TEMPERATURE DEPENDENCE OF THE YIELD STRENGTH OF TUNGSTEN WITH DIFFERENT MICROSTRUCTURES

V. S. Kravchenko, I. V. Sheina,
V. S. Gnuchev, E. É. Zasimchuk,
V. K. Kharchenko, and A. N. Vashchenko

UDC 539.04

The use of products made of tungsten in various areas of technology is due to a need for strength in a high-temperature region where other metals and alloys are unfit for use. It was shown in [1] that the high-temperature strength and ductility of tungsten depends substantially on grain size. In this connection, it is of considerable interest to examine the temperature dependence of the mechanical properties of a tungsten cermet with different grain sizes and to determine the physical nature of this dependence.

The investigation was carried out on commercially pure tungsten obtained by high-temperature rolling and annealing of sintered billets. The tensile tests were held on high-temperature unit VTU-2V [2] within the temperature range 20-1500°C. The diameter of the working section of the specimens was 4.0 mm, length 20 mm. The specimens were heated by radiation at a rate of 1 deg/sec and soaked before tension for 10 min. Strain rate was equal to $1 \cdot 10^{-3}$ sec^{-1}. We determined ultimate tensile strength σ_u, yield strength $\sigma_{0.2}$, elongation δ, and reduction in area ψ from the test results.

The specimens were etched before testing until the structure was revealed so that they could be classified according to structural type [1].

We conducted studies using the method of x-ray two-crystal spectrometry to determine the degree of perfection of the crystal structure of the grains. This method allowed us to obtain a topograph and record the angular distribution of reflection intensities of individual grains on recorder chart paper [3].

We studied the fracture surfaces of the specimens by electron fractography using a "Stereoskan S4-10" electron microscope at a relatively low magnitication (180-1600 ×) and an accelerating voltage of 39 kV. We studied the distribution (where present) of nonmetallic inclusions and voids on cleavage facets obtained at room temperature.

Grain diameters were measured by the secant method [1] to obtain quantitative characteristics of the microstructure.

Figure 1 shows the results of the mechanical tests in Hall–Petch coordinates [4, 5]. For test tempera-

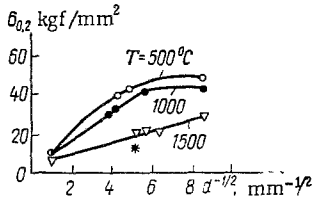

Fig. 1. Dependence of yield strength $\sigma_{0.2}$ on grain size of commercially pure tungsten at different temperatures (*variable grain size with patches of small grains).

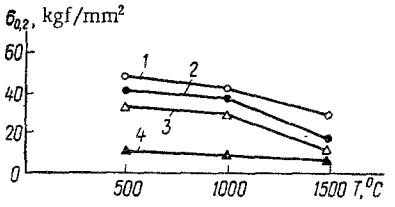

Fig. 2. Temperature dependence of yield strength $\sigma_{0.2}$ for structures of different types: 1) fine-grained ($d^{-1/2} \approx 8.5$); 2) medium-grained ($d^{-1/2} \approx 5$); 3) medium-grained ($d^{-1/2} \approx 4$); 4) coarse-grained ($d^{-1/2} \approx 1$).

Institute of Strength Problems, Academy of Sciences of the Ukrainian SSR, Kiev. Translated from Problemy Prochnosti, No. 2, pp. 54-57, February, 1979. Original article submitted June 12, 1977.

0039-2316/79/1102-0175$07.50

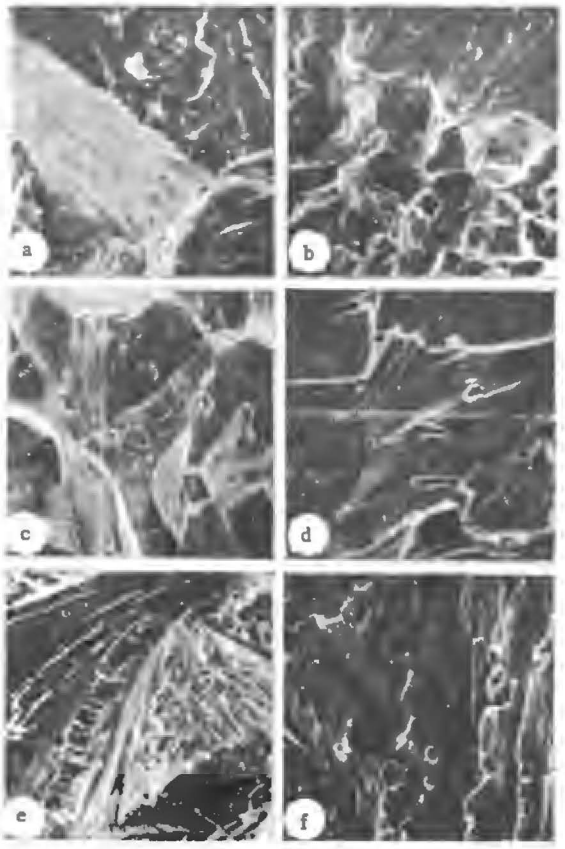

Fig. 3. Cleavage surface of tungsten specimens: a) medium-grained structure (×1600); b) coarse grain in fine-grained structure (×310); c) fine-grained section (×770); d) section of coarse grain (×1600), e) nonmetallic inclusions in fine-grained structure (×1300); f) structure of sintered tungsten.

tures of 500 and 1000°C the dependence of yield strength on average grain size shows the discontinuity characteristic of commercially pure materials [4]. At 1500°C the Hall–Petch relation is satisfied for the entire grain-size range investigated (3.6 μm to 0.3-0.5 mm), although in the region of average grain sizes there is a tendency toward smaller values of yield strength. The most substantial decrease in yield strength $\sigma_{0.2}$ is seen in specimens with a variable structure, a circumstance connected with partial diffusion of segregated impurities along grain boundaries at this temperature. A similar effect of temperature on the role of boundary impurities in intermediate layers is also characteristic in relation to other structure-sensitive properties of the materials (e.g., rate and character of migration of high-angle grain boundaries) [6]. Studies of boundary migration during primary, secondary, and selective recrystallization of several metals of varying degrees of purity conducted earlier showed that the quantity of grain-boundary impurities is reduced sharply with a shift in the temperature range of the process to the side of higher temperatures.

However, an analysis of the effect of the impurity phenomenon alone on the yield strength value in the temperature function does not provide an explanation of the reason for the deviation of the test data from the Hall–Petch relation in the average-grain-size region at 1500°C. The temperature dependences of the yield strength of tungsten for structures of different types (fine-, medium-, and coarse-grained sizes in accordance with the classification proposed in [1]) testifies to the fact that at 1000°C the inflection in the curve is seen only for the medium-grained structure (Fig. 2). Evidently the investigated types of structure are characterized by a more profound difference than the most probable grain size.

To explain this process we examined the perfection of the intracrystalline structure of tungsten having all three types of structure using the method of two-crystal spectrometry. It was found that the degree of perfection of the crystalline structure of the individual grains is different for the types of structure examined. For the medium-grained tungsten, characterized by high strength and ductility properties, the width of the oscillation curve averages 22' for half its height, which is typical of polygonized tungsten [6].

The coarse- and fine-grained structures are distinguished by their more perfect crystalline structure: the half-width of the oscillation curves from grains of these structures is 5-10', which corresponds to the recrystallized condition of tungsten [6], apparently arising as a result of primary recrystallization. The fact that the fine- and coarse-grained structures appear as a result of primary recrystallization also explains the deviations from the nominal distribution of grains with respect to size established for these structures earlier [1].

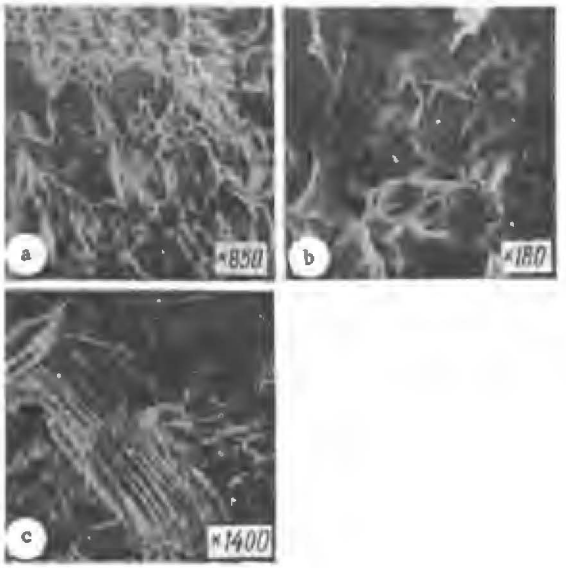

Fig. 4. Fracture surface of specimens after testing at 1500°C: a) medium-grained structure; b) coarse-grained structure; c) fine-grained structure.

The coarse- and fine-grained structures also have the same size voids on the cleavage facet surfaces of the investigated specimens (Fig. 3). As can be seen from Fig. 3a, which shows the surface of the cleavage facet of the medium-grained specimen, the voids in the tungsten matrix are uniformly distributed and most of the ones in the rolling direction are no larger than 1 μm.

The voids in the coarse- and fine-grained specimens are also distributed uniformly, but they are about 3 μm in size. This common feature is particularly well illustrated in Fig. 3b-d, which shows a section combining a coarse-grained and fine-grained structure. In certain cases nonmetallic inclusions are visible on the cleavage surface of the fine-grained specimens (Fig. 3e).

It should be noted that in cases where recrystallization is observed during the sintering process, voids are also seen on the cleavage surface of sintered specimens, and in most cases they are equiaxed (Fig. 3f).

Evidently, one of the reasons for the recrystallization of tungsten during the rolling process is a certain increase in local stresses in the metallic matrix in the vicinity of voids and inclusions [7].

The differentiation in void sizes in tungsten according to type of structure may be indicative of a difference in the chemical composition of the sintered billets, which would explain the different tendencies of the billet materials to recrystallize.

Using the method of radio-frequency mass spectrometry we observed a high content of low-melting elements (K, Na, etc.)* in the tungsten with coarse voids. The vapor pressure of these elements in the voids evidently prevents welding of the latter during rolling.

The uniform distribution of the nonmetallic inclusions observed in different-size grains is evidence of the fact that they are fairly effective barriers to the migration of high-angle boundaries and thus cannot play a significant role in forming the undesirable variable-grained structure [1]. The formation of such a structure during recrystallization is probably connected with the action of several factors: the presence of insoluble impurities, the number of recrystallization nuclei, the presence of especially fine particles unresolvable by the methods used here, etc.

Formation of a variable-grained structure in a material contaminated with uncontrolled impurities and process additions is always accompanied by primary recrystallization due to the extremely uneven distribution of these impurities in the volume of the material.

Thus, the appearance of the variable-grained structure in contaminated tungsten is a result of primary recrystallization. In this connection, the occurrence of the above process in tungsten destined for practical use is very undesirable.

Moreover, it is well known that recrystallization in tungsten, as in other bcc metals (molybdenum, niobium) results in a substantial reduction in strength and ductility properties at both low and high temperatures.

*The analysis was conducted by I. I. Nemoshkalenko and V. P. Mel'nichenko.

However, the mechanism of embrittlement of these materials during recrystallization is still not entirely clear.

It may be proposed that the appearance of sections with a perfect structure (recrystallization centers) during tungsten recrystallization occurs by the mechanism of a critical stage of polygonization described in [8]. In the event of multiple disintegration of subboundaries the superposition of stresses arising due to a displacement in dislocations of opposite sign toward each other may result not only in the formation of high-angle boundaries, but also in the nucleation of microcracks if the stresses cannot be relaxed by some other means. Under dynamic conditions (as during rolling) crack opening is facilitated, as is well known.

The completed fractographic studies do not conflict with the proposed mechanism of tungsten embrittlement. Figure 4 shows fractographs of medium-, coarse-, and fine-grained specimens after testing at 1500°C.

As can be seen, the medium-grained specimen has a typically tough fracture surface (Fig. 4a). At the same time, the coarse-grained specimen has a mixed type of fracture – sections of tough and brittle fracture together, the latter arising from microcracks (Fig. 4b c).

The presence of microcracks is evidently attributable also to the lesser temperature dependence of yield strength at temperatures above 1000°C in specimens with coarse- and fine-grained structures formed by recrystallization.

Thus, the three types of structures of commercially pure tungsten manifest by different temperature dependences of the mechanical properties are distinguished not only quantitatively (average grain size and character of grain distribution according to size) but qualitatively: while medium-grained tungsten has a partially polygonized intragranular structure, the fine- and coarse-grained tungsten specimens have a primary recrystallization structure. This was established with x rays according to the half widths of the peaks of the double-reflection curves.

Among the reasons for the recrystallization of the commercially pure tungsten during high-temperature rolling are the presence of nonmetallic inclusions and the high concentration of impurities (K, Na, Ti, etc.) in the sintered billets. The vapors of these impurities, accumulated in voids, impede healing of the latter during rolling and create conditions favorable to the appearance of local overstresses in the matrix, which in turn facilitates the formation of recrystallization centers.

LITERATURE CITED

1. V. S. Gnuchev, E. É. Zasimchuk, K. N. Kas'yan, et al., "Mechanical properties and structural nonuniformity of tungsten cermet," Probl. Prochn., No. 12, 14-18 (1977).
2. V. K. Kharchenko, "Unit VTU-2V for the study of the short-time strength of refractory materials to 3100°K," Novye Pribory, Ustanovki, i Oborudovanie dlya Nauchnykh Issledovanii, 7, 102-104 (1968).
3. V. V. Zholud', E. É. Zasimchuk, V. S. Kravchenko, and E. A. Maksimenko, "Applicability of x-ray diffraction topography to the study of deformed structures in single crystals," Ukr. Fiz. Zh., 14, No. 6, 904-909 (1969).
4. V. I. Trefilov, Yu. V. Mil'man, and S. A. Firstov, Physical Bases of the Strength of Refractory Metals [in Russian], Naukova Dumka, Kiev (1975).
5. L. K. Gordienko (editor), Superfine Grains in Metals [Russian translation], Metallurgiya, Moscow (1973).
6. E. É. Zasimchuk, Polygonization, Recrystallization, and the Thermal Stability of the Properties of Materials [in Russian], Naukova Dumka, Kiev (1976).
7. E. É. Zasimchuk, V. S. Kravchenko, and V. K. Kharchenko, "Critical polygonization stage in single crystals of tungsten deformed in compression and its role in strength changes," Probl. Prochn., No. 1, 69-73 (1978).
8. J. Friedel, Dislocations, Pergamon (1964).