Influence of annealing temperature on microstructure and microhardness of V–Cr–Ta–Zr alloy

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Abstract. The effect of stabilizing annealing temperature on the features of microstructure and microhardness of V–Cr–Ta–Zr alloy after thermomechanical treatment by regime II was studied. Main stages of relaxation processes are revealed. Characteristic microstructure parameters and microhardness values are determined for each relaxation stage.

1. Introduction

One of the ways to increase the mechanical properties of vanadium alloys is the use of thermomechanical treatments (TMT), which provide a modification of the structural-phase state [1 – 7]. In work [7], it was shown that TMT by mode II (TMT II) of V–Cr–Ta–Zr-system alloy increases its strength values at room and elevated temperatures. A necessary step in optimizing TMT modes is to study the effect of stabilizing annealing temperature on the structural-phase state.

In this work, we studied the effect of annealing temperature on microstructure parameters and microhardness of V–Cr–Ta–Zr alloy after TMT II.

2. Experimental materials and procedures

We used V–Cr–Ta–Zr vanadium alloy produced by SC "VNIINM", whose chemical composition in weight (wt. %) and atomic (at. %) percent is given in Table 1.

Table 1. Chemical composition of V–Cr–Ta–Zr alloy.

| Element | V | Cr | Ta | Zr | O | C | N |
|---------|---|----|----|----|---|---|---|
| wt. %   |   | 6.80 | 6.10 | 0.79 | 0.052 | 0.031 | 0.009 |
| at. %   | base | 6.99 | 1.80 | 0.45 | 0.174 | 0.138 | 0.034 |

Samples of this alloy were subjected to TMT II, which includes homogenizing one-hour annealing at 1400 °C and several rolling cycles (ε ≈ 20 – 30 %) at room temperature with intermediate one-hour annealing at 600 – 700 °C [7]. To study the thermal stability after final deformation cycle of TMT II, the samples were subjected to annealing in the temperature range from 700 to 1500 °C. Microhardness (Hₐ) was determined by the Vickers method on a Neophot 21 instrument with a load of 0.5 N and exposure time of 15 seconds. At least 10 prints were made for each structure state.

Electron backscatter diffraction (EBSD [8]) analysis was performed on scanning electron-ion microscope FEI Quanta 200 3D (30 kV) with the analytical system Pegasus in the hexagonal point staging mode. The program “TSL OIM data collection” has automatically indexed the Kikuchi
patterns formed by backscatter electrons. Collected data was processed using the “TSL OIM analysis” software. Structural studies were carried out on Philips CM 30 TWIN transmission electron microscope (300 kV).

3. Results and discussion

Grain structure of the alloy after TMT II before the stabilizing annealing (Figure 1) was studied in [7]. According to EBSD-analysis (Figure 1 a), it is characterized by grains, elongated in the rolling direction (RD), with a width of 3 to 35 μm and a length of 50 μm to several hundred μm. Gradient color is observed inside such grains, which indicates the presence of low-angle misorientation boundaries. According to the results of transmission electron microscopy, the microstructure is characterized by high heterogeneity. There are areas almost free of dislocations and areas where the scalar dislocation density (ρd) reaches $10^{11}$ cm$^{-2}$. Heterophase structure of the alloy is characterized by the presence of three main fractions of second phase particles (Figure 1 b, c): large (1 – 2 μm), medium (50 – 400 nm) and nanoscale (2 – 10 nm). It is established that these particles of different fractions are zirconium-based carbides and oxycarbonitrides.

![Figure 1. Microstructure of V–Cr–Ta–Zr alloy after TMT II before stabilizing annealing [7].](image)

After annealing at 700 °C (Figure 2 a), structural state is similar to that which is formed immediately after TMT II (Figure 1 a). But at the same time, the dislocation density decreases to $(3 – 7)\cdot10^{10}$ cm$^{-2}$. Minor changes in the microstructure after annealing at 800 °C (Figure 2 b) are revealed by the appearance of areas with less pronounced gradient color or without it. In our opinion, this is a result of recovery processes, which are accompanied by a decrease in the dislocation density to $(2 – 4)\cdot10^{10}$ cm$^{-2}$. After annealing at 900 °C (Figure 2 c), near-equiaxed grains with sizes from 3 to 20 μm with a gradient-free color are formed against the background of the initial band structure. The dislocation density in such grains does not exceed $10^{10}$ cm$^{-2}$. Thus, at 900 °C primary recrystallization processes are activated.

Annealing at 1000 °C is accompanied by a sharp intensification of primary recrystallization processes in the entire volume of the samples (Figure 2 d). This leads to the formation of almost equiaxed grains with gradientless color, the dimensions of which are in range from 3 to 30 μm. The dislocation density in these grains does not exceed $10^{10}$ cm$^{-2}$. At the same time, on the background of these grains, individual large grains elongated in RD (width 20 – 30 μm, length 50 – 300 μm) are preserved in the material, inside which gradient color is still observed. Such grains are a legacy of the initial structural state.

Processes of collective recrystallization begin to occur at 1100 °C (Figure 2 e). In addition, at this temperature the relaxation of elongated initial grains into large fragments occurs, the dimensions (width 20 – 30 μm, length 40 – 70 μm) of which are several times larger than grain sizes (5 – 20 μm) after primary and collective recrystallization. There is no gradient color inside large and small grains, which indicates the relaxation of the defect substructure.
A further gradual increase in annealing temperature up to 1400 °C inclusive is accompanied by processes of collective recrystallization (Figure 2 e – h), at which the grain sizes increase in a monotonic manner. At the same time, the division into two main fractions of grains is preserved: small (10 – 25 μm) and large (30 – 50 μm).

After annealing at 1500 °C, coarse grains up to 100 μm in size appear on the background of relatively small grains (10 – 20 μm). The scatter between small and large grains increases significantly, which manifests itself during statistical processing, the results of which indicate the presence of two main fractions, characterized by intervals of 15 – 40 μm and 60 – 100 μm. Thus, in the process of annealing at 1500 °C, secondary recrystallization is activated, which leads to substantial grain growth.

Table 2 presents the results of microhardness measurement before and after stabilizing annealing in the range from 700 °C to 1300 °C.
Accompanied by recovery processes ($\rho_d$ reduced to (3 – 7)$\times$10$^{10}$ cm$^{-2}$ and (2 – 4)$\times$10$^{10}$ cm$^{-2}$) anneals at 700 and 800 °C lead to a decrease in the microhardness values by ≈ 20 % and 30 %, respectively. A substantial (more than 40 %) decrease in $H_u$ after annealing at 900 °C is associated with the onset of active relaxation of defect substructure until the appearance of small defect-free grains, the dislocation density of which is 10$^{10}$ cm$^{-2}$. The completion of the primary recrystallization processes at 1000 °C is accompanied by a decrease in the microhardness to 1.5 GPa, which is ≈ 60 % of the initial values (Table 2). After heat treatments in the range from 1100 °C to 1300 °C, the values of $H_u$ are at the same level (Table 2) within the measurement error (∼ 10 %). Dislocation density after such annealing does not exceed 10$^{10}$ cm$^{-2}$. It was established that the main processes of microstructure transformation are associated with collective recrystallization, which results in the redistribution of the volume fractions of large and small grains.

The study of the effect of annealing temperature on the stability of heterophase structure is one of the topical areas for further research.

4. Summary

As a result of investigation of annealing temperature effect on microstructure and microhardness values of V–Cr–Ta–Zr alloy, the main stages of relaxation processes were revealed. Annealings at temperatures of 700 – 800 °C are accompanied by recovery processes. Heat treatment at 900 °C is characterized by the onset of primary recrystallization, the active phase of which proceeds up to 1000 °C inclusive. An increase in the annealing temperature in the range of 1100 – 1400 °C is accompanied with collective recrystallization processes. The temperature of secondary recrystallization onset is 1500 °C.

Acknowledgements

The authors are grateful to Professor A.N. Tyumentsev, Professor V.M. Chernov and M.M. Potapenko for facilitating experimental research and discussing the results.

Thermomechanical treatments and mechanical tests have been performed using the financial support from RFBR (Grant No. 18-08-00213_A). The microstructure studies were carried out within the framework of the Fundamental research program of the state academies of sciences for 2013 – 2020, research line III.23. The investigations were conducted using the equipment of Tomsk Material Engineering Center of Collective Use of Tomsk State University.

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