

Investigation of early stage deformation mechanisms in a metastable β titanium alloy showing combined twinning-induced plasticity and transformation-induced plasticity effects

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Abstract

As expected from the alloy design procedure, combined twinning-induced plasticity and transformation-induced plasticity effects are activated in a metastable β Ti–12 wt.% Mo alloy. In situ synchrotron X-ray diffraction, electron backscatter diffraction and transmission electron microscopy observations were carried out to investigate the deformation mechanisms and microstructure evolution sequence. In the early deformation stage, primary strain/stress-induced phase transformations ($\beta \rightarrow \omega$ and $\beta \rightarrow \alpha''$) and primary mechanical twinning ($\{332\}\langle 113 \rangle$ and $\{112\}\langle 111 \rangle$) are activated simultaneously. Secondary martensitic phase transformation and secondary mechanical twinning are then triggered in the twinned β zones. The $\{332\}\langle 113 \rangle$ twinning and the subsequent secondary mechanisms dominate the early-stage deformation process. The evolution of the deformation microstructure results in a high strain-hardening rate (~ 2 GPa), bringing about high tensile strength (~ 1 GPa) and large uniform elongation (> 0.38).

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1. Introduction

During the past few decades, interest in titanium alloys has been continuously increasing due to their combination of properties such as high strength [1–6], low density, biocompatibility [7–10] and good corrosion resistance [11,12]. However, both their low ductility (uniform elongation typically less than 0.20) and their lack of strain-hardening when compared with steels or Co–Cr alloys [11,13] limit their use in advanced applications where superior combinations of strength and ductility are required.

A strategy dedicated to a general improvement of the mechanical properties based on transformation-induced plasticity (TRIP) or twinning-induced plasticity (TWIP) has been widely investigated in the case of steels [14–16]. An optimization of mechanical properties can be also achieved in Ti-based alloys, when controlling the metastability of the β matrix through its chemical composition [4,5,13,17,18] that strongly influences the martensitic start (M_s) temperature [5,13,19] and the critical resolved shear stress (CRSS) for twinning. Indeed, it has been reported that several deformation-induced phase transformations, i.e. ω phase and α'' phase precipitation, and two twinning modes, $\{112\}\langle 111 \rangle$ type and $\{332\}\langle 113 \rangle$ type, could be activated under different stress conditions (strain rate, temperature, etc.) in some specific Ti alloys

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[3,20–25]. These mechanisms can be activated in various alloys exhibiting the shape memory effect [19], superelasticity [18–20,23,24,26] or strain-hardening behaviour [3,4,27,28].

Recently, an electronic design approach for the development of a new family of titanium alloys exhibiting a combination of high ductility and improved strain-hardening rate has been proposed and exemplified in the binary Ti–12 wt.% Mo grade [3,27]. The chemical formulation of such alloys was designed following the Morinaga model based on the cluster DV- $X\alpha$ method by mapping electronic parameters Bo (bond order) and Md (d -orbital energy) [29,30]. This map is of great interest since it can be used as a tool to design new titanium alloys exhibiting specific improved performances. So far, this kind of stability diagram has specially been used for the design of the last generation of low modulus/high strength alloys, such as the “Gum metals” series [31].

In the case of the high strain-hardening rate in Ti–12 wt.% Mo alloy, the design approach aims at activating simultaneously various deformation modes [27]. It is well known that, in titanium alloys, the main deformation mechanism evolves from dislocation glide to mechanical twinning then to martensitic transformation when the β phase chemical stability decreases. Domains corresponding to the transition between these two last deformation modes have then been targeted on the Bo/Md map [27]. Experimental validation of this design procedure was reached with the Ti–12Mo alloy exhibiting true stress–true strain values at necking, of ~ 1000 MPa and 0.38, respectively, with a large strain-hardening rate close to the theoretical limit [27,32]. These values of strength and elongation were hardly reached before in body-centred cubic (bcc) alloys. Both mechanical twinning and stress/strain-induced phase transformations were observed in deformed samples, in good agreement with the theoretical predictions. However, the sequence of deformation processes, involving both TWIP and TRIP, is still unclear and needs to be investigated in more detail to understand the role of the synergy between these mechanisms in the improvement of strain-hardening. As a consequence, the present work focuses on the characterization of the deformation mechanisms of the Ti–12Mo alloy strained from 0 to 0.08 owing to in situ synchrotron X-ray diffraction (SXRD), electron backscattered diffraction (EBSD) mapping and transmission electron microscopy (TEM).

2. Experimental

The binary Ti–12 wt.% Mo alloy was processed by the self-consumable melting technique, giving an ingot of 9 kg. The chemical composition of the raw ingot is listed in Table 1. Plates of 10 mm in thickness were cut, followed by solution treatment (ST) at 1173 K for 30 min and water quenching. The specimens were then cold-rolled down to 0.5 mm thick sheets, corresponding to a reduction level of 95%. These sheets were finally recrystallized at 1173 K

Table 1
Ingot chemical composition (wt.%).

Mo	Fe	H	O	N	C	Ti
12.3	0.02	0.004	0.06	0.004	0.011	Balanced

for 30 min and water quenched to restore a fully β state. The mentioned heat treatments were carried out in tubular furnaces under vacuum (10^{-7} mbar) to prevent oxidation.

In situ SXRD data were collected at the high resolution beam line ID31 of the European Synchrotron radiation Source (ESRF), Grenoble, France, from a tensile sample with gauge width of 4 mm and 0.5 mm in thickness. The incident X-ray wavelength was 0.4 Å. Data collection was performed over the angular range 2–14°, with a step size of 0.005°. Nine scanning stages were carried out at room temperature, starting from the unloaded state, then for increasingly loaded states up to a strain of 0.08 and finally after unloading. Analysis of the diffraction patterns consisted in identifying the phases corresponding to the different peaks and then estimating the lattice parameters owing to Pawley refinement using TOPAS [33]. Estimation of the intensity of specific peaks was also carried out.

Cyclic tensile loading/unloading steps were applied to a prior polished sample, bringing about some surface relief related to the activated deformation mechanisms.

Finally, specimens deformed to various strains were also prepared for EBSD and TEM. Prior to the EBSD observations, samples were first mechanically polished down to 1 μm and then chemically polished with a solution of H_2O_2 and OP-S (oxide polishing suspension from STRUERS, a colloidal silica suspension with a pH of 9.8 and a grain size of $\sim 0.04 \mu\text{m}$). EBSD scans were performed using a field emission gun scanning electron microscope operating at 15 kV, with step sizes ranging from 0.1 to 0.05 μm . A JEOL 2000FX transmission electron microscope operating at 200 kV was also used. Thin foils were prepared by the twin-jet electropolishing technique using a solution of 4% perchloric acid in methanol.

3. Results

3.1. Tensile behaviour

The uniaxial tensile loading curve of the solution-treated Ti–12Mo specimen is shown in Fig. 1. The true strain/true stress curve exhibits a large uniform elongation close to 0.4 as well as a significant strain-hardening rate, much larger than in the case of the conventional titanium alloys [34,35]. The corresponding strain-hardening rate ($d\sigma/d\varepsilon$) also represented in Fig. 1 illustrates a multi-stage deformation process as classically observed in microstructures exhibiting multiple plasticity phenomena [14–16,36]. From the elastic limit to $\varepsilon = 0.1$, there is first a monotonic increase of the strain-hardening rate that reaches a maximum value at ~ 2000 MPa, which is close to the theoretical limit of such an alloy (E/50) [27,32]. Three stages of

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