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On the mechanism of superelasticity in Gum metal

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Abstract

The deformation mechanisms of the β -Ti alloy, Gum metal, were investigated with the aid of in situ synchrotron X-ray diffraction (SXRD) and transmission electron microscopy (TEM). SXRD showed that Gum metal undergoes a reversible stress-induced martensitic (α'') phase transformation. Oxygen increases the resistance to shear by increasing *C'* and limits the extent of α'' growth. Prior deformation aids α'' formation of by providing nuclei, such as $\{112\}<111>$ twins and stress-induced ω plates. The formation of twins and ω plates, both observed in TEM, are believed to be a result of a low G_{111} in this alloy. Features similar to the "giant faults" seen previously were observed in TEM; their formation is believed to be a result of $\{112\}<11>$ shear. © 2008 Acta Materialia Inc. Published by Elsevier Ltd. All rights reserved.

Keywords: Gum metal; Martensitic phase transformation; Titanium alloys; Synchrotron radiation; Transmission electron microscopy

1. Introduction

The recently developed β -titanium alloy Gum metal (Ti-36Nb-2Ta-3Zr-0.3O, wt.%) possesses high strength, a low elastic modulus, a high yield strain and high ductility [1]. These "superproperties" are reportedly achieved when the following electronic "magic numbers" are simultaneously satisfied: (i) a compositional average valence electron number (e/a) of ~ 4.24; (ii) a bond order (Bo) of ~ 2.87; and (iii) a *d*-electron orbital energy (Md) of ~ 2.45 eV. In addition, this alloy requires cold working and > 0.7 at.% oxygen. Gum metal is a candidate material in a range of applications, including automotive components and biomedical implants.

It is claimed that Gum metal plastically deforms without dislocation glide [1,2] as its ideal shear strength is comparable with its actual strength, such that plastic deformation can occur by ideal shear, explaining the existence of the "giant faults" observed in transmission electron microscopy (TEM). Since the ideal strength can be estimated from

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the single crystal elastic constants, C_{ij} [3], these are considered to be key to explaining this observation, Eq. (1). The C_{ij} of several binary titanium alloys have been calculated using the ultra-soft pseudo-potential method with a generalized approximation to density functional theory [4], indicating that the shear modulus, $C' = (C_{11} - C_{12})/2$, approaches zero when e/a is around that of Gum metal (4.24). The ideal shear strength τ_{max} is given by [3]:

$$\tau_{\max} = 0.11G_{111} = 0.11\frac{3C_{44}(C_{11} - C_{12})}{(C_{11} - C_{12}) + 4C_{44}}$$
(1)

where G_{111} is the shear modulus along < 111 > on $\{011\}$, $\{112\}$ or $\{123\}$.

90% cold-worked Gum metal shows non-linear elasticity [1,5]. Recently we have observed a stress-induced reversible phase transformation during such pseudoelasticity using in situ synchrotron X-ray diffraction (SXRD) [6]. Further, we have observed dislocations in cold-worked specimens [7] and shown that $(C_{11} - C_{12})$ is > 32 GPa, implying τ_{max} is > 2 GPa. Taking this into account, along with the data in Table 1, it appears that in body-centred cubic (bcc) alloys with low ideal shear stresses, the lattice is fundamentally unstable and will undergo a stress-induced phase

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Table 1 Comparison of C_{ij} and τ_{max} (GPa) of Gum metal [6] with those of other bcc superelastic alloys, adapted from Kim et al. [9].

		C'	C_{44}	$\tau_{\rm max}$
EP0.9	[6]	16	28	2.1
PH0.9	[6]	17.5	29	2.2
Ti–Ni	[8]	17-19	35-39	2.4
Ti–22Nb–6Ta	[9]	8	21.3	1.1
Cu-Al-Ni	[10]	7–8	100	1.2
Cu–Zn	[11]	8	90	1.3
Cu–Zn–Al	[12]	5.8	86	0.9
Au–Cu–Zn	[13]	3–5	60	0.6
Au–Cd	[14]	3-3.8	42	0.5
Ni–Al	[15]	14.6	132	2.3

transformation, before deformation can proceed via ideal shear; the alloys in Table 1 have ideal shear stresses similar to or lower than Gum metal, but are known to deform via a stress-induced martensitic phase transformation.

In the present study, the new diffraction peaks observed previously using SXRD [6] are identified with a phase and the deformation structures are analysed using TEM. It is possible that the unusual behaviour observed in Gum metal may be due to inhomogeneities at the nanoscale produced by the blended elemental powder (EP) processing route. Therefore, we evaluate here material produced by different process routes to examine this issue. In addition, published reports of the single crystal elastic constants show significant scatter, which may be due to variations in alloy composition [7,6,16]. Further, similar alloys are known to deform by either slip or stress-induced martensite, depending on β stability. Therefore we also examine the effect of oxygen and β stability (Mo_{eq} [17]) on the transformation. Cold working was originally reported to be required to achieve the low modulus in the alloy, and so the effects of cold work and texture are examined.

2. Experimental methods

Pure elemental powders (EPs) were mixed and compacted by cold isostatic pressing at 392 MPa, sintered at 1300 °C for 16 h in a vacuum of 10^{-4} Pa and furnace cooled. The billet was hot forged at 1150 °C and rolled at 800 °C into a 16 mm diameter bar. The surface oxide was mechanically removed prior to solution heat treatment at 900 °C for 30 min (specimen condition EP0.0, where the suffix denotes the amount of cold work). Cold work was performed by: (i) rolling a 10 mm thick cross-section of the bar to 1 mm thickness (EP0.9); and (ii) swaging of the 16 mm diameter bar into a 4 mm diameter bar, followed by cold rolling into a 1 mm sheet (EP3.5).

Plasma-sprayed powder was hot isostatically pressed (HIPed) at 140 MPa and 920 $^{\circ}$ C in a mild steel can, solution treated at 1000 $^{\circ}$ C for 30 min and water quenched (specimen PH0.0). A second billet was then cold rolled to a strain of 0.9 (PH0.9).

In the ingot metallurgy (IM) route a 1.2 kg button of compacted elemental raw materials was plasma melted using a 50 kW He torch in an evacuated Cu hearth furnace. The button was inverted and remelted three times. An extrusion billet machined from the button was solution treated at 850 °C for 60 min, coated with glass lubricant, canned in mild steel and heat treated at 975 °C for 105 min prior to extrusion to 12 mm rod (IM0.0). The compositions of the specimens are given in Table 2.

In addition to the specimens listed above, some Ti–29Nb–13Ta–4.6Zr sheet was acquired. This alloy was prepared by levitation melting and vacuum arc remelting, hot forging at 900 °C in air and solution treatment at 790 °C for 60 min, followed by water quenching. In order to compare to the extruded microstructure of Gum metal, a near- β alloy, Ti–5Al–5Mo–5V–3Cr, was extruded in the β condition, with an extrusion ratio of 10:1.

Dog-bone-shaped specimens with a 10 mm gauge length and 1.75 mm² cross-section were machined and mechanically polished. The tensile axis of the worked materials (PH0.9, EP0.9, EP3.5 and IM0.0) was parallel to the rolling/extrusion directions. Loading tests were performed at ID15B at the European Synchrotron Radiation Facility, Grenoble, France, using an X-ray energy of 88 keV $(\lambda = 0.1415 \text{ Å})$ and a $0.5 \times 0.5 \text{ mm}$ incident beam (Fig. 1). A Pixium detector at a sample-detector distance of 648 mm was used to collect diffraction rings. Sampling times were ~ 0.4 s. XRD rings were segmented and intensity $-d_{hkl}$ diffraction profiles obtained from a 10° region from grains with plane normals parallel to the loading direction using the software program FIT2D [18]. All specimens were deformed at an initial strain rate of 5×10^{-4} s⁻¹. Loading–unloading tensile tests were per-

Table 2

Measured chemical compositions of the Ti–Nb–Ta–Zr–O alloys (wt.%). All alloys had Md = 2.5 eV and Bo = 2.87.

	Nb	Та	Zr	0	V	Fe	Ni	Cr	e/a	Moea
D. C. [1]	25.0	0.1	2.1	0.22					1.2.4	10.4
Ref. [1]	35.8	2.1	3.1	0.32	-	-	-	-	4.24	10.4
EP	36.9	2.0	3.0	0.30	0.09	_	0.2	0.1	4.26	11.0
PH	35.7	2.1	3.2	0.42	0.14	0.08	0.08	0.1	4.25	10.9
IM	35.0	2.1	3.1	0.30	0.01	0.03	0.01	0.02	4.23	10.3



Fig. 1. Schematic of the experimental setup used to perform the SXRD measurements.

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