Micromechanisms of damage in a hypereutectic Ti–6Al–4V–B alloy

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Article info

Article history:
Received 17 December 2008
Received in revised form 16 April 2009
Accepted 21 April 2009

Keywords:
Titanium alloys
Microstructure
Acoustic emission spectroscopy
Interrupted tensile test

Abstract

The tensile stress–strain response and fracture in a hypereutectic Ti–6Al–4V–1.7B alloy were investigated by employing interrupted tensile tests combined with acoustic emission measurements, with the aim to identify the cause for the observed low ductility in this alloy. These tests were complemented with microscopy. The alloy contains TiB whiskers of different length scales, the majority of which include micro-whiskers (~5–10 μm length) and a few primary-whiskers (~200–300 μm length). Although the fracture of both types of whiskers occur during deformation, the former leads to a gradual decrease in the secant modulus whereas initiation of the latter leads to a drastic drop in the modulus along with failure of the specimen, limiting the ductility.

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1. Introduction

Titanium alloys are important aerospace structural materials because of their high mechanical performance, low density and ability to withstand elevated temperatures up to ~600 °C [1–3]. Many Ti alloy compositions and their microstructural design have been optimized over the past few decades and additional improvements have been sought through the composite route. Therefore, considerable attention was paid to fiber-reinforced Ti alloy matrix composites (TMCs) during 1980s and 1990s [4,5]. While TMCs exhibit some outstanding properties, large costs associated with fiber processing limit their widespread application. Additionally, they suffer from internal residual stresses arising from thermal expansion mismatch between the SiC fibers and the matrix, property anisotropy, notch sensitivity and multiple matrix cracking during fatigue loading [1,6–10]. Consequently, alternate routes to develop high performance Ti alloys and their composites are sought. One of these is the in situ composites approach.

Significant improvements to mechanical properties of Ti–6Al–4V (all alloy compositions are in weight percent), a widely used aerospace grade alloy, can be achieved by adding B. While minor additions of B (less than 0.1 wt.% B) refines the microstructure dramatically, addition of more than 1.0 wt.% B improves mechanical performance significantly [11–15]. This is attributed to in situ precipitation of TiB, which enhances the stiffness and strength. Amongst a number of candidate ceramic reinforcements, TiB possesses the most appropriate balance of thermomechanical stability, good mechanical properties and similar thermal expansion coefficients as well as ease of in situ processing due to its insolubility in the matrix [16–19]. More importantly, TiB precipitates in whisker morphology due to orthorhombic crystal structure, which is relatively more effective from a strengthening viewpoint compared to the approximately equiaxed shapes observed in most other reinforcements [16]. As a result of these factors, it is possible to achieve significant increase in stiffness and strength of Ti alloys with small amounts of B addition, produced by employing the powder metallurgy route [1,4,16,18]. Boron-modified titanium alloy were classified as alloys below the eutectic limit and as composites above the eutectic limit due to transition from ductile to brittle failure behavior [20].

A variety of techniques such as conventional casting, powder metallurgy, rapid solidification, and mechanical alloying have been used to produce the Ti–B alloys, and the final microstructural characteristics sensitively depend on the processing method and parameters [4,16]. Most of the earlier processing efforts in these alloy systems adopted the conventional casting route where there is little control over the sizes of TiB. In fact, the primary needles grow very large in size and make these composites extremely brittle. On the other hand, powder metallurgy (P/M) techniques have distinct advantages over the liquid metallurgy processes, which include control over the size, morphology, spatial distribution, and volume fraction of the reinforcement. Recently, modified Ti alloys with small additions of B were successfully produced through the P/M route [11,13,17].

The focus of this work is a hypereutectic Ti–6Al–4V–1.7B alloy (referred as Ti64–1.7B here afterwards) produced by P/M route. It has been shown in previous work that 1.7B addition causes significant enhancements to the room temperature Young’s modulus,
E (by ~24%), yield strength, YS (by ~21%) and ultimate tensile strength, UTS (by ~31%) of Ti64 [15,21]. Notably, these properties are maintained at elevated temperature also. Microstructural characterization of this alloy shows the presence of TiB needles/whiskers of different length scales [17,22]. Majority of these whiskers are 1–10 μm long and are referred as micro-whiskers whereas a few primary-whiskers (~200–300 μm long) are also present. A significant portion of strengthening in B modified alloys was found to be from the load-sharing mechanism by the micro-whiskers. Analytical modeling suggests that the observed strengthening is reasonably consistent with predictions based on continuum and dislocation models appropriate for the size scale effects of the various micro-constituents [23,24].

However, the plastic strain at failure of Ti64–1.7B is only 2.5–3.5%; significantly low as compared to the high (>10%) ductility of annealed Ti64 [2,25,26]. Clearly, imparting significant improvement to ductility (above 5%) while retaining the higher strength and stiffness is a key challenge. An understanding of the evolution of damage with deformation and identification of the responsible micromechanism is necessary to achieve this goal, which is the main objective of the work reported in this paper. Systematic interrupted tensile tests coupled with microstructural observations on Ti64–1.7B were conducted and analyzed along with acoustic emission measurements for this purpose.

2. Material processing and characterization

The material used in this work is produced via pre-alloyed powder metallurgy approach as described in Ref. [17]. This powder has been prepared by the liquid metallurgy process. The melting procedure involves induction skull (made of Ti alloy) melting of appropriate amounts of the raw materials (Ti, Al–V master alloy and TiB, which completely dissolves in the liquid melt and forms in situ TiB during solidification) in a water-cooled copper crucible. Alloy powder with a nominal composition of Ti–6Al–4V–1.7B is screened to obtain ~100 mesh size fractions (150 μm mesh opening size) and about 1 kg of it is packed inside a thick-walled (6.35 mm) Ti64 can with a 70 mm diameter and 130 mm length and vacuum out-gassed at 300 °C for 24 h. The can is subsequently coated with glass for lubrication and for minimizing the oxidation damage, heated to 1200 °C, soaked for 1 h, and then subjected to blind-die compaction (BC) in an extrusion chamber heated to 260 °C. The billet height is reduced by about 30% at a ram speed of 6.35 mm s\(^{-1}\). The compact was held at a pressure of 1400 MPa for 180 s, and subsequently air-cooled to room temperature (RT). Extrusion of the compacted billets is performed next after skin machining to remove the α case, using the following process schedule: heating to 1100 °C, soaking for 1 h, round-to-round extrusion using 16.5:1 conical die (effective strain = 280%), 6.35 mm s\(^{-1}\) ram speed, and air-cooled to RT. The starting microstructure consisted of ~8 vol.% TiB in an equiaxed α + β Ti64 matrix. The TiB existed in two distinctly different length scales: around 7.5 vol.% are micro-whiskers of 5–10 μm length and 1–3 μm diameter, whereas coarse primary TiB particles comprising of 0.5 vol.% have 200–300 μm length and aspect ratio of ~5.

3. Experiments

Structural damage was assessed by measuring mechanical properties during interrupted tensile testing. Flat, dog bone-shaped samples were cut from Ti64–1.7B rods of 20 mm diameter using electro-discharge machining (EDM). The recast layer after EDM was removed by grinding and polishing. Samples had a gage length of 35 mm and a cross-section of 8 mm (width) × 2 mm (thickness). Uniaxial loading was applied at a rate of 50 N s\(^{-1}\), and the strain was measured with an extensometer over a gage length of 25 mm. Damage was quantified by measuring the secant modulus, \(E_s\), during periodic sample unloading after the onset of inelastic deformation. Microstructural damage on sample surfaces was characterized with the scanning electron microscope (SEM) over three different strain ranges of 1–1.5%, 1.5–2.5%, and 2.5% to failure strain. Fracture surfaces were also examined in SEM.

Acoustic emission (AE) tests were performed following the standard method of ANSI/ASTM E 610–77 [27] on similar test samples loaded under uninterrupted uniaxial tension at a loading rate of 16 N s\(^{-1}\). A single piezoelectric AE transducer attached to the sample allows detection of transient elastic waves generated by failure events such as whisker micro-cracking during loading and deformation [28]. Documented AE signal characteristics included: the specified voltage level that must be exceeded before the signal is detected and processed (the AE preset threshold); the number of times the signal amplitude exceeds the preset threshold (the AE count); the time interval between the first and the last time the threshold is exceeded for a given AE event (the event duration); the maximum signal amplitude within the event duration (the peak amplitude); the number of events (also called hits) and the total energy of each AE event. Damage mechanisms were determined by careful comparison of the AE signal characteristics with the observed mechanical and microstructural damage.

4. Results and discussion

4.1. Interrupted tensile tests and microscopy

A typical interrupted tensile stress–strain response of Ti64–1.7B is shown in Fig. 1. The yield strength is more than 1 GPa and the ultimate tensile strength approaches 1.5 GPa. Progressive widening of the stress–strain hysteresis with strain indicates increasing damage accumulation with plastic strain. The variation of the secant modulus, \(E_s\), calculated from the unloading curves and normalized by the Young’s modulus, E, is shown as a function of the total strain, \(\varepsilon_T\), in Fig. 2. No perceptible drop in \(E_s/E\) was measured for applied strains less than ~0.5%. Beyond strain of ~0.5%, the variation of \(E_s/E\) with \(\varepsilon_T\) can be divided into three distinct regimes as marked in Fig. 2. Regime A spans 0.5% ≤ \(\varepsilon_T\) ≤ 2.1% and is characterized by a marginal drop in modulus. The variation of \(E_s/E\) with \(\varepsilon_T\) in this regime appears to be linear with a slope of ~0.027. In regime B, a precipitous drop in \(E_s/E\) by ~10% (from ~95% to 85%) is noted over a relatively short strain span of 2.1% ≤ \(\varepsilon_T\) ≤ 2.4%. Regime C continues to sample failure at 3.6%, and is characterized by a modest drop in modulus with a slope of ~0.036.
SEM images of the interrupted tensile test specimens, tested to different $\varepsilon_T$, show the evolution of cracks in micro- as well as primary-whiskers. Fig. 3 shows the micrographs of cracks at $\varepsilon_T \sim 1.3\%$ and $3.2\%$ in the micro-whiskers whereas Fig. 4 shows the cracks at corresponding strains in case of primary-whiskers. The average size of cracks observed in the primary-whiskers is much larger than that of cracks developed in micro-whiskers. It is observed from the images that both in the primary-whiskers as well as the micro-whiskers, cracks that develop at the initial stages of plastic regime are typically perpendicular to the direction of loading. However cracks in micro-whiskers start earlier, i.e. at lower strain compared to that in primary-whiskers as is evident from Fig. 5. These cracks in the micro-whiskers lead to the gradual reduction of the normalized $E_s$ within $\varepsilon_T \sim 0.5\%–2.0\%$, corresponding to regime A of $E_s/E$ vs. $\varepsilon_T$ plot (Fig. 2). It has also been observed that the cracks that developed in the earlier stages of loading in the micro-whiskers do not exhibit branching within the whiskers at later stages of deformation but widen with increasing strain (Fig. 6) and new cracks develop perpendicular to the tensile direction. Apart from that, cracks in micro-whiskers rarely propagate to the matrix during failure and only contribute in reducing the load-sharing effect of micro-whiskers. On the other hand, not only the number of newly developed cracks in the primary-whiskers increases with strain (Figs. 4 and 2) but also the initial cracks branch out in all the directions. These branching effects are also observed in the fractographs which show extensively fractured whisker with the transverse cross-section divided into a number of crack surfaces propagating in different directions (see for example Fig. 7). Thus

**Fig. 2.** Variation of secant modulus normalized with elastic modulus ($E_s/E$) and crack per whisker (CPW) with total strain ($\varepsilon_T$).

**Fig. 3.** Evolution of cracks in micro-whiskers at various strains $\varepsilon_T = (a) 1.3\%$ and (b) $3.2\%$. Arrow indicates the loading direction.

**Fig. 4.** Evolution of cracks in primary-whiskers at various strains $\varepsilon_T = (a) 1.3\%$ and (b) $3.2\%$. Arrow indicates the loading direction.
rapid growth of cracks in the primary-whiskers leads to the precipitous reduction in the $E_s/E$ with the advancement of $\varepsilon_T$ in regime $B$ (Fig. 2). Fig. 2 also shows the change in the number of cracks per whisker (CPW), counted and averaged using a large number of micrographs, for primary-whiskers as well as the total number of whiskers, with increasing strain in the sample. It is evident from the graph that with increasing $\varepsilon_T$, CPW increases very rapidly in primary-whiskers and hence a sudden drop in $E_s$ is noticed.

Along with the cracks in the whiskers, other microstructural features such as matrix cracking and propagation of whisker cracks into the matrix and crack widening in micro-whiskers (Fig. 6) are also observed. This helps in explaining the progressive widening of the tensile stress–strain hysteresis observed in Fig. 1, which is possibly due to the fact that micro-crack open during the loading part, leading to plastic flow of the matrix that is at the crack-tip locations. During unloading, these blunted tips resist closure of the cracks leading to a hysteresis. Larger the applied stress maximum, higher will be the blunting and hence higher will be the hysteresis width. The reduction in the normalized $E_s$ within $\Delta\varepsilon_T \sim 2.2–3.5\%$, i.e. regime $C$ of Fig. 2 is probably because of these features and also linking of these cracks in the whiskers through the matrix.

4.2. Acoustic emission

The various acoustic emission signatures of Ti64–1.7B composite with respect to the strain till failure are shown in Fig. 8. In the number of counts vs. $\varepsilon_T$ plots (Fig. 8a), only the bursts are recorded discarding the background continuous signal. These bursts are directly proportional to micro-cracks in the material because of the fact that the material shows Kaiser effect [29], i.e. in every cycle, the acoustic emission starts only when the stress value crosses the maximum stress value of the previous cycle. Fig. 8b and c represents the variation of number of hits and duration of events respectively with $\varepsilon_T$.

It is evident from all the graphs of AE tests that the entire strain range can be broadly divided into three distinct regimes. Most of the events at regime I for $\varepsilon_T$ below about 0.5% (i.e. stresses below about 550 MPa) have a very low number of counts (Fig. 8a) and hits (Fig. 8b) as well as very low duration (Fig. 8c). This could be the signals coming from damage of TiB whiskers that are on the free surface, which are probably damaged by polishing and fail at low loads. There are just a couple of such events below 0.5% strain as is evident from the duration plot, but they become dominant above $\varepsilon_T \sim 0.5\%$. The next regime (regime II) starts at $\varepsilon_T \sim 0.5\%$ and continues up to $\varepsilon_T \sim 1.3\%$ (i.e. a stress level of 550–1200 MPa). This population of events has low but observable and steadily increasing activities of counts and hits with the durations being increased to 1500–2500 relative units. The steady drop in $E_s/E$ in regime A of Fig. 2 suggests that the activities in AE during the first two regimes represent failure of the micro-whiskers. Beyond $\varepsilon_T \sim 1.3\%$, the AE plots show a drastic rise in the number of counts, hits as well as duration of events, indicating the start of regime III. This is due to the increase in cracks in micro- and primary-whiskers as well as branching of the cracks and linking of the cracks in different
whiskers through the matrix that causes failure of the specimen. It should be noted at this point of time that the three regimes of Fig. 2 as obtained from the drop in normalized $E_S$ with $\varepsilon_T$ does not have a 1:1 correspondence with the three regimes as found from the AE tests. Thus regime III of Fig. 8 corresponds to the later part of regime A along with regimes B and C of $E_S/E$ vs. strain plot of Fig. 2.

5. Conclusions

SEM images of Ti64–1.7B confirm the presence of TiB whiskers of different sizes in the Ti64 matrix, a major fraction of which include micro-whiskers ($\sim$5–10 $\mu$m in length) of volume fraction $\sim$7.5%. Apart from that, a few primary TiB whiskers of length $\sim$200–300 $\mu$m are also present. All these whiskers play significant roles in the strength and failure of the composite. For example, cracking in micro-whiskers start early, i.e. at lower strains, which leads to the gradual drop in $E_S/E$ during the first regime. Also, micro-whiskers mostly help in increasing the strength of the composite by load-sharing mechanism. On the other hand extensive cracking and branching of those cracks in the primary-whiskers with increasing strains lead to a precipitous drop in $E_S/E$ as found in the second regime. All these cracks in the whiskers propagate through the matrix in the third regime that again leads to a steady drop in $E_S/E$ and ends up with the catastrophic failure of the specimen that limits ductility. Thus for increasing ductility of this composite it is important to limit the size and number of the primary-whiskers in the composite.

Acknowledgements

The authors are thankful to Asian Office of the Aerospace Research & Development (AOARD) for supplying the materials and sponsoring the project. IS and UR are also grateful to The Boeing Company for their support.

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