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Microstructural control and mechanical properties of dual-phase TiAl alloys

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This paper summarizes our recent work on the effects of microstructural features on the mechanical properties of TiAl alloys prepared by powder and ingot metallurgy. TiAl alloys based on Ti–47Al–2Cr–2Nb (at%) were alloyed with small amounts of Ta, W, and B additions for control of alloy phases and microstructure. The alloys were processed by hot extrusion above and below T_α, followed by short- and long-term heat treatments at temperatures to 1350°C in vacuum. The microstructural features in the lamellar structures were characterized by metallography, SEM and TEM, and the mechanical properties were determined by tensile tests at temperatures to 1000°C. The tensile elongation at room temperature is mainly controlled by the colony size, showing an increase in ductility with decreasing colony size. The yield strength, on the other hand, is sensitive to the interlamellar spacing. Hall–Petch relationships hold well for both yield strength and tensile elongation at room and elevated temperatures. TiAl alloys with refined colony size and ultrafine lamellar structures possess excellent mechanical properties for structural applications at elevated temperatures. © 1998 Elsevier Science Limited. All rights reserved

Key words: A. titanium aluminides, based on TiAl, B. mechanical properties at ambient and high temperatures, yield stress, ductility, C. extrusion, heat treatment, D. microstructure.

INTRODUCTION

The mechanical properties of dual-phase TiAl alloys are sensitive to both alloy composition and microstructure.[1–8] The two different types of microstructure commonly formed in TiAl alloys are lamellar structures generally observed in cast conditions, and duplex structures formed in thermomechanically treated conditions. The lamellar structures with coarse grain sizes ($>500\,\mu$m) exhibit adequate fracture toughness but usually poor tensile ductility at ambient temperatures; on the other hand, the duplex structures with their fine grain size ($<50\,\mu$m) show adequate tensile ductility but poor fracture toughness. More importantly, the duplex structures exhibit poorer strength and creep resistance at elevated temperatures, as compared with the lamellar materials.[3,9,10]

For the past decade or more, considerable effort has been devoted to microstructural design of two-phase TiAl alloys with balanced properties for potential structural applications.[1–13] In particular, a great deal of research has been focused on various thermomechanical treatments of lamellar TiAl alloys in order to improve their room-temperature properties through reductions of lamellar colony size and interlamellar spacing.[6] Liu *et al.*[2] attempted to control the microstructure of Ti–47Al–2Cr–2Nb (at%) by hot extrusion of alloy powder at temperatures above and below T_α, the α transus temperature (1320°C). They obtained a refined colony size of 65 μm for TiAl alloy powder hot extruded at 1400°C. Tensile tests at room-temperature indicated that the tensile ductility of this alloy with a refined fully lamellar structure was higher than that of the same alloy with a fine duplex structure produced by hot extrusion at 1250°C (below T_α). Subsequent work to similarly control the microstructure of hot extruded ingot materials has further demonstrated that a room-temperature tensile ductility as high as 5% can be achieved for lamellar structures with a colony size as fine as 22 μm.[11,14,15]

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This paper summarizes our recent work on control of microstructures and mechanical properties of TiAl alloys based on Ti–47Al–2Cr–2Nb produced by hot extrusion above and below T_α. The microstructural parameters such as colony size (or grain size), lamellar spacing, and intercolony phase were controlled by material processing and heat treatment, and microstructural features were characterized by optical, scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The mechanical properties were determined by tensile testing at temperatures up to 1000°C. Our studies reveal that the average colony size and the interlamellar spacing are the two key microstructural parameters for controlling the tensile properties of lamellar TiAl alloys at room and elevated temperatures.

EXPERIMENTAL PROCEDURES

Table 1 lists the alloy compositions and hot extrusion conditions for the TiAl alloys prepared by ingot metallurgy (IM) and powder metallurgy (PM). The base composition of Ti–47Al–2Cr–2Nb (at%) was modified with additions of 0·15B, 0·2W and/or 1Ta, as well as with ±1% variation of the Al concentration. Boron and tungsten were added for refining the lamellar structure and improving its stability.[16–18] Tungsten and tantalum were also reported to have the beneficial effect of enhancing creep and oxidation resistance at elevated temperatures.[3,19,20] The two alloys containing 47% Al, T2-73 and T2-74, were prepared by hot extrusion of rapidly solidified alloy powders at 1150 to 1400°C. The five alloys containing 46 to 48 at% Al, TIA-20 to 25 were prepared by arc melting and drop casting into copper molds, using commercially pure charge materials. The alloy ingots were then canned in molybdenum billets and hot extruded at 1350 to 1400°C.

The hot extruded alloys were heat treated at different conditions (see Table 2), including short-term treatment for 2 h at temperatures from 900 to 1350°C and long-term treatment for 5040 h at 800°C. Buttonhead type tensile specimens (gage section: 9·53 mm length × 3·18 mm diameter) were prepared from hot-extruded and heat-treated materials by electro-discharge machining, followed by surface grinding. The specimens were mechanically polished to remove surface scratches (using 00-grade SiC papers) prior to testing. Tensile tests were performed on an Instron testing machine at temperatures to 1000°C in air at a crosshead speed of 0·25 cm min^{-1} (strain rate $4{\cdot}4 \times 10^{-3}$ s^{-1}). In order to calibrate small strains obtained at room temperature and 300°C, the tensile strains measured from a strip chart were corrected on the basis of strain measurement using a clip-on Instron strain gage at room temperature. The larger tensile strains at 800 and 1000°C were determined mainly from broken specimens.

Microstructural features in selected specimens were studied by optical microscopy, SEM, and

Table 2. Heat treatments and microstructural parameters of lamellar TiAl alloys hot extruded at 1400 or 1350°C

Alloy number	Heat treatment	Colony size (μm)	Interlamellar spacing (nm)
T2–73[a]	900°C/2 h	65	100
	1320°C/2 h	62	390
	1350°C/2 h	450	140
	800°C/5040 h		120
T2–74[a]	900°C/2 h	60	86
	800°C/5040 h	114	
TIA-20[b]	900°C/2 h		225
	1320°C/2 h	33	440
	1350°C/2 h	182	
TIA-21[b]	900°C/2 h		160
	1320°C/2 h	31	300
	1350°C/2 h	150	
TIA-21[a]	900°C/2 h	25	141
	1320°C/2 h	35	
	1350°C/2 h	99	
TIA-23[a]	900°C/2 h	22	
	1320°C/2 h	258	
TIA-24[a]	900°C/2 h	23	
	1320°C/2 h	30	
TIA-25[a]	900°C/2 h	26	105
	1320°C/2 h	135	
	1350°C/2 h	148	

[a]Hot extruded at 1400°C.
[b]Hot extruded at 1350°C.

Table 1. Alloy composition of TiAl alloys prepared by powder metallurgy (PM) and ingot metallurgy (IM)

Alloy number	Alloy composition (at%)	Alloy preparation	Hot extrusion temperature (°C)
T2-73	Ti–47Al–2Cr–2Nb	PM	1150, 1250, 1400
T2-74	Ti–47Al–2Cr–1Nb–1Ta	PM	1150, 1250, 1400
TiA-20	Ti–47Al–2Cr–2Nb–0·15B	IM	1350
TIA-21	Ti–47Al–2Cr–1·8Nb–0·2W–0·15B	IM	1350, 1400
TIA-23	Ti–46Al–2Cr–2Nb–0·15B	IM	1400
TIA-24	Ti–48Al–2Cr–2Nb–0·15B	IM	1400
TIA-25	Ti–46Al–2Cr–1·8Nb–0·2W–0·15B	M	1400

TEM. Metallographic samples were polished and etched in a solution of 100 ml H_2O, 8 HNO_3, and 0·5 HF to reveal lamellar structures. TEM disks were prepared by twin-jet electro-polishing in a solution of 6% perchloric acid, 60% methanol, 33·5% butyl cellusolve, and 0·5% glycerin at a temperature of −20°C and at 32 V. Thinned foils were then examined using a Hitachi S4100 scanning electron microscope operating at 5–15 kV, and a Philips CM30 transmission electron microscope.

RESULTS

All IM and PM materials were successfully hot extruded above and below T_α (1320°C) without difficulty. The hot extrusion above T_α (e.g. 1400°C) produced fully lamellar structures, while the extrusion below T_α (e.g. 1250°C) resulted in duplex structures. As shown later, the alloys with refined lamellar structures exhibited attractive mechanical properties, and their microstructural features and mechanical properties were thus the focus of a more detailed characterization.

Figure 1 shows optical and TEM microstructures typically observed in PM alloys T2-73 and -74 with refined lamellar structures produced by hot extrusion at 1400°C and stress-relieved for 2 h at 900°C. Since the detailed characterization of microstructures was reported elsewhere,[2,14,21] only the key microstructural features will be summarized here. The PM alloys showed refined colony size (60 to 65 μm) and ultrafine interlamellar spacing (≤100 nm). These values are much finer than those reported in literature (colony size = 300 to 600 μm) and interlamellar spacing (1 to 5 μm). One unique feature of this lamellar structure is that the α_2 platelets are long and straight, and quite regularly spaced. Furthermore, the correspondence of α_2/γ ratio is almost 1:1, with only the widest γ lamellae having γ/γ twins inside.

Figures 2 and 3 are SEM and TEM micrographs for the IM alloys TIA-21 and -25 hot extruded at 1400°C and stress-relieved for 2 h at 900°C. Inter-colony structures, such as equiaxed γ grains, are visible along certain colony boundaries in SEM micrographs. It appears that the ultrafine lamellar structures in these IM materials are less stable as compared with those in the PM alloys. As shown in Fig. 3(a), the poor stability of α_2 platelets results in annihilation of α_2 platelets and the generation of more γ/γ boundaries in TIA-21 containing 47% Al while the alloy cools from the hot extrusion temperature. For TIA-25 containing 46% Al, clusters

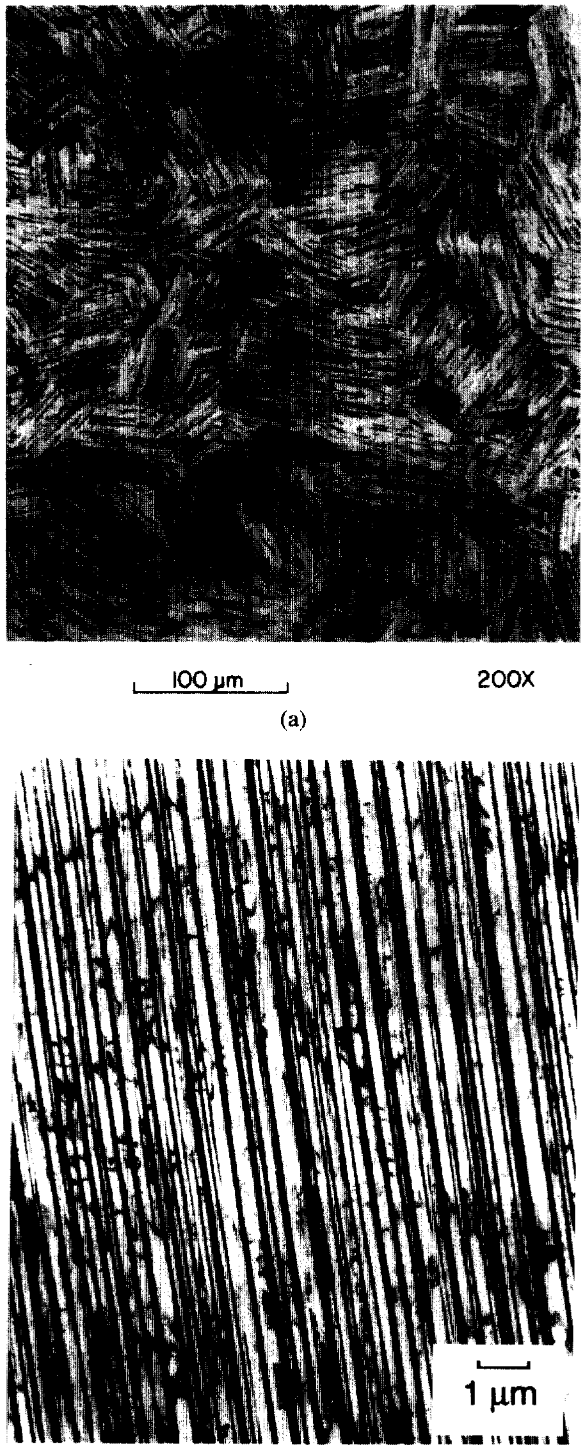
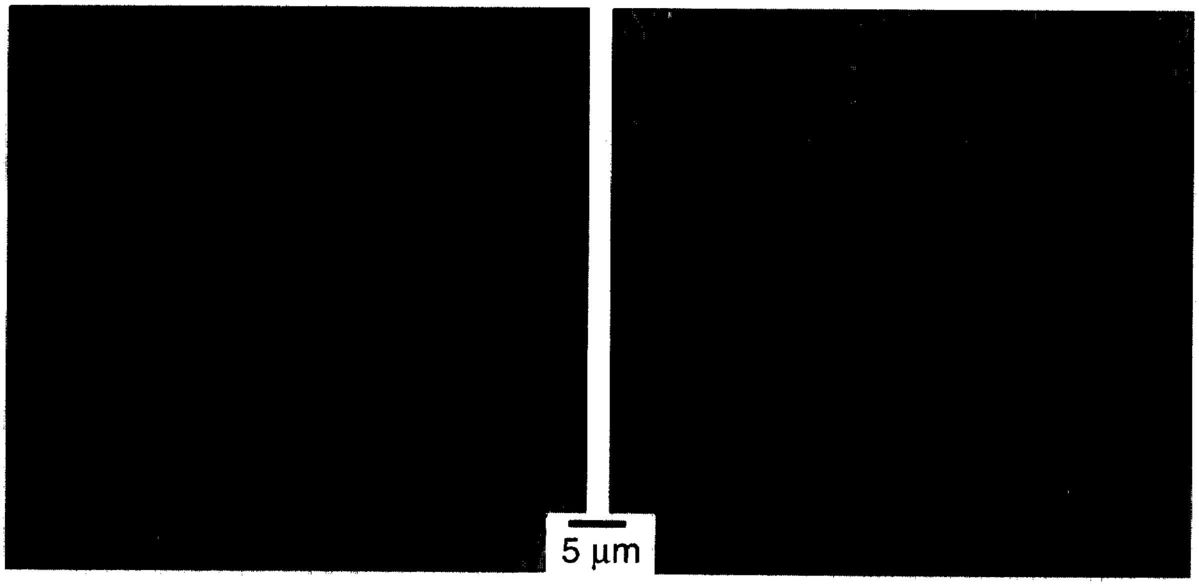


Fig. 1. Microstructures of PM Ti–47Al–2Cr–2Nb hot extruded at 1400°C and stress-relieved for 2 h at 900°C: (a) optical micrograph, and (b) TEM micrograph showing ultrafine lamellar spacing.

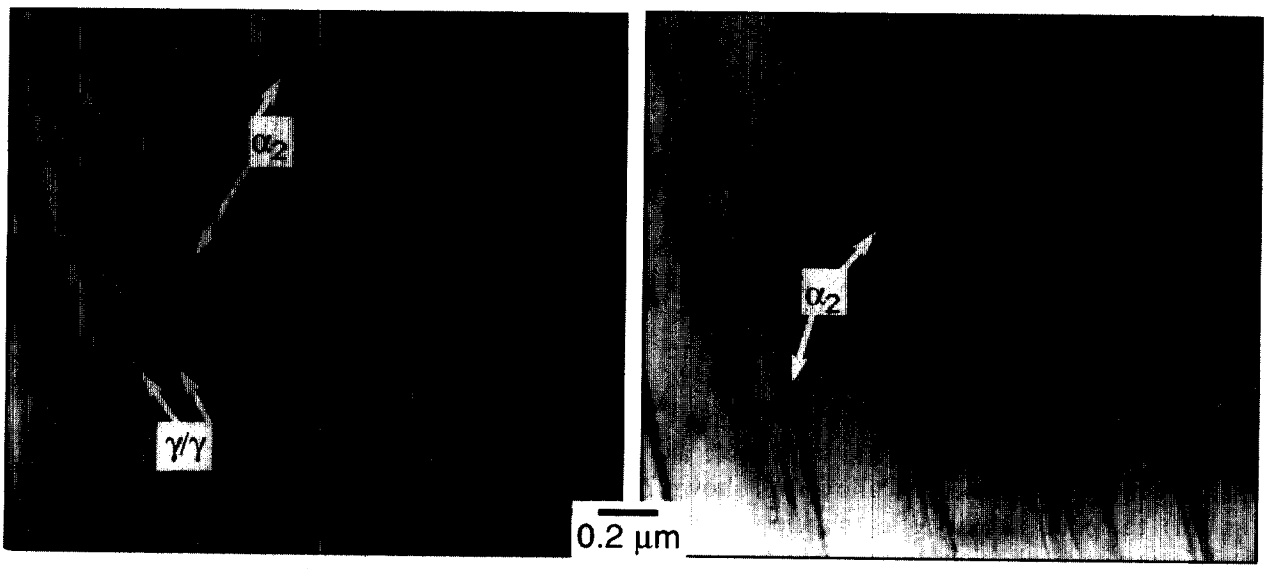
of fine α_2 platelets are observed in Fig. 3(b). Careful TEM examination of TIA-25 suggests the possibility that the clustering of α_2 platelets was developed as a result of decomposition of wide α_2 platelets in the 46% Al alloy during cooling down from hot extrusion. Further studies are certainly required to completely understand the microstructural evolution in these IM alloys. The interlamellar spacing measured by TEM and the colony size measured from optical photographs are summarized in Table 2.

Figure 4 compares the tensile properties as a function of test temperature for T2-73 with both lamellar and duplex structures produced by hot extrusion at 1400 and 1250°C, respectively. The ultrafine lamellar structure showed a slight decrease in yield strength at temperatures to 800°C and a steep decrease above that temperature. The yield strength of this structure remains as high as 577 MPa (83·7 ksi) even at 1000°C. In comparison, the fine duplex structure exhibited a significant decrease in strength below 600°C and a sharp decrease above 600°C. As a result, this structure had a yield strength of only 172 MPa (25 ksi) at 1000°C. Clearly, the TiAl alloy with a ultrafine lamellar structure is much stronger than the same alloy with a fine duplex structure. The room temperature ductility of the lamellar structure is also higher that that of the duplex structure. This is quite unexpected because the duplex structures



(a) (b)

Fig. 2. SEM micrographs of (a) IM TIA-21 (Ti–47Al–2Cr–1·8Nb–0·2W–0·15B) and (b) IM TIA-25 (Ti–46Al–2Cr–1·8Nb–0·2W–0·15B) produced by hot extrusion at 1400°C and stress-relieved for 2 h at 900°C.



(a) (b)

Fig. 3. TEM micrographs of (a) IM TIA-21 and (b) IM TIA-25 produced by hot extrusion at 1400°C and stress relieved for 2 h at 900°C.

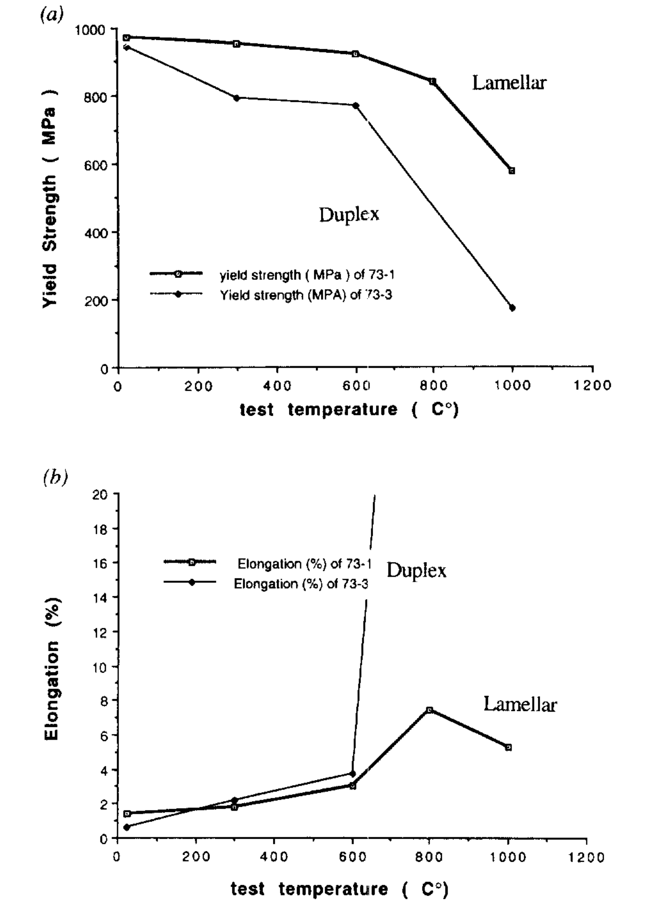


Fig. 4. Comparison of tensile properties of PM T2-73 (Ti–47Al–2Cr–2Nb) alloy with a lamellar structure (hot extrusion at 1400°C) and duplex structure (hot extrusion at 1250°C).

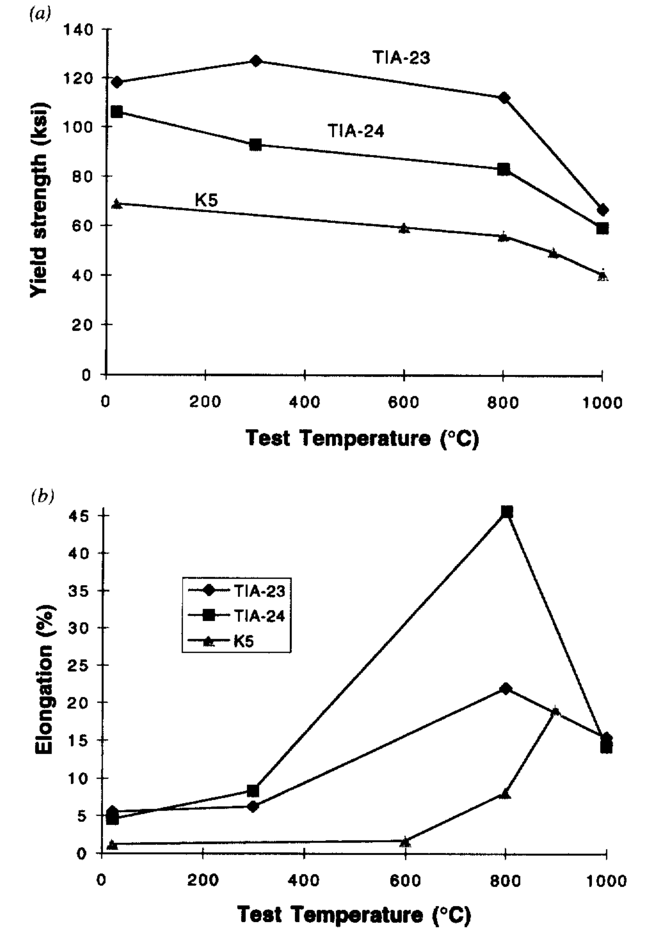


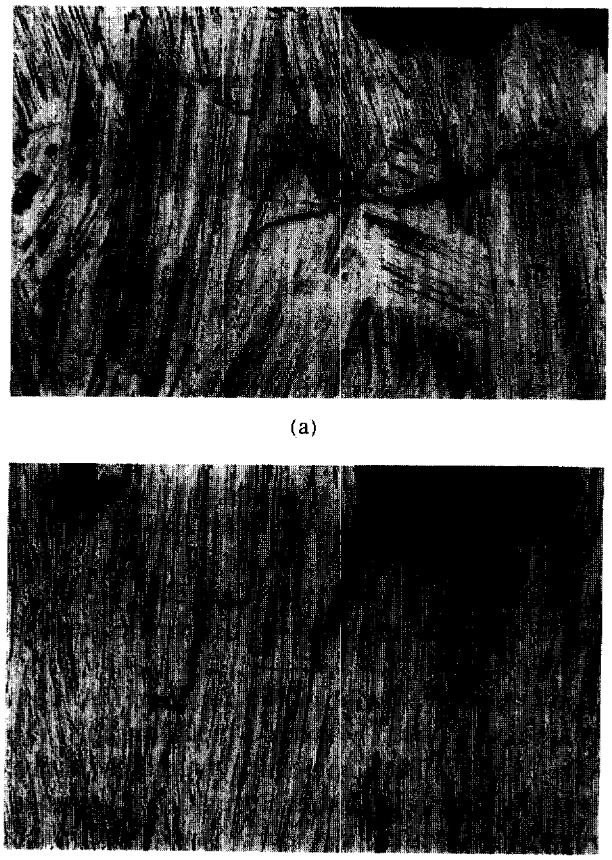
Fig. 5. Comparison of tensile properties of TIA-23 (Ti–46Al–2Cr–2Nb–0·15B) and TiA-24 (Ti–48Al–2Cr–2Nb–0·15B) with an advanced TiAl alloy K-5 (Ti–46·5Al–2·1Cr–3Nb–0·2W).[5]

generally show a higher tensile ductility than that of lamellar structures.[1–8] The ductility of both structures increases with temperature to 600°C, and above that temperature the ductility increases sharply for the duplex structure but not for the lamellar structure.

The mechanical properties of the TiAl alloys with refined lamellar structures are further influenced by both minor alloy additions and changes in the aluminum concentration. Our studies show that the yield strength increases with the addition of 0·2% W and with decreasing the aluminum concentration from 48 to 46%.[11] Fig. 5 compares the tensile properties as a function of test temperature for IM TIA-23 containing 46% Al and TIA-24 containing 48% Al. The plot indicates that the 46% Al alloy is substantially stronger than the 48% Al alloy at all test temperatures. Since both alloys have about the same colony size, the difference in strength appears more related to the lamellar structure differences within the colonies. The tensile ductility of these two alloys is similar, except that the 48% Al alloy is much more ductile at 800°C. Figure 5 also compares the tensile properties of TIA-23 and 24 with an advanced two-phase TiAl alloy, K-5 (Ti–46·5Al–2·1Cr–3Nb–0·2 W), prepared by casting and isothermal forging.[5] This comparison indicates that TIA alloys with the refined colony structures and ultrafine lamellar structures are much stronger and more ductile than the K-5 alloy. Note that both TIA-23 and -24 exhibit tensile ductilities as high as ≈5% at room temperature. As shown later, the good ductility of these TIA alloys is related to their fine colony size (22 to 23 μm).

The fracture behavior of the lamellar TiAl alloys was studied by both SEM and metallography. The alloys with refined lamellar structures showed essentially translamellar fracture at room temperature, with individual lamellar colony facets visible on fracture surfaces. There is no change in fracture mode from room to 800°C, but fine nodules, presumably formed by dynamic recrystallization, were observed on surfaces fractured at 1000°C. Microcracks with orientations both parallel and perpendicular to lamellar platelets were observed near the fracture surfaces (Fig. 6). As evidenced in Fig. 6(a), colony boundaries appear to be quite effective in stopping the propagation of microcracks.

The tensile properties of IM and PM TiAl alloys were analyzed in terms of both colony size and interlamellar spacing. Figure 7 is a plot of the room-temperature yield strength as a function of $(\lambda)^{-1/2}$, where λ is the average interlamellar spacing. The plot shows that there is a good linear relation between the yield strength and $\lambda^{-1/2}$, with R (correlation coefficient) = 0·93. A similar relationship is also clear for the 800°C yield strength, with R = 0·89 (Fig. 8). The tensile ductilities at room and 800°C were analyzed using both the colony size (d) and interlamellar spacing (λ). We found no clear correlation between the ductility and λ. Figures 9 and 10 show the plot of the room temperature and 800°C ductility, respectively, as a function of $d^{-1/2}$. As indicated by the plots, a reasonable correlation is found at both temperatures. At room temperature (Fig. 9), the two data points with their ductility values <2%, obtained from PM alloys hot extrusion at 1400°C and stress-relieved for 2 h at 900°C, are significantly lower than those predicted from the linear relationship. It is possible that additional defects, such as porosities, in the PM materials lower their tensile ductility,[2] as compared with IM materials. On the other hand, the data point (3·6%) obtained from PM T2-73 annealed for 2 h at 1320°C is not lower than those of the IM materials, suggesting that this heat treatment was sufficient to anneal out such defects in the PM materials. At 800°C (Fig. 10), the data point (46%) obtained from the TIA-24 alloy containing 48% Al is significantly higher than other data points.



(a)

(b)

Fig. 6. Microcracks near the fracture surface of TIA-21 hot extruded at 1400°C and stress-relieved for 2 h at 900°C: (a) tensile fracture at room temperature, and (b) tensile fracture at 800°C.

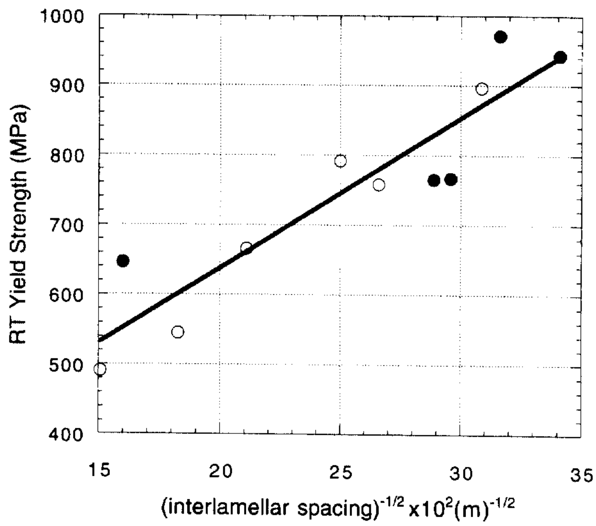


Fig. 7. Plot of the room-temperature yield strength of IM and PM TiAl alloys against (interlamellar spacing)$^{-1/2}$. Open symbols for IM alloys and closed symbols for PM alloys.

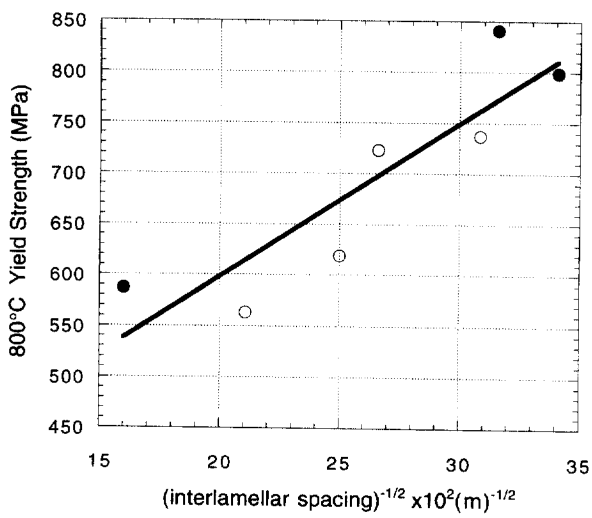


Fig. 8. Plot of the 800°C yield strength of IM and PM TiAl alloys against (interlamellar spacing)$^{-1/2}$. Open symbols for IM alloys and closed symbols for PM alloys.

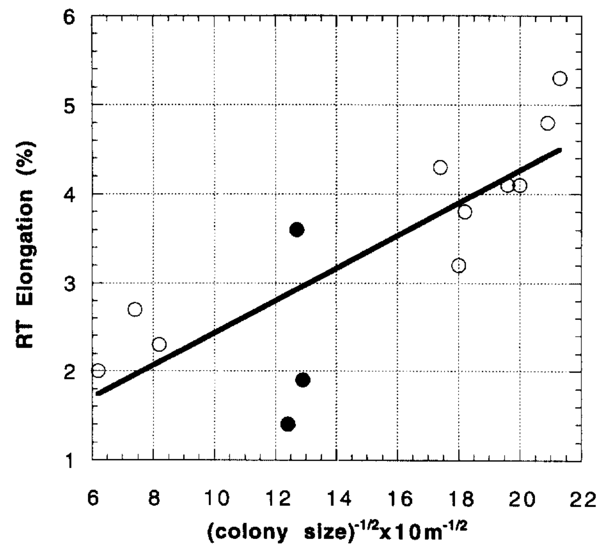


Fig. 9. Plot of the room-temperature elongation of IM and PM TiAl alloys against (colony size)$^{-1/2}$. Open symbols for IM alloys and closed symbols for PM alloys.

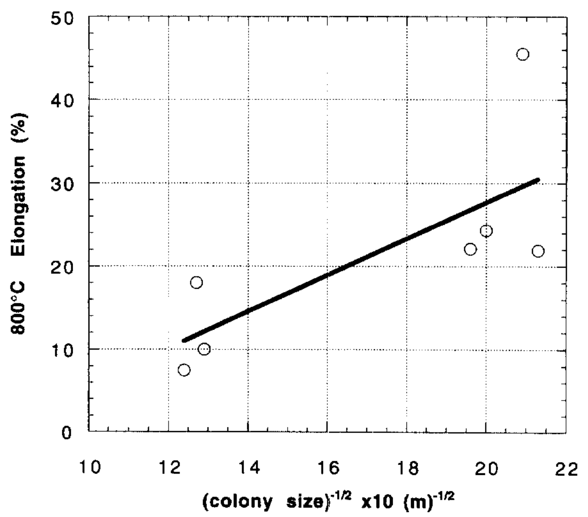


Fig. 10. Plot of the 800°C elongation of IM TiAl alloys against (colony size)$^{-1/2}$.

DISCUSSION AND CONCLUSION

The Hall–Petch equation (eqn (1)) has been applied extensively to explain the effect of grain size on yield strength of mainly single-phase alloys in polycrystalline forms,[22–24]

$$\sigma_y = \sigma_0 + k_{gb} d^{-1/2} \quad (1)$$

where σ_0 and k_{gb} are material constants, which are related to the frictional stress against the motion of dislocations within grains and the Hall–Petch slope constant associated with the strength of grain boundaries (σ_{gb}), respectively. In the case of two-phase TiAl alloys with lamellar structures, the yielding is expected to involve transmitting plastic flow across lamellar interfaces via multiple dislocation pileups against interfaces within each grain. Based on this consideration, the frictional stress term, σ_0, can be simply written as:

$$\sigma_0 = \sigma_0' + k_{lm} \lambda^{-1/2} \quad (2)$$

where σ_0' and k_{lm} are the intrinsic frictional stress and a slope constant associated with the strength of lamellar interfaces (σ_{lm}). Inserting eqn (2) into eqn (1) results in eqn (3):

$$\sigma_y = \sigma_0' + k_{lm} \lambda^{-1/2} + k_{gb} d^{-1/2} \quad (3)$$

By assuming that $\sigma_{lm} \cong \sigma_{gb}$ and $d >> \lambda$ (that is, $k_{lm}\lambda^{-1/2} >> k_{gb} d^{-1/2}$ for the case of ultrafine lamellar structures), eqn (3) can be simplified as:

$$\sigma_y \cong \sigma_0' + k_{lm} \lambda^{-1/2} \quad (4)$$

The plots in Figs 7 and 8 strongly indicate that eqn (4) is effective in predicting the yield strengths at room and 800°C for IM and PM TiAl alloys with refined lamellar structures. The measurement of the slopes of the linear relations in these figures gives

$k_{lm} = 0{\cdot}22$ MPa m$^{-1/2}$ for room temperature, and
$k_{lm} = 0{\cdot}15$ MPa m$^{-1/2}$ for 800°C.

The yield strength of duplex structures can be well described by the Hall–Petch equation for grain size (eqn (1)).[7,25–27] In this case, the measured k_{gb} was reported to be about 1 MPa m$^{-1/2}$. Recently, eqn (4) was verified by theoretical calculations based on multiple dislocation pileup models by Sun.[28] Also, Kim *et al.* observed a linear relationship between room-temperature fracture toughness and λ for TiAl alloys with lamellar structures.[5,29]

Figures 9 and 10 indicate that the Hall–Petch equation based on colony size is applicable to TiAl alloys with refined lamellar microstructures tested at room temperature and 800°C. This observation suggests that microcracks with a length comparable to the colony size are stable, and that final fracture of such alloys is then controlled by the propagation of these stable microcracks. Examinations of microcracks in the specimens fractured at room temperature and 800°C support this assertion (see Fig. 6).

So far, all studies indicate that TiAl alloys with refined colony sizes and ultrafine lamellar spacings possess superior mechanical properties for structural applications at elevated temperatures. These present studies show that the TiAl alloys with colony size $<50\,\mu m$ and lamellar spacing <200 nm exhibited a yield strength above 760 MPa (110 ksi) at temperatures to 800°C, with a tensile ductility of 3 to 5% at room temperature (e.g. see Fig. 5). The lamellar alloys in hot extruded and stress-relieved conditions have a fracture toughness of more than $22\,MPa\,m^{-1/2}$, as measured by three-point bend tests at room temperature.[2] The toughness increased to $>50\,MPa\,m^{-1/2}$ when the alloys were heat treated at 1320°C and above.[14]

It was a logical concern that lamellar structures with refined colony sizes might not have good creep resistance, because of possible sliding along colony boundaries at elevated temperatures. In view of this, the creep behavior and creep mechanism of these lamellar alloys with refined colony sizes and ultrafine interlamellar spacings have recently been studied by Wang *et al.*[10] and Hsiung *et al.*[30–32] Wang *et al.*[10] showed that the PM lamellar TiAl alloys exhibit outstanding creep resistance. The lamellar alloys far exceed the creep strength of other TiAl alloys reported previously, despite their finer colony sizes and lamellar structures. The multiplication of lattice dislocations within both γ and α_2 may be very limited due to the restriction imposed by the ultrafine interlamellar spacing. The detailed TEM study by Hsiung *et al.*[30–32] provides evidence that the glide of interfacial dislocations on both γ/α_2 and γ/γ interfaces becomes an active deformation mode. The pileup of dislocations at such interfaces triggers mechanical twinning, resulting in stress relaxation at the interfaces.

In summary, our studies demonstrate that the mechanical properties of TiAl alloys with refined lamellar structures are mainly controlled by two key parameters, i.e. colony size and interlamellar spacing. The tensile elongation at room and elevated temperatures increases with decreasing colony size, and the yield strength decreases with increasing interlamellar spacing. In both cases, the Hall–Petch type of equations is applicable for lamellar TiAl alloys. The TiAl alloys with refined colony sizes and ultrafine interlamellar spacings possess excellent mechanical properties for structural applications at elevated temperatures.

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