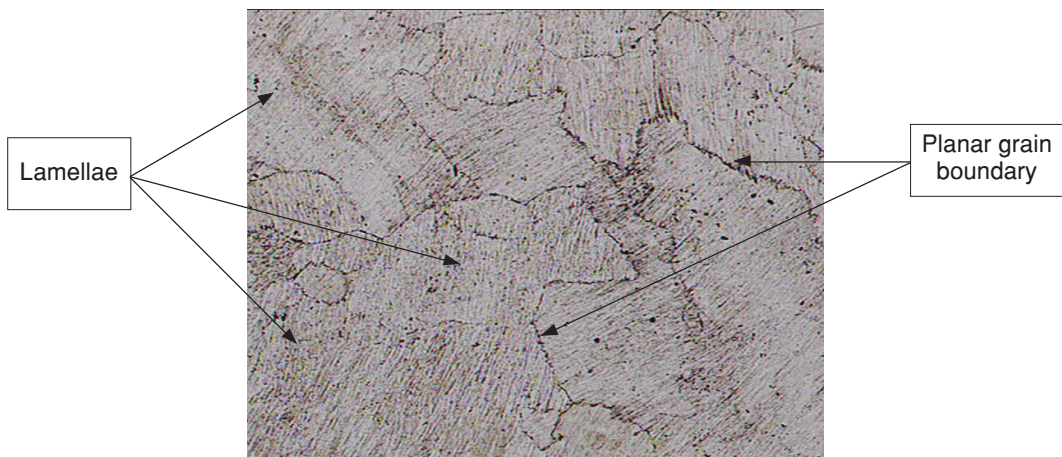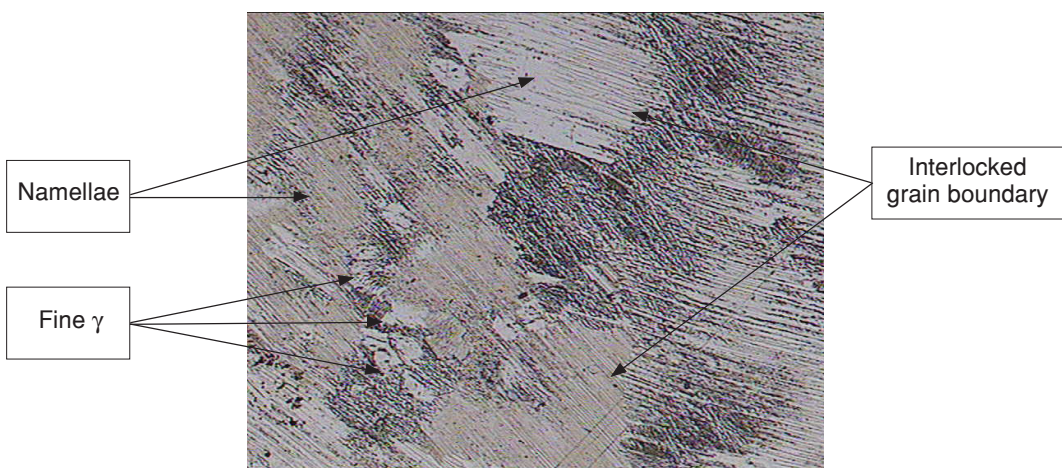
Figure 2   Optical micrograph showing microstructure of (a)Ti-45Al, (b)Ti-48Al and (c) Ti-50Al at magnification of 50x
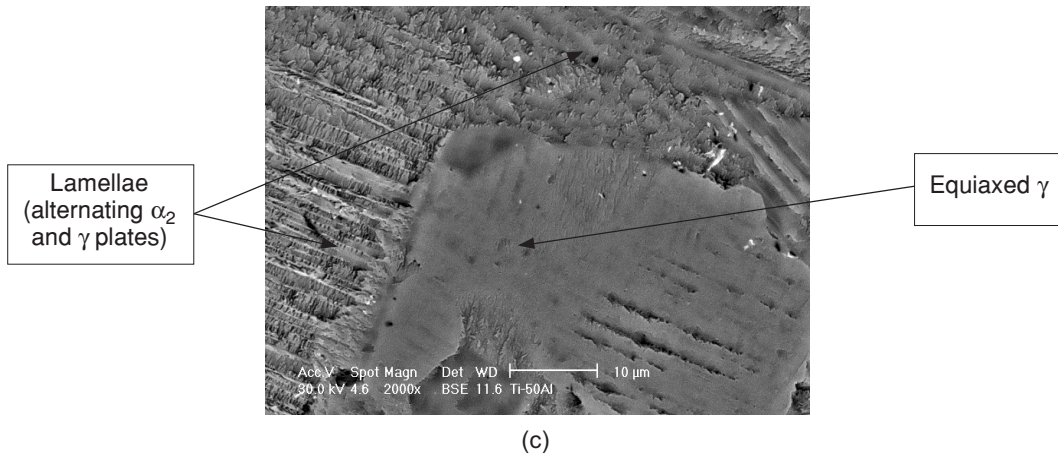

(a)

(b)

(c)

Figure 3   Scanning electron (back scattered electron mode) micrograph showing microstructures of (a) Ti-45Al, (b) Ti-48Al and (c) Ti-50Al at magnification of 2000x

interdendritic γ phase. The solidification of these two phase alloys, based on the phase diagram, occurs through two peritectic reactions, $L + \beta \rightarrow \alpha$ and $L + \alpha \rightarrow \gamma$, which is normally incomplete due to limited diffusion caused by the formation of a solid envelope of the peritectic phase, avoiding the physical contact between the reactants. For both peritectic reactions, high cooling rate or melt under cooling inhibit the formation of the pro-peritectic solid phase; the incompleteness of the peritectic reactions will cause the remaining liquid to progressively enrich in aluminum and solidify, at lower temperatures, as interdendritic γ phase [8].

3.2   Phases and Crystal Structures

Figures 4(a), 4(b) and 4(c) show the XRD test results of the samples. All samples showed hexagonal and tetragonal crystal structures which represent Ti_3Al and TiAl respectively.

3.3   Microstructural Features

3.3.1   Grain Size

Determination of the grain size of near gamma titanium aluminide is most probably the second most important metallography measurement after the lamellae spacing. The grain size is measured using the intercept method. The detail description of this method is described elsewhere [9]. Table 2 shows the grain size of lamellae grain size.
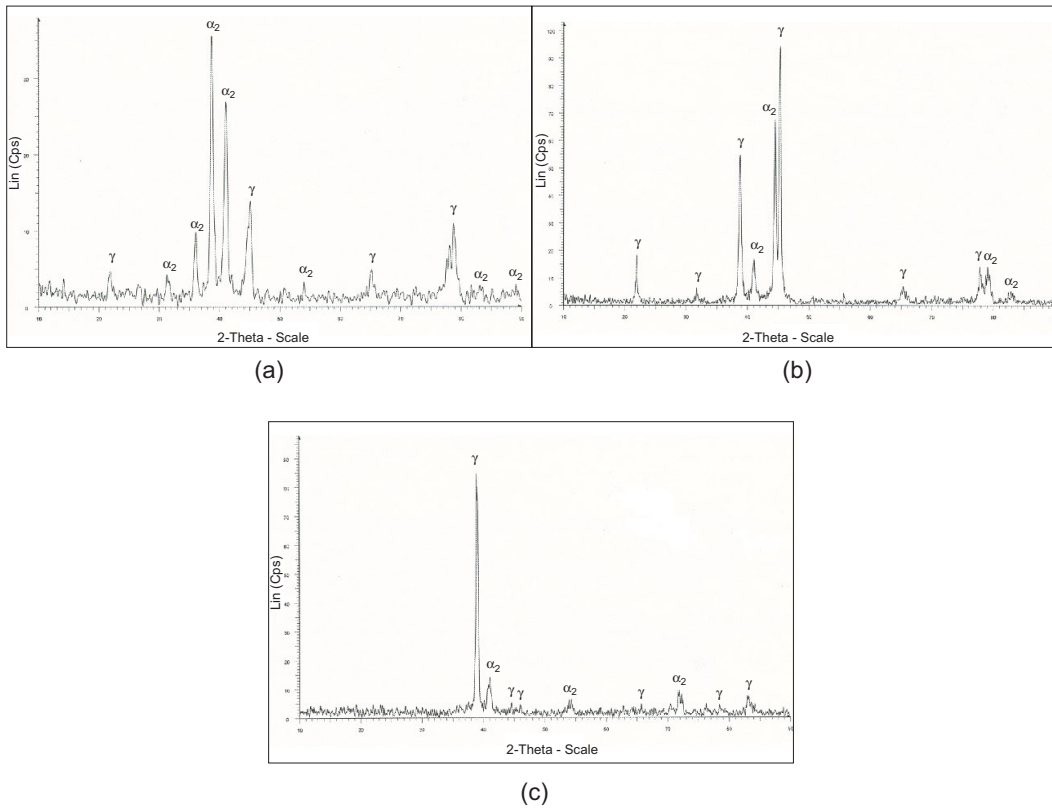
(a)   (b)

(c)

Figure 4   X-ray diffraction results of (a) Ti-45Al, (b) Ti-48Al and (c) Ti-50Al

Table 2   Lamellae grain size measured using intercept method

Samples	Grain size (μm)
Ti-45Al	500 – 800
Ti-48Al	300 – 700
Ti-50Al	100 – 500

3.3.2   Volume fraction

Determination of the volume fraction of a particular phase or constituent in a microstructure is one of common stereological measurements [9]. The volume fraction is measured using point counting method. The detail description of this method is described elsewhere [9]. Table 3 shows the volume fraction measured for single gamma phase (fine/equiaxed) in the samples.

Table 3   Volume fraction of single gamma phase measured using point count method

Samples	Volume fraction (%)
Ti-45Al	0
Ti-48Al	2 – 5
Ti-50Al	30 – 50

3.3.3 Grain Boundary Morphology

Grain boundary of near gamma titanium aluminides is usually in a condition of planar or interlocked. The grain boundary morphology of the samples is characterized using the microstructure micrographs with higher magnification. Table 4 shows the grain boundary morphology of the samples.

Table 4   Grain boundary morphology exhibited by the samples

Samples	Morphology
Ti-45Al	planar
Ti-48Al	interlocked
Ti-50Al	interlocked

It is observed that sample Ti-45Al, Ti-48Al and Ti-50Al exhibit lamellae, nearly lamellae and duplex structures respectively. XRD results showed that the crystal structure in all three samples was hexagonal and tetragonal which indicated that Ti_3Al and TiAl phases present. The gamma phase does not exist as single phase in the lamellae structure but it exists as alternating gamma plates (lamellae phase). The gamma exists as fine grain at the grain boundaries and as alternating plates in nearly lamellae structure. As for the duplex structure, the lamellae and gamma phase can easily be identified even at the lower magnification. It exists as equiaxed grains and alternating plates. The duplex structure is also known as the bi-modal structure [1].

Basically, the microstructure of near gamma alloy is distinguished by analysing the size of single gamma phase. When there is no presence of single gamma phase, the structure is called lamellae. If the amount of the gamma phase is small and located mainly at grain boundaries, it is referred as nearly lamellae and if it is almost similar size with lamellae colonies, it is called duplex structure. These microstructural characteristics are greatly influenced by the aluminum content.

Grain size [10, 11], volume fraction [10] and grain boundary morphologies [12] are important microstructural parameters which influence the mechanical properties of near gamma titanium aluminides. It is proposed that fine lamellae grain exhibits better mechanical properties compared with coarse lamellae grain. However, very small grain (<100 μm) is usually associated with low creep strength [10]. As for the

volume fraction of the constituent phases, it is insensitive to creep strength [10]. On the other hand, grain boundary morphology plays a major role in influencing the mechanical properties. Near gamma alloy usually has a planar or interlocked grain boundary. The interlocked grain boundary is reported to be very effective for improving creep resistance in heat resistance austenitic steels and cobalt base superalloys. A number of factors such as restriction of grain boundary sliding, slow crack growth due to crack deflection and a greater crack path can possibly explain the improvement of creep resistance due to the interlocked grain boundaries [12].

The lamellae structure has coarse lamellae grain of about 500 to 800 μm and with planar grain boundaries. So, it is clear that this single phase alloy have no significant engineering properties. Therefore, these alloys were never studied or extensively developed for engineering applications.

The nearly lamellae structure exhibits almost similar grain size (300-700 μm) with the lamellae structure. Besides that, this structure has also an interlocked grain boundaries. This recently emerging class of alloy Ti-(46-48)at % Al is reported to exhibit the highest tensile ductility, as high as 2% at room temperature [12, 13] and has better fracture toughness [14] and creep properties [12] compared to other class of near gamma alloys in the as-cast condition. Thus, extensive studies have been done to improve further this reliable alloy by altering the microstructures. Currently, most efforts have been focused on these alloys which are slightly lean in aluminum and microalloyed with several third elements. These have led to very complex alloys with the general compositions (in at.%) as follows:

$$Ti – (45–49)Al + (0.3–10)X$$

with X designating alloying elements, such as Cr, Nb, V, Ta, Si, B and C [15].

The duplex structure has more fine lamellae grain (100-500 μm) compared to the nearly lamellae structure. However, the gamma grains have almost equal size to lamellae grains (100-300 μm). It is reported that gamma grains reduce the tensile ductility, which is an important criteria for structural applications. The single phase gamma alloy (more than 52% at Al), which has equiaxed gamma grains exhibits poor ductility. As stated earlier, the low ductility of this single phase gamma alloy is not even improved by alloying additions [3]. This indicates that gamma grains have significant effect on the ductility of the alloys. Figure 5 shows the equiaxed microstructure of as-cast gamma alloy as reference [13].

There is a well known microstructure which exhibits very good creep strength. It is called the fully lamellae microstructure. Typical heat treatment to produce it is homogenization in the disordered α phase field at 1400°C for 2 hours in vacuum and slowly cooled to 1000°C for an aging treatment of 4 hours [16]. Figure 6 shows the fully lamellae microstructure.

The purpose to highlight this microstructure is to distinguish it with the lamellae microstructure which is exhibited by as-cast single phase near gamma alloy

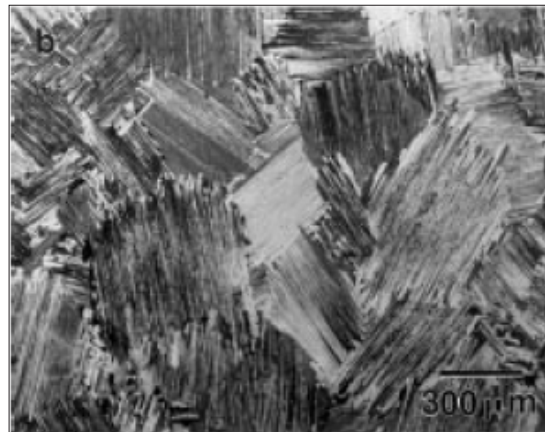**Figure 5**   Optical micrograph shows the equiaxed microstructure of as-cast single phase gamma alloys [13]
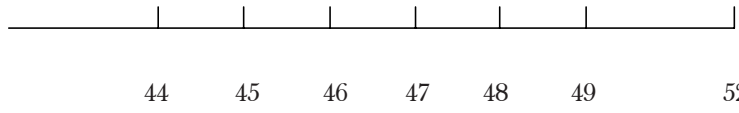

Figure 6   Optical micrograph shows the fully lamellae microstructure [17]

(Ti-45Al). Both lamellae and fully lamellae structures have 100% lamellae phase but there is a significant difference between them. The fully lamellae structure has finer lamellae grains compared to the lamellae structure. Another significant difference is that fully lamellae has an interlocked grain boundaries, whereas the lamellae structure has planar grain boundaries.

There is another important matter that has to be cleared here to avoid confusion. As stated earlier, near gamma alloys $(\alpha_2+\gamma)$ are divided into single phase (lamellae microstructure) and two phase (nearly lamellae and duplex) alloys. However, it is very common among researchers to classify these two phase alloy as gamma alloys. The common classification among researchers; 'gamma alloys are divided into single phase (equiaxed microstructure) and two phase (nearly lamellae and duplex microstructure) alloys. This classification have become common and widely accepted [5]. At the beginning, researchers had no interest on the near gamma alloys because

of the lamellae structured alloys which has no significant engineering properties. Therefore, researchers instead moved to the single phase gamma alloys (equiaxed microstructure). At this point, researchers found that if the Al content is reduced below 52at% to form Ti_3Al as a second phase besides the TiAl, the resulting two phase alloys show ductilities of a few percent, which are acceptable for applications [3]. Besides that, two phase alloy also has better creep strength. Thus, researchers had classified this new emerging alloys as two phase gamma alloys and it has been widely accepted. However, there were some researchers who classified the two phase alloys as near gamma alloys [7]. Both classifications have been accepted and are used world-wide. As far as this paper is concerned, the two phase alloys will be classified as near gamma alloys. Table 5 shows the classification of near gamma alloys. The sketched microstructure is only for comparison purpose and does not represent any quantitative measurements.

Table 5 Classification of near gamma titanium aluminides

Alloy	Near gamma		
Number of phase	Single	Two	
Microstructure	Lamellae	Nearly lamellae	Duplex
Sketching			
Aluminium content (at%)	44 45	46 47 48	49 52

4.0 CONCLUSIONS

(1) Ti-45Al, Ti-48Al and Ti-50Al exhibits lamellae, nearly lamellae and duplex microstructure respectively.
(2) All the samples showed the presence of hexagonal, Ti_3Al and tetragonal, TiAl.
(3) The lamellae grain size decreases whereas the volume fraction of single gamma phase increases as the amount of aluminum in the samples increases (45at.% to 50at.%).

(4) Ti-45Al showed planar grain boundary whereas Ti-48Al and Ti-50Al showed interlocked grain boundaries.

ACKNOWLEDGEMENTS

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