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# Substructure of recovered Ti-23Nb-0.7Ta-2Zr-O alloy

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**Abstract:** Ti-23Nb-0.7Ta-2Zr-O (TNTZO, molar fraction, %) alloy is a newly developed multifunctional $\beta$-type titanium alloy. This alloy was reported to deform via a unique dislocation-free deformation mechanism, and thus its recovery and recrystallization behavior should be further studied and compared with traditional BCC metals. The substructures of the cold-swaged and the annealed TNTZO alloy were investigated by transmission electron microscopy(TEM) to study the recovery of the cold-swaged TNTZO alloy. The results show that no dislocation can be distinguished from the cold-swaged TNTZO alloy due to the heavily-accumulated strains, whereas high density dislocations were observed in the cold-swaged TNTZO alloy after annealing at 800 ℃ for 5 min. During annealing, recovery proceeds through the redistribution and annihilation of the dislocations, which are considered to result from the plastic deformation and the recovery process of the cold-swaged TNTZO alloy is similar to that of ordinary BCC metals.

**Key words:** $\beta$ titanium alloy; substructure; recovery; dislocation

## 1 Introduction

Ti-23Nb-0.7Ta-2Zr-O (TNTZO, molar fraction, %) alloy is a newly developed multifunctional $\beta$-type titanium alloy. This alloy simultaneously possesses ultra-low elastic modulus, ultra-high strength, superplastic nature permitting cold plastic working without work hardening at room temperature, and Invar and Elinvar properties over a wide temperature range [1−2]. The changes in the microstructure of the TNTZO alloy during plastic deformation were investigated previously, which was characterized to be of transgranular macroscopic crystal rotation, giant planar faults with local distorted areas, and nano-order lattice disturbances along specific crystallographic orientation. SAITO et al[1−6] have thought that all these features cannot be explained by traditional dislocation theory, so they propose a specific dislocation-free plastic deformation mechanism. It is well known that recovery and recrystallization are associated with dislocation rearrangement and annihilation in traditional BCC metals deformed via dislocation slip. Recovery involves individual dislocation motions within the existing grains whereas recrystallization involves movements of grain boundaries that eliminate the deformed structure entirely[7]. Therefore, it is thought that recovery and recrystallization behaviors of the TNTZO alloy deformed without any aid of dislocations should be further investigated. In this work, the substructures of the cold-swaged and the recovered TNTZO alloy were investigated by transmission electron microscopy(TEM) to study the recovery of the cold-swaged TNTZO alloy.

## 2 Experimental

The as-received material is the round rods of the TNTZO alloy after cold-swaging with 90% reduction in area. Samples with the dimensions of *d*4 mm×5 mm were carefully cut out from the rods by wire-cutting machine, and then subjected to a heat treatment in argon atmosphere at 800 ℃ for 5 min followed by brine quenching rapidly. Thin foils for transmission electron microscopy(TEM) observations were cut into small disks of 0.3 mm in thickness, mechanically polished to 80 μm in thickness and electropolished by twin jet electro-polishing machine in a 6% perchloric acid+30% butanol +64% methanol solution chilled to about −30 ℃ at 30 V. Then, the foils were examined by the JEOL JEM−2100F Field- Emission-Gun TEM operating at 200 kV. Samples prepared above were cross-section ones, which were perpendicular to the swaging direction of the rods.

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## 3 Results and discussion

Optical microstructure of the 90% cold-swaged TNTZO alloy is a characteristic marble-like structure composed of assemblies of fine filamentary structures [1−2]. Fig.1 shows the TEM image of the so-called marble-like structure of the cold-swaged TNTZO alloy, in which large amounts of deformation bands are observed. The dark contrast along the boundary results from the heavily-accumulated strains induced by intense deformation. No single dislocation or dislocation pile-ups could be distinguished from the deformation bands of the cold-swaged TNTZO alloy due to the heavily-accumulated strains.

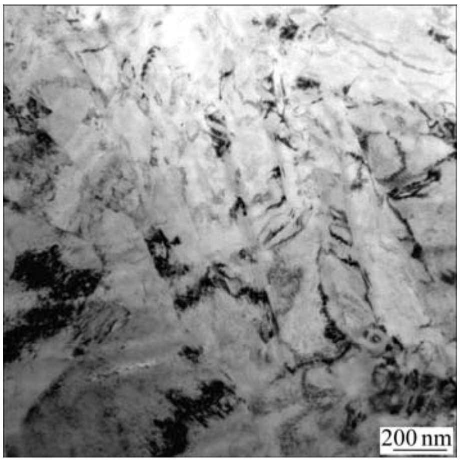


**Fig.1** TEM image of cold swaged TNTZO alloy

After annealing at 800 ℃ for 5 min, changes of microstructure occur in the cold-swaged TNTZO alloy, which is characterized to be of large amount of dislocations and subgrains, indicating recovery is progressing, as shown in Fig.2. Fig.2(a) shows the initial stage of recovery of the cold-swaged TNTZO alloy. It can be clearly seen that subgrains forms in the interior of the deformation bands. A cell with low stored energy is being locally created by coalescence (cell *a*). The dark contrast along the boundary is broadened compared with that in the cold-swaged TNTZO alloy. The deformed structure is still observed in the alloy. Fig.2(b) shows many subgrains that do not invade the adjacent band but grow inside its own band. There are a large number of dislocations accumulated in the unrecovered deformation bands. Fig.2(c) shows a wavy low angle grain boundary, where a high density of dislocations can be found and a high strain can be accumulated in the vicinity of the low angle boundary.

Dislocation networks and arrays are also observed in the cold-swaged TNTZO alloy after annealing at 800 ℃ for 5 min, indicating occurrence of the motion of dislocations, as shown in Fig.3. It can be seen that

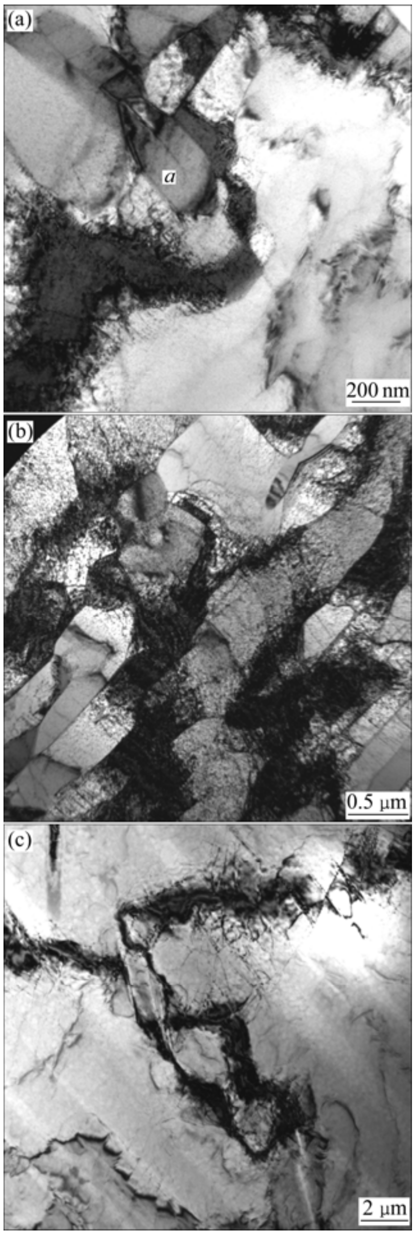


**Fig.2** TEM images of cold-swaged TNTZO alloy annealed at 800 ℃ for 5 min

polygonization occurs in the recovery process of the cold-swaged TNTZO alloy. Dislocation boundaries are formed normally to the primary slip planes and dislocation-free regions are separated from one another by dislocation boundaries.

Recently detailed studies performed by KURAMOTO et al[8] and GUTKIN et al[5] on the plastic deformation of the multifunctional $\beta$ titanium alloys have shown that planar nanoscopic areas of local shear are typical elements of the defect structure of the deformed alloys, which can be effectively modeled as dipoles of non-conventional partial dislocations with arbitrary, non-quantized Burgers vectors. However, in the present study, a large number of dislocations are easily observed in the partially annealed TNTZO alloy. It can

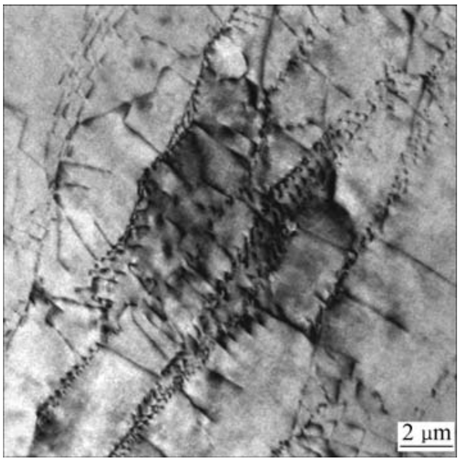


**Fig 3** Bright-field TEM image showing general feature of dislocation structure in cold-swaged TNTZO alloy annealed at 800 ℃ for 5 min

be deduced that these dislocations result from the heavy deformation during the cold-swaging, although no single dislocation or dislocation pile-ups could be distinguished from the deformation bands due to the heavily-accumulated strains. This is contrary to the statement of no dislocation activity during deformation proposed by SAITO et al[1−2].

TEM observations indicate that recovery occurs when the cold-swaged TNTZO alloy is annealed at 800 ℃ for 5 min. Recovery of the cold-swaged TNTZO alloy proceeds through the agglomeration and the annihilation of dislocations. During annealing, excess edge dislocations of the same sign, which are driven by elastic interaction, slip on the planes and then climb to form new tilt boundaries normal to the slip plane and possessing no long-range stress fields. A typical feature of the dislocation sub-boundaries formed in this way is their low curvature and therefore low mobility. The regions of the crystals separated by these low-angle boundaries are free of dislocations and represent blocks or polygons. This progress of redistribution of dislocations results in the formation of subgrains in the cold-swaged TNTZO alloy. All these features of the sub-structure observed in the cold-swaged TNTZO alloy after annealing at 800 ℃ for 5 min are similar to those in ordinary BCC metals in the recovered state.

In addition, orientation inspection by EBSD shows that the cold-swaged TNTZO alloy presents a predominant 〈110〉 fiber texture in the axial direction [9]. The formation of this tensile texture is well established for BCC metals. It is also predicted for materials with 〈111〉 slip systems by polycrystal plasticity simulations based on plasticity models such as Taylor-Bishop-Hill[10]. All these results seem to be in disagreement with dislocation-free deformation mechanism proposed by SAITO et al[1−2] and GUTKIN et al[5]. So it seems that deformation of the TNTZO alloy occurs via the traditional dislocation glide on slip systems, rather than the non-crystallographic mechanism.

## 4 Conclusions

1) A large number of deformation bands are observed in the cold-swaged TNTZO alloy. No single dislocation or dislocation pile-ups can be distinguished from the deformation bands of the cold-swaged TNTZO alloy due to the heavily- accumulated strains.

2) High density dislocations occur in the cold-swaged TNTZO alloy after annealing at 800 ℃ for 5 min. Recovery proceeds through the redistribution and annihilation of the dislocations during annealing, and the recovery process of the cold-swaged TNTZO alloy is similar to that of ordinary BCC metals.

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