Originally published as:

Cao, G. H., Kloeden, B., Rybacki, E., Oertel, C. G., Skrotzki, W. (2008): High strain torsion of a TiAl-based alloy. - Materials Science and Engineering A - Structural Materials Properties Microstructure and Processing, 483-484, 512-516

DOI: 10.1016/j.msea.2006.12.157

High strain torsion of a TiAl-based alloy

G.H. Cao¹⁺, B. Klöden¹⁺⁺, E. Rybacki², C.-G. Oertel¹, W. Skrotzki^{1*}

¹ *Institut für Strukturphysik, Technische Universität Dresden, 01062 Dresden, Germany*

² *Geoforschungszentrum Potsdam, Telegrafenberg, 14473 Potsdam, Germany*

Keywords: TiAl, torsion, microstructure, texture, superplasticity

Abstract

A two-phase lamellar and globular TiAl-based alloy (Ti-45Al-4(Cr, Nb, Ta, B)) with different textures has been deformed in torsion in a Paterson-type rock deformation machine at a temperature of 1173 K under 400 MPa argon confining pressure. The shear stress – shear strain curves with increasing shear strain show a decrease or increase of shear stress in the lamellar and globular case, respectively, to a common steady state value. Activation analysis at high strains yields stress exponents of about 1.8 and an activation energy of 3.29 eV. With increasing shear strain the lamellar structure breaks down and a two-phase fine-grained globular structure develops by recrystallization with a grain size in the order of 2 microns. The weak initial fibre textures at high strains are randomized. The mechanical, microstructural and textural features indicate a transition from dislocation creep to superplastic flow.

* Corresponding author:

E-mail address: werner.skrotzki@physik.tu-dresden.de (W. Skrotzki)

Present addresses:

+ 206 Metals Development, Ames Laboratory, Iowa State University, Ames, IA 50011-3020, USA

++ Fraunhofer-Institut für Fertigungstechnik und Angewandte Materialforschung,
Winterbergstr. 28, 01277 Dresden, Germany

1. Introduction

Two-phase (TiAl (γ) and Ti₃Al (α_2)) titanium aluminides have a potential as high temperature structural materials due to their attractive properties such as low density, high elastic modulus, good creep and oxidation resistance. Small amounts of different alloying elements (Cr, Nb, Mn, V, Si, B, C) improve the room temperature ductility, formability, tensile strength, creep and oxidation resistance [1]. Most of the previous works on hot-working of TiAl-based alloys have focused on the processing related aspects, however, only a few studies are available on details of the recovery and dynamic recrystallization (DRX) processes [2-8]. Therefore, it is the aim of the present work to investigate the microstructure evolution and creep behaviour during large strain torsion of Ti-45Al-4(Cr, Nb, Ta, B). It is the advantage of torsion that cylindrical samples from the middle to rim have a strain gradient allowing strain dependent investigations of the microstructure and texture on the same sample. Moreover, effects of large strain, i.e. dynamic recrystallization, can be explored without significantly changing the geometry of the specimen [9]. Based on these studies, possible creep mechanisms of the TiAl alloy used are elucidated.

2. Experimental and data processing

The Ti-45Al-4(Cr, Nb, Ta, B) alloy developed at Plansee AG (Reutte, Austria) with an ingot diameter of 180 mm was thermomechanically processed in the following way. One ingot was extruded with an extrusion ratio 20/1 at 1543K and annealed in high vacuum at 1613K for 2h followed by furnace-cooling (sample A). Another ingot after the first extrusion experienced a second extrusion with extrusion ratio 4/1 at 1273K (sample B). Cylindrical

samples with diameter and height of 10 mm were deformed in torsion at 1173K in a Paterson-type rock deformation machine [9, 10] under 400 MPa argon confining pressure. The sample assembly was enclosed in a thin steel jacket. The measured torque was corrected for apparatus compliance and jacket strength (accuracy $\approx 3\%$). Temperature was monitored by a thermocouple located 3 mm above the top of the specimen and kept constant within about $\pm 2\text{K}$.

Within a solid cylinder deformed in torsion the shear strain γ and shear strain rate $\dot{\gamma}$ increase linearly with radius r from zero along the central axis to a maximum at the outer mantle surface at radius R according to

$$\gamma_r = \Theta r/l \quad (1)$$

and

$$\dot{\gamma}_r = \dot{\Theta} r/l, \quad (2)$$

with Θ = twist angle, $\dot{\Theta}$ = twist rate and l = length of torsion cylinder. The twist rate was kept constant yielding a maximum initial shear strain rate of $2 \cdot 10^{-4} \text{s}^{-1}$. Due to sample shortening during torsion the shear strain rate slightly increased with shear strain (at maximum 4%). A report on this effect, known as Swift effect [11], will be published separately. The maximum shear strain γ was about 3.5. The shear stress τ increases non-linearly with radius. It can be estimated if some assumptions are made on the flow behaviour of the material. In the following it is assumed that the material obeys a power law constitutive relationship

$$\dot{\gamma} = A \tau^n \exp(-Q/kT), \quad (3)$$

where n and Q are the stress exponent and creep activation energy, respectively, and k is Boltzmann's constant. The pre-exponential factor A is a material dependent constant, implicitly containing a possible strain dependence for non-steady state behaviour. Following this approach, the shear stress at any given radius can be calculated from the measured torque M using an appropriate value of n and accounting for the Swift effect:

$$\tau_r = \frac{M(3+1/n)}{2\pi R^3} \left(\frac{r}{R}\right)^{1/n}. \quad (4)$$

It should be mentioned that the maximum stress was always calculated at the outer surface R by measuring sample shortening and calculating the actual R at any given strain/length assuming constant volume deformation.

The stress exponent n and the activation energy Q were determined by twist rate and temperature steps at several twists using the relations

$$n = \frac{\Delta \ln \dot{\Theta}}{\Delta \ln M} \quad (5)$$

$$Q = nk \frac{\Delta \ln M}{\Delta(1/T)}. \quad (6)$$

The temperature was stepped $\pm 20\text{K}$ to above and below the actual test temperature and the twist rate was reduced in two steps to 4/7 and 2/7 of the original rate. Heating and cooling rates were about 30K/min. Uncertainty in determination of n and Q is at maximum 20%. More experimental details may be obtained from [9, 10].

Thin foils for transmission electron microscopy were prepared by spark erosion of a column 3 mm in diameter. From this pin 0.5 mm thick discs were cut by diamond saw and mechanically grinded to a thickness of about 60 μm . Electron transparent foils were prepared by jet electropolishing using an electrolyte consisting of 65 methanol, 30 butanol and 5 perchloric acid (in vol.%) at a voltage of 30 V and temperatures between -20 and -30°C. TEM bright field (BF) imaging was performed on a Philips CM 200 electron microscope equipped with a LaB₆ cathode operated at an acceleration voltage of 200 kV.

The initial texture of the whole samples (global texture) was measured by neutron diffraction. Local texture measurements were done by high-energy synchrotron radiation at different radii of the deformed cylinders on a pin of 1 mm diameter taken along the radial direction, i.e. at different shear strains. Details are reported elsewhere [12].

3. Results and discussion

The shear stress – shear strain curves $\tau(\gamma)$ are shown in Fig. 1. For sample A the curve is characterized by a peak stress at low shear strain followed by strain softening towards a steady state shear stress above a shear strain of about 3. This behaviour is typical for materials exhibiting dynamic recrystallization (DRX) [13, 14]. During DRX the lamellar structure (colony size about 100 μm) breaks down and grain refinement takes place resulting in a steady state equiaxed γ grain structure with a grain size of about 2 μm (Fig. 2). Simultaneously, dissolution and spheroidization of the α_2 laths lead to a new equilibrium state. In the case of the fine-grained globular sample B strain hardening takes place to the steady state shear stress of sample A. The recrystallized grain size seems to be constant at about 2 μm or even slightly increases (Fig. 3). Moreover, transformation of Ti_3Al to TiAl via Ti_2Al intermediate phase takes place [15].

The stress exponent decreases with shear strain in sample A from 4.2 to 1.9, while in sample B it is constant at about 1.8 (Tab. 1). The activation energy at large strain is about 3.29 eV.

During torsion the weak initial fibre textures are randomized (Fig. 4).

These results suggest that grain refinement by DRX leads to superplastic flow characterized by grain boundary sliding with diffusional accommodation [17]. The activation energy for creep lies between that of Ti and Al self-diffusion in TiAl (2.59 eV and 3.71 eV) [16]. Similar conclusions have been drawn by [17-21]. On the other hand, in the coarse-grained sample A before DRX, creep is dominated by dislocation climb with a stress exponent of 4.2 [22].

Different mechanisms have been proposed for DRX of titanium aluminides. (i) Due to maintenance of the deformation texture and continuous strengthening of the texture, strain-

induced grain boundary migration has been assumed for compressed Ti-49Al [3]. (ii) Due to non-thermal equilibrium and metastability of the microstructure, phase boundary bulging takes place in Ti-45Al-10Nb [5]. (iii) Due to the low stacking fault energy of titanium aluminides, nucleation and growth takes place in highly deformed regions of Ti-47Al-2Mn-2Nb + 0.8 vol.% TiB₂ [4]. (iv) Due to shear processes in Ti-45Al-(5-10)Nb +X (X = other metallic and non-metallic elements) traversing into the inter-colony γ grains (Fig. 2a), strain accommodation in these grains is provided by mechanical twinning and dislocation glide and climb leading to the formation of subgrain boundaries. Rearrangement of the twin structures and sub-boundaries leads to recrystallization [8]. There are indications that all of these mechanisms have operated in sample A, too, but their contribution to DRX is not clear.

Acknowledgements

G.H. Cao thanks the Alexander-von-Humboldt Foundation for a fellowship. Thanks are also due to DESY-HASYLAB for beamtime and financial support of travel and accommodation. The two starting materials were kindly provided by Dr. H. Kestler (Plansee AG, Reutte, Austria). The neutron texture measurements by Prof. H.-G. Brokmeier (GKSS Geesthacht) and the technical assistance by Mr. T. Reiter and Mrs. H. Müller (TU Dresden) are gratefully acknowledged.

References

- [1] F. Appel, M. Oehring, in: C. Leyens, M. Peters (Eds.), *Titanium and Titanium Alloys*, WILEY-VCH, Weinheim, Germany, 2003, pp. 89-105.
- [2] C. Hartig, H. Fukutomi, H. Mecking, K. Aoki, *ISIJ Int.* 33 (1993) 313-320.
- [3] H. Fukutomi, K. Aoki, A. Nomoto, S. Ikeda, C. Hartig, *Mat. Trans. JIM* 35 (1994) 794-799.
- [4] B. Skrotzki, T. Rudolf, A. Dlouhy, G. Eggeler, *Scripta Mater.* 39 (1998) 1545-1551.
- [5] W.J. Zhang, U. Lorenz, F. Appel, *Acta Mater.* 48 (2000) 2803-2813.
- [6] F. Appel, M. Oehring, J.D.H. Paul, Ch. Klingenberg, T. Caneiro, *Intermetallics* 12 (2004) 791-802.
- [7] R.M. Imayev, V.M. Imayev, M. Oehring, F. Appel, *Met. Mat. Trans.* 36A (2005) 859-867.
- [8] F. Appel, C.J. Buque, S. Eggert, U. Lorenz, M. Oehring, J.D.H. Paul, in : Y.-W. Kim, H. Clemens, A.H. Rosenberger (Eds.), *Gamma Titanium Aluminides 2003*, TMS (The Minerals, Metals & Materials Society), Warrendale, Pennsylvania, USA, 2003, pp. 319-326.
- [9] M.S. Paterson, D.L. Olgaard, *J. Struct. Geol.* 22 (2000) 1341-1358.
- [10] E. Rybacki, M.S. Paterson, R. Wirth, G. Dresen, *J. Geophys. Res.* 108, B2, 2089 (2003) ETG 8 -1-24.
- [11] H.W. Swift, *Engineering* 163 (1947) 253-257.
- [12] W. Skrotzki, B. Klöden, R. Tamm, C.-G. Oertel, U. Garbe, E. Rybacki, *Textures Microstruct.* 35 (2003) 163-173.
- [13] F. Montheillet, P. Gilormini, J.J. Jonas, *Acta Metall.* 33 (1985) 705-717.
- [14] S. Gourdet, F. Montheillet, *Acta Mater.* 51 (2003) 2685-2699.

- [15] G.H. Cao, W. Skrotzki, T. Gemming, J. Alloys Comp. 417 (2006) 169-172.
- [16] Y. Mishin, C. Herzig, Acta Mater. 48 (2000) 589-623.
- [17] T.G. Nieh, J. Wadsworth, O.D. Sherby, Superplasticity in Metals and Ceramics, Cambridge University Press, Cambridge, United Kingdom, 1997.
- [18] J.A. Jiménez, O.A. Ruano, G. Frommeyer, S. Knippscher, Intermetallics 13 (2005) 749-755.
- [19] J. Sun, J.S. Wu, Y.H. He, Z. Metallkde 91 (2000) 472-476.
- [20] R. Bohn, T. Klassen, R. Bormann, Intermetallics 9 (2001) 559-569.
- [21] A. Bartels, H. Kestler, H. Clemens, Mater. Sci. Eng. A 329-331 (2002) 153-162.
- [22] S. Bystranowski, A. Bartels, H. Clemens, R. Gerling, F.P. Schimnsky, G. Dehm, H. Kestler, Intermetallics 13 (2005) 515-524.

Figure captions

Fig. 1: Shear stress - shear strain curves for samples A (a) and B (b). The shear stress changes at certain shear strains are due to shear strain rate and temperature jumps as well as to unloading in order to measure the Swift effect.

Fig. 2: TEM BF image of the initial (a) and deformed microstructure at $\gamma = 0.6$ (b) of sample A.

Fig. 3: TEM BF image of the initial (a) and deformed microstructure at $\gamma = 3$ (b) of sample B.

Fig. 4: Texture represented as (110) pole figures: (a) initial texture of sample A, (b) initial texture of sample B, (c) texture of sample A at $\gamma = 3$, (d) texture of sample B at $\gamma = 3$. (Intensity maxima are given in multiples of a random distribution, grey areas are below unity; (a) and (b): ED = extrusion direction in centre of pole figures, (c) and (d): ND = shear plane normal/torsion axis, SD = shear direction, TD = transverse direction)

Tables

Table 1: Results of the activation analysis at different shear strains γ : n = stress exponent

($\Delta n_{\max} = \pm 0.2$), Q = activation energy ($\Delta Q_{\max} = \pm 0.07$ eV).

	A	B
$n_1 (\gamma)$	4.2 (0.1)	1.8 (0.1)
$n_2 (\gamma)$	3.2 (0.7)	1.7 (2.1)
$n_3 (\gamma)$	1.9 (3.2)	
Q in eV (γ)	3.15 (2.8)	3.44 (2.8)

Figures

Fig. 1

(a)

(b)

Fig. 2

(a)

(b)

Fig. 3

Fig. 4