JOURNAL OF MATERIALS SCIENCE **35** (2000) 147–151

An analysis of the role of grain size on superplasticity of γ titanium aluminides

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The superplastic data for several microcrystalline and submicrocrystalline TiAl alloys has been analyzed to establish the rate controlling mechanism. The results show that the lattice diffusion controlled slip-accommodated grain boundary sliding mechanism is operative for the entire grain size range, 150 nm–20 μm. The detail of the nature of α_2 phase, i.e. ordered or disordered, does not influence the kinetics of superplastic flow. The optimum superplastic temperature decreases with the decrease in grain size. The optimum superplastic flow stress shows an intrinsic inverse dependence on the grain size. This grain size dependence of the optimum superplastic flow stress can be explained as the stress required to nucleate dislocations from grain boundary edge during slip accommodation of grain boundary sliding. Superplasticity in nanocrystalline TiAl remains an intriguing possibility because it has the potential for increasing the optimal superplastic strain rate or alternatively, decreasing the superplastic forming temperatures. © *2000 Kluwer Academic Publishers*

1. Introduction

Titanium aluminides have emerged as the intermetallic with potential for application in gas turbine engines [1, 2]. Significant amount of research has led to optimization of compositions for most desirable combination of strength and ductility. Most of these alloys have 45–48 at % Al with ternary or quaternary alloying additions. Also, the microstructure can be significantly altered by thermomechanical treatment to obtain optimum mechanical properties [2]. Superplastic forming is an attractive near net shape forming technique for difficult-to-form materials [3]. A number of titanium aluminide alloys have exhibited superplasticity [4–14]. The optimum superplastic temperature changes with grain size for microcrystalline titanium aluminides. More recently, results on superplasticity of submicrocrystalline titanium aluminides have been reported. All the reported studies are summarized in Table I. The optimum superplastic temperature is plotted against grain size in Fig. 1. This shows the possibility of low temperature superplasticity in titanium aluminides. It also raises a fundamental question. The optimum superplastic temperature ranges from 700–1280 °C. It should be noted that in ($\gamma + \alpha_2$) TiAl alloys, the α_2 phase undergoes phase transformation at ∼1110 °C to disordered α phase. An analysis of the entire reported data set is required to evaluate any possible influence of $\alpha_2 \rightarrow \alpha$ transformation on the kinetics of superplastic deformation.

Technologically, two experimental features have important implications: the optimum superplastic temperature and the optimum superplastic strain rate. There is an obvious desire to lower the superplastic temperature and shift the superplastic strain rates to higher levels. The fact that superplasticity is a grain size dependent phenomenon has led to extensive discussion on the possibility of low temperature or high strain rate superplasticity in nanocrystalline materials. There is some evidence of these trends in nanocrystalline materials [15, 16]. Although the possibility of superplasticity in nanocrystalline titanium aluminide has been discussed [17], so far no experimental evidence has been reported. Very recently, Mishra *et al.* [18] have reported a large compressive plasticity in nanocrystalline titanium aluminide, however, the stress exponent was ∼5–6 at the strain rates and temperatures they investigated.

The purpose of this paper is three-fold: (a) to present an analysis of the superplastic data on microcrystalline and sub-microcrystalline titanium aluminides, (b) to discuss the mechanism of superplasticity in titanium aluminides, and (c) to evaluate any influence of $\alpha_2 \rightarrow \alpha$ phase on the kinetics of superplasticity. The present analysis differs from the conventional analysis in that it deals with the optimum superplastic data. As is shown later, one advantage of this approach is that the results can be used to develop a predictive map of optimum superplastic conditions, which is not possible from

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TABLE I A summary of superplastic conditions and parametric dependencies for γ titanium aluminides

		Superplastic conditions				Parametric dependence	
	Al content (at %)	Temp. (°C)	Strain rate (s^{-1})	Elongation (%)	Grain size (μm)	m	Q (kJ/mol)
TiAl-Cr [4]	47	1200	5.4×10^{-4}	383	18	0.57	—
TiAl [5]	50	1025	8.3×10^{-4}	250	5	0.43	—
TiAl [6]	50	850	8.3×10^{-4}	260	0.4	—	—
TiAl-Nb-Cr [7, 8]	47	1280	8×10^{-5}	470	20	0.5	395
TiAl-V [9]	39–43	1147	3×10^{-4}	600	6	0.8	—
TiAl [10]	48	1050	5.6×10^{-3}	550	0.85	0.5	350
TiAl-Nb-Cr [11]	48	1280	2×10^{-4}	475	20	0.5	389
TiAl-Nb-Cr [12]	46	1200	1×10^{-3}	980	4	—	—
TiAl [13]	46	900	1.3×10^{-3}	720	0.2	0.48	—
	46	900	6.4×10^{-4}	680	1.5	0.52	—
TiAl-Si [14]	46	700	2×10^{-4}	—	0.15	0.44	—
	46	800	4×10^{-4}	—	0.28	0.57	—

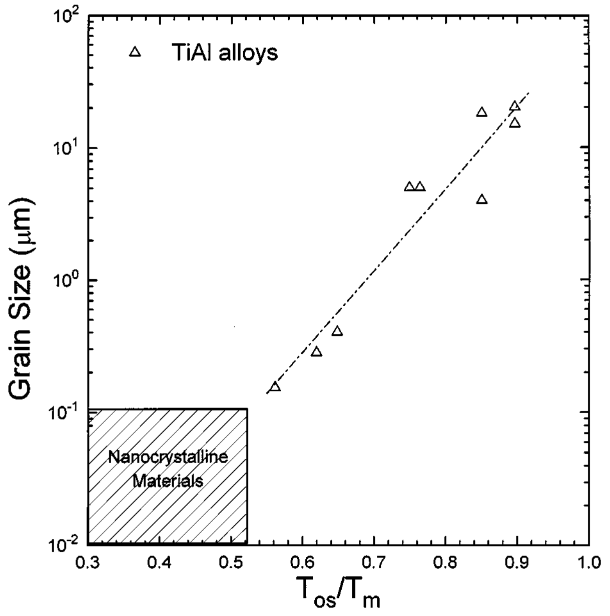

Figure 1 The variation of normalized optimum superplastic temperature with grain size for a number of TiAl alloys. The present trend indicates the possibility of low temperature superplasticity in nanocrystalline TiAl.

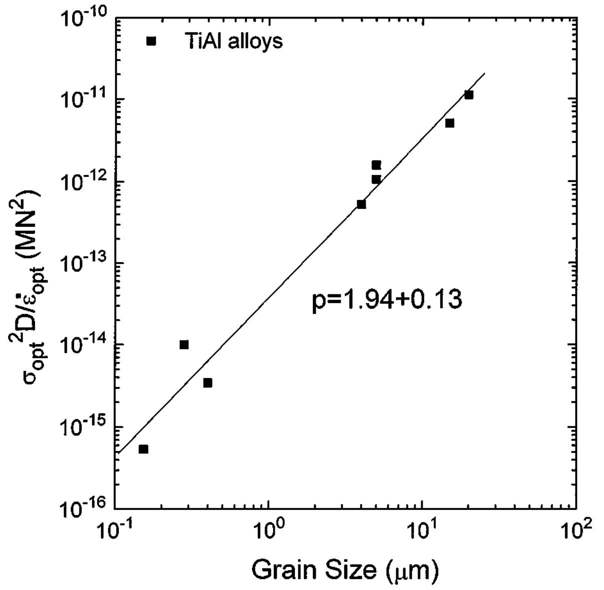

Figure 2 A plot of $(\sigma_{\text{opt}}^2 D)/\dot{\varepsilon}_{\text{opt}}$ against grain size shows a grain size dependence of ~2.

the general constitutive relationships for superplastic deformation.

2. Analysis of superplastic data on microcrystalline and submicrocrystalline titanium aluminides

The reported data (Table I suggests a strain rate sensitivity of 0.5 (i.e., stress exponent of ~2) in the range of optimum superplastic strain rate ($\dot{\varepsilon}_{\text{opt}}$). We choose the flow stress at which maximum elongation or maximum strain rate sensitivity is observed as the optimum superplastic stress, σ_{opt}. Same criterion is used to define the optimum superplastic temperature. These optimum superplastic parameters are used for further analysis.

Fig. 2 shows a plot of an optimum superplastic parameter $(\sigma_{\text{opt}}^2 D)/\dot{\varepsilon}_{\text{opt}}$ against the grain size. We have used an activation energy of 380 kJ mol^{-1} ($D = 1 \times 10^{-4} \exp(-380000/RT)$ m^2 s^{-1}). The choice of activation energy is based on the reported values of activation energy (see Table I) and some previous analysis of superplasticity in titanium aluminides [19]. We note that the chosen value of 380 kJ mol^{-1} is much higher than the activation energy for lattice self-diffusion of Ti in TiAl, which is 291 kJ mol^{-1} [20]. Similar discrepancy can be noted in the creep deformation of γ-TiAl alloys. Oikawa [21] and Es-Souni *et al.* [22] have reported activation energy values in the range of 350–400 kJ mol^{-1}. Although, the origin of this discrepancy is not clear at this time, a value of 380 kJ mol^{-1} provides a good temperature compensation for the entire data. Cheng *et al.* [19] have interpreted this value to be associated with the lattice diffusion of the slowest moving species in TiAl. Fig. 2 gives a grain size dependence of ~2 for the entire data set, from 150 nm to 20 μm grain size. This is similar to the grain size dependence observed by Ameyama *et al.* [10] in the grain size range of 0.85–2.6 μm. This gives a correlation for optimum superplasticity in $(\gamma + \alpha_2)$ titanium aluminides as

$$\frac{\sigma_{\text{opt}}^2 D}{\dot{\varepsilon}_{\text{opt}} d^2} = 4 \times 10^{-2} \text{ MPa}^2. \qquad (1)$$

The parametric dependencies can be summarized as: (a) a stress exponent of 2, (b) an activation energy of about

380 kJ mol^{-1}, and (c) an inverse grain size dependence of 2. It should be noted that no discontinuity in the correlation is observed at $\alpha_2 \rightarrow \alpha$ transformation temperature ($\sim$1383 K). The implication is that the nature of α_2 or α i.e. ordered or disordered, does not influence the mechanism of superplasticity. Also, the mechanism of superplastic flow is same over the entire temperature range investigated so far. From the observed parametric dependencies the superplastic mechanism appears to be *lattice diffusion-controlled slip-accommodated grain boundary sliding* (see a review by Sherby and Wadsworth [23] for a compilation of mechanisms). It appears that the slip accommodation in γ phase is a critical step during superplasticity of TiAl alloys. The activation energy for the superplastic flow being close to that of lattice diffusion suggests that dislocation climb is involved during the accommodation process. It should be noted that none of the existing models based on slip accommodation predict the combination of parametric dependencies observed for TiAl alloys, i.e. stress exponent of 2, grain size dependence of 2 and activation energy for lattice diffusion [23]. These parametric dependencies are, however, quite similar to those observed in a number of disordered superplastic alloys [23]. Mishra *et al.* [11] have noted that the dimensionless constant for TiAl alloys is higher than the value for the disordered Ti alloys. This is intriguing because one would expect the kinetics to be slower in ordered alloys on a normalized basis. This aspect needs further investigation.

Another interesting aspect involves the optimum flow stress during superplasticity of TiAl alloys. We note that the data for 150–280 nm grain size was generated in compression and flow stresses were quite high (260–540 MPa), although the stress exponent was close to 2. It raises an intriguing question regarding cavitation in tension. Lower stresses during superplasticity are preferable to avoid cavitation during tensile deformation. To get an idea of the stress dependence of optimum superplasticity in the reported data for titanium aluminide, the optimum flow stress is plotted against $(d/b)^2(\dot{\varepsilon}_{\mathrm{opt}}/D)$ in Fig. 3a. The increase in flow stress with the parameter $(d/b)^2(\dot{\varepsilon}_{\mathrm{opt}}/D)$ is a combination of grain size, optimum strain rate and optimum superplastic temperature. We need to separate the effect of grain size, optimum strain rate and temperature on the optimum flow stress. For this, the optimum flow stress is plotted against diffusivity compensated strain rate $(\dot{\varepsilon}_{\mathrm{opt}}/D)$ in Fig. 3b. Linear regression of the data in Fig. 3a and b gives

$$\left(\frac{d}{b}\right)^2 \frac{\dot{\varepsilon}_{\mathrm{opt}}}{D} = 4 \times 10^{20} \sigma_{\mathrm{opt}}^2, \text{ and} \tag{2a}$$

$$\frac{\dot{\varepsilon}_{\mathrm{opt}}}{D} = 1.6 \times 10^{9} \sigma_{\mathrm{opt}}^4. \tag{2b}$$

From these relationships we can obtain,

$$\sigma_{\mathrm{opt}} = 5 \times 10^5 \frac{b}{d}. \tag{3}$$

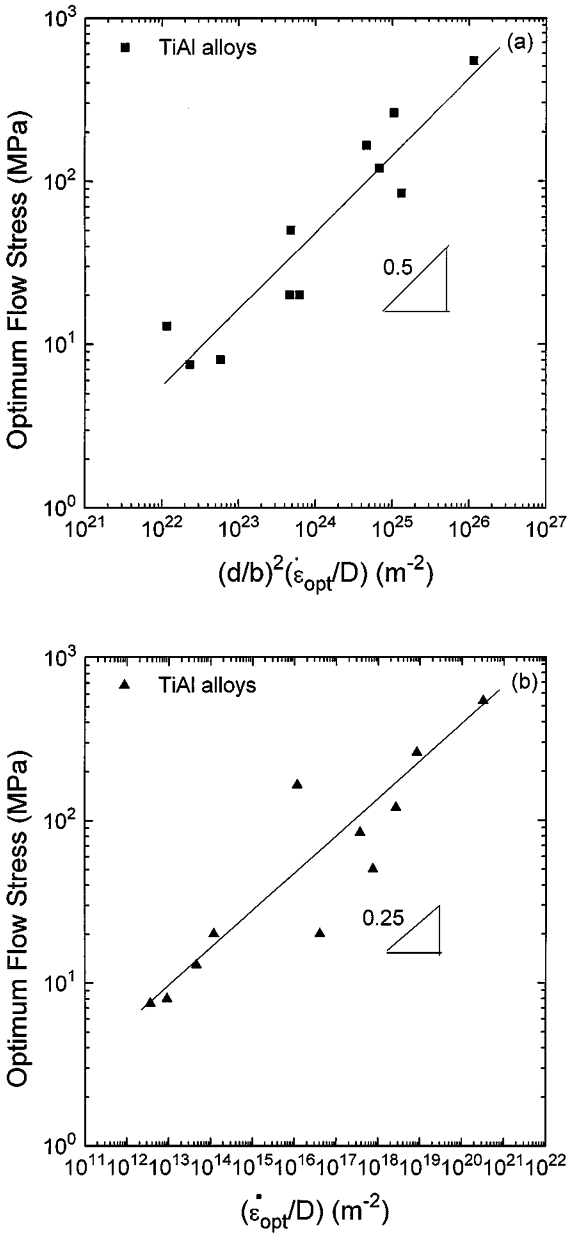

Figure 3 The variation of optimum flow stress for superplasticity with (a) a superplastic parameter $(d/b)^2(\dot{\varepsilon}_{\mathrm{opt}}/D)$, and (b) diffusivity compensated strain rate. A comparison of both these plots gives a relationship between optimum flow stress and grain size.

It is important to note that the above correlation suggests an intrinsic optimum flow stress dependence on the grain size. This assumes particular significance because as noted earlier the slip accommodation plays an important role in superplasticity of TiAl alloys.

An approximate estimate of stress required to nucleate dislocation during slip accommodation of grain boundary sliding can be made in the following manner. Fig. 4 shows a schematic of the geometry of dislocation generation from the grain boundary edge. Hirth and Lothe [24] have given a relationship for dislocation generation by Frank-Read source in lattice, which after simplification can be written as

$$\tau = \frac{\mu b}{4\pi L(1-\nu)}\left(\ln\frac{L}{b} - 1.67\right) \tag{4}$$

where L is the distance between the pinning points, ν is the Poisson's ratio and τ is the shear stress required

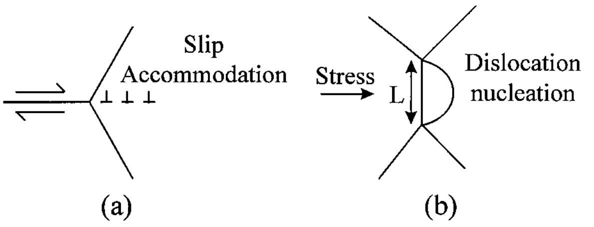

Figure 4 A schematic of dislocation nucleation from grain boundary edge during superplasticity.

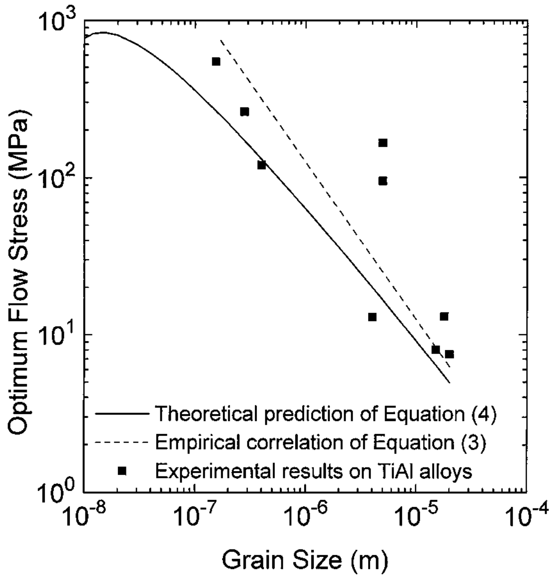

Figure 5 A comparison of the experimental and theoretical flow stress for optimum superplasticity.

to generate the dislocations. For superplastic deformation, the tensile stress needed to nucleate dislocation for slip accommodation can be calculated by approximating $L = d/3$, $\nu = 0.33$ and $\sigma = \sqrt{3}\tau$. The choice of $L = d/3$ is based on the fact that the edge of the tetrakaidecahedron grain is 1/3 of the distance between the faces of the grains [25]. We realize that Equation 4 does not have (a) strain rate dependence (which is an integral part of high temperature deformation), (b) temperature dependence other than the modulus and (c) the details of dislocation generation from grain boundaries. These effects can be significant as the flow stress at high temperatures is known to be strain rate and temperature dependent in the superplastic region. However, in the absence of any other model incorporating these effects, Equation 4 can be used to check qualitatively if a grain size dependent flow stress is expected for superplasticity. The theoretical flow stress from Equation 4 is plotted against grain size in Fig. 5 as the solid line. The empirical correlation in Equation 3 is included as the dash line. Two important features emerge from this. First, the theoretical flow stress is inversely proportional to the grain size in 100 nm–20 μm range. This matches with the experimental observation. Second, the magnitude of flow stress is close to the experimental values. We note that some scatter exists because the experimental data need to be compensated for the differences in the optimum strain rate and test temperature. Nevertheless, it clearly shows an intrinsic grain size dependence of the flow stress during superplastic flow in TiAl alloys.

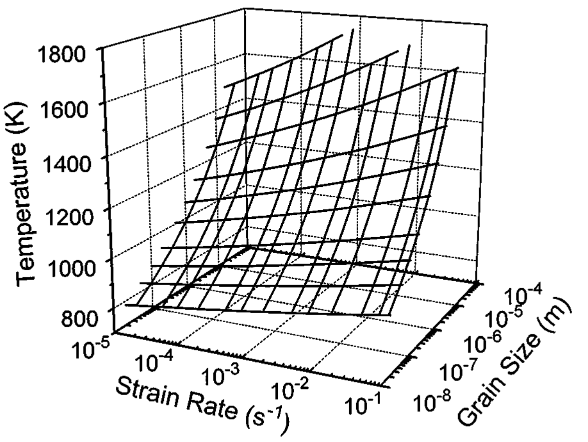

Figure 6 A three-dimensional map depicting optimum superplastic condition for TiAl alloys. Note that by decreasing the grain size, one can lower the optimum superplastic temperature and/or increase the optimum strain rate.

The intrinsic grain size dependence of flow stress might have an important implication on the possibility of superplasticity in some nanocrystalline materials. Combining Equations 1 and 3 gives,

$$\frac{D}{\dot{\varepsilon}_{\mathrm{opt}} d^4} = 2.6 \times 10^6 \qquad (5)$$

This relationship can be used to construct a three dimensional map depicting the optimum superplasticity domain for TiAl alloys in terms of temperature, strain rate and grain size. Fig. 6 shows such a three dimensional map. It shows the possibility of attaining low temperature superplasticity as well as high strain rate superplasticity at lower grain sizes. Clearly, more experimental results are required to check out these predictions.

3. Concluding remarks

The mechanism of superplastic deformation of microcrystalline and submicrocrystalline TiAl alloys is same. The optimum temperature appears to correlate well with grain size. Microstructural refinement lowers the optimum superplastic temperature for γ-TiAl alloys close to that of Ti-6Al-4V alloys. The empirical correlations obtained in the present analysis can be used to obtain the optimum superplastic conditions for a given grain size. The inverse dependence of optimum superplastic flow stress on grain size can be linked with slip accommodation during grain boundary sliding.

Acknowledgement

The authors gratefully acknowledge the support from the National Science Foundation through the grants NSF-DMR-93-00217 and NSF-DMR-96-30881.

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Received 18 February 1998
and accepted 21 June 1999