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Distinctive test for tungsten wires with different splitting properties

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Abstract

Deformation is often localized into split induced Lüders bands upon the free-end torsion of heavily drawn and annealed doped tungsten at the ambient. The Lüders bands nucleate at very low true shear strains ($\gamma < 0.01$), when the samples are annealed below 1400 °C. This work reports on the Lüders band nucleation in primary recrystallized samples (annealed at 1800 °C) having an elongated homogeneous fine grained structure. The deformation without mechanical damage increased ($0.09 < \gamma < 0.27$) in this condition, when the material was slightly softer and the grain width was slightly larger. The diffuse Lüders bands contained macro-cracks having a continuous helical trace on the free surface in the quasi homogenous part of the band. In addition to the macro-cracks, also a great number of micro-cracks were formed connecting grain boundary triple lines along the width of the elongated primary recrystallized grains, although many grain boundary segments of this type remained micro-crack-free. The results may be taken as a hint for the importance of the orientation dependent grain boundary decohesion as well as for the effects of higher incompatibility stresses at the vicinity of triple lines, when they are sufficiently close to each other.

Keywords: Doped tungsten; Split propensity; Grain boundary decohesion; Working damage; Incompatibility stresses

1. Introduction

It is of basic importance in the technology of incandescent filaments that properly processed heavily drawn tungsten can be coiled onto a fine mandrel at the ambient. Although this kind of formability is in agreement with the finding that the brittle to ductile transition temperature of heavily drawn tungsten is below −10 °C in bending [1], it is well established that coilability requires a more dedicated test. The most usual decision is to perform a standard coiling operation with a given mandrel to wire ratio. In addition, the coils of non-sag tungsten remain ductile with respect to stretching at the ambient also upon stress relief performed at 1500 °C. (In other words: non-sag tungsten preserves its torsional formability upon stress relief, as stretching of coils induce primarily torsion-like strains in the wire.)

This remarkable behaviour has been ascribed to the following microstructural features: (a) high drawing strains result in an elongated fibrous grain structure with ultra fine size [1] and (b) the grain structure remains fine and elongated also during stress relief, because the microstructural coarsening is very sluggish, as lamp grade tungsten is hardened through rows of ultra fine sized potassium bubbles [2].

It has long been known that the technological exploitation of this kind of deformation induced ductility is sometimes hindered by secondary effects. One may distinguish two sorts of them. The first group is closely connected with the break of coils at very low stretching strains initiated through chemically induced localized heterogeneity in the fibre structure [3,4]. The second group concerns decohesion along a chain of longitudinal grain boundaries. (This kind of cracking is often referred to as splitting in the industry.) The wires often split also upon coil manufacture or upon stretching of stress relieved coils. Splits often appear also

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in wires which have been split-free in "as-drawn" condition. Since it is typical that the splits do not yield to separation of the coils into distinct parts, the splitting upon coil manufacture or coil stretching should be considered as a peculiar from of working damage that is typical for tungsten having an elongated ultra fine or fine grain structure. It is also well known that the split propensity is sensitive to the details of the thermo-mechanical processing route [1,5].

The microstructural background of the split propensity of non-sag tungsten has been studied by means of "bend-and-stretch" test [6] and knife-edge compression test [7,8]. It turned out that the dispersed potassium phase plays only a minor direct role in splitting, while the role of the grain structure can be characterized [8–10] as follows:

(i) The correlation between split load and transversal mean linear intercept is less pronounced. Thus, the longitudinal boundaries should play only a secondary role in crack nucleation, although the major part of the macro-crack propagates along them.
(ii) The split load seems to be governed by the frequency of the transversal boundaries. Therefore, the *formation of transverse boundaries is considered as the primary reason for the marked decrease of split load upon stress relief anneals* conducted between 800 and 1200 °C.

The aim of the present work was to reveal the mechanism of fracture nucleation along the originally longitudinal grain boundaries by means of free-end torsion in case of heavily drawn and annealed doped tungsten having an elongated fine grained primary recrystallized microstructure.

2. Experimental

The evolution of the working damage has been followed up upon free-end torsion at the ambient. The deformation has been carried out either by means of an Amsler torsion testing machine or by means of a similar torsion tester without torque detection. The axial load of the sample amounted to 10% of its ultimate tensile stress. The samples were 50 mm long pieces of lamp grade tungsten having a diameter of 173 μm. The sample surface has been carefully cleaned through a two-step heat treatment. The first one was performed at 1200 °C for 10 min in high vacuum having an adjusted $P(O_2)$ pressure of 10^{-4} mbar, while the second one was carried out at 1300 °C or 1800 °C for 1 h in ultra high vacuum having a residual $P(O_2)$ partial pressure less than 10^{-12} mbar. These treatments were carried out in an ultra high vacuum chamber used for Auger electron spectroscopy. The behaviour of samples after an annealing at 1800 °C depended on the processing route. Therefore, we will refer to samples in this condition as sample A and B in order to stress the importance of their different proprietary thermo-mechanical processing routes. The proof stress at 1% conventional plastic strain in tensile test at the ambient amounted to 1.4 GPa and 1.6 GPa in batch A and B, respectively.

The dislocation density has been estimated by means of a dedicated high-resolution X-ray diffractometer for line profile analysis [11–13]. It amounted to 10^{10} cm^{-2}, when the annealing has been performed at temperatures from 1200 to 2500 °C [14]. The contrast of the grain boundaries on transmission electron micrographs was typical for equilibrium grain boundaries in the annealed samples (Fig. 1) [15]. We may, therefore, conclude that the microstructure of the annealed samples has the inevitable features of a primary recrystallized microstructure, although the aspect ratio of the grains is high (Fig. 2), due to the influence of the potassium bubbles rows on continuous primary recrystallization. The scanning electron micrograph in Fig. 2 shows the free surface of a B type sample that was sub-

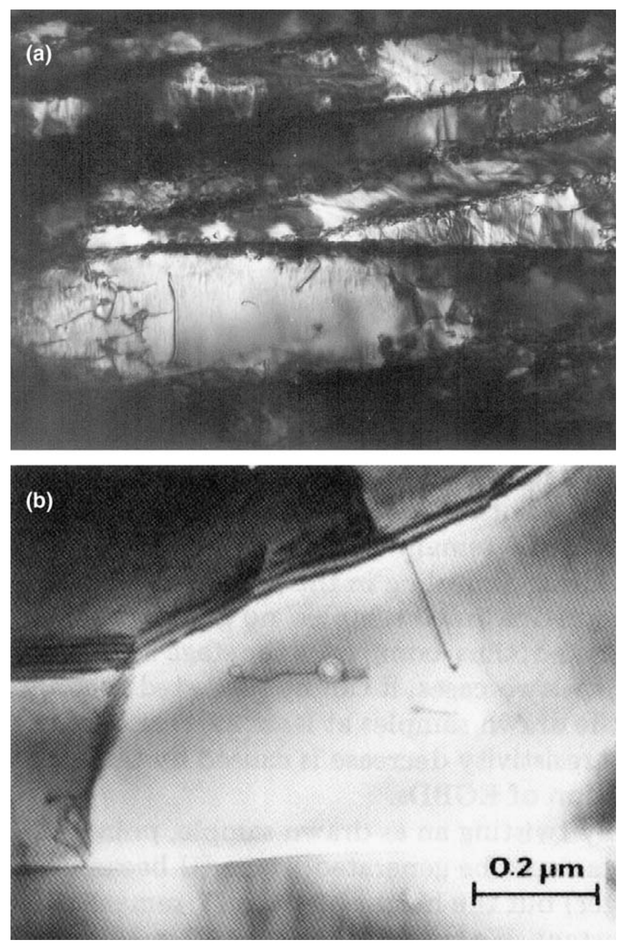

Fig. 1. The contrast of the boundaries on the transmission electron micrographs depends markedly on the annealing temperature. The boundaries are "thick", if the annealing temperature is below 1000 °C, while above this temperature the boundaries are strain free showing only contrast lines corresponding to equal depths below the free surfaces of the foil. The thick black contrast along the grain boundaries is due two kinds of dislocations (a) lattice dislocations accumulated along the grain boundaries and (b) lattice dislocations incorporated into the grain boundary as extrinsic grain boundary dislocations.