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Features of formation of nanocrystalline state in internal-oxidized V-Cr-Zr-W and V-Mo-Zr system alloys during deformation by torsion under pressure

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Abstract. The results of investigation of features of nanostructural state formed during deformation by torsion under pressure in high-strength vanadium V-Cr-Zr-W and V-Mo-Zr systems alloys are presented. It was found that after deformation at number of revolutions $N = 1$, samples are characterized by high anisotropy of defect and grain structure. Inside grains, limited by high-angle boundaries, the formation of two-level structure states was revealed: fragmentation of the above grains on nanofragments from 5 to 20 nm in size with a dipole nature of low-angle misorientations and high (hundreds of degrees per micron) elastic curvature of crystal lattice. Formation of the above structural states leads to a 3-fold increase in microhardness values. Further increase in deformation degree leads to fracture of samples of vanadium alloy V-Mo-Zr with a high volumetric content of fine-disperse oxide phase. At the same time V-Cr-Zr-W-system alloy with a lower concentration of Zr and, as a result, a lower volume fraction of fine particles remains ductile.

1. Introduction

Investigation of the specificity of nanostructural states formation in metallic materials of different classes under severe plastic deformation today is still one of the rapidly developing areas of modern physical materials science. According to modern concepts, the behavior of nanostructural metallic materials in the fields of external influences and their physical and mechanical properties are largely determined by features of nonequilibrium structural states formed inside and regularities of their evolution in the process of generation and propagation of defects at different scale levels [1 – 5].

Analysis of published data shows that the study of such issues is advantageously carried out on pure FCC metals and alloys. Much smaller number of experimental works is devoted to studying of features of formation of nanostructural states in metals and alloys with BCC lattice during large plastic deformations. Meanwhile BCC metals are the basis for creation of a broad class of structural materials for use in extreme conditions (high temperatures and pressures, aggressive media, etc.). Due to high chemical activity of these materials relative to interstitial impurities, their alloys often become

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heterophase [6] with opportunities for implementation of solid-solution, dispersion, precipitation, etc. types of strengthening. So, one of the actual trends is the investigation of features of formation of nanostructural states in metallic materials with BCC lattice with different strengthening types.

In current work the investigation of features of nanostructural states and parameters of microhardness of high-strength internal-oxidized vanadium V-Cr-Zr-W and V-Mo-Zr systems alloys with different volume fraction of fine-disperse second phase particles after severe plastic deformation by torsion on Bridgman anvils was carried out.

2. Experimental materials and procedures

In this work vanadium alloys of V-Cr-Zr-W (V - 1.17 Zr - 8.75 Cr - 0.14 W - 0.01 C - 0.02 O - 0.01 N) and V-Mo-Zr (V - 9 Mo - 1.5 Zr - 0.07 C) (wt. %) were used. Before plastic deformation in Bridgman anvils V-Cr-Zr-W alloy was processed by thermo-mechanical treatment (TMT) by scheme [7] which provides the dispersion of initial coarse phase. Further, oxygen doping during chemical-heat treatment (CHT) [8, 9] was conducted on alloys V-Cr-Zr-W and V-Zr-Mo to achieve oxygen concentration of 1.34 and 2.7 (at. %) respectively. Then one-hour annealing was conducted at 1400 °C. Deformation of disc samples with thickness $h = 0.2$ mm and diameter of 8 mm was produced by torsion under high pressure (7 GPa) at room temperature and the number of revolutions of the anvil $N = 1$. Thickness of specimens after deformation was 0.15 mm. Values of shear ($\gamma \approx 2\pi NR/h$) and true logarithmic ($e \approx \ln\gamma$) deformation depending on the distance from the center of the deformable disk (R) changes from $\gamma \approx 16$, $e \approx 2.8$ at $R = 0.5$ mm to $\gamma \approx 110$, $e \approx 4.7$ at $R = 3.5$ mm.

Structural studies were conducted by transmission electron microscope Philips CM-30 STEM-TWIN (300 kV). Thin foils were prepared in sections normal to anvils plane. For their preparation, copper layer was electrolytically precipitated on samples after deformation. Flat samples in the above sections were cut with electrospark machine and mechanically thinned to a thickness of 100 microns. Further thinning was carried out by bilateral sputtering by argon ions at accelerating voltage of 5 kV.

Methods of analysis of high continuous and discrete misorientations [10, 11] were used to study the characteristics of defect structure of volume and boundaries of grains. Microhardness (H_μ) was measured by Vickers method on «Neophot 21», with a load of 0.5 N and 15 seconds of exposure in section perpendicular to anvils plane (AP) at different distances from torsion axis (TA).

3. Results and discussion

Figure 1 shows the bright-field (figure 1 a) and dark-field (figure 1 b) electron microscopic images of V-Cr-Zr-W alloy microstructure after CHT and subsequent deformation on Bridgman anvils at $N = 1$.

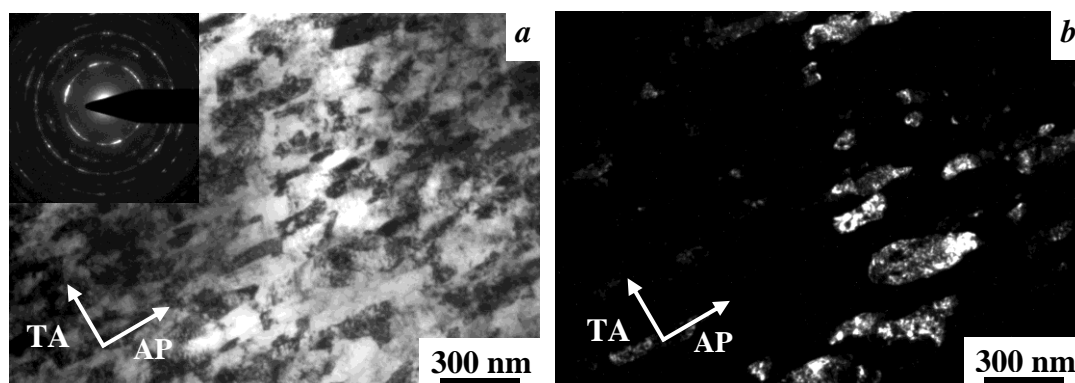


Figure 1. V-Cr-Zr-W alloy microstructure ($N = 1$, $e \approx 4.7$). Bright-field (a) and dark-field (b) images taken in cross section perpendicular to anvils plane.

Formation of similar structural state was also found in V-Mo-Zr-system alloy after such treatment (figure 2 a). The analysis revealed that characteristic dimensions of crystallites of both alloys in directions parallel to the anvils plane are in the range from 150 to 600 nm, in rare cases reaching 1

micron or more in the alloy V-Mo-Zr, whereas in perpendicular to them (parallel to the torsion axis) crystallites sizes does not exceed 100 nm (figure 1 and figure 2 a). Thus, in the studied alloys the formation of structural state with pronounced grain structure anisotropy is observed.

In addition to anisotropy of grain sizes, high anisotropy of discrete low-angle and high-angle misorientations was revealed that determine [10, 11] characteristics of grain and subgrain structure, as well as continuous misorientations that characterize parameters of dislocation-disclination substructure of volume and boundaries of crystallites. A schematic representation of anisotropic structural state is presented on figure 2 b. It was found that values of crystal lattice curvature tensor components of χ_{31} and χ_{21} type reach 20 – 30 degrees/ μm (with maximum up to 40 degrees/ μm) which two or three times exceed values of χ_{13} and χ_{23} type components (5 – 15 degrees/ μm). Thus, the intensity of all these misorientation types in the directions lying in anvils plane greatly exceeds crystal lattice misorientations in the direction normal to this plane, parallel to the torsion axis (figure 2 b).

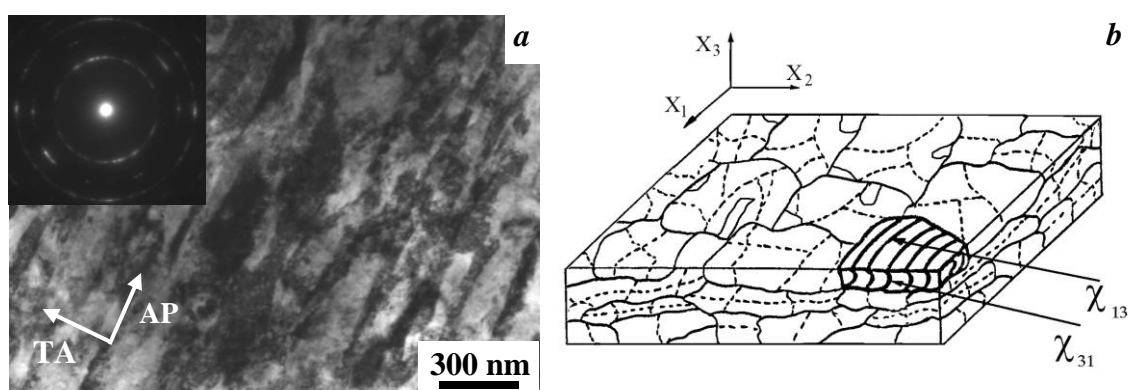


Figure 2. a – Bright-field microstructure image and corresponding microdiffraction of V-Mo-Zr- system alloy ($N = 1$, $e \approx 3$). Section perpendicular to anvils plane. b – Scheme of anisotropic structural state of material after deformation by torsion under pressure [11].

Previous, similar structural features were detected in V-Ti-Cr-system alloy after deformation by torsion under pressure [11], and it was shown that observed microstructure anisotropy is a consequence of high anisotropy of fields of displacements and rotations due to scheme of the method.

An important feature specific to studied in this paper vanadium alloys of V-Zr-Cr-W and V-Mo-Zr systems is the fact that after deformation by torsion at $N = 1$ in both central and peripheral parts of specimens the formation of two-level structural states is observed. Figure 3 is an example of electron microscopic dark-field images obtained at different goniometer tilt angles.

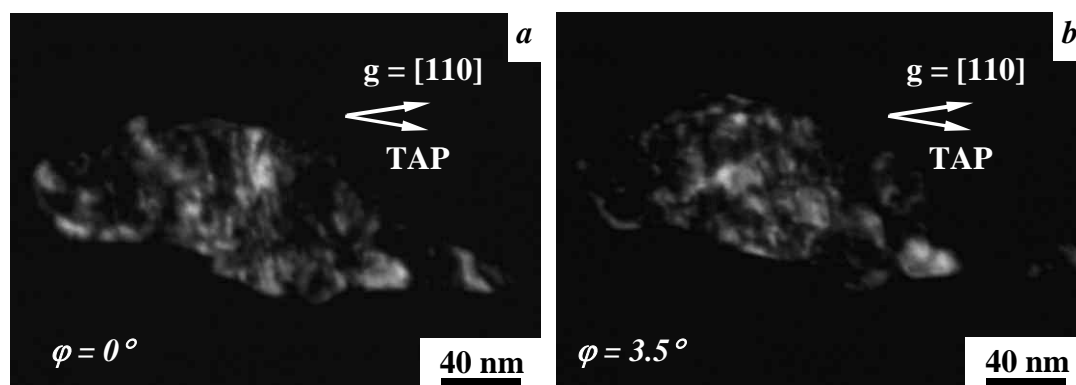


Figure 3. Example of dark-field analysis of defect structure inside nanocrystal of V-Mo-Zr- system alloy ($N = 1$, $e \approx 3$). Section perpendicular to anvils planes. φ - specimen tilt angle in goniometer. TAP – goniometer tilt axis projection.

This analysis revealed that characteristic dimensions of nanofragments are 5 – 20 nm and values of crystal lattice curvature reaches up to several tens and sometimes even hundreds of degrees/ μm .

Formation of a similar two-level structural state in V-4Ti-4Cr-system alloy after deformation by torsion at $N = 1$ was detected only at peripheral area, in which $e \approx 4.7$, and filling of the entire volume of disk sample by structural state of this type was observed only when $N = 5$ [12]. Figure 4 a is a diagram of defect substructure with a dipole character of misorientations, reflecting the main features of the two-level structural state.

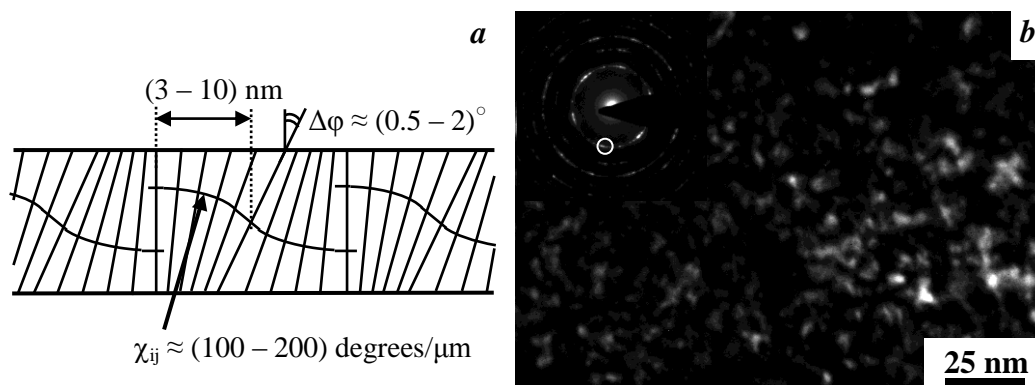


Figure 4. a – scheme of the defect substructure with a dipole character of misorientations [12]. b – dark-field image of the fine-disperse phase of Zr-(O-N-C)-based complex oxycarbonitride in V-Cr-Zr-W alloy ($N = 3$, $e \approx 4.7$)

In addition to differences in values of plastic deformation at which begins the formation of two-level structures, vanadium alloys with internal oxidation are characterized by more intense strengthening, which manifests in microhardness measurements (table 1), before the deformation values were 1.5 GPa for all these alloys.

Table 1. Values of vanadium alloys microhardness at $N = 1$ subject to distance from center of disks (R), H_{μ} , GPa

R , mm	V-Cr-Zr-W	V-Mo-Zr	V-Ti-Cr [13]
0.5	3.17	3.63	3.21
3.5	4.01	4.17	3.44

Figure 4 b shows an example of dark field electron microscopic image of nanoscale (from 3 to 10 nm) FCC particles in V-Cr-Zr-W alloy. It was found that these particles are Zr-(O-N-C)-based complex oxycarbonitride with a lattice parameter of $\approx 4.28 \text{ \AA}$. An important feature of identification of these particles in BCC alloys is the realization of Bain orientation relationship [14], according to which the microdiffraction pictures reflexes of [200]-type of FCC particles and [110]-type of the BCC matrix coincide or are close enough.

Vanadium alloys used in current work are characterized by different content of the volume fraction (f) of second phase disperse particles. The concentration of Zr in V-Mo-Zr-system alloy allows oxidation with the achievement of a significantly greater f . The results of evaluations conducted on the basis of data of the elemental composition shown that as a result of chemical-heat treatment the maximum values of f , mainly represented by fine particles of ZrO and/or ZrO₂ (figure 4 b), reaches about 0.027 and 0.054 respectively in alloys of V-Cr-Zr-W and V-Mo-Zr systems.

In our opinion, the high density of distributed over the volume Zr-based fine particles, and possibly, a high concentration of oxygen in solid solution, are the main reasons of so intense evolution of microstructure and a sharp changes in H_{μ} . These factors combine to provide a high localized stress concentration at the nanoscale level while reducing the relaxation ability of the material.

It should be noted that a further increase in deformation degree on Bridgman anvils leads to intense embrittlement and destruction of samples of internal-oxidized V-Mo-Zr-system alloy: material crumbled into fine powder. At the same time V-Cr-Zr-W-system alloy, with a smaller volume fraction of fine particles remains plastic and deforms to 5 and more revolutions by torsion under pressure.

Unfortunately, in the framework of this article even rough estimates of contribution of any particular type of strengthening, just as was done for fine-crystalline V-4Ti-4Cr alloy [15], are not possible to be made. The reason is the necessity to integrate a large number of interrelated factors (grain and subgrain size, particle size and density, dislocation density, etc.), which, to date, have not been fully studied in conceptions of strengthening of nanostructural states of heterophase systems.

4. Summary

Comparison of presented data with the results of [13] shows that the volume fraction of fine-disperse second phase particles, apparently influences the intensity of two-level-type structural states formation. The evolution of these states actually determines microhardness of analyzed vanadium alloys, including V-4Ti-4Cr. Furthermore, the maximum values of microhardness of these alloys are achieved when the whole bulk of material is presented by two-level-type state at deformation degree typical for each alloy.

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