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A Comparison of Grain-Boundary and Matrix-Crystalline Fracture in Ti-Mo Wires by Scanning Electron Microscopy

Experimental evidence as to the nature of grain-boundaries has been sought for more than a century using, for the most part, indirect methods which normally measure various properties of the material in question rather than the boundary itself. For many years, the grain-boundary was even considered to be a structureless, vitreous layer [1] having an unknown thickness. While the contemporary view of the grain-boundary as a transition from the lattice of one grain to that of the other was seriously considered some 40 years ago [2], this model has only recently been convincingly demonstrated experimentally [3-6]. The structure of grain-boundaries is not completely understood today [6-9], and in particular the boundary thickness is generally not known. A study of fracture uniquely associated with a grain-boundary (as compared with matrix fracture) may be expected to cast light on grain-boundary structure. Such a comparison is, however, difficult to achieve except for specially grown bicrystals, and there seem to be no reports of fracture comparisons of this type in the literature.

During routine operation of an ultra-high vacuum unit, titanium-molybdenum sublimation filaments which failed were observed to have crystallised into large-grained segments with grain-boundaries meeting the wire normal to the axis, as a result of high temperature strain-anneal. It was immediately recognised that because of the embrittlement of the strain-annealed wires, this accidental occurrence presented a good opportunity to study grain-boundary fracture in relation to the fracture of intracrystalline regions along the wire, using the scanning electron microscope. The present study outlines the results of a comparison of fractures in these wires.

High purity Ti-Mo sublimation pump filaments 1.5 to 2 mm in diameter were heated under normal gettering operations in high vacuum using an AC voltage. Under prolonged heating, grains averaging 2 mm in length, with boundaries approximately normal to the wire axis, were formed. Short-time anneals were also performed which produced a fine-grained polycrystalline structure with an average grain size of 0.4 mm.

With a wire section gripped with two pairs of needle-nose pliers, brittle fractures could be induced either at the grain-boundaries of the large-grained wires, or in the grain section between boundaries. The fine-grained wires also fractured in a semi-brittle manner, and served as a means of comparison with the crystalline fractures.

Observations of freshly cleaved surfaces were made in a Cambridge Stereoscan scanning electron microscope operated in the secondary electron mode, using an accelerating potential of 20 kV. Fracture surface orientations were identified using X-ray diffraction techniques.

Spectrographic analysis of various wire samples indicated a molybdenum content ranging from 10 to 14 wt %. 0.004 wt % Mg was observed as the sole impurity element. Electron probe microanalysis in the region of the grain-boundaries in a number of the samples also indicated a homogeneous alloy, with no noticeable segregation.

Fig. 1 shows a typical length from a Ti-Mo wire sample following crystallisation. It is to be noted that the grain-boundaries are aligned mostly normal to the local wire axis, and there are several examples of grain-boundary sliding. Cleavage fracture within the individual crystals was observed to occur along planes having an angle of approximately 60° to the local wire axis.

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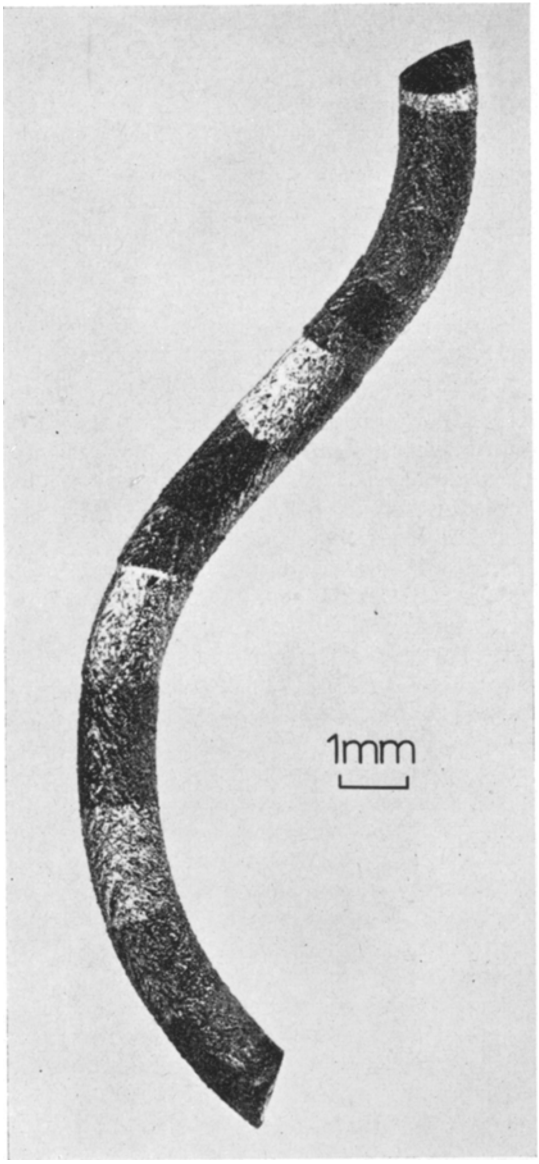

Figure 1 Optical micrograph of crystalline Ti-Mo wire. Grain-boundaries are clearly defined and there are several obvious examples of grain-boundary sliding.

Observations of the matrix fracture surfaces by Weissenberg X-ray diffraction photographs indicated the cleavage planes to be the (001) plane, and indicated the lattice structure to be body-centered cubic, with a lattice parameter of 3.26 ± 0.01 Å. This structure is consistent with the β-isomorphous phase [10]. In addition, electron microscopy of severely stressed wire cleavage slices also indicated some evidence of the α' (hcp) phase [10].

Fig. 2 illustrates the appearance of a wire sample fractured partially along the grain-boundary (top), with axial cleavage occurring at a later stage. The grain-boundary fracture surface is observed to be an exceptionally smooth fracture completely free of recognisable irregularities compared with the associated matrix fracture surfaces which are characterised by irregular or discontinuous cleavage along the $\{001\}$ planes. The grain-boundary fracture surface in fig. 2 was observed to be nearly exactly coincident with (111), and the wire axis was therefore coincident with $[111]$.

In fig. 3 is shown a magnified view of another grain-boundary fracture surface showing very fine irregularities which appear to be the result of stress concentrations or structural irregularities in the grain-boundary which give rise to a local detachment or cleavage of the lattice below the boundary plane. The continuity and exceptional smoothness of the grain-boundary cleavage fracture suggests that the grain-boundary has a fairly regular atomic arrangement. Since the boundary plane in fig. 3 was (320), and

Figure 2 Scanning electron micrograph of Ti-Mo crystalline wire sample fractured partially across a grain-boundary. The grain-boundary plane is observed on the extremely smooth area. The arrow indicates the normal to the boundary, coincident with [111] and the wire axis.

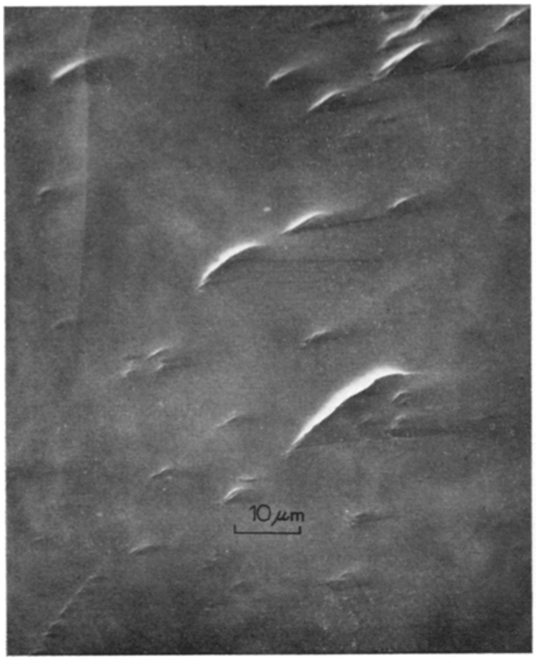

Figure 3 Scanning electron micrograph showing a magnified view of a typical grain-boundary fracture surface.

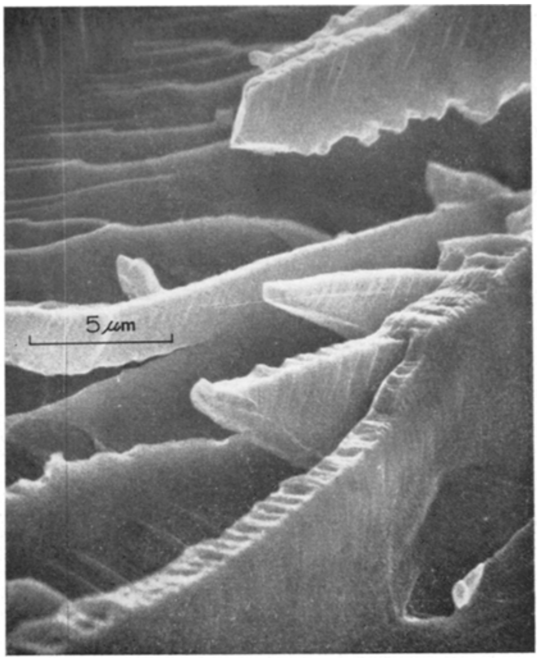

Figure 4 Scanning electron micrograph of matrix cleavage fracture in crystalline Ti-Mo wires.

bearing in mind that several other grain-boundary fracture surfaces were observed to be approximately (111) or (320) planes, it is suggested that the grain-boundaries may be essentially coincidence lattice planes having a high degree of perfection and uniformity. Unfortunately, it was not possible to measure the crystal rotations characterising the grain-boundaries, to check whether the boundaries were indeed of coincidence type [3].

While it is obvious from fig. 2 that the fracture surface associated with grain-boundary cleavage differs markedly from that of the matrix, fig. 4 amplifies this difference, and shows for comparison with fig. 3 a typical fracture surface associated with matrix cleavage. The angle of observation in fig. 4 is approximately 15° from a plane parallel to the cleavage plane (001) as compared with a more normal view of a grain-boundary fracture surface as shown in fig. 3. It is observed in fig. 4 that the brittle cleavage fracture is irregular, and not confined to large areas of a single (001) plane section. The fracture is characterised by cleavage flakes having a plate-like appearance as shown in fig. 4, with the surfaces of the plates coincident with the {001} planes.

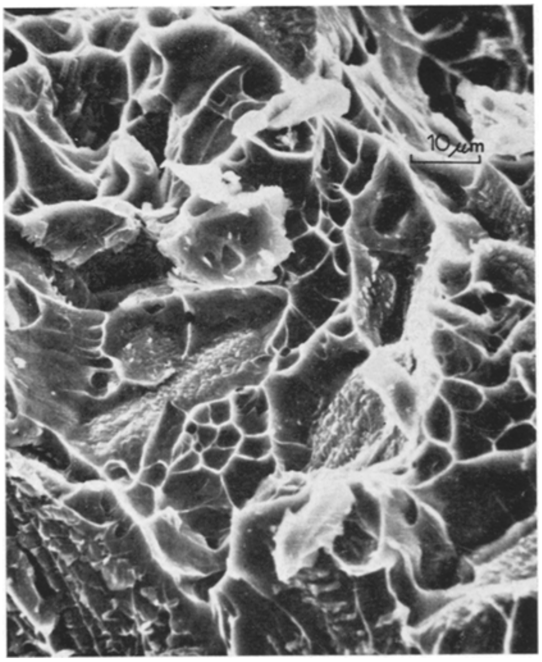

Figure 5 Scanning electron micrograph of typical polycrystalline fracture surfaces in Ti-Mo wires.

In fig. 5 is shown a typical view of the fracture surface for a fine-grained Ti-Mo wire sample. The cratered appearance of the surface in fig. 5 is indicative of a semi-ductile characteristic. The network-type appearance of the fracture cups in fig. 5 might possibly be associated with dislocation sub-boundaries, a suggestion which would be consistent with the ductile character associated with these features. It was impossible to associate these features with the actual grain-boundaries since the grain sizes were very much larger than any of these characteristics. Little evidence for crystallographic cleavage such as in fig. 4 is apparent in fig. 5, and plainly the degree of crystallographic perfection is considerably less than that in the highly annealed large crystal specimens, as shown in fig. 1.

If the reader will now examine in a comparative retrospect figs. 3 to 5, it should be obvious that distinctly autonomous characteristics exist for grain-boundary fracture surfaces, crystalline cleavage fracture surfaces in the matrix, and the fracture morphologies associated with fine-grained Ti-Mo wires. In this regard, it might be revealing to subject similar crystalline wires to corrosive atmospheres or related environmental features and to re-examine the associated fracture surfaces. In particular, the mechanism of grain-boundary stress corrosion might be elucidated by such a procedure, especially in the light of the presently demonstrated capabilities of the scanning electron microscope.

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