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Abstract

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RECRYSTALLIZATION DIAGRAM OF VANADIUM

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The study of recrystallization processes and the construction of recrystallization diagrams for metals and alloys, relating grain size to the degree of deformation and to the temperature of subsequent annealing, are necessary for establishing regimes of thermomechanical treatment and for selecting the operating temperatures of alloys. The processes occurring during recrystallization treatment and secondary recrystallization have a great influence on the properties of refractory metals and their alloys because of the high sensitivity of these materials to grain size (⌒1). The properties of vanadium, as well as of other refractory elements of group V of the periodic system (⌒2), depend strongly on grain size. A recrystallization diagram for vanadium has not yet been constructed. However, vanadium, being the lightest of the refractory monomorphic metals, is of great interest as a basis for alloys with high specific strength. In the literature there are only some data on the recrystallization of cold-deformed calciothermic and electrolytic vanadium ([3-]6). There are no literature data at all on the recrystallization of carbothermic vanadium, the method of producing which was developed in the USSR (⌒7).

We have constructed a first-order recrystallization diagram for cold-deformed (rolled) carbothermic vanadium refined with cerium (Fig. 1, see insert to p. 609). The initial carbothermic vanadium contained a significant quantity of interstitial impurities and could not be cold-worked. To increase its plasticity, the vanadium was refined with cerium according to a procedure developed in the laboratory of rare metals and alloys of our institute (⌒8). After refining with cerium in an arc furnace with a nonconsumable tungsten electrode in an atmosphere of purified helium (at a pressure of 0.9 atm), the vanadium contained 0.07% oxygen, 0.21% carbon, 0.01% nitrogen, about 0.2% metallic impurities, and could be cold-rolled without fracture to a high degree of deformation.

To eliminate the coarse-grained cast structure, the ingot was preliminarily cold-forged (degree of deformation 50%) and annealed in vacuum at 1200° for 1 hour. After forging and annealing, the vanadium possessed a polyhedral structure with

Fig. 1. Recrystallization diagram of first-grade vanadium

Figure 1: Fig. 1. Recrystallization diagram of first-grade vanadium

an average grain diameter of about 63 μ. In the metal structure there also occurred complete redistribution of carbides (the solubility of carbon in vanadium at room temperature is less than 0.2% ([4-]6)). The material treated by the above method was the starting material for constructing the recrystallization diagram. After cutting, the specimens were cold-rolled without intermediate anneals to the following degrees of deformation: 5, 10, 15, 25, 50, 75, and 95%. Cold rolling of vanadium at a degree of deformation above 25% leads to refinement of the grain and to the formation of a sharply pronounced deformation texture (Fig. 2a, see insert to p. 609).

The hardness H_{V5} of the initial vanadium (120 kg/mm^2 after forging and annealing at 1200°) increases with increasing degree of cold deformation; however, vanadium work-hardens weakly. At 5% deformation its hardness is 136 kg/mm^2, at 25%–156 kg/mm^2, and at 50-95%–165 kg/mm^2. The deformed specimens were annealed in a vacuum of 10^{-5} mm Hg for 1 hour.

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Fig. 2. Microstructure of vanadium (200×): a –deformed by 75% (rolling); b –deformed by 75% and annealed at 850°; c –deformed by 10% and annealed at 1100°; d –deformed by 75% and annealed at 1300°.

Fig. 3. X-ray diffraction patterns of vanadium: a –initial; b –deformed by 50%; c –deformed by 50% and annealed at 800°.

at 750-1300°. The temperature of the onset of recrystallization was determined by methods of microstructural and X-ray analysis, as well as by the hardness method. The polished sections were etched electrolytically in a 5% solution of nitric acid at a current density of 5-7.5 A/cm^2. The average grain diameter was determined by the intercept method on the transverse section of the polished specimen at the point of intersection of the diagonals.

Fig. 1. Recrystallization diagram of first-grade vanadium

The X-ray patterns were recorded with copper radiation, with an exposure of 3-4 hours, by the sliding-recording method from a flat polished section immediately after annealing.

The temperature of the onset of recrystallization of vanadium decreases with increasing degree of deformation. At 5% deformation, recrystallization begins only at 900°. When the degree of deformation is increased to 50%, the temperature of the onset of recrystallization falls to 800°, and at 75-95% it falls to 775°. The change in the temperature of the onset of recrystallization as a function of the degree of deformation is plotted on the recrystallization diagram with a dashed line (Fig. 1). Selected X-ray patterns are shown in Fig. 3. The recrys-

Fig. 4. Hardness of cold-deformed vanadium after annealing at various temperatures (the dashed line shows the line of the beginning of recrystallization)

Figure 2: Fig. 4. Hardness of cold-deformed vanadium after annealing at various temperatures (the dashed line shows the line of the beginning of recrystallization)

tallization process during treatment leads to complete elimination of the rolling texture and to the filling of the entire volume of the metal with new recrystallized grains having an average diameter on the order of 20 μ (Fig. 2b). It should be noted that at small degrees of deformation (<15%), apparently because the growth rate of the recrystallized grain exceeds the rate of formation of nuclei, coalescence recrystallization begins before recrystallization during treatment is completed; this leads to the formation of a coarse-grained structure (40–50 μ) immediately after the onset of recrystallization. An analogous phenomenon was observed in magnesium ([9]). In addition, at small degrees of deformation a certain nonuniformity of the structure formed over the cross section of the specimen is observed.

When cold-deformed vanadium is annealed up to 1050°, grain growth scarcely occurs. Coalescence recrystallization begins at 1100°. Annealing at 1100° leads to sharp grain growth at a deformation degree of 10% (Fig. 2b). When the annealing temperature is raised to 1200°, the "critical" degree of deformation shifts by 5%, and when it is raised to 1300°,

probably to an even smaller degree of deformation. At large degrees of deformation the grain grows much less. Beginning with 25% deformation, the average grain diameter, when the annealing temperature is raised to 1200°, increases from 20 μ to 40–50 μ. More substantial coarsening of grains at large degrees of deformation is observed only at 1300° (Fig. 2e). Recrystallization of cold-deformed vanadium leads to a reduc-

Fig. 4. Hardness of cold-deformed vanadium after annealing at various temperatures (the dashed line shows the line of the beginning of recrystallization)

tion in its hardness; moreover, the higher the degree of deformation, the more rapidly the hardness decreases to 120 kG/mm^2. During collective recrystallization, an increase in the annealing temperature causes a further decrease in the hardness of highly deformed specimens (to 100-110 kG/mm^2), which is evidently associated with grain growth. The effect of the degree of deformation and the annealing temperature on the hardness of vanadium is shown in Fig. 4.

On the basis of the data obtained, it may be considered that the optimum treatment regime ensuring a fine-grained structure and good mechanical properties of vanadium is cold deformation with a reduction of 50–95% and annealing at temperatures of about 850–1050° in an inert atmosphere.

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Note: Figure translations are in progress. See original paper for figures.