SHORT COMMUNICATIONS

-Hill, New York, 1953.

73.

Journal of the Less-Common Metals
Elsevier Sequoia S.A., Lausanne – Printed in The Netherlands

The effects of strain rate and pressurization on the ductile–brittle transition temperature of polycrystalline sintered tungsten

The ductility of polycrystalline body-centred cubic transition metals is well known to be sensitively dependent on temperature and many physical and metallurgical factors such as purity, grain size, strain rate and, for iron, chromium[1] and molybdenum[2], pressurization. The transition with decreasing temperature, T, from ductility to brittleness, defined as an absence of detectable macroscopic plastic deformation, we[3] shall refer to as *the* transition temperature, T_T. It is generally found that T_T increases with increasing strain rate, $\dot{\varepsilon}$, but a satisfactory formulation of this dependence based on theories of yielding and fracture does not appear to exist. Recently[4], we have postulated a phenomenological correlation between T_T and $\dot{\varepsilon}$ for molybdenum in which the transition occurs at a constant stress, σ_C. To apply this model it is also required that the stresses for yield, σ_Y, and brittle fracture, σ_F, are approximately continuous, known functions of T and $\dot{\varepsilon}$ in the transition region. The principal aim of this investigation was to see whether this simple model was applicable to sintered tungsten.

A difficulty in the study of brittle fracture of this material is the occurrence of "low-stress failures[5]". These are sometimes avoided (quite frequently at temperatures not more than 50°K below T_T) and brittle fractures take place at stresses at which it is predicted the material would yield, if it were ductile. At the transition temperature brittle fracture thus occurs at the yield stress and it appears that immediately below T_T this is, at least, approximately so.

Another aim of the investigation was to determine the effects, if any, of pressurization at 14 kbars on the ductile–brittle transition temperature. If polycrystalline specimens of the other Group VIA b.c.c. transition metals, chromium[1] and molybdenum[2], are subjected to a hydrostatic pressure of the order of 15 kbars irreversible effects on the mechanical properties at atmospheric pressure, *e.g.* lowering of T_T, are sometimes found.

Tensile test pieces of $\sim$18 mm gauge length and $\sim$2 mm gauge diameter were ground from two batches of $\frac{1}{4}$ in. diameter Sylvania sintered tungsten rod. One batch of over thirty specimens was used to investigate the effects of pressurization on T_T, and the other of over fifty specimens to study in detail the effect of strain rate on T_T. All the specimens were recrystallized to a grain diameter of $\sim 50\,\mu$ by heat treatments of 1 h at $\sim$1750°C in vacua of 10^{-6} torr. Then, before testing, the specimens were electropolished in a 2% NaOH solution at $\sim$9 V.

In tungsten the important impurities are thought to be the interstitials and accordingly analyses were carried out for oxygen, nitrogen, hydrogen and carbon. The analyses figures are: N_2: 1–2 p.p.m., H_2: < 1 p.p.m. and O_2: 3–8 p.p.m. by mass spectrometry (A.E.I.'s MS 10); O_2: 26–87 p.p.m. by conventional vacuum fusion; and C $\sim$1 p.p.m. by neutron activation.

The investigation of the effects of pressurization at 14 kbars (carried out by Harwood Engineering Co.) was performed on one batch of specimens on an Instron testing machine at strain rates of approximately 10^{-4}, 4×10^{-4}, 10^{-3}, 2×10^{-3} and 10^{-2} sec^{-1}. Further tests, on the other batch of specimens which were all not pressurized, were carried out on a Hedeby universal tester at strain rates of about 10^{-5}, 10^{-4}, 5×10^{-4}, 10^{-3}, 2×10^{-3}, 5×10^{-3} and 10^{-2} sec^{-1}. Temperatures, other than ambient, were obtained by surrounding the specimen with a heated vegetable or silicone oil, maintained at a temperature constant to within ±1°K during a test.

For all strain rates the increase in yield stress and the decrease in ductility with decreasing test temperature characteristic of the b.c.c. transition metals were observed. Yield points were exhibited by only 17 of the 30 "definitely ductile" specimens of both batches and for the ductile specimens not showing a yield point the stress at the apparent proportional limit was taken to be the yield stress. For tests carried out at, especially, the lower strain rates difficulty was experienced in deciding from the autographic charts whether a specimen was "just ductile" or brittle. There were 28 "definitely brittle" specimens and 19 in the former category. As we have defined T_T as the lowest temperature at which macroscopic plastic deformation is detectable, our values of T_T, shown plotted as a function of $\dot{\varepsilon}$ in Fig. 1, are the upper limits of the transition temperatures. The values of the transition stress were 53, 57, 55, 55, 54, 61 and 57 kg mm^{-2} at strain rates of 10^{-5}, 10^{-4}, 5×10^{-4}, 10^{-3}, 2×10^{-3}, 5×10^{-3} and 10^{-2} sec^{-1} respectively, *i.e.* $57\ \pm4$kg mm^{-2}.

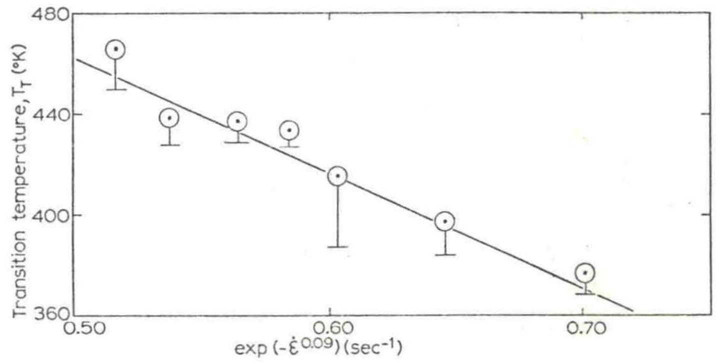


Fig. 1. The dependence of the ductile–brittle transition temperature, T_T, on strain rate, $\dot{\varepsilon}$, in recrystallized sintered tungsten of mean grain diameter 50μ.

The pressurization treatment failed to produce detectable irreversible changes in either the yield stress or the transition temperature at the 5 strain rates and accordingly it is concluded that 14 kbars is insufficient to alter irreversibly the mechanical properties of our sintered polycrystalline Sylvania tungsten. Hereafter no distinction will therefore be drawn between pressurized and unpressurized specimens. Tests on cast[6] specimens pressurized at ~30 kbars and on sintered (American) General Electric specimens[7] pressurized at 25 kbars also failed to reveal any effect.

Metallographic examination revealed non-propagating cracks, away from the fracture surfaces, only in ductile specimens. These microcracks appeared to be located mainly in the surface layer, $\sim100\ \mu$ deep, and were predominantly intergranular in character (Fig. 2). The size of these cracks ranged from $\sim20\ \mu$ to $\sim150\ \mu$, *i.e.* several grain diameters.

In this polycrystalline sintered tungsten the transition from ductile to brittle behaviour appears to occur at a constant stress of 57 ± 4 kg mm^{-2} for strain rates from 10^{-5} to 10^{-2} sec^{-1} as the transition temperature is raised from $\sim377°$ to $\sim465°$K.

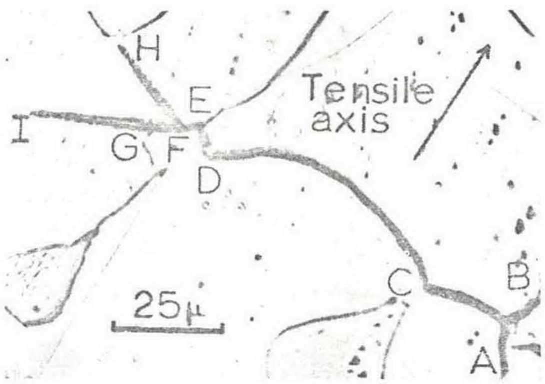
The existence of a constant stress, σ_C, at the transition enabled us to carry out an analysis of the dependence of T_T on the strain rate, $\dot{\varepsilon}$. Let us, as WRONSKI *et al.*[4] have done for molybdenum, assume relationships between stress and temperature[5] and strain rate[8], respectively, of the form:

$$\sigma_Y = A - BT \tag{1}$$

and

$$\sigma_Y = E\dot{\varepsilon}^F \tag{2}$$

where A and B are constant at a given strain rate and E and F are constant at a given temperature. B is approximately equal to 0.3 kg mm^{-2} °K^{-1} for our material at all strain rates in the transition region and E and F have been evaluated for sintered tungsten at 473°K to be ~68 kg mm^{-2} and ~0.09, respectively[8]. If we also



Composite micrograph of a curved surface.

Fig. 2. Non-propagating surface crack in a recrystallized sintered tungsten specimen which cleaved at 442°K at a strain rate of 5×10^{-3} sec^{-1} after 0.6% plastic deformation. Note that the crack follows grain boundaries along AB, AC, CDEF, FH, FG and is transgranular only along the segment GI. (In order to avoid etch-pitting, Murakami's reagent, which is a relatively poor etchant for tungsten, was employed.)

assume that (in the transition region) F is a constant, that the variation of E with temperature is given by eqn. (1), and make use of the identity:

$$\left(\frac{\partial\sigma}{\partial\dot{\varepsilon}}\right)_T \left(\frac{\partial T}{\partial\sigma}\right)_{\dot{\varepsilon}} \left(\frac{\partial\dot{\varepsilon}}{\partial T}\right)_\sigma = -1 \tag{3}$$

we derive for the relationship between T and $\dot{\varepsilon}$, for constant σ the expression:

$$\exp(-\dot{\varepsilon}^{0.09}) = K(E_0 - 0.3T_T) \tag{4}$$

where K and E_0 are constants. Figure 1 shows T_T plotted against $\exp(-\dot{\varepsilon}^{0.09})$ and it is seen that, although not as good for molybdenum[4], there is a fair agreement between the data and the model.

The constant stress at the transition temperature suggests that this is a critical stress for some mechanism in the fracture process. It appears that the transition coincides with a change in the critical stage in this process, a hypothesis supported by the observation of microcracks in ductile but not in brittle specimens. If the cracks observed in the ductile region are of the type that cause fracture, crack propagation is the critical stage in ductile cleavage. Because microcracks are not observed in the brittle specimens away from the fracture surfaces it appears quite possible that the first crack to be nucleated will propagate.

If, at T_T, stresses for crack nucleation, σ_N, and propagation, σ_P, are equal and equal to the yield stress, then:

$$\sigma_C=\sigma_N=\sigma_O+k_N(Y\gamma)^{\frac{1}{2}}l_C^{-\frac{1}{2}}=\sigma_P=k_P(Y\gamma_B)^{\frac{1}{2}}l_C^{-\frac{1}{2}} \tag{5}$$

where σ_O, k_N, k_P are constants, Y is Young's modulus (4.04×10^{12} dyne cm^{-2}), γ and γ_B are the surface energies for crack nucleation (true surface energy) and crack propagation across a grain boundary respectively, and l_C the crack length, which is probably related to the grain diameter. Let us assume that l_C equals the grain diameter, l, and that k_P is $(2/\pi)^{\frac{1}{2}}$, which is given by the simplest (Griffith–Orowan–Irwin) model of surface crack propagation. γ_B then evaluates to $\sim 6 \times 10^4$ erg cm^{-2}, which compares reasonably with surface energy values estimated by HULL *et al.*[9], *e.g.* $\sim 3 \times 10^4$ erg cm^{-2} at 350°K for propagation of cracks in tungsten single crystals, in which blunting of the cracks is thought to occur above $\sim$150°K.

Below 150°K their values of γ were near the theoretical estimates, *e.g.* on GILMAN's model[10]: 4.7×10^3 and 3.3×10^3 erg cm^{-2} for {100} and {110} cleavage, respectively[11]. Assuming the smaller value to be correct and taking[12] k_N to be $\sim$2.4 it is also possible from eqn. (5) to estimate the lattice friction stress, σ_O. It is seen to be $\sim$22 kg mm^{-2}, which is in excellent agreement with the value estimated by HULL *et al.*[9] at a strain rate of 5×10^{-4} sec^{-1} using a different analysis.

The work described forms part of an investigation supported by the Science Research Council. It was initiated with Prof. A. A. JOHNSON by one of us (ASW) during the tenure of a visiting professorship at the Polytechnic Institute of Brooklyn. The award was made as part of the Polytechnic's Science Development Programme founded by the National Science Foundation. The authors acknowledge the provision of laboratory facilities at Bradford by Prof. D. L. SMARE and ACC the award of a University of Bradford scholarship. The investigation was made possible through the skill of Mr. L. BOYAJIAN and Mr. E. JEFFREY who ground the tensile specimens.

School of Materials Science, A. C. CHILTON
University of Bradford, A. S. WRONSKI
Bradford (Gt. Britain)

1 F. B. BULLEN AND H. L. WAIN, in A. C. STICKLAND (ed.), *Physical Basis of Yield and Fracture*, Inst. of Phys. and Phys. Soc., 1966, p. 60.
2 H. G. MELLOR AND A. S. WRONSKI, *Ocean Eng.*, in press.
3 A. S. WRONSKI AND H. G. MELLOR, *J. Inst. Metals*, 97 (1969) 63.
4 A. S. WRONSKI, A. C. CHILTON AND E. M. CAPRON, *Acta Met.*, *17* (1969) in press.
5 A. WRONSKI AND A. FOURDEUX, *J. Less-Common Metals*, *8* (1965) 149.
6 H. G. MELLOR AND A. S. WRONSKI, unpublished results, 1968.
7 G. DAS AND S. V. RADCLIFFE, *Trans. Japan. Inst. Metals*, 9 (Suppl) (1968) 334.
8 A. WRONSKI AND A. FOURDEUX, *Phil. Mag.*, *10* (1964) 969.
9 D. HULL, P. BEARDMORE AND A. P. VALENTINE, *Tech Rept. A.F.M.L.-TR-66-369*, Vol. II, 1967.
10 J. J. GILMAN, in G. T. HAHN (ed.), *Fracture*, Wiley, London, 1959, p. 193.
11 A. WRONSKI AND A. FOURDEUX, *J. Less-Common Metals*, *6* (1964) 413.
12 E. SMITH, *Acta Met.*, *14* (1966) 985.

Received November 13th, 1968