INCLUSIONS IN TUNGSTEN AND ITS ALLOYS

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Removing the impurities from tungsten increases its ductility, and thus a study of the effect of nonmetallic inclusions on the brittle fracture of tungsten is of particular importance [1].

Previous studies of the erosion of cast commercially pure tungsten and its alloys in a single strong discharge by low-voltage spark showed, along with vaporization of the tungsten matrix, formation of cracks, and melting (with over 0.1 wt. % C), with erosion of some types of inclusions when the sample is the anode. Four types of inclusions were observed in cast commercially pure tungsten – three types of primary and secondary carbides, sharp-angled fairly coarse "glasses" (based on SiO_2), and large round inclusions (based on Al_2O_3) that are easily distinguished on etched or unetched microsections at magnifications of ×200 with standard or oblique illumination, while inclusions of the fourth type, evidently different in composition, are distinguishable only in polarized light. It is difficult to identify the inclusions due to their small size (less than 1-4 μ) and difficult to observe them in microscopic examinations [2].

In this work we used the erosion of inclusions under the influence of discharge for two types of investigations: 1) spectral analysis of the material in the inclusion extracted in the plasma during the partial destruction of the inclusions; 2) microprobe analysis of the inclusion retained in the metal, the outlines of which on the microsection are clearly revealed due to the erosion under the influence of the discharge.

The characteristic types of erosion of inclusions in tungsten are shown in Fig. 1. The outward appearance of secondary carbides and "glasses" based on SiO_2 (Fig. 1a) does not change after a single spark discharge, although melting would be expected. There is a considerable group of inclusions not distinguishable on the original microsections with direct or oblique illumination that evaporate or melt under the influence of the discharge, due to which hydrodynamic ejection of the inclusion material occurs, with formation of coronas where the material is deposited in the form of iridescent films of condensed vapors of the inclusion material (Fig. 1a) or spattered droplets (Fig. 1b). Some inclusions are evidently eroded to a lesser extent and retain their original shape but are clearly distinguishable in areas of the microsection subject to the effect of the discharge due to large evaporation of the metal at the inclusion—matrix interface (Fig. 1c).

Microprobe analysis with the MS-46 analyzer showed that the iridescent border around the inclusion (Fig. 1a) consists of approximately 30% silicon, while the inclusion is rich in aluminum (along with oxygen). The inclusions surrounded by spattered droplets (Fig. 1b) contain ~30% Mg, with some aluminum (Fig. 1c, curve 3) and in some cases silicon or calcium (Fig. 1b). The fine inclusions undergoing slight erosion (Fig. 1c) consist mainly of aluminum and oxygen. The size of these inclusions does not exceed 5 μ, which makes it impossible to determine the stoichiometric composition from the data obtained by microprobe analysis. However, it is possible to distinguish which are the principal elements and which are additional elements.

Spectral analysis of material from the inclusion extracted in the plasma during a single low-voltage discharge was conducted with the ISP-51 spectrograph, with focused discharge at the gap of the spectrograph in air and in an atmosphere of high-purity helium [3]. The quantities of oxygen, hydrogen, and nitrogen were determined in a helium atmosphere. With a single discharge (C = 320 μF, L = 10 μH) the vaporization of tungsten is so slight that even highly sensitive lines WI 4008.75 Å and WI 4659.9 Å are almost lacking, which greatly facilitates the identification of partially destroyed inclusions. The inclusions frequently contain O, Ca, Al, C, Ba, H, K, and Mg.

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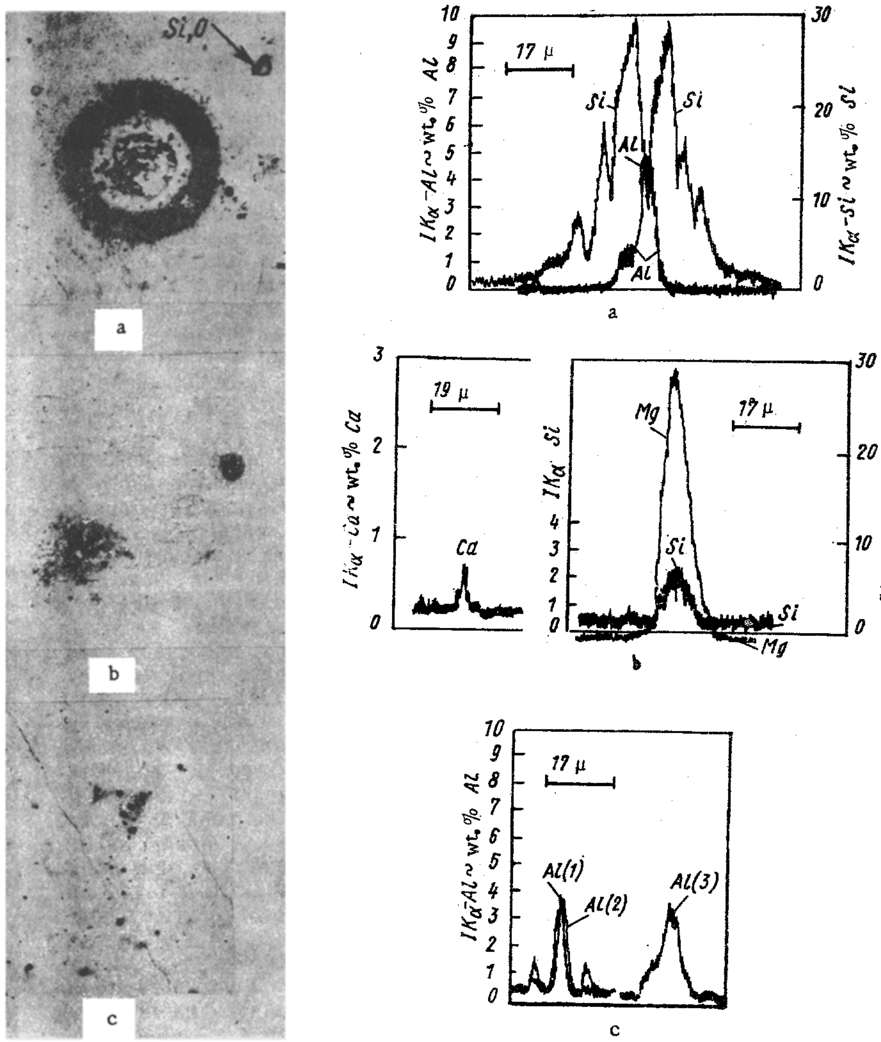

Fig. 1. Erosion of inclusions in tungsten under the influence of a single discharge (×340) and distribution of elements in them. a) "Glass" in an inclusion containing aluminum and silicon; b) inclusion containing magnesium, silicon, aluminum, and calcium; c) inclusion containing aluminum and oxygen.

In tests of different samples of tungsten alloys in spark discharge (~0.5 sec with the same parameters) the erosion of tungsten and the inclusions increases – lines of tungsten appear in the spectrum and the intensity of lines of other elements increases, particularly oxygen, with a weak line of nitrogen NII 3995 Å, and in some cases lines of silicon.

There are differences in the patterns of elements observed in the spectrum of the spark discharge and in the microprobe analysis of the inclusions. Thus, there are no lines of silicon in the spectrum of the spark, while the microprobe analysis shows a substantial number of eroded and undamaged inclusions based on silicon. At the same time, it is impossible to obtain data on the presence of hydrogen and nitrogen by means of microprobe analysis.

Comparison of the results of chemical, gas, spectral, and microprobe analyses and data on the microstructure leads to the conclusion that the inclusions in tungsten are very different in composition. Carbides, and also the largest inclusions of the oxide type, mainly silicon dioxide (sharp-angled "glasses") or aluminum oxide (rounded), are the most resistant to erosion. Inclusions of more complex composition, also containing silicon, aluminum, calcium, and magnesium, barium, and potassium, are more subject to erosion. The lines of hydrogen and nitrogen observed in the spectra of the inclusions confirm the previous assumption by us and by other investigators of the existence of inclusions of complex composition such as oxynitrides, carbonitrides, and oxyhydrides.

A relationship can be traced between the data from spectral analysis of inclusions and the results of spectral and chemical analysis of massive samples for aluminum, silicon, calcium, potassium, barium, and magnesium. It is interesting that the spectra of the inclusions show no lines of the metals contained in tungsten – iron, titanium, zirconium, molybdenum, niobium, and others. They are evidently in solid solution or enter into the composition of more erosion resistant inclusions in small quantities.

The destruction of fine relatively low-melting inclusions in the grain boundaries may be one reason for the intensive formation of cracks in the grain boundaries of fine-grained tungsten [2].

CONCLUSIONS

1. A single strong discharge in an atmosphere of high-purity helium or air induces erosion of nonmetallic inclusions in tungsten, which permits spectral analysis of the inclusion material extracted in the plasma and also facilitates observation of inclusions on microsections and microprobe analysis of them.

2. The most erosion resistant inclusions in tungsten are carbides and oxide inclusions such as SiO_2 and Al_2O_3. Inclusions of more complex composition, containing silicon, aluminum, magnesium, calcium, barium, and potassium together are eroded more easily. Metallic impurities (titanium, iron, zirconium, and others) are found in the solid solution or in negligible quantities in high-melting impurities. Along with carbides and oxides, there are some inclusions with hydrogen and nitrogen, probably of the type of carbonitrides, oxynitrides, or oxyhydrides.

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