STRUCTURAL EVOLUTION AND HARDENING OF VANADIUM UPON SHEAR UNDER PRESSURE

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Abstract. The evolution of the structure and hardness of vanadium deformed by shear under pressure at room temperature is investigated. With a true deformation $e<1$, its localization was found, leading to the formation of a band structure. It is shown that the localization of deformation delays the transition to the QMS structure during subsequent deformation. The causes of the formation of deformation bands in vanadium are discussed. It has been established that dislocations make the main contribution to the hardening of vanadium during pressure shear at the initial stage ($e<1$), and large–angle boundaries of deformation origin during further deformation. The values of the parameters of the Hall–Petch type equation are obtained.

Keywords: *vanadium, deformation, shear under pressure, hardness, structure, deformation bands, Hall–Petch equation*

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INTRODUCTION

The study of the evolution of the structure of bcc transition metals during plastic deformation has been of constant interest to researchers for several decades [1–6]. The most significant differences between bcc metals and others are the strong temperature dependence of the yield strength and a fairly high stacking fault energy (SFE) [1, 7]. A high SFE facilitates the processes of cross slip and climb, which contribute to the formation of cellular structures. Whereas the temperature dependence of the yield strength complicates the rearrangement of dislocations into energetically more favorable configurations, such as cellular structures or subgrains, at low deformation temperatures. The deformation of all bcc metals is carried out by the appearance and movement of screw dislocations. It is known [2] that the mobility of screw dislocations is significantly lower than that of edge dislocations. At relatively low temperatures, the mobility of screw dislocations is controlled by the formation of a double kink, since the propagation of the kink occurs more easily than the movement of the entire dislocation line. Above a certain temperature, called the critical$T$c, the dislocation easily overcomes the Peierls barrier under the influence of thermal activation. In this case, the difference in the mobility of screw and non-screw segments decreases. The mechanical behavior of BCC metals then becomes similar to the behavior of crystals with an FCC lattice [8].

It is believed that the evolution of the structure of fcc metals depends on the ETL, in contrast to bcc, where this dependence is very weak [9]. On the contrary, according to Trefilov [1], the evolution of the dislocation structure of bcc metals is consistent with the ETL: the ETL of group V metals (V, Nb, Ta) is lower than that of neighboring elements from group VI (Cr, Mo, W), and at the same time, the former experience difficulties in forming a cellular structure with narrow boundaries during deformation.

At the same time, a number of studies have shown that niobium and vanadium are characterized by low values of T_c and Peierls stress, which leads to relatively high dislocation mobility and makes the deformation behavior of these BCC metals similar to that of FCC metals [10 – 14]. The evolution of the niobium structure during severe plastic deformation by high-pressure torsion (HPT) at room temperature has been studied in many works [15 – 18]. As in other metals with sufficiently high SFE (Fe [19, 20], Mo [21], Ni [22]), during Nb deformation, a cellular structure is first formed, in which rotational modes develop during severe plastic deformation and a transition to collective disclination effects occurs [23]. As a result of HPT, a disoriented submicrocrystalline (SMC) structure is formed. The observed slowdown in hardening in Nb is associated with the development of dynamic recovery [18] or migration of grain boundaries under the influence of internal stresses caused by deformation [16, 17]. It should also be noted that in niobium, during deformation at room temperature, neither pressure-induced phase transformations nor twinning occur. In vanadium, the lower SFE (100 and 200 mJ/m ^{2} for V and Nb, respectively [24]) increases the probability of twinning, in particular, with increasing impurity content [25].

The evolution of the V structure during deformation leading to the formation of the SMC state has not yet been studied in sufficient detail [26 – 28]. The aim of this work is – to study the patterns of changes in the structure of vanadium deformed by HPT at room temperature, and its strengthening depending on the degree of deformation.

MATERIAL AND EXPERIMENTAL TECHNIQUE

Vanadium with a purity of 99.98 wt.% was studied. The samples were cut from a rolled sheet annealed in vacuum at a temperature of 1100°C for 1 hour. The size of predominantly equiaxed grains was several millimeters (Fig. 1a). No clear texture was recorded in the plane of the sample; the structure of each sample before deformation was formed by several large grains of different orientations (Fig. 1).

Fig. 1. Initial structure of the studied vanadium, a – orientation map in inverse pole figure (IPF) colors; b – IPF. EBSD analysis data.

Deformation was performed by high-pressure torsion in Bridgman anvils without lateral support [29, 30]. The pressure during deformation was 6 GPa. The anvil rotation angle (φ) varied from 0° (compression without shear) to 3600° (10 anvil rotations). Before deformation, the samples had a disc shape with a diameter of 5 mm and a thickness of 0.3 mm. The true strain (e) was calculated taking into account the sample thickness before and after deformation, the anvil rotation angle, and the distance from the sample center [21]. The investigated range of true strain was 0.4 – 10.7. The error in determining the true strain did not exceed $\Delta e = 0.2$.

Vickers hardness measurements were carried out along two mutually perpendicular diameters on each sample with a step of 0.25 mm at a load of 0.5N using a "Qness GmbH" hardness tester, Austria. The results obtained on different samples were averaged over true strain intervals of 0.4. The hardness measurement error was 7%.

The structure of the deformed vanadium was studied using a JEM 200CX transmission electron microscope (TEM), Japan, at an accelerating voltage of 160 kV. The studies were conducted at a distance of 1.5±0.2 mm from the center of the sample. Foils for TEM were prepared by mechanical thinning followed by electrolytic polishing. Samples deformed by HPT with an anvil rotation angle of no more than 15° were additionally examined using a QUANTA 200 Pegasus scanning electron microscope (SEM), Netherlands, with an electron backscatter diffraction (EBSD) attachment. EBSD analysis was performed at an accelerating voltage of 20 kV, with a scanning step of 0.2 – 4.0 µm. During the analysis, points with a confidence index (CI) below 0.1 and grains containing 4 or fewer pixels were excluded from consideration.

The average size of structure elements was determined from the results of SEM studies using the linear intercept method and TEM studies using the inverse diameter method with an error of 5%.

RESULTS

The evolution of vanadium structure during SPD is shown in Fig. 2 – 7. Compression deformation led to the formation of a dislocation cellular structure (Fig. 2a) and deformation bands (uncompensated mesobands [31]), which are observed at different scale levels (Fig. 2b, 3a, b). The misorientation at the band boundaries occurs abruptly, with values ranging from 15° to 55° (scanning line *1* , Fig. 3b). In the cellular structure, the misorientation changes gradually, but even at a considerable distance (hundreds of microns) within one initial grain, the accumulated misorientation does not exceed 13° (scanning line *2* , Fig. 3c).

Fig. 2. TEM image of the microstructure of vanadium deformed by compression (e =0.4): a - bright field; b - dark field in the $(21\bar{1})$ reflection; c - microdiffraction and interpretation scheme, zone axes [135] and [132].

Fig. 3. Microstructure of vanadium deformed by compression (e = 0.4): a - orientation map in IPF colors with a schematic of HAGBs; b, c - orientation change along scanning lines *1* and *2* respectively, A – misorientation relative to the initial point; B – misorientation of adjacent points. SEM.

Analysis of microdiffraction patterns (Fig. 2c) shows that the deformation bands are not in twin orientation with respect to the matrix. The histograms of the distribution of structural elements by misorientation angles (Fig. 4a, b) show that after SPD with $e < 1$ (compression and φ=5°), the fraction of low-angle boundaries (LAGBs) exceeds 20%, and in the high-angle boundary (HAGB) region, there are no distinct peaks corresponding to twin misorientations (60° [25, 32]). Thus, in vanadium at the initial stages of deformation, along with dislocation slip, localization of deformation is observed. However, twinning as a possible deformation mechanism was not detected.

Fig. 4. Histograms of the distribution of structural elements by misorientation angles: a - e = 0.4 (compression); b – e = 0.7 (φ=5°); c - e = 1.4 (φ=15°). Based on EBSD analysis data.

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Fig. 5. Microstructure of vanadium deformed with e = 1.4 (φ =15°, a, b) and e = 2.5 (φ =45°, c); a – TEM bright-field image; b, c – dark-field images in reflections of (110) BCC type and microdiffraction patterns in the insets, in Fig. 5c arrows indicate microcrystallites.

Deformation bands are present in the structure even after greater deformation (Fig. 5a, b). During SPD with e =1.4 (φ=15°), fragmentation of the bands occurs, but no formation of new bands without fragmentation is observed. After this deformation, the fraction of HABs sharply increases and, accordingly, the shape of the misorientation angle distribution changes (Fig. 4). The fraction of LABs after SPD with e =1.4 drops sharply to 5%, and in the region of large angles, the distribution shape practically corresponds to that of an ensemble of randomly oriented grains [33] (Fig. 4c).

From the experimental EBSD data on kernel average misorientation maps – between a selected point (pixel) and all neighboring pixels in the second coordination sphere (Fig. 6), the density of geometrically necessary dislocations was calculated. For the calculation, the following expression was used [34]:

$$\rho_{GND} = (a\,\theta_{KAM})/(bx), \qquad (1)$$

where a – is a constant (for BCC a = 2), b – is the Burgers vector, x – is the scanning step, θ_{KAM} – is the kernel average misorientation, defined as the average misorientation angle. Calculations showed that at the initial stage of deformation, the dislocation density increased by an order of magnitude: the value of ρ_{GND} changed from $0.3 \cdot 10^{14}$ 1/m^2 after e =0.4 to $7 \cdot 10^{14}$ 1/m^2 after e =1.4.

Fig. 6. Kernel average misorientation distribution maps obtained from EBSD analysis: a – e = 0.4 (upsetting);
b – e = 0.7 (φ = 5°); c – e = 1.4 (φ =15°).

Fig. 7. UFG structure of vanadium and microdiffraction patterns (in the insets): a – e = 7.8 (3 revolutions); b – e = 9.8 (10 revolutions); TEM dark-field images in the reflection of (110) type.

The first submicron-sized structural elements having HABs of deformation origin, microcrystallites, were detected in the vanadium structure after deformation e =2.5 (indicated by arrows in Fig. 5c). Continued deformation leads to an increase in the number of microcrystallites and a decrease in the proportion of dislocation cells. After deformation e =7.8, dislocation cells with low-angle boundaries are no longer observed, i.e., the formation of SMC structure occurs (Fig. 7a). However, up to e =9.8, microcrystallites within deformation bands maintain similar orientations (Fig. 7b). This indicates that the formation of band structures in the initial stages of deformation has a significant effect on the formation of the SMC structure: within the initial band, rotations of adjacent microcrystallites are hindered.

Based on the results of SEM and TEM studies, histograms of the size distribution of structural elements were constructed (Fig. 8), average sizes (d_{avg}) were determined, and the dependence of d_{avg} on true strain was plotted (Fig. 9a).

Fig. 8. Histograms of size distribution of structural elements for vanadium deformed by SPD: a – e =0.4; b – e =1.4; c – e = 5.1; d – e = 8.3.

In Fig. 8b, it can be seen that as a result of deformation with e =1.4 (15°), the structural elements (more than 90%) are refined to submicron sizes, and individual large elements that form the "tail" of the distribution observed after deformation with e <1 (Fig. 8a) disappear. In Fig. 9a, the relationship also shows that in vanadium, strong structural refinement occurs rapidly: the average size of structural elements is 170 nm after deformation e = 4. Then, when a significant number of microcrystallites form in the structure (deformation e >5), their size stabilizes at 140 – 150 nm. In this case, the size distribution histograms practically do not change with increasing true strain (Fig. 8c, d).

Fig. 9. Dependence of hardness (●) and average size of structural elements (▲) of vanadium on true strain (a) and determination of contributions to its strengthening (b, c); b - establishing the parameters of the Hall – Petch relationship, c - dislocation strengthening.

Figure 9a shows the dependence of vanadium hardness on true strain. It can be seen that compression under a pressure of 6 GPa (e =0.4) leads to almost 2-fold strengthening of vanadium. With an increase in true strain up to e =7.0, the hardness continuously increases, and then in the range of 7.0 – 9.3, it practically does not change. At a strain of more than 9.3, further increase in hardness is observed. The maximum achieved hardness value is 3.4 GPa, which exceeds the values of 2.4 – 2.5 GPa obtained in works [26–28] during HPT deformation with 10 anvil turns of vanadium with purities of 98.8, 99.9, and 99.8 wt.%, respectively. The authors of these works observed strain reaching a steady state, and in work [28] they noted the development of dynamic recovery when increasing the anvil rotation angle beyond 5 turns.

In works [26 – 28], the smallest achieved grain size was 500 – 330 nm. Probably, the formation of a more dispersed structure in the present work made it possible to obtain higher hardness values compared to those reported in the literature. It should be noted that in these works, the minimum deformation was ¼ of a turn, which apparently did not allow the authors to observe the formation of band structures. According to our study, after deformation with an anvil rotation angle of 45° (1/8 turn) and more, obvious deformation bands are no longer observed (Fig. 5c, Fig. 7); their former presence is evidenced by similar orientations of microcrystallites within the former band. The possible evolution through the formation of band structures may be evidenced by the fact that in work [26], even after deformation by 10 anvil turns, elongated grains were observed.

DISCUSSION

As noted in the Introduction, vanadium and niobium are characterized by low values of critical temperature and Peierls stress, which determines the high mobility of dislocations at relatively low temperatures. However, the structural evolution during severe plastic deformation (SPD) in these materials is different. In vanadium, along with the formation of a cellular structure, deformation bands are formed, which were not observed in niobium [18]. In addition, in niobium, deformation at the UFG structure stage practically does not lead to further hardness increase, whereas in vanadium with UFG structure, hardness increases during deformation e >9.3 (Fig. 9a). Thus, it can be assumed that in V the effect of dynamic recovery is less than in niobium. It should be noted that the graph of hardness versus true strain for vanadium is similar to that for iron [20]: the initial stage of deformation with rapid hardening is replaced by an interval in which hardening is practically absent, and then there is a further increase in hardness (Fig. 9a). However, in iron during SPD deformation at room temperature, deformation bands are not formed. Possibly, the cessation of strain hardening at large deformation of niobium, due to the development of dynamic recovery, is associated with a higher stacking fault energy (SFE) value than that of iron and vanadium.

The formation of deformation bands in vanadium may be associated with the peculiarities of the "vacancy – dislocation" interaction. In work [35], a deformation model for BCC metals was developed, according to which there is a deformation temperature T_0, below which the interaction of a dislocation with a vacancy is hindered. According to the model, for Fe T_0=229 – 245 K, and for V T_0=396 K. Additionally, the vacancy migration energy calculated in work [35] is significantly higher in V than in Fe (125.4 and 73.3 kJ/mol, respectively). Thus, during deformation at room temperature (300 K) in vanadium, dislocation climb should be hindered because, firstly, the deformation temperature is below T_0 and, secondly, the vacancy migration energy has a high value. This leads, at the initial stages of deformation (e <1), to the inhibition of cellular structure formation and creates conditions for deformation localization, manifested in the formation of band structures. Such small deformations during pressure shear are realized only at small angles of anvil rotation and correspond to the beginning of the " $H - e$ " relationship in Fig. 9a, where there are few values. Thus, the experiment conducted in this work does not allow for a detailed study of the evolution of band structures; one can only assert that during deformation, their fragmentation occurs. However, such localization of deformation significantly influenced the subsequent evolution of the structure, since rotations of structural elements within the band were hindered. As a result, the formation of the SMC structure in vanadium occurred at a significantly greater deformation than in iron (7.8 and 5.3 [36]). It should be noted that the formation of the first microcrystallites in V and Fe occurs at similar values of true deformation (2.5 and 3.0 [36], respectively). The increase in true deformation required for the transition to the SMC structure stage, due to the presence of various types of band structures, has been previously noted in other materials, for example, in structural steel with a lath martensite structure [37], in austenitic stainless steel, Ti and Zr prone to pressure transformations [38 − 40], as well as during cryogenic deformation of iron and nickel, where deformation twinning occurred at the initial stages [41, 42].

It has been shown in [9] that the main contribution to the strengthening of pure metals during SPD comes mainly from grain boundaries and dislocations. Grain boundary strengthening is determined by the Hall –Petch equation [9]:

$$\Delta\sigma_{GB} = k\, d^{-1/2}, \qquad (2)$$

where d – is the grain size (or subgrain, but in this case the power exponent changes to –1), k is a constant for a given material. To evaluate the mechanical properties of nano- and submicrocrystalline materials, hardness values are commonly used as flow stress [43 – 45]. To establish the parameters of the equation " $H - d^{-n}$ ", the dependence of ln($H - H_0$) on ln d (Fig. 9b) was plotted. The value of 1 GPa, corresponding to the microhardness of undeformed coarse-grained V, whose structure is shown in Fig. 1, was taken as H_0. Fig. 9b shows that after deformation with $e > 1$, the experimental values are approximated by a straight line. The calculation leads to a Hall –Petch type equation, in which k = 0.7 (GPa μm $^{1/2}$), and $n = 0.51$, which is close to the value of 1/2, characteristic of a grain structure. The hardness values of vanadium deformed with $e < 1$ do not fit on the obtained dependence (Fig. 9b), i.e., the hardness of vanadium increases much faster than can be expected according to equation (2). In the present work, it has been experimentally established that at true strain with $e < 2$, a sharp increase in dislocation density occurs in vanadium.

The contribution of dislocations to strengthening can be estimated according to the expression [9]:

$$\Delta\tau = \alpha\, Gb\, \sqrt{\rho}, \qquad (3)$$

where α – is an empirical constant equal to 0.3; G is the shear modulus, 53.2 GPa; b is the Burgers vector, 0.262 nm for V [46]; ρ is the dislocation density, m $^{-2}$. The total dislocation density is the sum of the density of statistically stored dislocations (ρ_S) and the density of geometrically necessary dislocations (ρ_{GND}) [47]. As shown in studies [48, 49], during SPD the density of geometrically necessary dislocations can exceed the density of statistically stored dislocations by a factor of 3. In this case, material strengthening will be controlled by geometrically necessary dislocations. Substituting the ρ_{GND} data obtained from expression (1) into expression (3), it is possible to estimate how the contribution of dislocation strengthening changes at the initial stage of deformation up to e =2 (Fig. 9c). Analysis of Fig. 9 allows us to conclude that the main contribution to vanadium strengthening during SPD at the initial stage ($e < 1$) comes from dislocations, and with further deformation - from high-angle boundaries, the proportion of which after e =1.4 already exceeds 90% (Fig. 4). The dominant role of grain boundary strengthening has been noted previously. For example, in [50], an increase in the contribution of grain boundary strengthening was shown with the refinement of the SMC-structure elements of iron.

CONCLUSION

In vanadium deformed by high-pressure torsion, at the initial stage (at e <1), along with the formation of a dislocation cell structure, localization of deformation occurs, which leads to the formation of band structures - deformation bands.

The initial stage (e <1) is characterized by intensive strain hardening. In this case, hardness increases much faster than can be expected according to the Hall – Petch type equation. At greater deformation, strengthening follows the Hall-Petch equation with a power exponent close to -1/2, characteristic of grain structure. Thus, the main contribution to the strengthening of vanadium during SPD at the initial stage comes from dislocations, and with further deformation - from high-angle boundaries.

Comparison of vanadium and iron structure evolution during SPD deformation revealed the influence of deformation localization in band structure formation on achieving UFG state in vanadium. Deformation localization does not affect the formation of initial microcrystallites but promotes transition to UFG structure at higher true strain values, i.e., extends the stage of mixed-type structure existence.

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CONFLICT OF INTERESTS

The authors declare that they have no conflict of interest.

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