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Citation: AIP Conference Proceedings **1783**, 020066 (2016); doi: 10.1063/1.4966359
View online: http://dx.doi.org/10.1063/1.4966359
View Table of Contents: http://scitation.aip.org/content/aip/proceeding/aipcp/1783?ver=pdfcov
Published by the AIP Publishing

Influence of Temperature on Microstructure Parameters and Microhardness of Dispersion-Hardened V–Cr–Zr–W Alloy after Deformation by Torsion under Pressure

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Abstract. Study of microstructure transformation and microhardness changes of dispersion-hardened V–Cr–Zr–W alloy after severe plastic deformation by torsion on Bridgman anvils and subsequent heat treatments was conducted. Basic stages of relaxation processes were revealed: at 800°C recovery processes take place and primary recrystallization begins; at 900°C primary recrystallization intensifies; in range of 950–1050°C collective recrystallization processes activate; at 1200°C secondary recrystallization starts. Microhardness measurement and comparison of its values with structural states features were conducted. Strengthening mechanisms and their contribution at various stages of defect substructure relaxation are discussed. It is shown that increase of thermal stability of V–Cr–Zr–W alloy microstructure is a consequence of the formation of high density of thermally stable Zr (O–N–C)-based nanoparticles.

INTRODUCTION

Earlier it was shown [1] that after severe plastic deformation (SPD) by torsion on Bridgman anvils at revolution number (N) equal to 1, the anisotropic submicrocrystalline structural state is formed in the alloy under investigation. High-defect nanostructural state of two-level type was found inside crystallites. These microstructure changes are accompanied by significant increase in microhardness of the material.

In present study the investigation of temperature influence on microstructure parameters and microhardness of dispersion-hardened V–Cr–Zr–W-system alloy after plastic deformation by torsion under pressure was conducted.

EXPERIMENTAL MATERIALS AND PROCEDURES

Alloy V–8.75Cr–1.17Zr–0.14W–0.01C–0.02O–0.01N (wt %) (V–Cr–Zr–W) was used for investigation. Stages of deformation and prior treatments have been reported earlier in [1]. Specimens after deformation by torsion under pressure were annealed for one hour in vacuum at temperatures from 700 up to 1200°C.

Structural investigations were conducted by transmission (Philips CM-30 TWIN, 300 kV) and scanning (FEI Quanta 200 3D, 30 kV) electron microscopy. Methods of dark-field analysis of discrete and continuous misorientations [2, 3] were used during structural characterization.

Microhardness (H_μ) was determined by Vickers method in the cross section perpendicular to anvils plane with the use of Neophot 21 at a load of 0.5 N and 15 s exposure.

Advanced Materials with Hierarchical Structure for New Technologies and Reliable Structures 2016
AIP Conf. Proc. 1783, 020066-1–020066-4; doi: 10.1063/1.4966359
Published by AIP Publishing. 978-0-7354-1445-7/$30.00

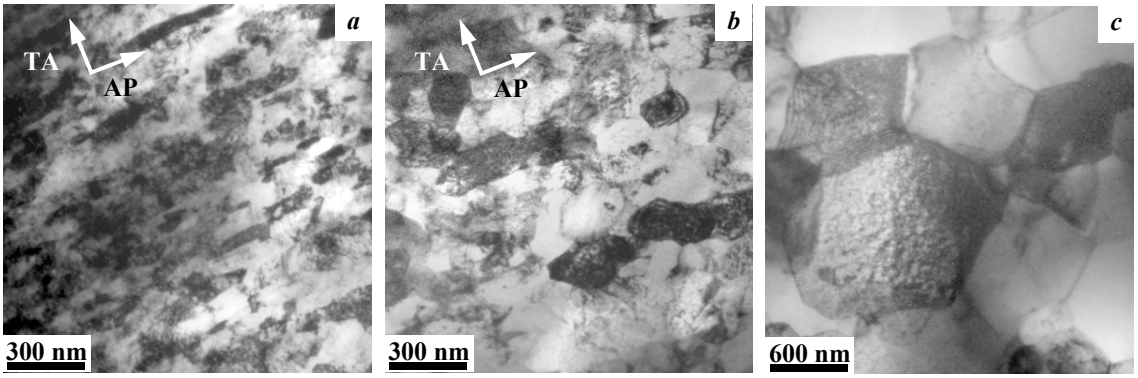

FIGURE 1. Microstructure of V–Cr–Zr–W alloy after deformation by torsion under pressure ($N = 1$) (a) [1] and subsequent annealing at temperatures of 800 (b) and 900°C (c). Transmission electron microscopy

RESULTS AND DISCUSSION

Figure 1a presents a bright field image of the studied alloy microstructure after deformation by torsion under pressure [1]. Anisotropic submicrocrystalline structural state formed in the process of deformation is characterized by grains with dimensions in directions parallel to anvils plane (AP) within 70–700 nm, while in the torsion axis (TA) direction they constitute 50–200 nm. In accordance with [2], the anisotropy of microstructure is the result of high anisotropy of fields of displacements and rotations during torsion under pressure. Inside submicron grains a two-level state [3] was found: nanofragments (5–20 nm) separated by low-angle (0.5°–2°) boundaries with elastic curvature of crystal lattice reaching several hundreds degrees per micron.

Heterophase structure of V–Cr–Zr–W alloy is mainly represented by complex Zr (O–N–C)-based oxycarbonitride particles [1], whose nanoscale (3 to 10 nm) fraction provides high effects of dispersion strengthening.

Annealing at 700°C had no effect on structural-phase state of the studied alloy. Bright-field images of microstructure are identical to those observed immediately after deformation by torsion under pressure (Fig. 1a).

Insignificant changes in grain-subgrain structure are observed after 800°C annealing. Against the background of initial structural state (Fig. 1a) crystallites with size of 50 to 250 nm and almost equiaxed shape (Fig. 1b) are formed. Sometimes initial anisotropic grains are completely fragmented with low-angle boundaries at almost equiaxed subgrains with sizes mentioned above.

Increasing the annealing temperature to 900°C leads to significant increase in size and changes of shape of crystallites (Fig. 1c). The bulk of the material is presented by nearly equiaxed grains (Fig. 1c) with dimensions (d) 0.7–1.7 μm, against their background there are smaller grains with characteristic dimensions 0.4–0.6 μm. Boundaries of the majority of large and small grains are mostly strained.

Annealing at 950 and 1000°C lead to the formation of identical structural states (Figs. 2a, 2b). Basically there are equiaxed grains, ranging in size from 1.2 to 2.6 μm and from 1.7 to 2.8 μm, respectively for the above annealing temperatures. Furthermore there are large grains greater than 5 μm in size. Significant difference of described structural states (Figs. 2a, 2b) from the structure after annealing at 900°C (Fig. 1c) is the presence of mostly flat grain boundaries. This feature indicates the activation of collective recrystallization type processes.

After annealing at 1200°C the bulk of material is represented by grains from 2 to 5 μm in size, against their background there are large grains greater than 15 μm (Fig. 2c). Such microstructure changes are associated with the start of intense secondary recrystallization processes.

It should be noted that in the investigated range of heat treatments from 700 to 1200°C, no changes were detected in phase composition and size of fine Zr (O–N–C)-based oxycarbonitride particles, which is a consequence of their high thermal stability.

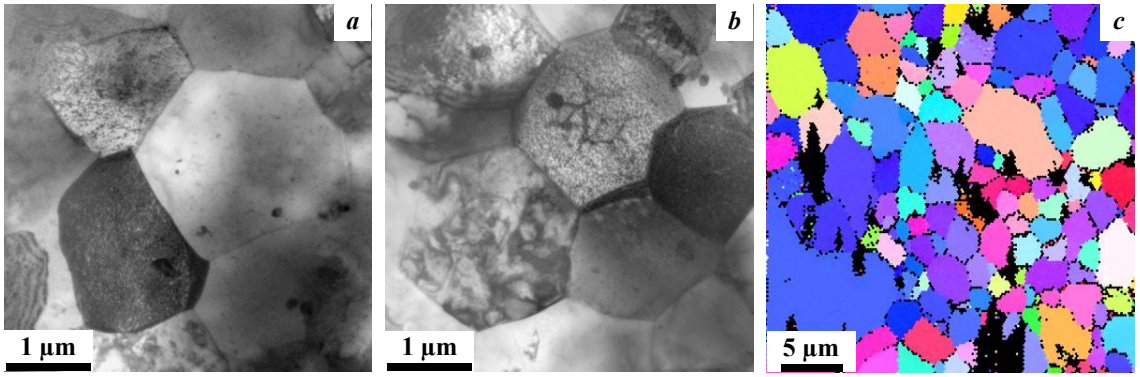

FIGURE 2. Grain structure of V–Cr–Zr–W alloy after deformation and annealing at temperatures of 950 (a), 1000 (b), 1200°C (c). Transmission electron microscopy (a, b). Scanning electron microscopy, EBSD analysis (c)

Described microstructure relaxation processes are accompanied by changes in microhardness values of V–Cr–Zr–W alloy. Microhardness of specimens before deformation (one hour annealing at 1400°C) amounted 1.5 GPa and after deformation by torsion under pressure for $N = 1$ its 2.3-fold increase up to 3.5 GPa is observed [1]. Figure 3a presents H_μ values after deformation and subsequent thermal treatments in the studied temperature range. It was found that annealing at 700, 800 and 900°C have no significant effect on average H_μ values. Increasing the temperature to 950°C leads to decrease in microhardness to 2.88 GPa. In the range 950–1050°C values of H_μ are in the range 2.8–2.9 GPa. After annealing at 1100 and 1200°C a decrease in H_μ values to 2–2.18 GPa is observed.

The analysis showed that in the temperature range from 900 to 1200°C microhardness decreases with the increase in average grain size (d) in accordance with Hall–Petch relation ($H_\mu = H_{\mu 0} + Kd^{-1/2}$) [4]. Basing on experimental data (Fig. 3b) corresponding Hall–Petch coefficients have been identified ($H_{\mu 0} = 0.94$ GPa, $K = 2.42$ GPa × μm$^{1/2}$). Experimental microhardness values and the results of theoretical calculations are presented on Fig. 3c.

It is well known [5] that strength parameters of heterophase materials are determined by several basic types of strengthening (deformation, solid solution, dispersion). Depending on conditions, predominance of a particular type or their cooperative implementation can be possible. After deformation by torsion under pressure, high microhardness level (3.5 GPa) of V–Cr–Zr–W alloy is a result of multiplicative implementation of the mechanisms described above. Increasing the temperature to 800°C promotes the relaxation of high-defect substructures. Despite the fact that after 900°C the activation of primary recrystallization is observed, the initial microhardness values are retained at the same level. This testifies to the low contribution of the deformation (substructural) strengthening type. In our opinion, the strength (hardness) in this case is determined mainly by disperse strengthening, the effectiveness of which depends on the characteristics of fastening of the defect substructure (dislocations) on nanosized particles of the second phase.

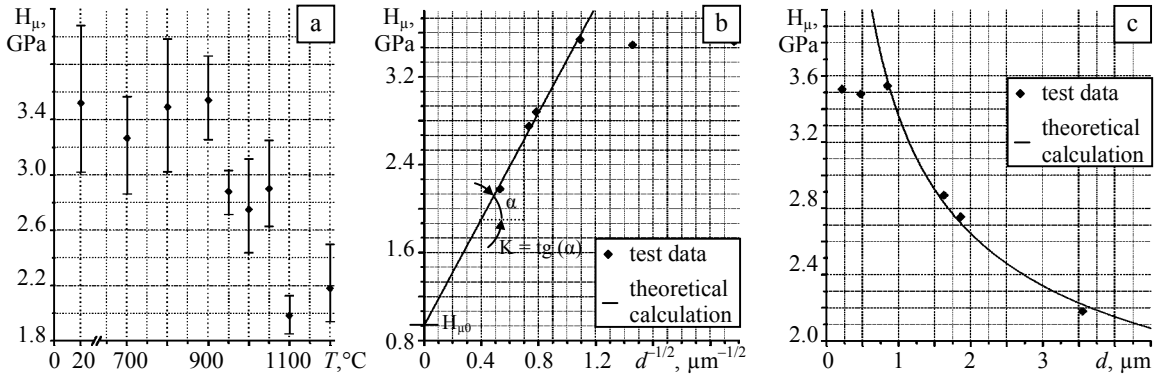

FIGURE 3. Microhardness of V–Cr–Zr–W alloy. Average microhardness values after deformation ($N = 1$) and subsequent annealing (a). (b) Hall–Petch coefficients definition ($H_{\mu 0}$ and K), (c) experimental and theoretical dependence of microhardness values from average grain size d

In the temperature range 950–1200°C intensive processes of primary and secondary recrystallization are observed (Fig. 2), which are accompanied by migration and change in grain boundaries length. Thus, microhardness values at this stage are mainly determined by the implementation of grain boundary type strengthening.

One of the most important results of this work is a significant increase in thermal stability of the investigated vanadium alloy. As it was shown (Fig. 3c), even after annealing at 1200°C the bulk of material is represented by fine grains of equiaxed shape close to 1–5 μm in size against which background coarse grains are found (10–20 μm). Earlier, such a state was observed in V–Ti–Cr-system vanadium alloy, deformed by multiple multiaxial forging, after annealing at 1000°C [6]. Significant increase in the thermal stability of V–Cr–Zr–W alloy is due to two main factors during the controlled modification of heterophase structure of the material in the process of thermo-mechanical treatment and chemical-heat treatment. Firstly, it is the formation of fine Zr (O–N–C)-based oxycarbonitride particles, characterized by improved thermal stability. Secondly, it is significant (more than 6-fold) increase in the volume fraction of uniformly distributed nanoscale (3–20 nm) particles.

The sharp decrease in microhardness values after annealing in the range of 950–1200°C (Fig. 3a), accompanied by an intense increase in grain size, is apparently due to the unlocking and activation of dislocation slip.

SUMMARY

The characteristic temperature ranges of relaxation processes implementation in V–Cr–Zr–W alloy after deformation by torsion under pressure and subsequent annealing were revealed. At 800°C polygonization and recovery processes are activated in the material. After 900°C, primary recrystallization processes, accompanied by rapid growth of grains, are observed. Further increase in temperature in the range 950–1050°C leads to intensification of collective recrystallization, the consequence of which is a significant increase in the proportion of equiaxed grains. At temperature 1200°C the secondary recrystallization activates, which resulted in significant increasing the size of individual grains, and significantly reduce microhardness values.

It has been shown that the increase in thermal stability of microstructure of V–Cr–Zr–W alloy is a consequence of the formation of high density of thermally stable Zr (O–N–C)-based nanoparticles.

ACKNOWLEDGMENTS

The work was conducted within the framework of the Program for Fundamental Scientific Research of Russian Academies of Sciences for 2013–2020 at partial financial support of Tomsk State University Competitiveness Improvement Program. Investigation was carried out using the equipment of the Tomsk State University.

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