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EFFECT OF GRAIN ORIENTATION ON THE DEVELOPMENT OF DISLOCATION SUBSTRUCTURES DURING COLD DEFORMATION OF PURE TANTALUM. LINK WITH STATIC RECRYSTALLIZATION AND RECOVERY.

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ABSTRACT- Recrystallization and recovery of pure polycrystalline tantalum are highly influenced by the intragranular dislocation structure developed during plastic deformation. A tantalum oligocrystal has been deformed by compression at room temperature. The resulting intragranular dislocation structures have been characterized using a FEG scanning electron microscope coupled with an EBSD system. Based on these experimental observations and crystal plasticity simulations, the development of dislocation substructures is related to the crystallographic stability of grain orientations.

INTRODUCTION: The annealing behavior of polycrystalline tantalum is directly linked to the microstructure developed during the deformation stage as shown by Sandim et al. [2005]. Depending on their initial orientation, grains behave differently and generate specific substructures. In the early study done by Vandermeer and Snyder [1979], tantalum single crystals with three different orientations along the α -fiber have been deformed by cold rolling ($\epsilon_{eq} = 1.6$) and subsequently annealed. Some grains developed a cell substructure whereas others exhibited a uniform dislocation density. Recrystallization occurred in grains with a well-defined cell substructure. Linking Vandermeer experimental observations to our crystal plasticity simulation results the development of cell substructure is related to the crystallographic stability of grain orientations during deformation, as already noticed by Kang [2007] in an IF steel. Cellular substructures develop in the unstable-orientation grains. The aim of this study is to relate the development of the intragranular structures to the plastic behavior (as characterized by crystal plasticity simulations), by observing the deformation microstructure of a tantalum oligocrystal of known initial grain orientations. In such an experiment both the orientation dependence of the substructure development and the influence of the grain boundaries and triple junctions can be assessed.

PROCEDURES, RESULTS AND DISCUSSION: A tantalum plate with large columnar grains has been cut to obtain a parallelepiped (28.2 mm x 5.9 mm x 5 mm)

containing 6 grains (Fig. 1). This parallelepiped is defined as the oligocrystal. The macroscopic frame used for defining grain orientations and the loading direction are also shown on Fig.1. Prior to deformation, all six sides of the sample have been polished to remove the hardened layer induced by cutting and to reduce the friction between the sample and the compression tools. The sample was compressed in the Z direction up to a true strain of 0.5 at an average strain rate of 0.01 s^{-1} . An additional Teflon layer was placed between the sample and the tool to reduce the friction furthermore. The microstructure was characterized before and after deformation using a FEG Scanning Electron Microscope (FEG-SEM) equipped with a fast Bruker EBSD system. The six crystal orientations in the macroscopic frame XYZ defined on Fig.1 are given in Table 1, in the form of sets of Euler angles defined according to Bunge's convention.

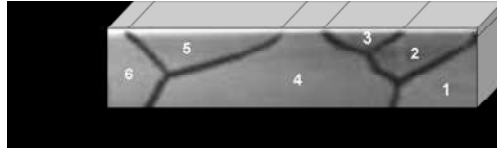


Fig. 1 Tantalum oligocrystal (with Grain ID and reference frame)

A Taylor-type crystal plasticity code was used to simulate the compression test for each crystal orientation present in the initial oligocrystal. No interactions between neighboring grains were considered. The usual 48 slip systems of the BCC structure were used with the same Critical Resolved Shear Stress (CRSS) for each. In these simulations, the grains are free to rotate, so that the grain orientation is changing during deformation. In the present work, the crystallographic stability of a grain orientation is defined from the misorientation angle between the initial and final orientations: when the angle exceeds a critical value of 5° , the orientation is considered as unstable.

Table 1: Grain Orientations, Stability And Intragranular Misorientations

Grain ID	Initial orientation			Stability	Initial KAM ($^\circ$)		Deformed KAM ($^\circ$)	
	φ_1	Φ	φ_2		average	StD	average	StD
1	77	43	304	Unstable	0.45	0.14	0.94	0.38
2	8	4	344	Stable	0.38	0.14	0.54	0.31
3	276	36	92	Unstable	0.5	0.15	1.41	0.69
4	228	52	139	Stable	0.71	0.26	1.13	0.45
5	18	40	354	Unstable	0.56	0.18	0.45	0.29
6	301	35	49	Unstable	0.5	0.15	0.71	0.32

The simulations reveal that both stable and unstable grain orientations are present in the oligocrystal (Table 1). The two cases of grain 1 and 2 are discussed here: grain 2 is stable, and grain 1 is unstable. An EBSD analysis (map size: $115 \mu\text{m} \times 85 \mu\text{m}$, step size: $0.29 \mu\text{m}$) was carried out in each grain far from grain boundaries to limit grain interaction effects. From EBSD data, the intragranular misorientations have been characterized using the Kernel Average Misorientation (KAM) parameter, as defined in

the OIM Analysis software. A fixed Kernel radius of $0.87\ \mu\text{m}$ was used. Table 1 gives the average value and the standard deviation of this parameter for each grain before and after deformation. Before deformation, the intragranular misorientations distributions are almost the same for all grains. The intragranular orientation spread is in the range of the EBSD accuracy considering the acquisition settings, i.e. $0.3\text{--}0.5^\circ$. For the stable orientation (grain 2), deformation induces only few changes: the histogram shifted to slightly higher values with a distribution somewhat larger. In the case of the unstable grain 1, the distribution completely shifts to a higher range of intragranular misorientations. A substructure starts forming, as revealed by plotting the Low Angle Boundaries (LAB) in the EBSD map of Fig. 2.

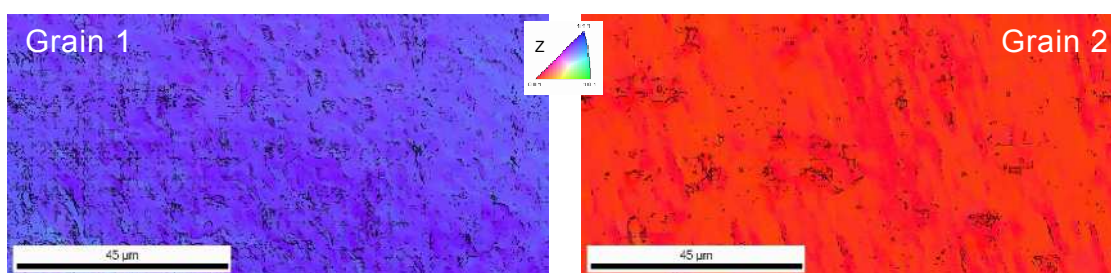


Fig. 2 Orientation map inside grain 1 and grain 2 showing LAB (1 to 5° misorientation angle in black).

The tendency to form substructures can be modified in the vicinity of grain boundaries and triple junctions. EBSD has therefore also been carried out in such areas. Depending on the nature of the boundary and on the grain orientations, the fragmentation process could be either totally different from the in-grain regions, or intensified by the presence of a grain boundary or a triple junction.

CONCLUSIONS: Grains with a crystallographic orientation which is unstable during deformation are more likely to develop dislocations substructures. Presence of grain boundaries and triple junctions modify or intensify this development depending on the nature of the grain boundaries and on the grain orientations. Further experiments increasing the strain and performing subsequent annealing treatments will be carried out to follow the substructure development and the related recrystallization mechanisms.

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