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Deformation-induced grain refinement to submicron or nanoscale has been achieved upon cold rolling in two metastable β -titanium alloys with different phase stability against $\beta \rightarrow \alpha'$ martensitic transformation. The microstructures after plastic deformation in both alloys, however, were inhomogeneous, consisting of nanoscale/submicron near-equiaxed β grains and elongated submicron β filaments. It is noteworthy that grains were more easily refined to the nanoscale (about 20–50 nm) in the β alloy with low stability in which the martensitic transformation and mechanical twinning were favoured. This indicated that at least two concurrent deformation modes had most likely contributed to the significant grain refinement. Complicated interactions between dislocation slip, martensitic transformation and twinning may have facilitated the refinement by dividing the original coarse grains into nanometre-sized blocks. On the other hand, less pronounced grain refinement to about 50–150 nm was found in the cold-rolled β alloy with high stability in which dislocation slip was the dominant deformation mode.

Introduction

Biocompatible β -titanium alloys containing non-toxic β -stabilizing alloying elements such as Nb and Ta have a great potential for biomedical implant applications due to their low Young's modulus and good formability [1]. However, the metastable characteristic of β phase leads to distinct deformation behavior and complex microstructure depending on its stability against deformation-induced martensitic transformation [2, 3]. The aim of this study is to identify microstructure evolution and grain refinement upon severe plastic deformation.

Experimental

Ti-Nb-Ta-In/Cr β -titanium alloys were prepared by arc-melting a mixture of pure elements under an argon atmosphere followed by suction casting into cylindrical cavities of a water-cooled copper mold. Samples of Φ 6 mm \times 12 mm were cold rolled into 1.8-mm thick plates by a total thickness reduction of 85%. Structural investigations were carried out by X-ray diffraction analysis, scanning electron microscopy and transmission electron microscopy. The room temperature mechanical properties were characterized under uniaxial compression loading at a strain rate of $1.2 \times 10^{-4} \text{ s}^{-1}$.

Results and discussion

With decreasing phase stability, microscopic deformation mechanism of metastable β -titanium alloys varies from slip of dislocations to deformation twinning and/or martensitic transformation (Fig. 1) [4]. Ti-Nb-Ta-In/Cr β alloys were chosen since the Ti-Nb-Ta-In (TNTI) and Ti-Nb-Ta-Cr (TNTC) alloys exhibited low and high stability against $\beta \rightarrow \alpha'$ martensitic transformation, as indicated in Figs. 2 (a) and (b), respectively.

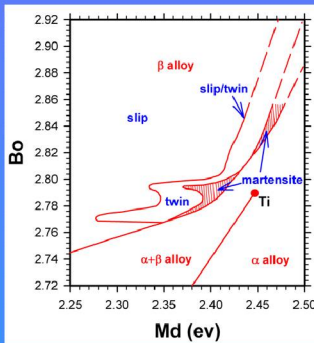


Fig. 1 β phase stability index diagram

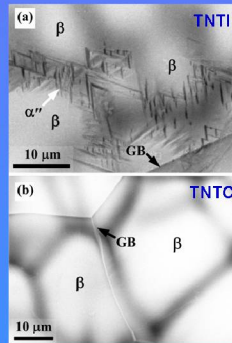


Fig. 2 SEM micrographs of as-cast TNTI and TNTC β alloys

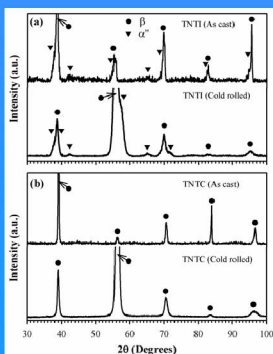


Fig. 3 X-ray diffraction profiles

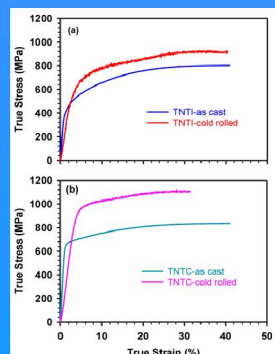


Fig. 4 Compressive true stress-strain curves

XRD analysis (Fig. 3) indicated that both bcc β and orthorhombic α' martensite were present in the as-cast TNTI β alloy, which is in agreement with Fig. 2 (a). Upon cold rolling, a remarkable peak broadening was visible, probably resulting from the contributions of both grain refinement and the imposed deformation strain. From the diffraction peak profile the grain size of the β phase was estimated to be about 24 nm by separating the strain effect. On the other hand, the bcc β phase was the only phase identified in the as-cast and cold-rolled TNTC samples, indicating that no $\beta \rightarrow \alpha'$ martensitic transformation happened upon cold rolling. The cold-rolled TNTC β alloy also exhibited a peak broadening from which the grain size of the β phase was estimated to be about 60 nm.

The compressive true stress-strain curves (Fig. 4) showed that the as-cast and cold-rolled TNTI β alloys exhibited lower yield strength and remarkable strain hardening compared with the TNTC β alloy. In addition, upon cold rolling a remarkable increase in strength by 35% was found for the TNTC β alloy compared with 13% for the TNTI β alloy, and this is believed to be due to various microscopic deformation mechanisms.

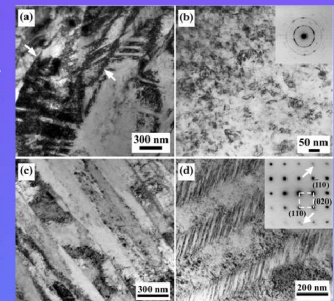


Fig. 5 TEM micrographs at severely deformed zone of compressive deformed as-cast β -Ti alloys. (a) and (b) TNTI; (c) and (d) TNTC

At the severely deformed zone of the compressive deformed as-cast β -Ti alloys, detailed TEM studies revealed inhomogeneous deformation microstructures for both TNTI and TNTC β alloys (Fig. 5). The original equiaxed β dendrites were severely deformed into a submicron filamentary structure (Figs. 5 (a), (c) and (d)). Submicron/nanoscale deformation twins were clearly visible in some of the filamentary structures. In addition, it is noteworthy that in the deformed TNTI alloy a pronounced grain refinement occurred in some of the β filaments to produce nanostructured β grains in the range of 20–50 nm, as evidenced in Fig. 5 (b). The nanostructured β grains exhibited a complicated moiré fringe pattern, which is similar to that of a nanostructured NiTi alloy processed by high pressure torsion (HPT) [5].

TEM investigations on cold rolled TNTI β alloy have also revealed inhomogeneous deformation-induced grain refinement, consisting of nanoscale near-equiaxed β grains and elongated submicron β filamentary structures, as indicated in Figs. 6 (a) and (b). Similar microstructure was observed in the cold rolled TNTC β alloy, however, grain refinement in this alloy was not as significant as that in the TNTI β alloy, implying that multi-deformation mechanisms contributed to the pronounced grain refinement.

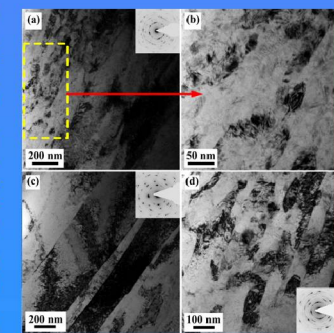


Fig. 6 TEM micrographs for cold rolled β -Ti alloys. (a) and (b) TNTI; (c) and (d) TNTC

Conclusions

Upon severely plastic deformation, inhomogeneous grain refinement was revealed in metastable β -titanium alloys with different phase stability against martensitic transformation. However, grain refinement to nanoscale was more easily achieved in the β alloy with low stability in which the martensitic transformation and mechanical twinning were favored.

References

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