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ABSTRACT

Superplasticity in a powder-metallurgy TiAl alloy (Ti–47Al–2Cr–2Nb) with a metastable microstructure has been studied. Samples were tested at temperatures ranging from 650 to 1100°C, and at strain rate ranging from 10^{-6} to 10^{-4} s^{-1}. An elongation value of over 300% was obtained at a strain rate of 2 x 10^{-5} s^{-1} and at a temperature as low as 800°C, which is close to the ductile-to-brittle-transition temperature. This is in contrast to the prior major observations of superplastic behaviors in TiAl in which typical temperatures of 1000°C have usually been required for superplasticity. It is proposed that the occurrence of superplasticity at 800°C in the present alloy is caused by the presence of a B2 phase. During superplastic deformation (grain boundary sliding), the soft β grains accommodate sliding strains to reduce the propensity for cavitation at grain triple junctions and, thus, delays the fracture process. The final microstructure consists of stable, equiaxed $\gamma+\alpha_2$ grains.

Key words: superplasticity, intermetallics, phase transformation, grain boundary, cavitation

INTRODUCTION

Gamma-TiAl (γ) alloys have considerable potential for use in advanced structures because of their good combination of elevated temperature strength and low density [1]. Intermetallic compounds, however, normally have low ductility at room temperature, which thus far has limited their applications. Methods to improve their ductility, while retaining their strength, have been major challenges [1, 2]. The poor intermediate and room–temperature ductility cause conventional manufacturing operations such as rolling, forging, drawing, or machining to be difficult for titanium aluminides. For these reasons, the development of superplastic TiAl to produce near-net-shape structural components is expected to be technologically attractive. In fact, some demonstration TiAl articles have already been manufactured by using superplastic forming techniques [3].

The Ti–Al phase diagram reveals that a TiAl–based alloy has several characteristics in common with hypereutectoid steels, materials that have been shown to be superplastic if appropriately thermomechanically processed. Thus, a Ti–Al alloy can be heated into a single–phase region at high temperature where the aluminum is completely in solution in the hexagonal close–packed structure of titanium (analogous to the solution of carbon in fcc austenite in iron–carbon steel); upon cooling, proeutectoid TiAl will precipitate (equivalent to precipitation of proeutectoid cementite in hypereutectoid steels), followed by a eutectoid reaction wherein a lamellar structure of TiAl and Ti_3Al is obtained (equivalent to pearlite in steel). If the alloy is then mechanically worked below the eutectoid temperature, the lamellar structure can be broken up to form two equiaxed, fine–grained phases (equivalent to strain–enhanced spheroidization in eutectoid steels). The above principle has been widely used to produce equiaxed, fine–grained microstructures in TiAl for superplasticity purposes. The thermomechanical processes were usually chosen to lie in the two–phase region where approximately 50 vol % of each of the two aluminides (TiAl and Ti_3Al) coexist. In fact, as listed in Table 1, superplasticity has only been observed in fine–grained, equiaxed, two–phase TiAl alloys [4-9], with the exception of the Ti–50Al alloy, which is single–phase [10].

Table 1 Summary of Data from Superplastic TiAl

Composition (at. %)	***d* (μm)**	**Temperature (°C)**	**$\dot{\varepsilon}$ (s^{-1})**	***m***	**Elongation (%)**	**Ref.**
Ti–43Al	5	1000–1100	10^{-5} to 2×10^{-2}	0.5	275	[4]
Ti–50Al	0.4	800	8×10^{-4}	~0.4	~225	[7, 10]
Ti–47Al–2Nb–1.6Cr–0.5Si–0.4Mn	20	1180–1310	2×10^{-5} to 2×10^{-3}	0.65	470	[11]
Ti–47.4Al	8	927	10^{-4}	NA	~400	[5]
Ti–47Al–3Cr	NA	1200	5×10^{-4}	0.57	450	[12]
Ti–43Al–13V	NA	800–1143	3×10^{-4} to 10^{-1}	NA	580	[9]

It is particularly noted in Table 1 that the reported temperature for the occurrence of superplasticity in TiAl is typically at or above 1000°C in either vacuum or an inert atmosphere. From a technological point of view, such a high temperature of 1000°C causes both tooling and oxidation problems. Also, the physical and mechanical properties of Ti–base alloys are known to be extremely sensitive to the presence of oxygen. A reduced forming temperature for TiAl is certainly desirable and attractive.

The ductile-to-brittle-transition (DBTT) temperature of γ–TiAl alloys is approximately 700–800°C and the tensile elongation at 800°C is typically less than 50% [13]. From a plasticity point of view, this is the temperature range at which lattice dislocations begin to activate. The activation of dislocations is necessary, since grain boundary sliding, which is the dominant deformation process in a fine-grained material, must be properly accommodated by dislocation slip within grains to avoid cavitation and, thus, fracture. This suggests that 700–800°C is probably the lowest possible temperature for the observation of superplasticity in a γ–TiAl alloy. In fact, Sikka [14] recently observed a 120% fracture strain in a powder-metallurgy TiAl alloy creep tested at 138 MPa and 760°C. The steady–state creep rate was measured to be approximately 6×10^{-7} s^{-1}. To the authors' knowledge, 760°C is the lowest temperature at which extended ductility has been observed in a TiAl alloy. In the present paper, we illustrate that, by manipulating the metastable phases present in a TiAl alloy, superplasticity can indeed take place at a temperature as low as 800°C in *air*.

EXPERIMENTS

The composition of the alloy used in the present study was Ti–47Al–2Cr–2Nb (in atomic %); the alloy was made by rapid solidification and subsequent powder extrusion was carried out at 1150°C [15, 16]. The extruded rods were then annealed at 900°C in a vacuum of ~10^{-4} Pa for 2 h. Sheet specimens were fabricated from the annealed material by electrical discharge machining. Tensile experiments were conducted at either a constant crosshead speed or constant strain rate. Samples were tested in air in a split, three–zone furnace. Temperature at the top, center and bottom of the specimen were controlled by individual thermocouples. The controlled temperature was less than ±1°C of the required temperature along the whole gage length. Samples were tested at temperatures ranging from 650 to 1100°C, and at strain rates ranging from 10^{-6} to 10^{-4} s^{-1}.

The microstructure of the P/M alloy was examined using a JEOL–200CX transmission electron microscope. TEM samples were first sliced from the alloy, and then lapped and thinned using SiC papers. Final thinning and perforation of TEM foils were completed by twin–jet electropolishing in a solution of 60% methanol, 35% butyl alcohol and 5% perchloric acid at ~15V and –30°C.

RESULTS

Microstructure

An optical micrograph showing a longitudinal view of the sample prior to testing is presented in Fig. 1. The grains are essentially equiaxed, indicating that the sample has undergone full recrystallization. The grain size appears to be fine and is in the range of about one micrometer. The microstructure is noted to be multiphase with a matrix of B2 phase (light gray) whose volume fraction is estimated to be about 50%. To resolve further the grain structure, a TEM micrograph (Fig. 2) reveals that the grain size is actually bimodal and consists of coarse grains (4 – 5 μm), which are mainly of B2 structure, and fine grains (0.5 – 1 μm), which are $\gamma + \alpha_2$ mixture. That is, although some supercooled B2 phase still remains in the alloy, a duplex structure composed of equiaxed γ and α_2 grains is formed, presumably via the $\alpha \rightarrow \gamma + \alpha_2$ transformation. (The details of the phase analysis have been described elsewhere [16].) Despite the fact that the grain morphology is inhomogeneous, the sizes of both B2 and $\gamma + \alpha_2$ grains are relatively fine, as compared to those in a typical two–phase superplastic TiAl–base alloy (> 5 μm).

The microstructure of the as–received sample (Fig. 2) is unstable; the B2 phase transforms readily into $\alpha_2 + \gamma$ at elevated temperatures. For example, the microstructure of the alloy annealed at 800°C for 1,500s is shown in Fig. 3. The volume fraction of B2 grains is noted to be greatly reduced. As a result of the phase decomposition reaction: B2 $\rightarrow \alpha_2 + \gamma$, the shape of retained B2 grains became irregular. The phase boundary curvature clearly indicates the direction of decomposition. The decomposition reaction becomes diminished with few B2 grains still remaining after aging at 800°C for 1 h. The microstructure after this heat treatment, shown in Fig. 4, contains mostly an equiaxed ($\alpha_2+\gamma$) duplex structure with an average grain size of about 1.5 μm.

In comparison, the microstructure of the sample deformed at 800°C to an elongation of 175% is presented in Fig. 5. The structure contains highly dislocated α_2 and γ grains (~2.5 μm) and many stacking faults. Also, the grain boundaries have become poorly defined, as a result of stress-induced phase decomposition and boundary migration, in comparison with those in the isothermally aged specimens (Fig. 4). The size of grains lies between the starting grain sizes of $\gamma + \alpha_2$ (0.5–1 μm) and B2 (4–5 μm). There is still some B2 phase but its volume content is virtually negligible. Occasionally, α_2/γ lamellar grains can be observed in the specimen. The presence of these lamellar grains indicates that a stress–induced $\alpha_2 \rightarrow \gamma$ transition may occur during testing. It is of particular interest to note that dislocation emission is often observed at grain triple junctions; this is shown in Fig. 6. The presence of a high dislocation density near grain triple junctions is probably induced by a grain boundary sliding process. Despite this high dislocation activity, limited cavitation was detected at these triple junctions, indicating a proper strain accommodation during deformation.

The microstructure of samples deformed at 1000°C is similar to that observed in samples deformed at 800°C, except the phase decomposition reaction: B2 $\rightarrow$ $\alpha_2+\gamma$ and the grain conversion into an equiaxed shape occur at a much faster rate. For example, a sample deformed at 1000°C to an elongation of 100% already exhibits a two-phase, equiaxed grain structure, as shown in Fig. 7. The fine particles present within grains are oxides produced *in situ* by oxidation. The grain size is about 8 μm which is much larger than that in the starting material.

Shown in Fig. 8 is the microstructure at the proximity of the fracture surface of a sample superplastically deformed at 1000°C. The tensile stress axis is marked. The alloy is noted to have cavitated extensively. The transverse interlinkage of some cavities is readily observed. The average length of these lenticular cavities (or cracks) is about 40-50 μm, but some cavities are over 100 μm in length. The irregular shapes of the cavities suggest a plasticity-controlled growth mechanism. It is also noted that sample oxidation took place during superplastic deformation. In fact, the surfaces of some internal cavities are even covered with oxide, suggesting an exposure to the external atmosphere. The oxide layer on this particular sample is 30 μm. The layer is evidently brittle and not tenacious.

Mechanical properties

The tensile true stress–true strain curve of the alloy typically shows that, after an immediate hardening stage the alloy continuously softens until the final fracture. Samples superplastically deformed to fracture at 800°C and 1000°C are shown in Fig. 10. The deformation was quite uniform at both temperatures and the total tensile elongation was over 300%. It is noted that 800°C is by no means the optimal temperature for superplasticity for the present alloy. In fact, as shown in Fig. 9, the ductility of the sample (unbroken) deformed at 1000°C is higher than that of the sample deformed at 800°C. 1000°C is the temperature above which the major observations of superplasticity in TiAl have been made.

TiAl alloys are known to be susceptible to oxidation at temperatures over 800°C. (Even at 800°C, the tested sample shows a slightly yellowish color.) For example, a 10 μm-thick oxide layer was obtained at 1000°C for one hour. This oxide layer is brittle, readily fractures under tension and, thus, degrades the superplastic elongation (Fig. 8). In fact, in the present study, we observed a dramatic reduction in elongation at T = 1100°C (elongation ~ 50%) which can be attributed to the fact that approximately 50% of the sample thickness was oxidized. A greater ductility is expected to be obtained at these high temperatures if tests were conducted in vacuum or an inert atmosphere, since previous studies indicated that the optimal temperature is about 1000–1100°C.

Strain rate change tests at a strain interval between each rate change over 10% were conducted to characterize the plastic flow behavior of the present alloy, as shown in Fig. 10. The strain rate sensitivity value, *m*, was measured from a log–log plot of strain rate versus flow stress, as shown in Fig. 11. It is obvious that strain rate and flow stress follow a power-law relationship. The *m* values of the alloy are calculated to be 0.5, 0.48, 0.43 for 800, 900, and 1000°C, respectively. Despite the slight differences, they have values of about 0.5, a typical value for many fine–grained superplastic alloys. An *m* value of 0.5 is also indicative of the fact that the dominant deformation mechanism is grain boundary sliding.

The activation energy, *Q*, for superplasticity is measured from a stress-compensated strain rate versus inverse of temperature plot, as shown in Fig. 12. The Q value is computed to be about 210 kJ/mol, which is lower than the activation energy for self diffusion of Ti in TiAl which is 291 kJ/mol [17]. It is also noted that 210 kJ/mol is lower than the activation energies observed for superplasticity in fine-grained, two-phase ($\gamma + \alpha_2$) TiAl alloys. In the latter case, reported Q values range from 395 kJ/mol [11] to 425 kJ/mol [4].

DISCUSSION

It is well recognized that grain boundary sliding (GBS) is the principal deformation mode in fine–grained alloys at high temperatures. During GBS, the sliding strain must be properly accommodated either by diffusional flow or by dislocation slip across neighboring grains. For TiAl, diffusional processes may prevail at T > 900°C, but are relatively sluggish at 800°C; slip accommodation is expected to dominate. Slip accommodation involves the sequential steps of glide and climb, with the slower of the two processes being rate–controlling. The principal equations for GBS (governed by either lattice or grain-boundary diffusion) accommodated by dislocation climb can be written as [18]:

$$\dot{\varepsilon}_{gbs} = A_{gbs}\left(\frac{b}{d}\right)^{p} D\left(\frac{\sigma}{E}\right)^{2} \qquad (1)$$

where $\dot{\varepsilon}_{gbs}$ is the strain rate for GBS; A_{gbs} is a constant; b is Burgers vector; d is the mean linear intercept grain size; p is the grain size exponent; D is the diffusivity (grain boundary or lattice); and E is the Young's modulus. On the other hand, the equation for GBS accommodated by dislocation glide has been proposed by Fukuyo *et al* [19] to be:

$$\dot{\varepsilon}_{gbs,glide} = A_{glide}\frac{D_{chem}}{d^{2}}\left(\frac{\sigma}{E}\right) \qquad (2)$$

where D_{chem} is the chemical diffusivity; and A_{glide} is a constant. That is, the strain rate is predicted to be linearly proportional to applied stress.

From the result that the strain rate sensitivity values are about 0.5 and the fact that dislocation density is high at grain triple junctions (Fig. 6), we may conclude that GBS accommodated by dislocation climb is the dominant deformation mechanism in the present study. However, it is pointed out that the microstructural features in the present alloy are quite different from those in conventional superplastic alloys, i.e. two-phase, fine, equiaxed grains. In particular, the present alloy is three-phase and has an inhomogeneous grain size distribution. As described earlier, the initial microstructure of the present alloy consists of α_2, γ, and ~50% β, with the β phase virtually disappearing in a superplastically deformed sample. The absence of the β phase suggests that the microstructural phase evolution may play an important role in superplastic deformation. Although the high–temperature strengths of α_2, γ and β phases are not available, β is expected to be softer than both α_2 and γ because of its open bcc structure. In other words, β can be deformed more readily than α_2 and γ.

During GBS, high local stresses at grain triple points are expected to be developed. This high local stress can be relieved by the emission of dislocations into adjacent grains, as illustrated in Fig. 13. Since dislocation slip (either climb or glide) is easier in β grains than in γ grains, the presence of the soft β is obviously beneficial in reducing the stress at triple junctions and, thus, delaying cavitation and fracture. The preferential deformation of β grains is also supported by the fact that the activation energy measured in the present study (200 kJ/mol) is much lower than those obtained from the study of superplasticity in fine-grained, two-phase ($\gamma+\alpha_2$) TiAl alloys (~400 kJ/mol). In the latter case, the Q values correspond to the activation energy for self diffusion in the γ phase. By contrast, the low 210 kJ/mol corresponds to the activation energy for self diffusion in the β phase. It is worth noting that Vanderschueren *et al* [9] have studied superplasticity in a β phase–containing TiAl at a fixed strain rate of 3×10^{-4} s^{-1}. Although not specifically reported by them, the activation energy for superplastic deformation can be deduced (assuming a strain rate sensitivity of 0.5) from their raw data to be about 300 kJ/mol. This Q value, although is higher than ours, is also lower than that observed from fine-grained, two-phase ($\gamma+\alpha_2$) TiAl alloys.

Since β is metastable, the stress at triple junctions can further induce phase transformation, especially at elevated temperatures. The transformation, as indicated earlier, is:

$$\beta \rightarrow \alpha_2 + \gamma \qquad (3)$$

which is displacive, involving only a simple lattice translation, and is primarily athermal. This phase transformation is expected to enhance plasticity (i.e. Transformational Plasticity [20]). It is further noted that the phase transformation described in Equation (3) produces both compositional as well as volume changes. The volume change from β to γ, for example, is calculated to be:

$$\Delta\Omega = (16.32-16.83)/16.83 = -3\% \qquad (4)$$

This is a relatively small value change and its overall effect on superplastic properties, and particularly upon elongation, is probably insignificant.

The observation of superplasticity has been reported in some β phase–containing TiAl alloys [9, 12]. For example, Masahashi *et al* [12] studied a $\gamma+\beta$ alloy (Ti–47Al–

3Cr) and claimed that the grain–boundary β phase acts like a lubricant which promotes grain–boundary sliding and grain rotation during superplastic deformation. On the other hand, Vanderschueren *et al* [9] argued that the presence of the β phase actually enhances the grain boundary cohesion and, thus, delays cavitation and fracture. In the present study, we believe that the role of β phase is primarily to improve the accommodation of sliding strain at grain triple junctions and, thus, delay cavitation and fracture. Despite these different viewpoints, all these studies clearly illustrated the beneficial role of β phase on the plasticity of TiAl at elevated temperatures.

According to the literature, the ductile–to–brittle–transition temperature for conventional TiAl is around 700–800°C [13, 21]. Our own experimental data also confirm that the ductility of the present TiAl alloy varies drastically at ~700–800°C. For example, we obtained the tensile elongation values of 40%, 60%, 120%, and 310% at testing temperatures of 650°C, 700°C, 760°C, and 800°C, respectively. The reduced ductility at decreasing temperatures is apparently associated with the propensity for cavitation and cracking at these temperatures.

It is pointed out that in comparison with all the experiments listed in Table 1, which were conducted in either vacuum or inert atmospheres, a 400% tensile elongation in *air* is quite impressive. It is especially worth noting that the present observation of superplasticity at ~800°C in TiAl is truly phenomenal. The traditional method to produce superplasticity at low temperatures is to produce a ultrafine (< 1 μm), stable, and equiaxed starting microstructure. This usually requires elaborate thermomechanical processes, such as Equal Channel Angular Extrusion [22]. By contrast, the present paper offers an intriguing concept to produce superplasticity at low temperatures by using a material with a metastable microstructure that contains B2 phase, even though the initial grain size and phase distribution are inhomogeneous. The metastable B2 transforms completely into $\gamma + \alpha_2$ phases after superplastic deformation. This removes concerns regarding the brittleness of B2 at room temperature.

CONCLUSION

Superplasticity was observed in a powder-metallurgy TiAl alloy (Ti–47Al–2Cr–2Nb) with a metastable microstructure. A unique aspect was the fact that it occurs at a temperature of as low as 800°C, which is close to the ductile-to-brittle-transition temperature. For example, an elongation value of over 300% was obtained at a strain rate of 2×10^{-5} s^{-1}. This is in contrast to typical temperatures of 1000°C, above which the major observations of superplastic TiAl have usually been reported. From the fact that the strain rate sensitivity is about 0.5, the dominant deformation mechanism is probably grain boundary sliding. However, microstructural examinations indicated that the starting microstructure of the present alloy actually consists of an inhomogeneous mixture of γ, α_2, and ~50% by volume of soft β grains. It is argued that, during superplastic deformation (grain boundary sliding), the soft β grains readily accommodate the sliding strain and also continuously convert into stable, equiaxed $\gamma+\alpha_2$ grains. This reduces the propensity for cavitation at grain triple junctions and, thus, delays the fracture process. The present discovery of low temperature superplasticity in TiAl has several important technological implications: examples include reduces tooling costs, reduced energy for forming, and minimized oxidation of products.

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FIGURE CAPTIONS

Fig. 1 Optical micrograph showing a longitudinal view of the sample prior to testing.

Fig. 2 TEM micrograph shows that the grain morphology of the alloy is actually bimodal.

Fig. 3 Microstructure of the alloy annealed at 800°C for 1,500 s.

Fig. 4 Microstructure of the alloy annealed at 800°C for 1,500 s.

Fig. 5 Microstructures of the sample deformed at 800°C to a 175% elongation: (a) poorly defined grain boundaries, and (b) highly dislocated grains.

Fig. 6 Dislocation emission is observed at grain triple junctions, indicating grain boundary sliding is accommodated by slip.

Fig. 7 A two-phase, equiaxed grain structure is developed in the sample deformed at 1000°C to an elongation of 100%

Fig. 8 Microstructure at the proximity of the fracture surface of a sample superplastically deformed at 1000°C.

Fig. 9 TiAl samples superplastically deformed at 800°C (middle) and 1000°C (bottom). An undeformed sample (top) is also shown for comparison.

Fig. 10 Strain rate change tests performed on the present TiAl alloys at 800°C.

Fig. 11 Strain rate sensitivity value is about 0.5 for TiAl deformed at 800, 900, and 1000°C.

Fig. 12 The activation energy for superplasticity is measured to be about 200 kJ/mol.

Fig. 13 The presence of a soft β grain adjacent to sliding α_2 / γ grains can reduce the high local stress at triple junctions and, thus, delay cavitation and fracture.

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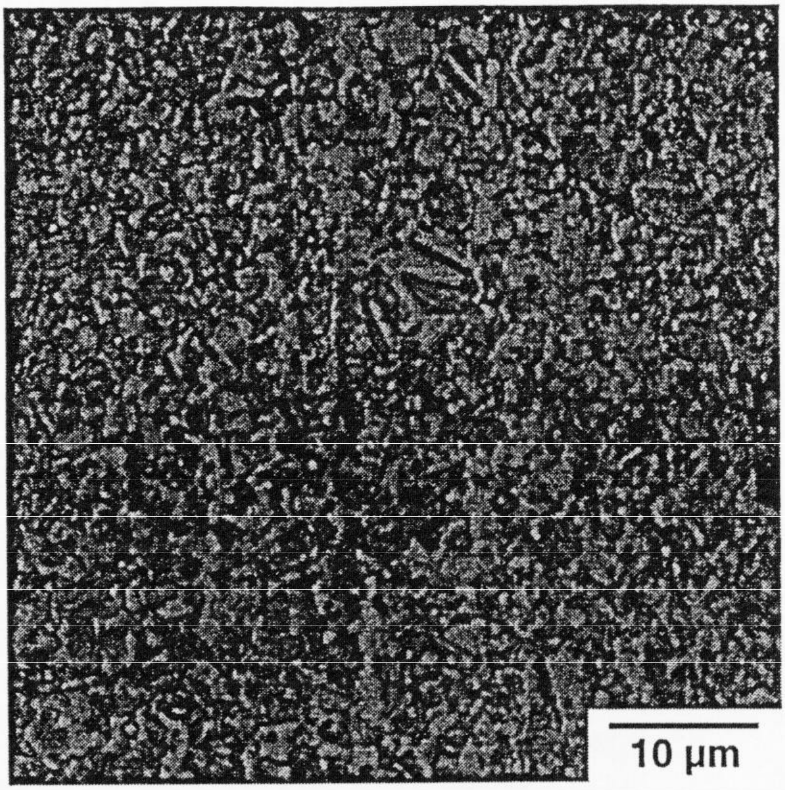

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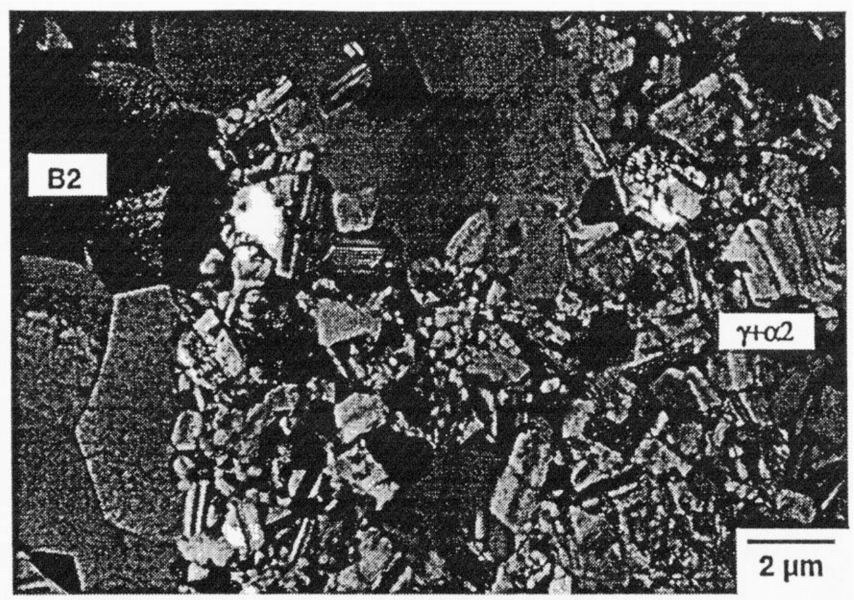


Fig. 2 TEM micrograph shows that the grain morphology of the alloy is actually bimodal.

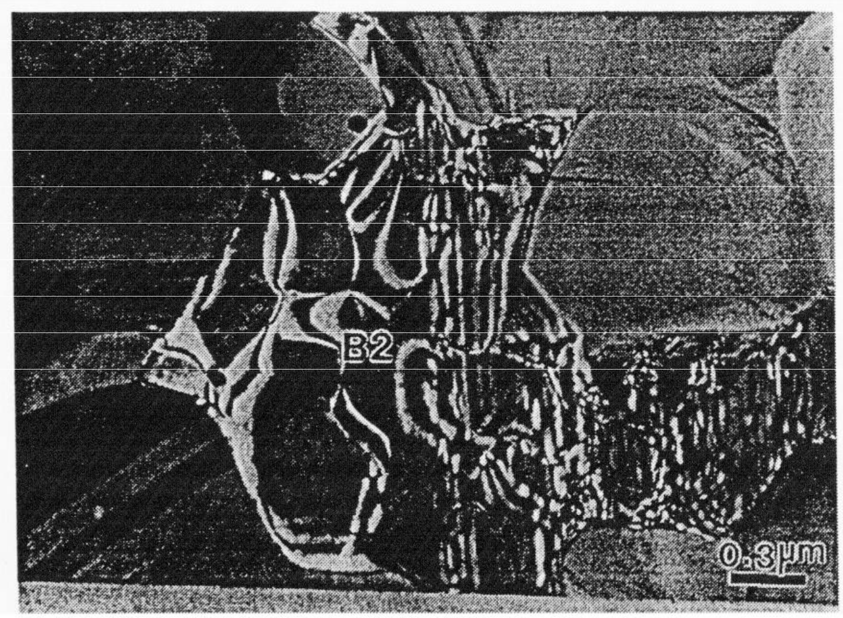


Fig. 3 Microstructure of the alloy annealed at 800°C for 1,500 s.

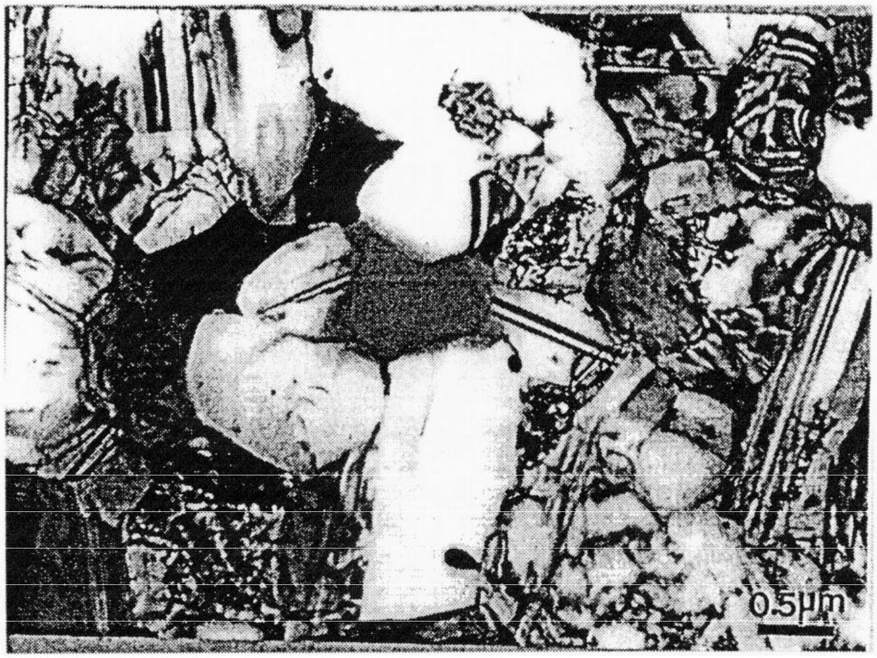
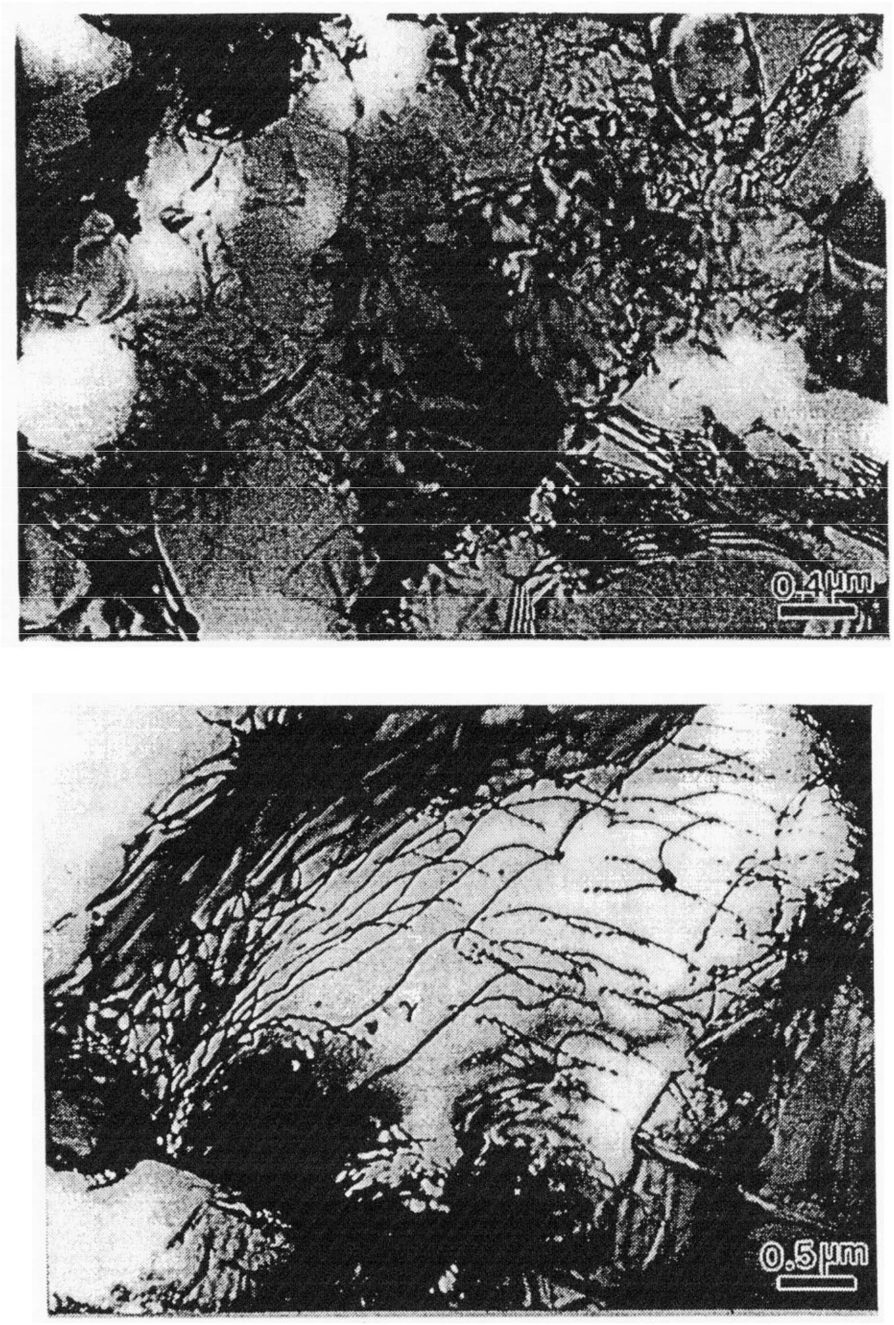


Fig. 4 Microstructure of the alloy annealed at 800°C for 1,500 s.



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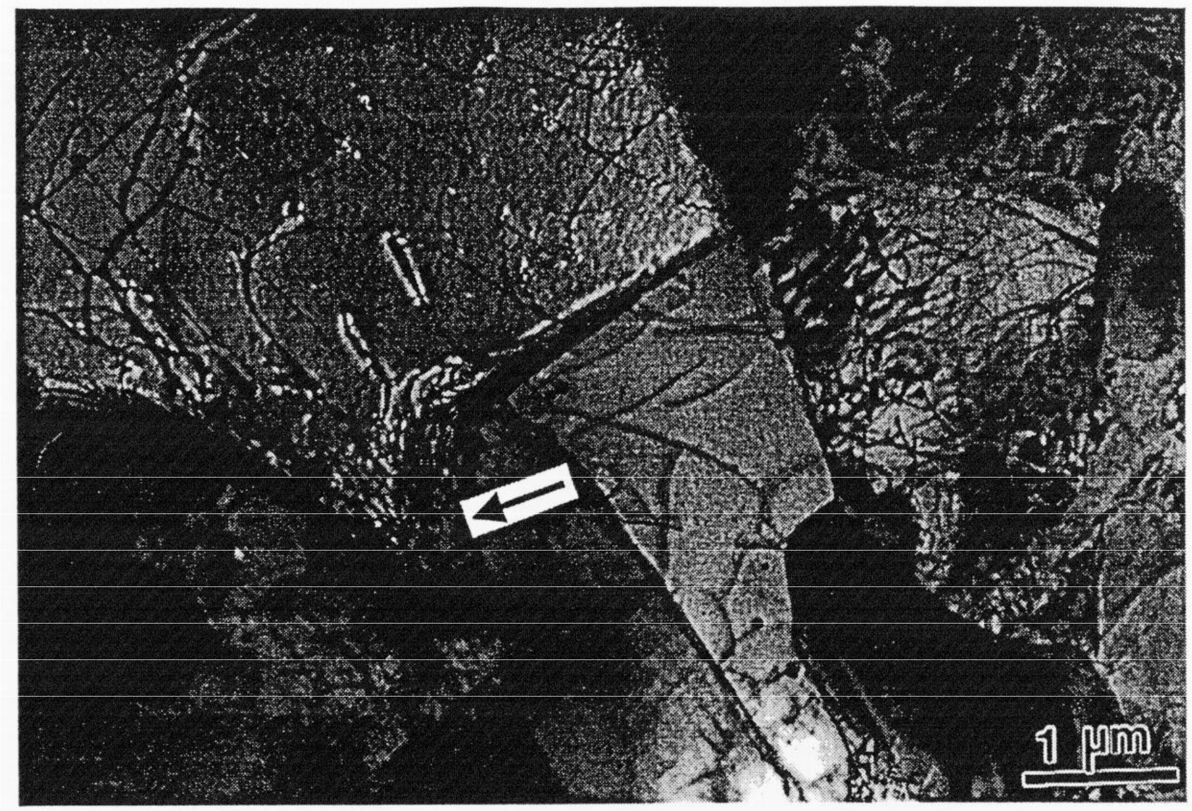

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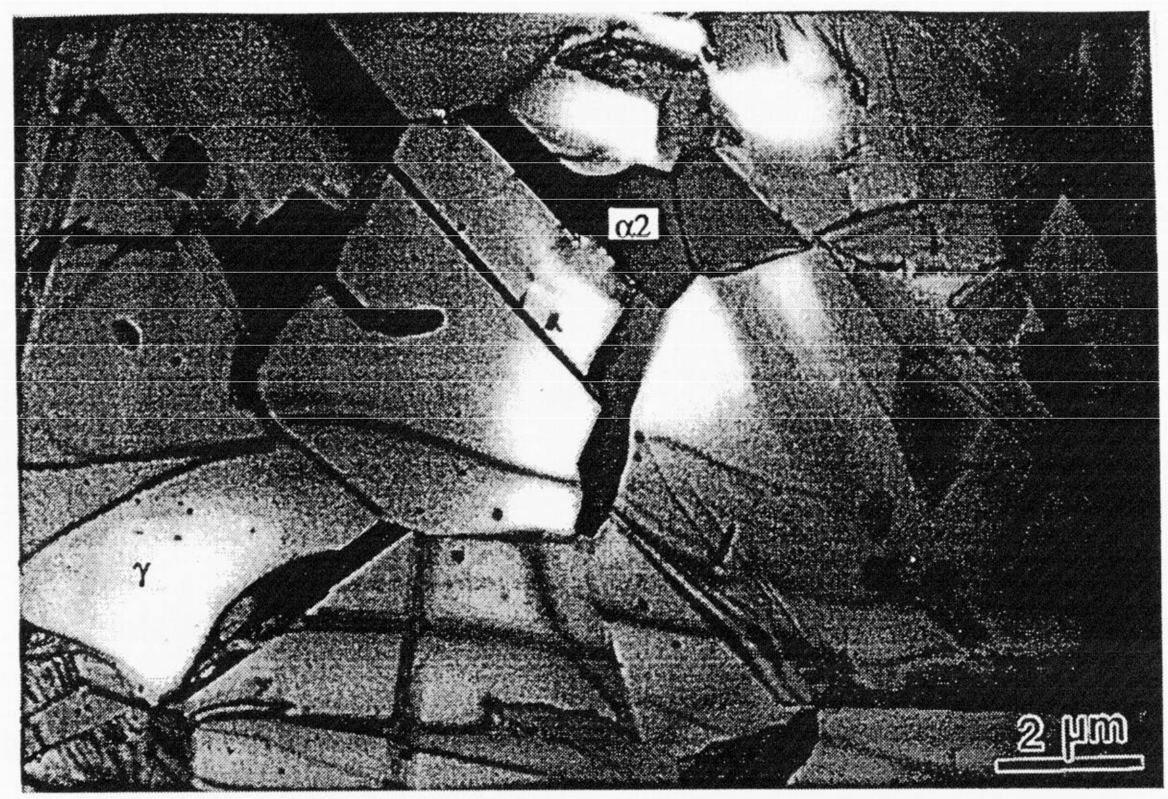

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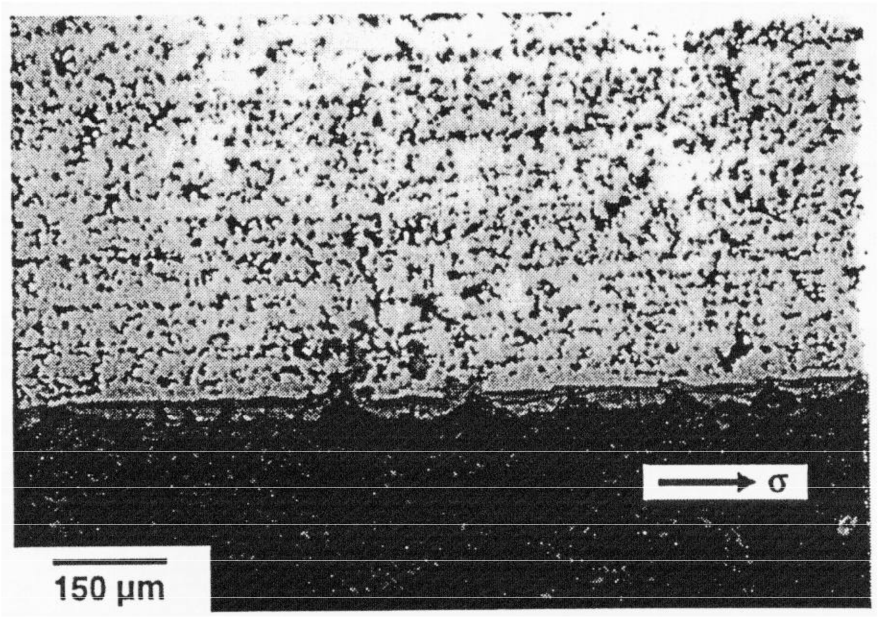

Fig. 8 Microstructure at the proximity of the fracture surface of a sample superplastically deformed at 1000°C.

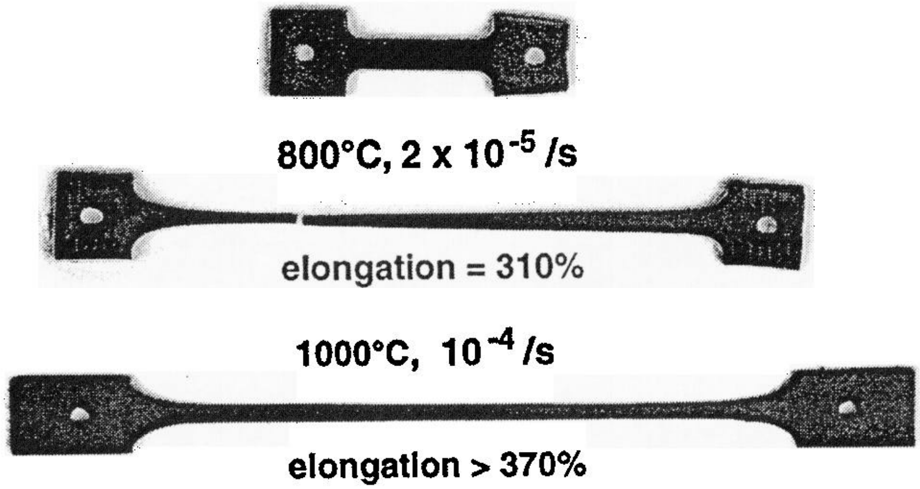

Fig. 9 TiAl samples superplastically deformed at 800°C (middle) and 1000°C (bottom). An undeformed sample (top) is also shown for comparison.

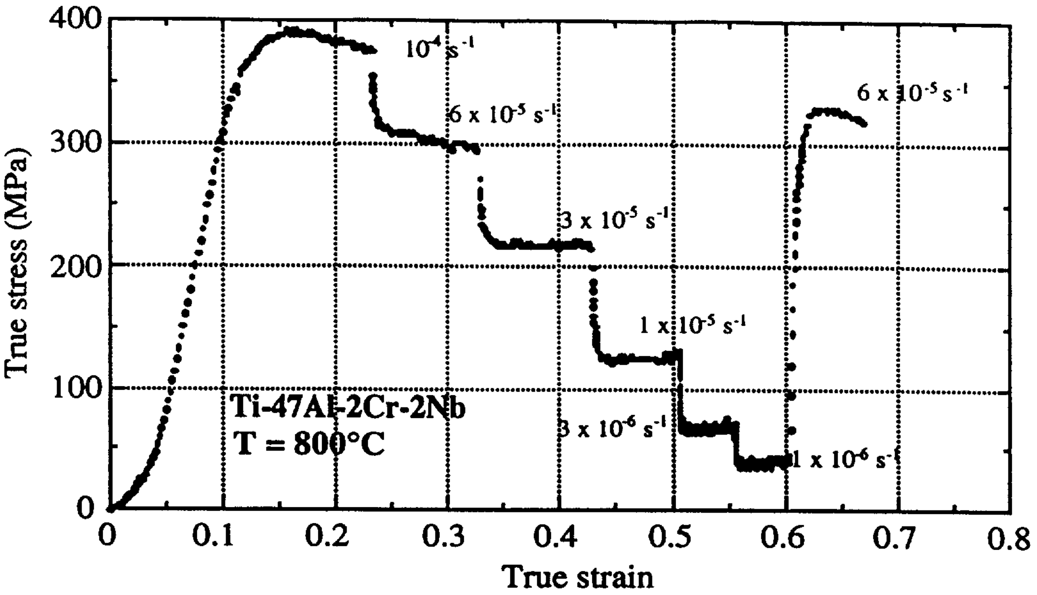

Fig. 10 Strain rate change tests performed on the present TiAl alloys at 800°C.

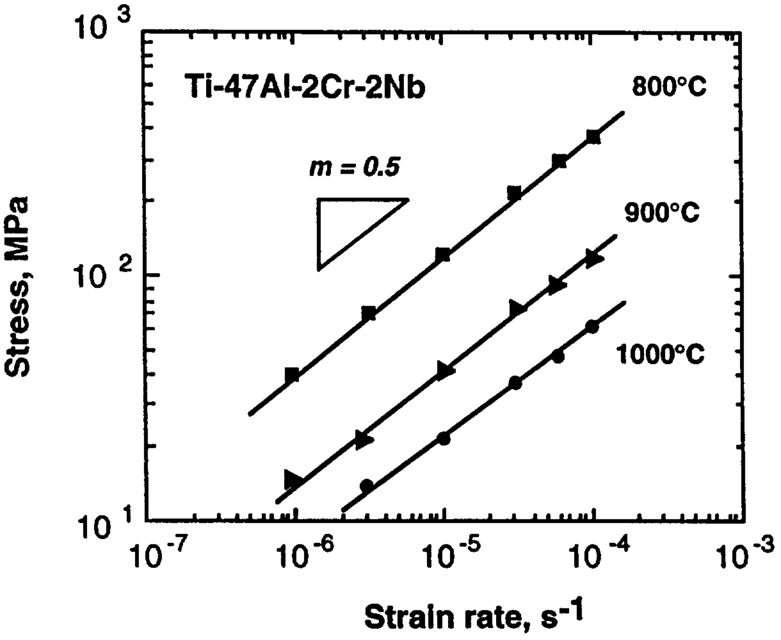

Fig. 11 Strain rate sensitivity value is about 0.5 for TiAl deformed at 800, 900, and 1000°C.

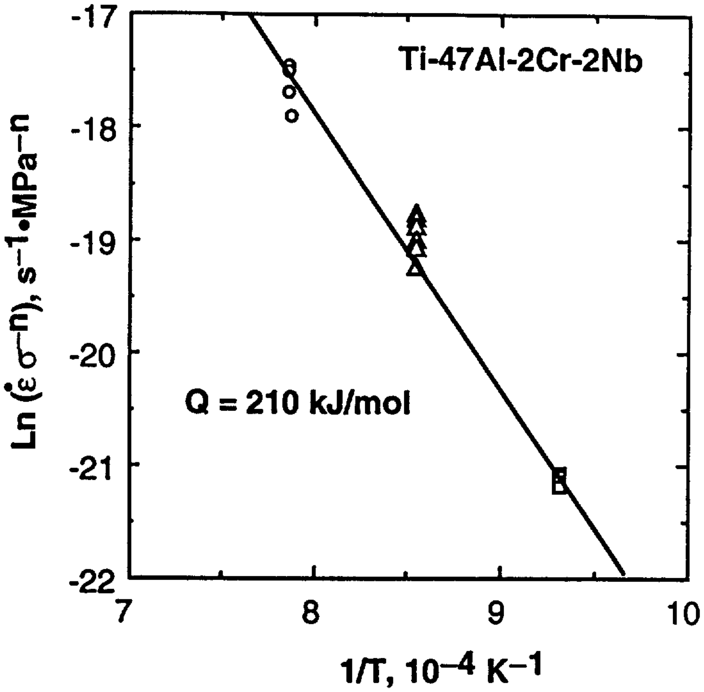

Fig. 12 The activation energy for superplasticity is measured to be about 210 kJ/mol.

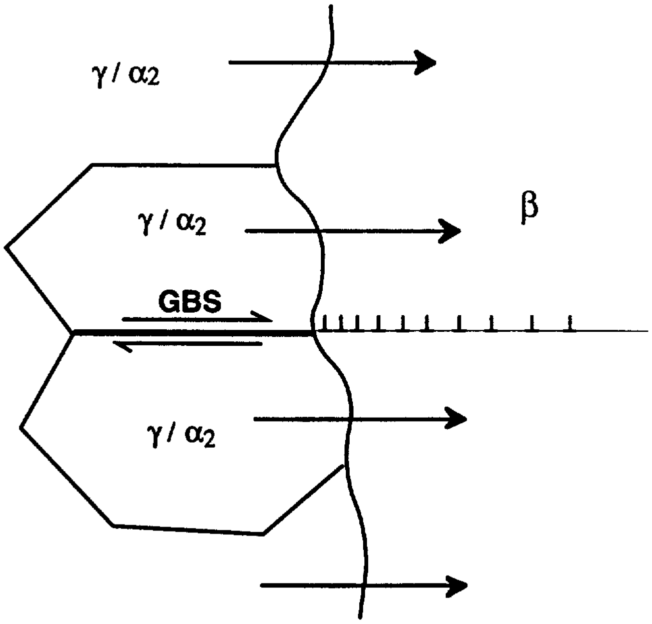

Fig. 13 The presence of a soft β grain adjacent to sliding α_2 / γ grains can reduce the high local stress at triple junctions and, thus, delay cavitation and fracture.

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