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ROOM AND ELEVATED TEMPERATURE MECHANICAL PROPERTIES OF PM TiAl ALLOY Ti-47Al-2Cr-2Nb*

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Abstract

A TiAl alloy powder with the composition Ti-47Al-2Cr-2Nb (at. %) was prepared by rotary atomization, followed by hot-extrusion and subsequent heat-treatments to produce refined lamellar structures and fine duplex structures. The mechanical properties of the TiAl alloy were determined at temperatures to 1000°C in air, and the microstructures were characterized by TEM, SEM, and electron microprobe analyses. The alloy with the refined lamellar structure showed excellent mechanical properties at both room and elevated temperatures. It exhibited a plastic strain of 1.4% and a yield strength of 971 MPa (140.9 ksi) at room temperature. The yield strength remained approximately constant up to 800°C and decreased to 577 MPa (83.7 ksi) at 1000°C. The transverse fracture toughness, estimated by three-point bend testing of chevron-notched specimens at room temperature, was 22.4 MPa $\sqrt{m}$. The refined lamellar structure contained long and straight alternating α_2 and γ platelets with an extremely fine interlamellar spacing (0.1 μm) and α_2-to-α_2 spacing (0.22 μm). The mechanical properties of the alloy have been correlated with the unique microstructures developed by hot extrusion.

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Introduction

Mechanical properties of two-phase γ titanium aluminide alloys are sensitive to microstructural features.(1-6) Near γ, duplex, near lamellar, and full lamellar structures can be produced in titanium aluminide alloys containing 46 to 50 at. % Al by control of heat treatment, thermomechanical processing, and cooling rate.(1-8) In the case of the lamellar structures, which possess good creep resistance at elevated temperatures(9) and fracture toughness at room temperature,(6,10) the mechanical properties are strongly dependent on colony size and interlamellar spacing, as evidenced from recent studies.(1-6) At present, most γ titanium aluminide alloys are prepared by various casting methods; however, the cast materials generally suffer from two technical difficulties, i.e., microscopic/macroscopic segregation and large colony size.(1-3,6,11) These are not considered to be problems for titanium aluminide materials produced by consolidation of rapidly solidified powder. The titanium aluminide alloys processed by powder metallurgy (PM) in the past were shown to contain high concentrations of interstitial impurities, typically several thousand ppm by wt.(12,13) Recent advances in powder production indicate no major problem in the production of titanium aluminide powders with interstitial contents below 1000 wt ppm.(14,15) Also, characterization of the mechanical properties provides evidence that this level of interstitials has no adverse effect in γ titanium aluminide alloys.(16)

The objective of this study is to process two-phase γ titanium aluminide alloys by PM, with the ultimate goal of improving their mechanical properties through careful control of microstructure. The alloy Ti-47Al-2Cr-2Nb (at. %) was selected in this study because of its balanced mechanical and metallurgical (such as oxidation resistance) properties.(5) Most PM titanium aluminide alloys prepared currently are consolidated mainly by HIPping.(12-14,16) In this study, the PM materials were consolidated by hot extrusion at various extrusion temperatures. Our studies have demonstrated that the mechanical properties of Ti-47Al-2Cr-2Nb can be dramatically improved by controlling the microstructural features through hot extrusion of rapidly solidified alloy powder.

Experimental Procedures

TiAl alloy powder with the composition Ti-47Al-2Cr-2Nb was produced by a rapid solidification technique, using the rotary atomization facility at Pratt & Whitney located in West Palm Beach, Florida. The powder weighing ~15 kg was canned in Ti cans and hot extruded at a ratio of 16 : 1 at temperatures below or above the α transus temperature, T_{α}. In order to control the microstructure and mechanical properties, the as-extruded rods with 2 cm diam were heat treated at temperatures of 800°-1350°C in a vacuum of ~10^{-4} Pa, followed by cooling in the furnace. Button-type tensile specimens with gage dimensions of 1.27 cm length x 0.32 cm diam were prepared first by electro-discharge machining (EDM) and then ground to the final dimensions. The specimens were heat treated at desired conditions in vacuum and then mechanically polished to remove surface scratches using 0-grade Emery papers. Tensile tests were performed on an Instron testing machine at temperatures to 1000°C in air at a crosshead speed of 0.25 cm per min. The specimens were heated inductively inside a Pt susceptor, and the test temperature was controlled using a Pt-10% Rh thermocouple located at the specimen center. Tensile properties were determined from a strip chart recorded at a speed of 12.7 cm/min. Fracture toughness was determined at room and 800°C temperatures in air by three-point bend testing of chevron-notched specimens with the dimensions 5 x 5 x 45 mm.(17) The fracture toughness was calculated from the area of the measured load-displacement curves. Fracture surfaces were examined by scanning electron microscopy (SEM) operated at 5 kV.

Microstructural features and phase compositions were studied by both transmission electron microscopy (TEM) and electron microprobe analyses. Microstructural analysis was performed on Philips CM12 (120 kV) and CM30 (300 kV) microscopes. Phase transition was monitored by differential scanning calorimetric (DSC) analyses of alloy samples at a heating rate of 20°C/min in an Ar atmosphere.

Results

Canned alloy powder was successfully consolidated by extrusion at temperatures above T_α (T_1) or below T_α (T_2 and T_3, with $T_1 > T_2 > T_3$). Wet chemical analyses indicated that the rod extruded at T_1 contained 800 wt ppm oxygen, 270 carbon, and 35 nitrogen. The DSC measurements showed that the T_α was around 1320°C. The $(\gamma + \alpha)$ field extends to 1220°C, which agrees well with that reported by Takeyama, et al.(18)

Figure 1 shows the microstructures of PM Ti-47Al-2Cr-2Nb in the hot-extruded conditions. Hot extrusion at T_1 in the α phase field resulted in a fully lamellar structure with a colony size (or grain size) of 65 μm [Fig. 1(a)]. Hot extrusion at T_2 and T_3 produced an extremely fine duplex structure, and the detailed grain structure can be seen by scanning electron microscopy [Fig. 1(b)]. The as-extruded materials have near theoretical density. There appears to have residual porosities in the material, the amount of which decreases with increasing extrusion temperature, with the least porosity in the material hot extruded at T_1.

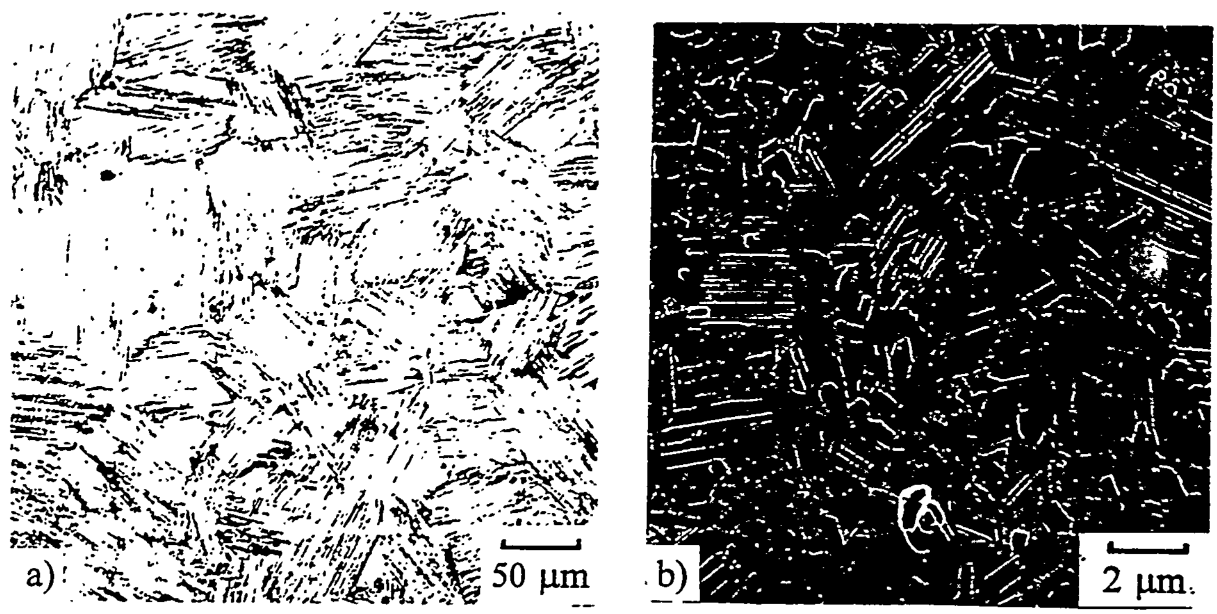


Fig. 1. Microstructure of PM Ti-47Al-2Cr-2Nb hot extruded at (a) T_1 (optical micrograph, 200x and (b) T_3 (SEM), 5000x.

The as-extruded materials were heat treated between 800° to 1350°C for up to 4 h. The heat treatment at ≤ 900°C did not cause any apparent change in the optical microstructure, while the heat treatment at higher temperatures resulted in the formation of more γ grain regions along colony boundaries in the refined lamellar structure produced by hot extrusion at T_1. The heat treatment of the T_3-extruded material for 4 h at 1325°C resulted in the formation of a similar fine lamellar structure, except that large cavities (5-21 μm in size) were observed in the heat treated material. Such porosity can easily be seen on tensile fracture surfaces and will be shown later.

The microstructural features in the T_1-extruded material were examined carefully by TEM. Figure 2 shows the refined lamellar structure, with α_2 platelets appearing essentially as dark lines due to a dynamic contrast effect. Note that the α_2 platelets are long and straight, and quite regularly spaced. Dislocation images are visible in the brighter γ regions of Fig. 2. The detailed analyses of interlamellar spacing and α_2/γ platelet widths are given in Table I. The average interlamellar spacing is extremely fine, only 0.1 μm. The correspondence of α_2/γ lamellaes is almost 1 : 1, with only the widest γ lamellae having γ/γ twins inside. Figure 3 shows, along the colony boundaries, the formation of coarsened γ lamellar bands and new equiaxed γ grains, which are so fine that they cannot be resolved in optical micrographs. The volume fraction of γ along the boundaries is estimated to be less than 5%, with many colony boundaries having interpenetrating lamellae and no coarse γ region at all. The heat treatment at

900°C for 2 h did not cause a significant change in the T_1-extruded lamellar structure and may have only slightly thickened the γ layer at colony boundaries. This heat treatment also introduced a few small regions of coarsened lamellae inside the colonies. A more detailed analysis of the lamellar structure and its coarsening processes during various heat treatments will be published elsewhere.[19]

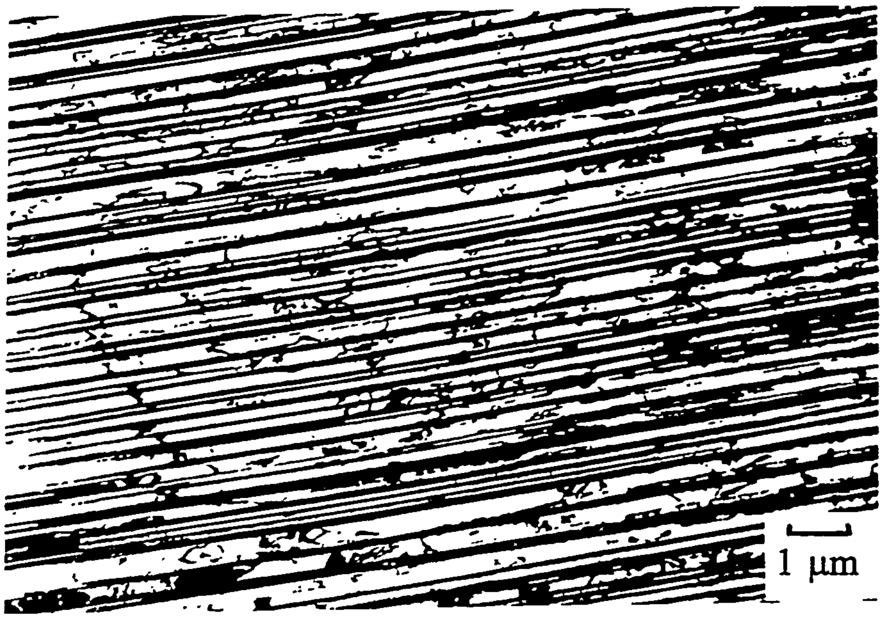


Fig. 2. A TEM micrograph showing fine α_2 platelets in PM material hot-extruded at T_1.

Table I. Quantitative Microstructural Data for PM Ti-47Al-2Cr-2Nb (at. %)
in As-extruded and Heat-Treated Conditions

Material Preparation	Microstructural Data
As-extruded at T_1	Colony size: 65 µm Width of colony-boundary γ layer: 1-3 µm Interlamellar spacing: 0.1 µm α_2-α_2 spacing: 0.22 µm γ lamellar width: 0.1-0.5 µm α_2 lamellar width: 20-76 nm α_2-γ layer ratio: ~1:1 Very few γ/γ boundaries
T_1 extrusion + 2h/900°C	Similar colony size: (~65 µm) Slightly thicker γ layer at colony boundaries Few coarsened γ spots within colonies
T_3 extrusion + 2h/1325°C	Colony size: ~59 µm Porosity size: 5-21 µm

The room-temperature tensile properties of PM Ti-47Al-2Cr-2Nb were determined in the as-extruded and heat treated conditions. The tensile data are summarized in Table II for both the fine duplex and the refined lamellar structures. The T_3-extruded material with a fine duplex structure showed no plastic strains in the as-extruded and in the extruded and heat treated conditions. The fracture strength increased with annealing temperatures to 1070°C, followed by a sharp decrease at and above 1200°C. The T_2-extruded material with a duplex structure showed $\leq$ 0.6% plastic strain in the as-extruded condition and heat treated conditions at temperatures $\leq$ 1050°C. The material lost its plastic ductility completely when heat treated at and above 1250°C. The yield and fracture strengths decrease with annealing temperature, and they showed a sharp decrease at $\geq$ 1250°C. The T_1-extruded material with a refined lamellar structure exhibited tensile ductilities in the as-extruded and heat treated conditions, with the greatest ductility (= 1.4%) obtained for 900°C heat treatment. It is interesting to point out that

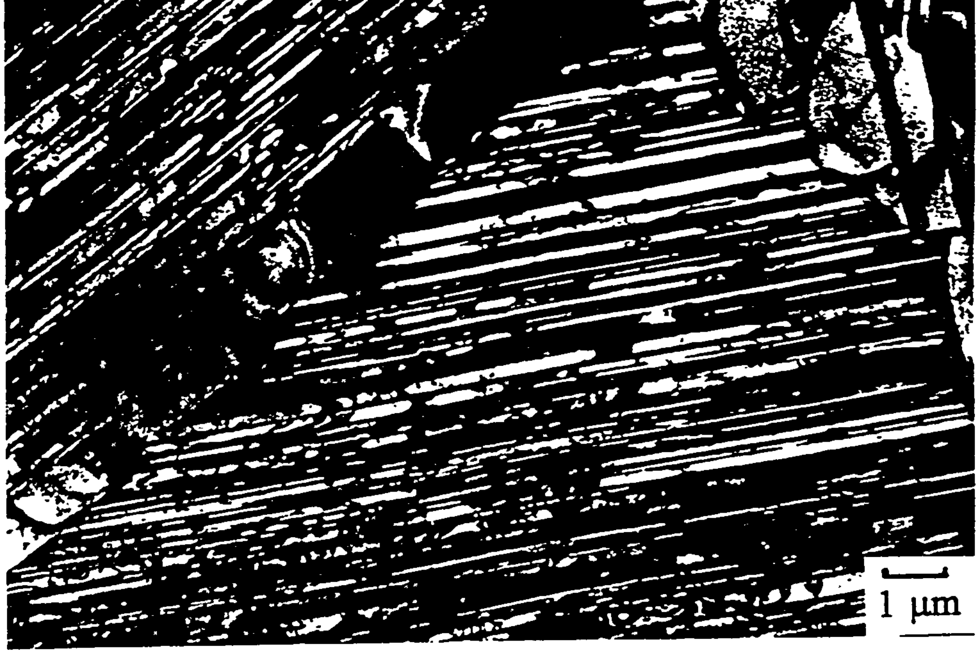


Fig. 3. TEM micrograph showing γ bands and grains formed along colony boundaries in T_1-extruded material.

Table II. Effect of Heat Treatment on Room-Temperature Tensile Properties of PM Ti-47Al-2Cr-2Nb (at. %) Hot-extruded at T_1, T_2 or T_3

Heat Treatment	Yield Strength (MPa)	Fracture Strength (MPa)	Plastic Strain[a] (%)
		T_3 Extrusion	
As-extruded[b]	–	701	0
4h/1000°C[b]	–	704	0
4h/1070°C[b]	–	972	0
4h/1200°C[b]	–	535	0
4h/1325°C[c]	–	430	0
		T_2 Extrusion	
As-extruded[b]	997	1029	0.5
2h/950°C[b]	945	949	0.6
2h/1050°C[b]	878	878	0.4
2.5h/1250°C[b]	–	398	0
2h/1350°C[c]	–	540	0
		T_1 Extrusion	
As-extruded[c]	992	992	0.6
2h/900°C[c]	971	1005	1.4
2h/950°C[c]	977	1017	1.0
2h/1000°C[c]	968	998	1.1

[a]Measured from strip chart [b]Fine duplex structure [c]Refined lamellar structure

the yield and tensile strengths (140-150 ksi) of the T_1-extruded material with the refined lamellar structure are comparable to those obtained from the fine duplex structures. Also, the room-temperature strengths remained almost constant without dropping when heat treated at temperatures to 1000°C. It is important to note that the lamellar structures produced by heat treating the materials extruded at T_3 and T_2 showed virtually no plastic strain, while the lamellar structures produced by hot extrusion at T_1 exhibited decent tensile ductilities in both the extruded and heat treated conditions, even though all the materials had similar refined lamellar structures and colony sizes (~59 μm).

The tensile properties of the T_1- and T_2-extruded materials heat treated to optimize room-temperature ductility were then determined as a function of test temperature up to 1000°C. The results are plotted against test temperatures in Fig. 4(a) and (b) respectively for the T_1-and T_2-extruded materials. The T_1-extruded and 900°C-heat treated material with the refined lamellar structure showed only a slight decrease in strength at temperatures to 800°C, and a substantial decrease above that temperature. The yield and ultimate tensile strengths of this material remain as high as 577 MPa (83.7 ksi) and 624 MPa (90.5 ksi), respectively at 1000°C. The tensile ductility of the material showed a moderate increase with temperature and reaches a maximum of 7.5% at 800°C. The T_2-extruded plus 950°C-heat treated material with the fine duplex structure exhibited a moderate decrease in strength at ≤ 600°C and a sharp decrease above 600°C. This material had a yield strength of 172 MPa (25 ksi) and an ultimate tensile strength of 203 MPa (29.5 ksi) at 1000°C, which were much lower than those obtained from the refined lamellar structure. This material showed a sharp ductile-to-brittle transition around 700°C, and the ductility increased to as high as 117.6% at 1000°C.

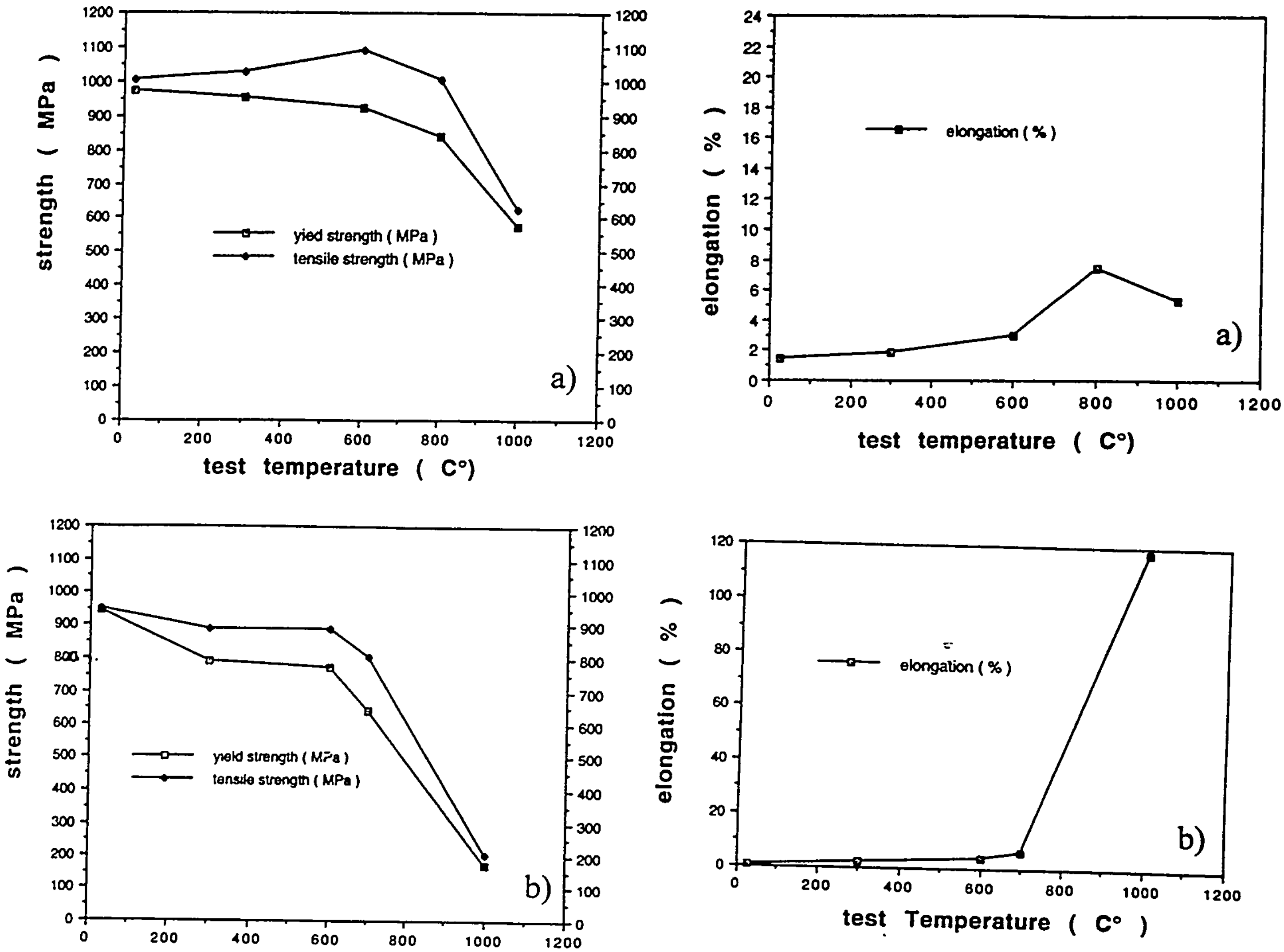
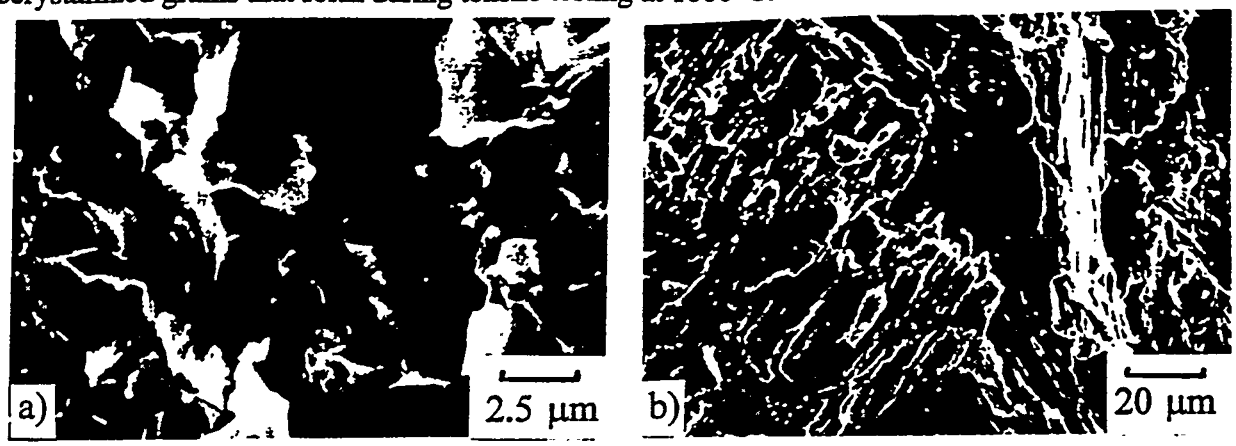


Fig. 4. Plot of tensile properties as a function of temperature for the materials (a) hot-extruded at T_1 and heat treated for 2 h at 900°C, and (b) hot-extruded at T_2 and heat treated for 2 h at 950°C.

The material with the fine duplex structure showed a mixed fracture mode [Fig. 5(a)] with cleavage fracture as the major fracture mode at room temperature. The material with the refined lamellar structure exhibited essentially translamellar fracture [Fig. 5(b)], with individual lamellar-colony facet visible on fracture surfaces. Large pores [Fig. 5(c)] as big as 15 µm are observed on fracture surfaces of the lamellar structure produced by T_3-extrusion plus 1325°C-heat treatment. The T_1-extruded and 900°C-heat treated material with the refined lamellar structure showed no change in fracture mode at temperatures to 800°C, while fine nodules were observed on 1000°C fractured facets (Fig. 6). These nodules are presumably dynamically recrystallized grains that form during tensile testing at 1000°C.



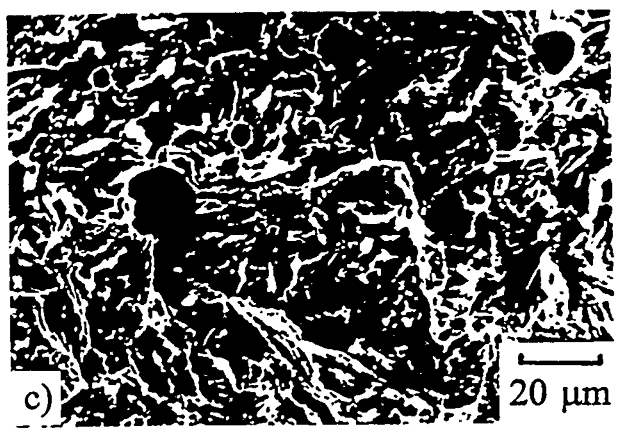


Fig. 5. SEM fractographs of PM materials fractured at room temperature. (a) Hot extrusion at T_3 and heat treatment for 2 h at 950°C, (4000x); (b) hot extrusion at T_1 and heat treatment for 2 h at 900°C, (500x); and (c) hot extrusion at T_3 and heat treatment for 4 h at 1325°C, 500x.

The fracture toughness of the materials hot extruded at T_1 or T_2 was determined by three-point bend testing of chevron-notched specimens at room temperature and 800°C in air. The material with the refined lamellar structure has a high toughness of 22.3-22.6 MPa $m^{1/2}$ while the material with the fine duplex structure has a low toughness of 9.3-9.7 MPa $m^{1/2}$ at room temperature. The fracture toughness for the refined lamellar structure increased to 40.1-41.4 at 800°C. The fracture modes by crack growth at room temperature are basically consistent with the fracture modes produced by tensile testing at room temperature (Fig. 5).

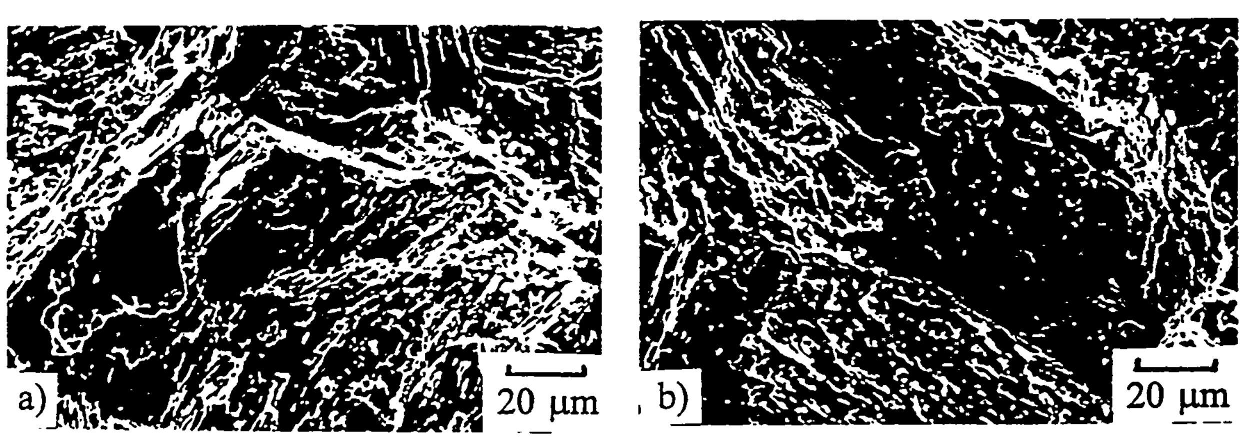


Fig. 6. SEM fractographs of PM material hot-extruded at T1 and heat treated for 2 h at 900°C (500x). Tested at (a) 600°C and (b) 1000°C in air.

Discussion

The α transus temperature, T_α, was determined to be 1320°C by DSC. This temperature is consistent with T_α reported by Takeyama and Kikuchi[18] (T_α = 1320°C for Ti-47Al-2Cr-2Nb) and Huang[5] (T_α = 1325°C for Ti-47Al-2Cr) but is lower than that reported recently by Fuchs[14] (T_α = 1363°C for Ti-48Al-2Cr-2Nb*). The heat treatment of the T_3-extruded duplex structure for 4 h at 1325°C resulted in a fine lamellar structure. This observation supports that the T_α of Ti-47Al-2Cr-2Nb should be slightly below but near 1325°C.

Hot extrusion at T_1 produced an essentially fully lamellar structure with a refined colony size of 65 µm. In comparison, Kim[6] recently observed a lamellar colony size of 200-400 µm for thermomechanically treated cast TiAl alloys containing W and other alloying elements. HIPping of Ti-48Al-2Cr-2Nb powder compacts by Fuchs[14] produced a near lamellar structure with the lamellar colony size of 200-250 µm. The interlamellar spacing in the T_1-extruded material was measured to be 0.1 µm in this work, which was much finer than that measured from cast TiAl alloys[6,7] with a spacing = 0.4 to 4.8 µm, dependent of alloy composition, thermomechanical treatment, and cooling rate. Fuchs[14] recently reported interlamellar spacings of 0.065-0.225 µm in PM Ti-48Al-2Nb-2Cr materials air-cooled from HIPping/heat-treatment temperatures. One unique feature of the refined lamellar structure produced by the T_1 extrusion is that the α_2 platelets are long, straight, quite regularly spaced within the fine lamellar structure, with the α_2 to γ lamellae ratio close to 1 : 1. As shown in Table I, the average α_2-α_2 spacing, measured from the center-to-center distance between two neighboring α_2 platelets, is 0.22 µm, which is roughly double the interlamellar spacing (= 0.1 µm). Based on this detailed microstructural characterization, the refined lamellar structure can be considered almost as a micro-laminate material consisting of alternating α_2 and γ platelets. In comparison, the α_2 platelets in fine lamellar structures produced by fast cooling of cast materials contain many irregular and short segments, as shown by Takeyama, et al.[7] Such imperfections in the lamellar structure reduce its beneficial effect on mechanical properties and cause degradation of the lamellar structure at high temperatures.[20]

For the PM materials produced by hot extrusion, the fine duplex structures had a tensile ductility less than that of the refined lamellar structure, as indicated in Table II. This is quite different from cast materials which generally show higher ductilities for fine duplex structures.[1-3,5,6] A possible reason for this property difference is that the duplex structures produced by hot extrusion at a lower temperature (e.g. T_3) contains more porosity, which reduces the room-temperature ductility. This reasoning is supported by the fact that the refined lamellar structure, produced by heat treating the T_3-extruded material for 4 h at 1325°C, still had a very low tensile ductility at room temperature (see Table II). The heat treatment does not remove the porosity produced at lower extrusion temperatures. As shown in Fig. 5(c), this material exhibited large pores (~15 µm) on the tensile fracture surfaces. Texture may be another factor which may affect the tensile ductility, and further studies are required to compare the texture difference between the material hot extruded at T_1 and the material heat treated at 1325°C after T_3 hot extrusion.

The T_1-extruded material showed excellent strengths at room and elevated temperatures. Table III compares the tensile properties of the extruded PM material with PM Ti-48Al-2Cr-2Nb prepared by HIPping[12] and the cast TiAl alloy (Ti-46.5Al-2.1Cr-3Nb-0.2W) prepared by thermomechanically treatment.[6] All the materials had refined lamellar structures. As shown in the table, the yield and tensile strengths of the extruded PM material are higher than those of the other two materials by more than 100% at all temperatures. Our characterization of the refined lamellar structure by TEM suggests that the superior strengths of the extruded material is due to a combination of the extremely fine colony size (65 µm) and interlamellar spacings (0.1 µm) together with the unique ultrafine morphology of the α_2 platelets, which are long and straight, and quite regularly spaced between γ platelets. In fact, the extruded lamellar material can be considered as a micro-laminate consisting of hard α_2 platelets and soft γ platelets. Our studies have demonstrated the importance of controlling fine structures within the general lamellar structures, and further investigation is certainly needed to develop a structure/property correlation for TiAl alloys with lamellar structures.

* The increase in Al concentration from 47 to 48% increases T_α by ~25°C.[5]

Table III. Comparison of Tensile Properties of TiAl Alloys with Refined Lamellar Structures Prepared by Casting or Powder Metallurgy

Alloy Composition (at. %)	Material[a] Preparation	Yield Strength (MPa)	Ultimate Tensile Strength (MPa)	Tensile Elongation (%)
	RM			
Ti-47Al-2Cr-2Nb	PM+HE+HT	971	1005	1.4
Ti-48Al-2Cr-2Nb	PM-HIP/HT	370	488	2.3
Ti-46.5Al-2.1Cr-3Nb-0.2W	MC+TMT	473	557	1.2
	600°C			
Ti-47Al-2Cr-2Nb	PM+HE+HT	922	1091	3.0
Ti-48Al-2Cr-2Nb	PM-HIP/HT	317[b]	527[b]	5.0[b]
Ti-46.5Al-2.1Cr-3Nb-0.2W	MC+TMT	408	525	1.7
	800°C			
Ti-47Al-2Cr-2Nb	PM+HE+HT	841	1005	7.5
Ti-48Al-2Cr-2Nb	PM-HIP/HT	322[c]	454[c]	55.6[c]
Ti-46.5Al-2.1Cr-3Nb-0.2W	MC+TMT	385	510	8.1
	1000°C			
Ti-47Al-2Cr-2Nb	PM+HE+HT	577	624	5.3
Ti-48Al-2Cr-2Nb	PM-HIP/HT	–	–	–
Ti-46.5Al-2.1Cr-3Nb-0.2W	MC+TMT	278	284	–

[a]PM=powder metallurgy; HE=hot extrusion at T_1; HT=heat treatment; HIP=hot isostatically pressed; MC=melting and casting; TMT=thermomechanical treatment

[b]Tested at 500°C   [c]Tested at 850°C

The tensile ductility of the T_1-extruded material with the refined lamellar structure increased steadily with temperature and reached a maximum at 800°C. However, the plot of ductility as a function of temperature gives no clear indication of ductile-to-brittle (DBT) transition, as shown in Fig. 4(a). Kim,(6) on the other hand, reported a DBT temperature of around 800°C for refined lamellar structures produced from thermomechanically treated cast TiAl alloys. The reason for this discrepancy is not known. The duplex structure produced by T_2 extrusion showed a sharp increase in ductility around 700°C, [Fig. 4(b)], consistent with the DBT temperature of 650°-700°C reported by Kim(6) and Fuchs(12) for a fine duplex structure. The sharp increase in ductility above 700°C is believed to be related to the onset of thermally-activated deformation processes.

The refined lamellar structure with the colony size of 65 µm, produced by T_1 extrusion plus 2 h/900°C heat treatment, gives an average fracture toughness of 22.4 MPa $m^{1/2}$ at room temperature. This toughness value was slightly higher than the toughness, K_Q (= 20.8 MPa $m^{1/2}$) for Ti-47.5Al-2.1Cr-3.0Nb-0.2W having a refined lamellar structure with a colony size of ~300 µm.(6) Recently, Chan and Kim,(2) and Kim(6) have correlated the fracture toughness at room temperature with grain or colony size. Their correlation would predict a fracture toughness of ~15 MPa $m^{1/2}$ for a colony size of 65 µm. The fracture toughness measured from the T_1-extruded refined lamellar structure is much higher than the predicted value. This may be due to the unique α_2 platelet structure as well as the fine lamellar spacings. In a recent paper, Kim(6) pointed out the importance of the characterization and control of lamellar structure (such as lamellar spacing) for improved mechanical properties. Our data support that concept and suggest additional microstructural parameters that are important. The fracture toughness of the T_1-extruded material with the refined lamellar structure increased from 22.4 to 40.8 MPa $m^{1/2}$ when the test temperature increased from room temperature to 800°C. The increase in toughness is consistent with the higher tensile ductility measured at 800°C. The measured toughness at 800°C agrees well with the data reported recently by Rogers and Bowen for a cast TiAl alloy with a coarse lamellar structure.(22)

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