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The range of applications of refractory metals is often restricted by their recyrstallization embrittlement [1-3]. Cracks in an embrittled material may lead to its premature failure; for example, the fatigue limit of technical molybdenum in a recrystallized condition sharply falls, to 20 MPa, in alternating bending tests. It is therefore important to study the high-temperature embrittlement of metals of this class and its effect on their fracture toughness.

In this paper the results are presented of an investigation into the high-temperature embrittlement of tungsten produced by the powder metallurgy method. An examination is made of the relationship between the recrystallization and fracture toughness of this material in a wide temperature range. The technical-purity tungsten investigated was a material of medium grain size, with a cellular deformation structure. The structural changes induced in it by recrystallization annealing were determined with a DRON-2.0 x-ray diffractometer provided with a BSV-11 tube and also by back-reflection x-ray photography with a controlled irradiated volume, using copper radiation.

Recrystallization annealing of the tungsten was performed in a vacuum at a temperature of 2200°C in the VURT-1 apparatus, developed at the Institute of Strength Problems, Academy of Sciences of the Ukrainian SSR [4]. The same apparatus was used for determining the fracture toughness of tungsten specimens. As a comparative fracture toughness parameter the critical coefficient of stress intensity  $K_{1C}$  was adopted [5].

High-temperature annealing induced recrystallization in tungsten specimens. After as little as 15 min of heating at 2200°C x-ray photographs showed clearly defined reflections from recrystallized grains, and a more perfect structure began to form. The numbers of reflections found in back-reflection photographs after recrystallization annealing for various periods of time at 2200°C are given below:

When the duration of annealing was increased to 90 min, the number of recrystallized grains markedly grew. Structural changes in local regions up to 1 mm in size were uneven in character. X-ray photographs contained sharp reflections formed by the annealed structure, but also revealed the existence of regions with considerable distortions.

Recrystallization annealing had a pronounced deleterious effect on the fracture toughness of the tungsten. Values of fracture toughness parameter  $K_{1C}$  for starting and recrystallized tungsten specimens are given in Table 1. The comparability of the values of  $K_{1C}$  at temperatures of 2000 and 2200°C is evidence that, as demonstrated by the results of x-ray

TABLE 1

| Tungsten                   | Crit. stress intensity coeff. $K_{1c}$ (MPa • $m^{1/2}$ ) at temp., $C$ , of |           |          |            |
|----------------------------|--|-----------|----------|------------|
|                            | 20   | 800       | 2000     | 2200       |
| Starting<br>Recrystallized | 28,5<br>7,5  | 44<br>7,5 | 7<br>7,5 | 7,5<br>7,5 |

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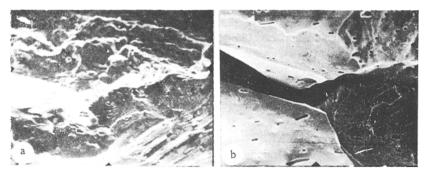


Fig. 1. Fracture surfaces of starting tungsten at temperatures of: a) 20, ×1300; b) 2200°C, ×1400.

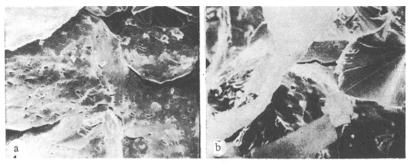


Fig. 2. Fracture surfaces of recrystallized tungsten at temperatures of: a) 20, ×1300; b) 2200°C, ×1350.

structural analyses, recrystallization processes did take place at these temperatures. The insensitivity of the parameter  $K_{1C}$  of recrystallized tungsten to thermal action confirms that the structural changes responsible for the fall in fracture toughness during high-temperature annealing were irreversible. Conditions favorable for brittle cleavage were created in the material at stresses independent of T; this finding is not in contradiction with the hypothesis advanced in [6].

To investigate details of the high-temperature embrittlement and its effect on the fracture toughness of tungsten, fracture surfaces of tungsten specimens were subjected to electron fractographic analysis with a Stereoscan-S4-10 scanning electron microscope. In order to attain optimum fracture conditions and obtain best-quality photographs, use was made of the maximum possible accelerating voltage, i.e., 30 kV.

Fractures were examined at magnifications in the range from 500 to 12,500 diameters, which is an optimum for a detailed study of the fracture of tungsten (Figs. 1 and 2). From the fractograms shown here it will be seen that, in accord with literature data on the character of high-temperature rupture of tungsten [1, 2, 7], the crack as a rule propagated along grain boundaries (Figs. 1b and 2a and b). A characteristic feature of the surfaces of starting tungsten specimens tested at 2200°C and of recrystallized tungsten specimens which failed at temperatures of 20, 2000, and 2200°C was the appearance of long grain-boundary cracks (Figs. 1b and 2a), which was indicative of a weakening of the grain boundaries. At temperatures below 2000°C no cracking of the starting tungsten was observed. The results obtained bear out the view expressed in the literature that the grain boundaries of tungsten constitute its "weak link," and that their strength falls as a result of isothermal heating [1, 2].

Thus, the high-temperature embrittlement of tungsten is characterized by the appearance of microcracks at grain boundaries, which leads to premature failure with only a partial utilization of the ductility properties of tungsten and is probably one of the reasons why brittle cleavage conditions are created at stresses which are independent of temperature.

On the basis of these physicomechanical investigations of the high-temperature fracture toughness of tungsten we can conclude that isothermal heating decreases the fracture toughness

of tungsten by weakening the bonds between its grains; prior recrystallization annealing brings about an irreversible fall in the fracture toughness parameter  $K_{1C}$  of tungsten and promotes the creation in this structure of brittle cleavage conditions at stresses which are independent of temperature.

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NEW GRADES OF TUNGSTEN-FREE TiC-Ni-Mo HARD METALS FOR MILLING CAST IRONS AND STEELS

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Experience with the production of tungsten-free hard metals (TFHMs) shows that their properties depend to a large extent on the way in which certain operations are carried out. By varying processing parameters and methods of preparation of titanium carbide, e.g., it is possible to markedly change its structure and properties without altering its composition.

Usually, to achieve effective mixing, increase the homogeneity of TFHM powders, and improve contact between the TiC particles and the binder, vigorous milling of mixtures is employed, as otherwise structurally heterogeneous alloys of high porosity and low strength

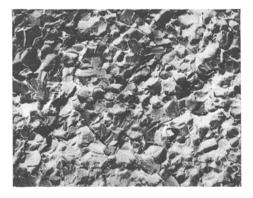


Fig. 1. Structure of fracture surface of TN-25-P alloy, ×3000.

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