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Generation of extreme grain aspect ratios in severely deformed tantalum at elevated temperatures

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ABSTRACT

Severe plastic deformation effectively produces nanostructured metals. During deformation these structures undergo repetitive dynamic recovery processes, limiting refinement and aspect ratio. Processing tantalum at elevated temperatures by high pressure torsion allows the synthesis of structures with extreme grain aspect ratios of almost ten. Although the overall restoration processes increase with deformation temperature, reflected in enlarged lamellae thicknesses, they are not effective enough to reduce the lamella length at elevated temperatures. Our results indicate that triple junction motion is impeded below a certain temperature while the efficiency of deformation-induced boundary migration diminishes with temperature, allowing to generate such elongated structures.

It is well established that severe plastic deformation of metals leads to extensive grain fragmentation and refinement, especially at low homologous temperatures. Of the various Severe Plastic Deformation (SPD) [1,2] techniques high pressure torsion (HPT) [3] is capable of producing ultrafine-grained (UFG) or nanocrystalline (NC) structures without cracking. The enormous grain refinement results from continuous storage and rearrangement of lattice defects into typical patterns like dislocation cells. Generally, with increasing strain the misorientation between the cells increases till the deformation structures consist mainly of high angle grain boundaries (HAGB, >15° misorientation). Further, the cell or grain size decreases with strain and tends to align with respect to the strain field, e.g. during HPT grains tend to elongate along the shear direction [4–7]. Such elongated microstructures provide interesting fracture toughness properties and are key to designing advanced, high-strength, tough materials [8–10]. However, grain size cannot be refined indefinitely and reaches a minimum saturation value after certain strains ($\varepsilon \sim 10$), which depend on the deformation temperature. This state is referred to as steady-state regime [11–14]. Grain boundary migration [15] and triple junction motion [16] lead to grain fragmentation or removal of thin, lamellar grains, and enable the structure to be in dynamic equilibrium with the deformation-induced refinement of the grains. Interestingly, such processes are not restricted to high strain regimes typically reached by SPD. Net removal of boundaries already commences above strains of $\varepsilon \sim 1$ [17] and a closer look on such low-strain structures suggest that similar processes are active. The movement of both, grain boundaries and triple junctions, are expected to be thermally activated processes and consequently occur at higher rates when the deformation temperature is increased. This is in line with observations of decreasing aspect ratios and the development of equiaxed structures at elevated deformation temperatures for a variety of materials [5,13,18,19]. Therefore, a significant reduction of the deformation temperature should allow synthesizing structures with pronounced aspect ratios. Quite on the contrary the present study shows an increase of the steady-state grain aspect ratio for tantalum when the deformation temperature is increased.

Within this study, quasi-constrained HPT was used to synthesize UFG or NC structures. Disks of technically pure (99.95%) tantalum from Plansee Austria with diameter of 8 mm and height of 0.8 mm were processed at different temperatures, ranging from 77 K to 873 K, under an applied pressure of 7.8 GPa for 10 revolutions with a constant rotational speed of 0.2 rot/min. This results in an equivalent von Mises strain of $\varepsilon_{eq} = 136$ at a radius of $r = 3$ mm, which is more than sufficient to achieve the steady state structure. Further details to the set-up used for heating and cooling may be found elsewhere [13]. Samples processed at elevated temperatures were immediately water quenched after deformation to avoid structural modifications resulting from static annealing. Additional isothermal annealing treatments on deformed samples showed no significant structure change for the deformation temperature range studied. Microstructure and microtexture of the different samples were recorded with electron backscatter diffraction (EBSD) using a Zeiss LEO 1525 field emission scanning electron microscope. A standard orientation imaging micrograph software was used for analysis of grain size and microtexture. All EBSD scans were obtained at a radius of $r = 3$ mm in radial (RAD) viewing direction, where the HPT deformed structure showed the highest aspect ratios. In addition,

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Table 1
Overview of the minor and major grain length (area weigthed), the resulting aspect ratio defined as the ratio of major to minor grain axis, microhardness *H*, and the fractions of low (2–15° misorientation) and high (>15° misorientation) angle grain boundaries of the various tantalum samples processed at different deformation temperatures T_{def}.

T_{def} [K]	T_{def}/T_m [−]	Major axis [nm]	Minor axis [nm]	Aspect ratio [−]	H [GPa]	LAGB/HAGB [%]
77	0.02	320	72	4.4	4.72	21/79
298	0.09	520	107	4.9	4.35	23/77
473	0.14	853	139	6.1	3.66	18/82
573	0.17	1238	153	8.1[a]	3.63	16/84
673	0.20	1375	160	8.6	3.42	17/83
773	0.23	1051	191	5.5	3.18	16/84
873	0.27	1333	221	6.0	3.00	17/83

[a] The aspect ratio of 8.1 is just the average value of the inhomogeneous structure. Taking into account only the region of heavily elongated grains, the average aspect ratio would be 10.7

EBSD scans were obtained in the tangential (TAN) direction at the same radius to account for the 3D nature of the grain structure. About 2000 grains were analyzed for each condition. For analysis, only data points with a confidence index larger than 0.05 were considered and a critical misorientation angle of 15° was set for identification of a grain. Additional TEM images were taken for two specific samples along the RAD direction to measure dihedral angles. Samples were prepared by conventional grinding and dimpling followed by ion polishing. The samples were analyzed in a Philips CM12 microscope. Microhardness measurements were carried out on all samples to correlate structural and mechanical properties with respect to the processing temperature. Minimum and maximum grain dimensions, the resulting aspect ratio and the microhardness of the various samples are summarized in Table 1.

Table 1 shows that the hardness values decreased steadily for increased HPT temperatures, in accordance with earlier studies where the minimum grain dimensions were reported to increase for higher deformation temperatures [13]. Moreover, the fractions of low and high angle grain boundaries (LAGB and HAGB) remained relatively constant for all samples, with just a slight increase of the LAGB fraction for lower deformation temperatures. This could be explained with an increase in the probability for dislocation storage at lower processing temperatures. Fig. 1 shows Inverse Pole Figure (IPF) images of the resulting microstructure for selected deformation temperatures. Images of representative microstructures for all deformation temperatures can be found in the supplementary section, Supplementary Fig. 1. Contrary to expectations, the aspect ratio, λ, continuously increased up to processing temperatures of 673 K before it decreased again (see Fig. 1 and Table 1). Samples deformed at 573 K and 673 K showed extreme aspect ratios with λ > 8 which is almost twice the value of λ ~ 4.5–4.8 for samples deformed at 77 K and 298 K. A closer look on the samples deformed at 573 K and 673 K show several exceptionally long grains of 4 μm and a thickness of only about 100 nm. Such extreme aspect ratios develop by a stronger increase of the major (long) axis compared to the minor (short) axis, Table 1. While the minimum grain axis is growing only slowly with temperature, the long grain axis increases by a factor of four for an increase in the deformation temperature from 77 K to 673 K. For processing temperatures larger than 673 K, the aspect ratio decreased again, as the long grain axis becomes smaller, while the minor axis increased further. To account for the 3D nature of the microstructures, the samples were additionally analyzed in the tangential direction, Supplementary Fig. 2. Interestingly, similar to the RAD direction, also in the TAN sections the long grain axis is not getting smaller at elevated processing temperatures. Although the changes are not as pronounced as in RAD direction, even in this direction a slight increase of the aspect ratio can be measured.

Further, tantalum samples deformed at RT were isothermally annealed up to 973 K for 30 min. Annealing of the samples induced a slight increase in hardness from 4.29 GPa to 4.43 GPa after 673 K annealing which could be attributed to dislocation source hardening [20], Fig. 2. Up to this temperature the microstructure remained stable, suggesting that dislocation annihilation dominates. For higher annealing temperatures the hardness decreased continuously until a pronounced drop to 1.92 GPa at 973 K. The EBSD measurements revealed first signature of globular grains at 773 K, shown by the circled regions in Fig. 2, and the aspect ratio started to decrease. For samples annealed at 873 K almost the whole structure consists of larger and globular grains of about 300–500 nm diameter, while only a few thinner lamellae with reduced aspect ratios were left. Annealing at 973 K led to a fully recrystallized material, with globular grains of several micrometers in size and different texture components, compare Figs. 2 and 3. The structural changes occurring at 773 K and 873 K are similar to the observations of enhanced migration of triple junctions during annealing of heavily cold rolled aluminium [21,22].

The results of increasing aspect ratios for tantalum deformed at elevated temperatures are surprising, as they are contradictory with earlier results [13]. Recent experimental studies demonstrate the importance of grain boundary (GB) migration [15] and triple junction (TJ) motion [16] on structural restoration. Although such processes are thought to be mainly mechanically driven at low temperatures, detailed experiments have shown that thermal activation [23] can assist them, leading to the expected reduction of the aspect ratio at elevated temperatures. The opposite behaviour observed in this study could be a result of different deformation mechanisms operating over the wide deformation temperature regime or a suppression of restoration mechanisms. A comparison of the ODF sections of the deformed samples shows the

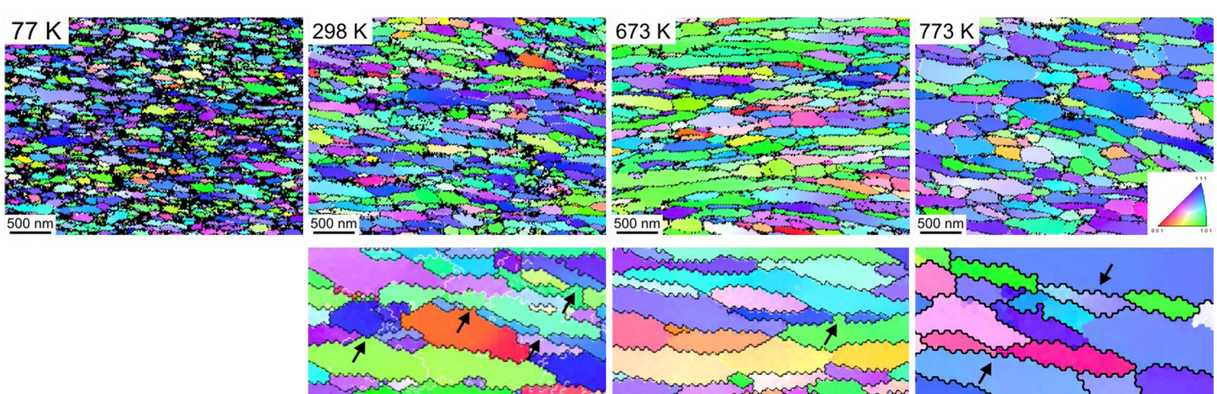

Fig. 1. Representative IPF colour maps showing the microstructure of the tantalum samples after HPT deformation at selected deformation temperatures. All maps were recorded in radial direction at a radius of r = 3 mm. For temperatures up to 673 K, the grain structure becomes increasingly elongated. Details (not to scale) show typical grain fragmentation events caused by migration of boundaries.