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Features of Change of V–4Ti–4Cr Alloy Hardness during Microstructure Evolution under Severe Plastic Deformation

Ivan A. Ditenberg[1, 2, 3, a)] and Alexander N. Tyumentsev[1, 2, 3]

[1] *National Research Tomsk State University, Tomsk, 634050 Russia*
[2] *Institute of Strength Physics and Materials Science SB RAS, Tomsk, 634055 Russia*
[3] *Siberian Physical-Technical Institute, Tomsk, 634050 Russia*

[a)] Corresponding author: ditenberg_i@mail.ru

Abstract. Features of changes of microhardness and nanohardness of V–4Ti–4Cr alloy at different stages of microstructure transformation during severe plastic deformations by torsion under pressure are presented. Microstructure features and mechanisms of its transformation affecting the hardness of studied alloy are discussed. Local temperature increase and activization of relaxation processes of nonequilibrium nanostructure states with high (hundreds of degrees/μm) elastic curvature of the crystal lattice are considered as main factors that define nonmonotonic character of changes in the microhardness at the stage of formation and evolution of the two-level structural states. In alloy under study at a value of true logarithmic deformation $e \geq 6.6$ the formation of areas consisting of nanocrystals several nanometers in size with a high density of large-angle boundaries and elastic curvature of the crystal lattice hundreds of degrees/μm was found. Hardness of the material ($H_{nano} \approx E/16$) differs little from its theoretical (limit) hardness.

INTRODUCTION

In [1, 2] was shown that at severe plastic deformation (SPD) of V–4Ti–4Cr alloy by torsion under pressure at room temperature several stages of microstructure transformation are observed. These stages are characterized by formation of certain types of structural states that vary in size of grains and subgrains, crystal lattice curvature values, level of local internal stresses and their gradients.

This paper presents the results of studying of features of changes of microhardness and nanohardness of V–4Ti–4Cr alloy during microstructure evolution under SPD.

EXPERIMENTAL MATERIALS AND PROCEDURES

Samples of vanadium alloy V–4Ti–4Cr (V–4.36Cr–4.21Ti–0.013C–0.011N–0.02O, wt %) in the form of disks, $h \approx 0.2$ mm of thick and 8 mm in diameter, were deformed by torsion under pressure of ~7 GPa at room temperature and number of revolutions of the anvil N = 1, 3 and 5. Specimen thickness after deformation was 0.15 mm. Estimates of values of shear ($\gamma = 2\pi NR/h$) and true logarithmic ($e = \ln\gamma$) deformation are shown in Table 1 together with microhardness values. Investigation of microstructure after various SPD stages was performed on transmission electron microscope Philips CM-30 TWIN (300 kV). Methods of preparation of thin foils are described in detail in [1, 2].

Microhardness (H_μ) was determined with the use of a diamond Vickers pyramid on Neophot 21 with loads of 0.102 and 0.051 kg and 15 s of exposure. Microhardness measurements were carried out in sections, parallel and perpendicular to the anvil planes, at different distances from the axis of torsion. Nanohardness was measured by Oliver-Pharr method by prints of Berkovich pyramid at nanoindenter Nano Test 600 with a load of 0.002 kg. Measurements were made in cross-section perpendicular to the plane of the anvil on the samples prepared by the above procedure. Depending on sample thickness, 3 lines of 20–30 prints were applied. Distance between lines and prints was 10 μm.

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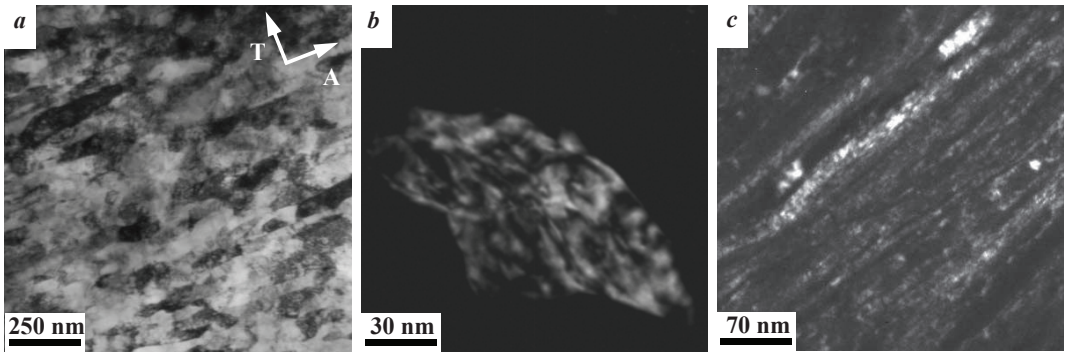

FIGURE 1. Brightfield (a) and darkfield (b, c) electron microscopic images of basic structure state types of vanadium alloy V–4Ti–4Cr at different stages of plastic deformation by torsion in Bridgman anvils at room temperature [1]

RESULTS AND DISCUSSION

In [1] three major phases of V–4Ti–4Cr alloy microstructure transformation under severe plastic deformation by torsion on Bridgman anvils at room temperature were identified.

On the first stage ($e \leq 3$), dislocation-disclinational mechanism of fragmentation and reorientation of crystal lattice provides the formation of submicrocrystalline structure (Fig. 1a). Grain sizes in the direction of the torsion axis (TA) does not exceed ≈100 nm, which is several times smaller than in directions parallel to anvil planes (AP), in which these dimensions are in the range (250–400) nm. The curvature of crystal lattice reaches values of a few tens of degrees/μm.

The second stage ($e \approx 3$–6) is associated with activization of quasi-viscous mechanisms of deformation and reorientation of crystal lattice that leads to the formation of two-level-type structural states (Fig. 1b). In accordance with [1], these states are characterized as nanograins of about 100 nm in size containing nanofragments (up to a few nanometers) with a dipole and multipole misorientation character and unusually high (hundreds of degrees/μm) elastic curvature of crystal lattice.

Third stage ($e \geq 6$) is characterized by development of collective effects in disclination substructure, that lead to group movement of nanodipoles and formation of vortex-type mezobands corresponding nanobands packs and regions of equiaxed nanocrystals from a few nm to several tens of nanometers in size with a dipole character of misorientation (Fig. 1c).

Table 1 shows the microhardness values (H_μ) of the studied alloy subject to degree of plastic deformation, determined by number of revolutions and distance from torsion axis.

TABLE 1. Microhardness and nanohardness of V–4Ti–4Cr alloy after SPD by torsion on Bridgman anvils

N	*R*, mm	γ	*e*	H_μ, GPa	H_μ	H_μ/G
Microhardness						
Initial (before SPD) state				1.5	*E*/85	0.032
1	0.5	21	3.0	3.2	*E*/40	0.068
5	0.5	104	4.6	3.7	*E*/34	0.080
1	3.5	147	5.0	3.4	*E*/37	0.073
5	3.5	735	6.6	4.8	*E*/26	0.102
Nanohardness H_{nano} ($N = 5$, $R \approx 3.5$ mm, $e \approx 6.6$)						
Mean value				4.8	*E*/26	0.102
Local nanobands pack area (Fig. 1c)				7.7	*E*/16	0.163

$G \approx 47$ GPa, $E \approx 127$ GPa [3]

Comparison of structural study results with microhardness values indicates that the formation of submicrocrystalline structure on the first stage provides more than a twofold H_μ increase (from 1.5 to 3.2 GPa) (Table 1).

As mentioned above, the second stage is characterized by the formation of two-level-type nanostructural states. Investigation of areas at different distances from the axis of torsion after deformation at different numbers of revolutions determined that with increasing of plastic deformation degree the proportion of crystals with two-level nanostructural states increases [1] and at $e \approx 6$ it becomes a typical state of the internal structure of almost all submicrocrystalls. At the same time, at the background of general trend of microhardness growth in the range of plastic deformation degrees $e \approx 3–5$, non-monotonous H_μ increase is observed (Table 1).

For an explanation of analogical quasi-stationary regime of hardening of pure Ta in [4] it is assumed the possibility of formation and plastic relaxation of nonequilibrium structural states with high values of elastic distortions. In the case of partial disclinations nanodipoles, this are processes of formation of nanodipoles of partial disclinations in the areas of elastic distortions localization and subsequent plastic relaxation involving quasi-viscous mechanisms of mass transfer. Meanwhile, repetition of the above processes is possible.

As shown in [5, 6], due to strong exponential dependence of the vacancies diffusion coefficient from temperature, it is an important parameter that determine the rate of plastic deformation in the conditions of implementation of the above quasi-viscous deformation mode. It is known [7] that in austenitic steel, in conditions of torsion under pressure, temperature may rise by several hundred degrees. Meanwhile, our evaluations showed [5, 6], that in nickel in the range from room temperature to 200°C, increase in deformation temperature on 100°C can lead to increase in local shear rate in nanobands by 3–4 orders of magnitude.

As can be seen from the above results and discussion of metallic materials deformation in Bridgman anvils, during analysis of regularities of formation of microstructure and mechanical properties subject to distance from torsion axis, in addition to plastic deformation degree dependence from this distance, it is necessary to take into account the possibility of formation of temperature gradients, determining the intensity of diffusion-controlled processes of plastic relaxation of forming nanocrystalline structures.

Non-monotonic dependence of microhardness from the degree of deformation is explained, apparently, by temperature influence. However, in this case, in addition to temperature gradients defining a different character of such dependence on the different distances from the center of the sample, it is obviously necessary to take into account the possibility of increasing temperature with increased deformation time.

The maximum values of microhardness of samples of Ta ≈ 5.9 GPa [4], whereas for the alloy V–4Ti–4Cr $H_\mu \approx$ 4.8 GPa (Table 1). Their normalization on the elastic moduli of materials almost eliminates such differences (Ta ≈ $E/32$, V–4Ti–4Cr ≈ $E/26$). Therefore, the above difference in absolute values of microhardness are mainly associated with differences in the values of the elastic moduli of materials.

Apart from the results of microhardness measurements, Table 1 shows the data of nanoindentation, that allows to obtain nanoindenter prints whose dimensions (a few microns) are much less than print sizes of microhardness indenter. These dimensions are comparable to the width of vortex-type mezobands of deformation localization [1] formed in the third step of nanostructuring of V–4Ti–4Cr alloy. Therefore, by using the nanoindentation method it is possible to measure the hardness value in local areas with different types of nanostructured state, including inside the mezobands above.

These mezobands are packs nanobands few nanometers in width with high angle boundaries, fragmented into nanocrystals of similar size in the longitudinal direction [1] Essentially here we manage to reach the limit option of samples microstructure nanostructuring – to obtain a nanocrystalline structure (Fig. 1 c) with the dimensions of the crystallites a few nanometers, a high density of high-angle boundaries and elastic curvature of the crystal lattice hundreds of degrees/μm.

The study showed (Table 1) that the average micro- and nanohardness values of vanadium alloy after deformation $e \approx 6.6$ are the same, but inside mezobands this value increases by more than 1.5 times, reaching values $H_{nano} \approx E/16$. In [8, 9] for mechanical testing by indentation the concept of theoretical (limit) hardness (H_{teor}) was introduced—"the maximum hardness of the material, which can be achieved provided that the stress causing plastic flow in the material under the indenter corresponds to the theoretical shear strength of the material". In [9] it is shown that in metals the "lower" limits of the theoretical hardness is $H_{teor} \approx E/10$. Thus, in deformation localization mezobands it is possible to obtain the nanostructure state in which the hardness of the material differs little from its theoretical (limit) hardness.

For example, in Ta [4], in contrast to the alloy V–4Ti–4Cr [1], there is no stage of the formation of vortex-type nanoband structures associated with collective effects in the disclination substructure, leading to group motion of

nanodipoles of partial disclinations in the fields of non-uniform moment stresses. In our opinion, this is associated with significantly lower homological deformation temperatures in more refractory Ta and, respectively, the lower activity of point defects and cooperative deformation mechanisms associated with them.

Apparently, in vanadium alloys conditions of implementation of quasi-viscous deformation mode by currents of non-equilibrium point defects and their complexes (the density of these defects, the rate of their migration, etc.) are sufficient to realize intensive processes of generation and collective propagation of nanodipoles of partial disclinations, but not sufficient for development of intensive relaxation processes like dynamic recrystallization such as in less refractory copper or nickel [1]. While in more refractory Ta these processes are limited by nanoscale level of relaxation of high elastic lattice curvature within nanobands several nanometers in width and provide formation of two-level nanostructural state with low-angle boundaries of nanobands [4].

SUMMARY

The evolution of microstructure of V–4Ti–4Cr alloy with increasing deformation degree consists in increase in volume fraction of two-level nanostructural states and results in 3-fold microhardness increase of deformed samples with maximum values $H_\mu \approx E/26$. It is assumed that a local temperature increase and activization of relaxation processes of nonequilibrium nanostructure states with high (hundreds of degrees/μm) elastic curvature of the crystal lattice define nonmonotonic character of changes in the microhardness at the stage of formation and evolution of the two-level structural states.

In V–4Ti–4Cr alloy at a value of true logarithmic deformation $e \approx 6.6$ collective effects were found in the system of nanodipoles of partial disclinations with the formation of deformation localization mezobands representing nanobands packs, which are distributed in noncrystallographic directions, forming a pronounced vortex structures up to the formation of loop configurations size up to several microns. Inside mezobands the limit version of nonequilibrium nanocrystalline structure is reached—nanocrystals several nanometers in size with a high density of large-angle boundaries and elastic curvature of the crystal lattice hundreds of degrees/μm. Hardness of the material ($H_{nano} \approx E/16$) differs little from its theoretical (limit) hardness.

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