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on the Strength of Metals and Alloys

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Supplement
to
Transactions of the Japan Institute of Metals
Vol. 9, 1968

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The effects of the application of hydrostatic pressure on the dislocation substructure and mechanical behavior observed subsequently at atmospheric pressure, and the influence of high pressure on flow and fracture have been investigated for polycrystalline powder metallurgy tungsten and similar material containing second phase additions of thoria and hafnium carbide with the principal objective of elucidating the factors controlling the pressure dependence of the mechanical behavior of a brittle *bcc* metal. Tensile tests were conducted from 25 to 250°C at atmospheric pressure on specimens subjected previously to pressures up to 25 kilobars and at room temperature during subjection to pressures up to 11 kilobars. The sub-structures were examined by optical and transmission electron microscopy.

The ductile-brittle transition behavior of recrystallised tungsten and a tungsten-1.9 vol% thoria alloy are essentially unaffected by pressure cycling up to 25 kilobars at atmospheric temperature. The results for tungsten are in keeping with the scarcity of impurity particles observed and the corresponding absence of pressure-induced dislocations. In tungsten containing additions of second phase particles (ThO_2 or HfC) a similar absence of induced dislocations was found for pressures up to 25 kilobars, but new dislocations were observed at particles after subjection to pressures of some 40 kilobars.

The stress-strain behavior of recrystallised tungsten at constant pressure and atmospheric temperature changes progressively as the environmental pressure is increased. Initially, fracture with no measurable plastic deformation persists, but the fracture stress increases with increasing pressure and, at sufficiently high pressures, a distinct yield drop followed by a substantial plastic deformation and work hardening is developed. The magnitude of the resulting lower yield stress (96,000 psi) is in agreement with the extrapolation to room temperature of the temperature dependence of the yield stress of recrystallized powder metallurgy tungsten as measured at higher temperatures at atmospheric pressure by several different workers. The dislocation substructures developed during plastic straining at high pressure are different from those reported previously for polycrystalline tungsten subjected to similar amounts of plastic strain at higher temperature and atmospheric pressure and the fracture mode is a combination of intergranular separation and transgranular cleavage. The implications of these results for the interpretation of the discontinuous yield phenomena in tungsten and the fracture process are discussed.

I. Introduction

When a single crystal or polycrystalline solid is subjected to external hydrostatic pressure, differential strains will be induced at any localized elastic discontinuities, such as precipitates, impurity particles or internal voids, due to their different compressibilities relative to the matrix. Although the calculated differential stresses in the region of the interface with the matrix appear too small to cause local plastic deformation, transmission electron microscopy observations on iron[1] and chromium[2] have demonstrated that such pressure-induced dislocations can be formed. The influence of these and other types of elastic discontinuity in inducing permanent changes in substructure and associated changes in mechanical behavior on subjection to hydrostatic pressure have been reviewed recently[3]. Such permanent changes have been designated variously as pressure cycling, pressurizing or pressurization effects.

The general effect of a superimposed hydrostatic pressure in enabling "brittle" materials to undergo substantial plastic deformation before fracture under uniaxial loading at pressure was established by Bridgman[4][5] but detailed studies of specific metals have been made only recently. Furthermore, as information is usually lacking as to the precise characteristics of the metallurgical history and structure of the metals studied, it is difficult to make valid comparisons between the results of different workers. Attention to the discontinuous nature of the increase in ductility which can occur in some metals with increasing hydrostatic pressure was first drawn to the non-cubic metals zinc and bismuth by Pugh and coworkers[6]. In the case of the body centered cubic metals (*bcc*), in several of which the nature of the

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(1) S. V. Radcliffe and H. Warlimont : Phys. Stat. Solidi, **7** (1964), 67.

(2) R. F. Garrod and H. L. Wain : J. Less-Common Metals, **9** (1965), 81.

(3) S. V. Radcliffe : *Structural Effects in Materials under Pressure*, Chap. 11 in *Mechanical Behavior of Materials Under Pressure* ed. H. Ll. D. Pugh, Elsevier Publishing Co., Amsterdam—in press.

(4) P. W. Bridgman : *Studies in Large Plastic Flow and Fracture*, McGraw Hill Co., New York, (1952).

(5) P. W. Bridgman : J. Appl. Phys., **24** (1953), 560.

(6) (a) H. Ll. D. Pugh and D. Green : *Behavior of Metals Under High Hydrostatic Pressure*, MERL Plasticity Rep. No. 128, National Engineering Laboratory, (1956).

(b) H. Ll. D. Pugh, J. Lees, K. Ashcroft and D. Gunn : Engineer (G. B.), **212** (1961), 258.

temperature dependent transition from ductile to brittle behavior is well established and partially understood, analogous pressure dependent transitions have been observed for molybdenum[7], chromium[10], and tungsten[5][8]~[11].

The above observations direct attention to the problem of the inter-relationship of the changes in ductility with pressure at room temperature and with temperature at atmospheric pressure. Unfortunately, the available data for tungsten are too limited for effective analysis—in particular, comparison between the various results and with the properties of tungsten at atmospheric pressure is difficult because of the different conditions and sources of tungsten used. Furthermore, the possible contribution of substructural changes arising during application of pressure on the behavior of *bcc* metals which are brittle at room temperature have not been examined in any detail with the exception of chrominum[12].

Accordingly, the present investigation was undertaken with the principal objectives of elucidating the effects of the simple application of hydrostatic pressure and the influence of pressure on plastic yielding and fracture at room temperature for polycrystalline tungsten as a metal of isotropic linear compressibility which is brittle under ambient conditions. Attention has been directed to working with recrystallized powder metallurgy (PM) tungsten of known history and characterised structure and to precise measurement of the pressure dependence of the tensile behavior, particularly with respect to possible discontinuous yielding at room temperature, a phenomena which cannot be investigated in recrystallised tungsten under ambient conditions due to premature fracture.

II. Materials and Proecdures

For the pressure cycling experiments, PM tungsten (99.9 % purity; Refractory metals Division, General Electric Co.) was obtained in the form of as-drawn wire which had been surface ground to 0.030 in diameter. Similar material containing additions of thoria (0.5 and 0.9 wt % ThO_2 i. e. 0.9 and 1.7 vol %) to provide controlled amounts and distributions of elastic discontinuities in the form of particles was obtained from the same source. In addition, two electron-beam melted alloys containing 1.4 and 0.4 vol % hafnium carbide were obtained in the form of 0.025″ in thick sheet (Lewis Research Center, NASA) which had been solution treated and cooled so as to precipitate the carbide as fine particles. For the tensile tests at atmospheric temperature and high pressure, PM tungsten was obtained from the same source as for the wire in the form of as-swaged rod, surface ground to 0.31 in diameter. Annealing and recrystallisation was carried out in a tungsten crucible in a tantalum strip furnace under a vacuum of 10^{-5} mm Hg. The wire specimens were annealed in batches of up to 50 to insure uniformity of treatment.

The subjection to single pressure cycles up to 25 kilobars for the metallographic and wire tensile specimens, was carried out as described previously[1]. For higher pressures, a stainless steel capsule filled with isopentane was used in a MIA-1 (hybrid belt or conical type) apparatus.

Tensile specimens were prepared from the annealed wires by electro-machining (1 in gage length) and tensile tests were carried out from room temperature to 200°C on an Instron machine at a constant crosshead speed of 0.025 in per min.

For the tensile tests at high pressure and room temperature, specimens of the button-end type (gage length 0.6 in and diameter 0.15 in) were prepared from the rod by centerless grinding. The specimens were recrystallized by annealing in vacuum at 2200°C for 1 hour; the resulting equiaxed grain size was 0.05 to 0.10 mm diameter. Subsequently, the specimens were electro-polished to remove any surface damage. The tests were conducted in a constant pressure tensile apparatus of the same basic design as that developed by Pugh and co-workers[6]. The apparatus, which is essentially a constant strain rate tensile machine contained within a high pressure chamber. The load on the specimen is measured by an internal load cell and the elongation and the reduction of area of the specimen are recorded as the test proceeds. The pressure fluid used was a solution of 10 % methyl alcohol in castor oil. A few experiments were conducted in the isopentane/*n*-pentane mixture. The strain rate was that corresponding to that of a crosshead speed of 0.003 in min^{-1} (0.005 min^{-1}).

Thin foils suitable for electron transmission microscopy were prepared from the larger tensile specimens by sparkmachining transverse discs approximately 0.010 in thick and 0.125 in diameter, followed by electropolishing. In the case of the 0.030 in diameter wires of the tungsten and the two-phase thoria alloys, a technique for foil preparation from longitudinal sections was developed in which uses a pressurised jet of electrolyte and precise positioning and oscillation of the specimen to give large areas suitable for electron transmission. The various foils were examined in a JEM 6A electron microscope using a goniometer stage (±20° tilt, 360° rotation) and operated at 100 kV. To minimise contamination problems, a 400 micron condenser aperture was used in conjunction with a useful beam current of 100 μA.

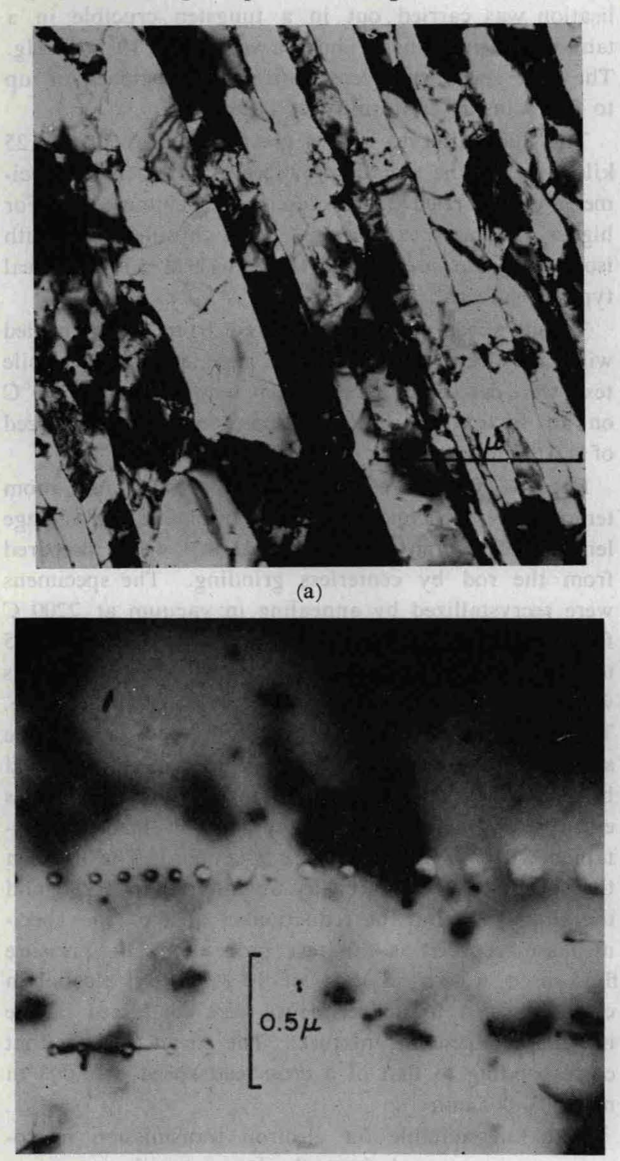
III. Results and Discussion

1. Recrystallisation behavior

For the tungsten wire, the successive changes in the initially "fibered" substructure with increase in temperature of isochronal (30 min) annealing were found

(7) J. R. Galli and P. Gibbs : Acta Met., **7** (1964), 775.
(8) A. Bobrowsky : Paper 64-WA/PT-29, *Symposium on High Pressure Technology*, Am. Soc. Mech. Eng., New York, 1965.
(9) T. E. Davidson, J. C. Uy and A. P. Less : Acta Met., **14** (1966), 937.
(10) L. D. Livshitz, Y. N. Ryabinin and B. I. Beresnev : Soviet Phys.-Tech. Phys., **10** (1965), 278.
(11) H. Ll. D. Pugh : Bulleid Memorial Lectures, Nottingham University Press, 1965.
(12) F. P. Bullen, F. Henderson and H. L. Wain : Phil. Mag., **9** (1964), 803.

to be in good agreement with those reported previously for temperatures up to 1600°C[13]—see Fig. 1(a). At higher annealing temperatures, migration of certain of



(a)

(b)

Fig. 1 Thin foil electron micrographs of longitudinal sections of PM tungsten wire (0.030 in diam) illustrating substructure developed on annealing at (a) 1000°C, and (b) 2200°C.

the boundaries of segmented fibers continues and the dislocation density within the grains becomes very low, although occasional isolated fragments of hexagonal networks still occur. By 2000°C, the optical grain size reaches 50 microns and remains essentially constant up to 2600°C, the highest temperature examined. The grains remain elongated up to that temperature, although the length to width ratio diminishes substantially.

Extensive searches for impurity second phase particles were carried out at all stages of annealing of the powder metallurgy tungsten. Such particles were observed very rarely and always in grain boundaries. In contrast with the few particles, a substructural feature which developed widely and increasingly with increasing annealing temperatures was the appearance of parallel rows of small features which were identified from electron diffraction contrast experiments as small voids within the foils (Fig. 1(b). Further discussion of the origin of this feature will appear elsewhere.

For the tungsten—1 wt % thoria alloy, secondary recrystallisation is incomplete after annealing at 2000°C, although the dislocation density within the grains is low. The thoria particles vary considerably in distribution, size and shape; the longest ones ('rod' shaped and approximately 1 micron long by 0.5 micron wide) tend to be aligned with their length parallel to the direction of the wire axis. Occasionally, the shape and spacing of adjacent particles indicated that they represented large original particles presumably fractured during the wire processing. At 2600°C, the matrix grains are larger and more equiaxed, with some indication of a less oriented array of rod particles i. e., of rearrangement of the particles. The thoria particles in the recrystallized 0.5 wt % ThO_2 alloy also exhibit a wide range of size, but are much more rounded in shape. The thin-foil structure of the precipitated W-HfC alloys exhibited a large grain size and low dislocation density in a similar manner to the thoria alloys.

2. Terminal behavior after subjection to hydrostatic pressure

Tensile tests at atmospheric pressure and temperatures from 25° to 250°C on tungsten wire specimens annealed at 1310°, 1600°, and 2200°C were conducted before and after subjection of the wires to pressures up to 25 kilobars. The lower annealing temperatures were included to examine the possible influence of initial dislocation density and distribution on pressure-induced

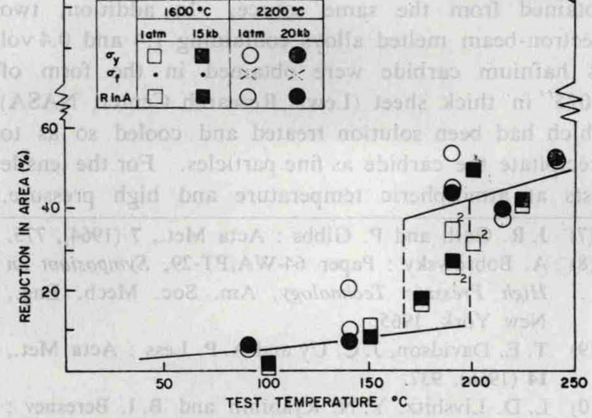


Fig. 2 Temperature dependence of yield stress, fracture stress and reduction in area for PM tungsten annealed at indicated temperatures and cycled to indicated pressures. All tests conducted at atmospheric pressure.

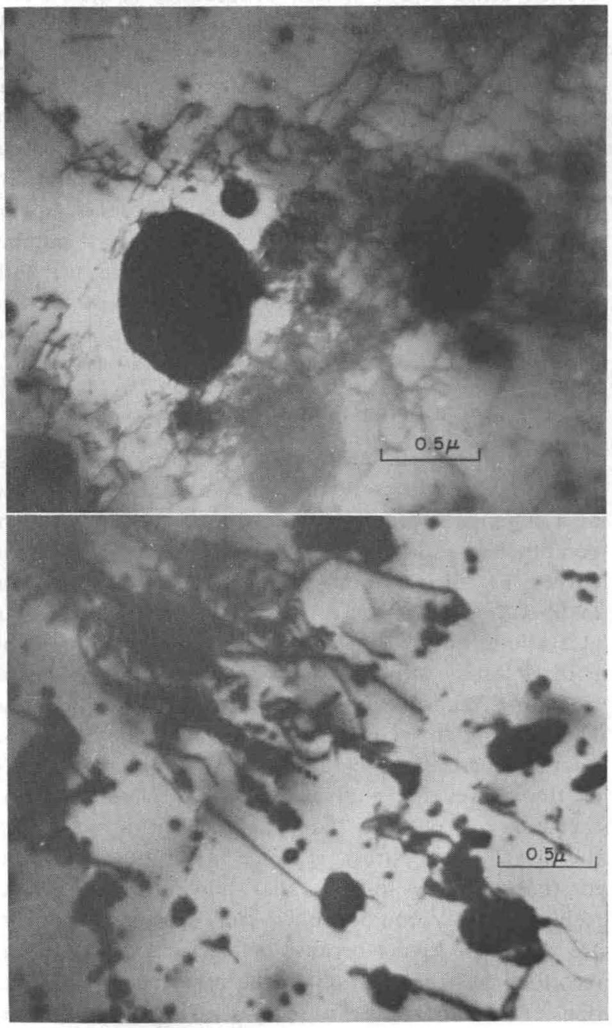
(13) E. S. Meieran and D. A. Thomas : Trans. AIME, **233** (1965), 937.
(14) A. Wronski and A. Fourdeux : J. Less-Common Metals, **8** (1965), 149.

changes in structure and mechanical behavior. Similar tests were made on W-1 wt % ThO_2 specimens annealed at 2000°, 2200° and 2600°C. The results for these various conditions of both materials show that the tensile stress-strain behavior and the ductile-brittle transition temperature, T_d, for a given condition are unaffected, within the accuracy of measurement, by subjection to pressures in the range used here. The nature of the results is illustrated in Fig. 2 by those for tungsten annealed at 1600° and 2200°C.

For the PM tungsten, a response to pressurizing had been expected if appropriate elastic discontinuities (particles or voids) were present and sufficient pressure could be applied to induce dislocation-generation by differential compression between the discontinuity and the matrix[3]. However, in the case of the particular material used here, the structure seen in thin foils showed that the only possible sources of elastic discontinuity (since second phase particles were almost completely absent) were the strings of small voids or gas bubbles. In many instances, the voids in the as-recrystallized tungsten were associated with isolated dislocations or hexagonal networks impeding the movement of dislocations. From dislocation contrast experiments on the larger voids, a few examples were seen of loop segments close to the void-matrix interface and it appears likely that these may have formed as the result of local plastic strain to relieve the complex stresses associated with the void growth. However, despite the existence of the voids acting as small elastic discontinuities, no evidence of additional pressure-induced dislocations was found in foils from the pressurized tungsten. In the case of foils prepared from the recrystallized and pressured W-1 wt % ThO_2 alloy, again no new dislocatins were observed to have formed. Thus, the mechanical behavior and substructural characteristics resulting from subjection to pressures up to 25 kilobars are in accord.

While it was understandable that the voids in the 'pure' tungsten could be too small to result in sufficient local differential strain, the absence of pressure-induced changes in the tungsten containing 1 wt % (1.9 vol %) ThO_2 was more surprising, in view of the maximum in the yield suppression as a function of the amount of second phase observed in Fe-Fe_3C alloys containing between 1 and 2 vol % of carbide[1][3]. Although the morphology and distribution of the second phase, in addition to its volume proportion, has been shown to be an important factor[1][3], it appeared most likely that in the present case, the applied pressure was too low in relation to the differential compression of the *W* and ThO_2, and the flow stress of the matrix—leading to no or insufficient generation of mobile dislocations.

It is of interest to note here that in a recent limited study[15] of the effects of pressure cycling up to 15 kilobars on the ductile-brittle transition temperature, T_d, in bend specimens of annealed commercial purity PM tungsten sheet and a tungsten −0.5 % hafnium −0.02 % carbon alloy sheet, decreases in T_d of 25° and 50°C, respectively, were reported. While the 25°C change is close to the accuracy with which changes in T_d can be measured, the larger change appears significant. In the present work, transmission microscopy examination of "high purity" PM tungsten from different manufacturers has demonstrated some variation in the impurity particle content, although it appears probable from the above results that the range of variation is in general insufficient to lead to any major change in the mechanical behavior of tungsten as the result of pressure cycling. However, it should be recalled that Bullen and co-workers[12][16] have demonstrated that a substantial increase in the plastic strain to fracture can occur after pressurization to only 10 kilobars for the normally brittle *bcc* metal chromium if certain impurity particles are present under what appear to be rather critical conditions. While the most probable reason for the much stronger effects of the simple application of pressure on the properties of iron and chromium compared with tungsten is the lower flow stress of the matrix, this deduction cannot be verified in the absence of specific knowledge of the relative compressibilities of the various phases involved.



(a)
(b)

Fig. 3 Thin foil electron monographs showing dislocation arrays induced in (a) 0.9 vol % ThO_2 alloy, and (b) 1.4 vol % HfC alloy after a pressure cycle to 40 kilobars.

(15) A. C. Schaffhauser : Annual Progress Rept, ORNL-3870 (1966), Oak Ridge National Laboratory, U. S. Atomic Energy Commission.

(16) F. P. Bullen and H. L. Wain : *Yield and Fracture*, (Oxford Conference, Sept. 1966) Institute of Physics and Physical Society, London (1967), p. 60.

In order to examine further the influence of the proportion and type of second phase particles and the maximum applied pressure on the response of tungsten to pressure cycling, specimens containing 0.9 and 1.7 vol % ThO_2, and 0.4 and 1.4 vol % HfC were subjected to pressures up to some 40 kilobars. Due to the restricted size of the specimen chamber available for the higher pressures, the observations of any effects were limited to transmission electron microscopy. The results showed that no changes in dislocation substructure were developed in any of these two-phase alloys for pressures up to 25 kilobars. The result for the 0.4 vol % HfC alloys is unexpected in view of Schaffhauser's observation of a decrease in T_d for an alloy of similar composition after subjection to only 15 kilobars. A possible explanation for the apparent disagreement may lie in a different precipitate distribution and amount, since these would depend on the particular thermal treatment adopted. In contrast to the results for the lower pressures, the two alloys which were subjected to a pressure of approximately 40 kilobars—0.9 vol % ThO_2 and 1.4 % HfC—both exhibited dense arrays of new dislocations in the vicinity of the second-phase particles, as illustrated in Fig. 3. The arrays at the thoria particles are distributed fairly uniformly and locally, in a manner simillar to that observed previously for carbide particles in iron[1]. In contrast, at the hafnium carbide particles the dislocations show many helical and loop arrays which are unidirectional. The fact that these differences in the type of dislocation array persist for the small number of particles similar size which are visible in the foils indicates that the different compressibilities of the two compounds (both of cubic structure) plays a significant role in determining the nature of the array formed.

3. Tensile behavior of tungsten at pressure

The results of the measurements of the tensile stress-strain characteristics of the recrystallized PM tungsten as a function of environmental pressure at room temperature are shown in Fig. 4 and 5. The stress-strain curves (Fig. 4) show only an increase in elastic strain to fracture with increase in pressure to 3 kilobars, but at 5 kilobars discontinuous yielding occurs followed by both plastic straining and work-hardening before fracture. With further increase in pressure to 8 and 11 kilobars, the yield drop persists and the strain to fracture increases progressively. The reproducibility of the curves for a given pressure is close—with the exception of one of the 8 kilobar runs in which the lower yield stress is some 4 % less than that for the two other tests at that pressure. (While the results for only one of the two runs made at 11 kilobars are shown in Fig. 4 because of electrical faults in the recording instrumentation in the other run, the reductions of area at fracture were in good agreement). The reduction of area at fracture increases correspondingly with pressure above 3 kilobars (Fig. 5) with no indication of a transition from brittle to ductile behavior over the pressure range investigated. Also shown in Fig. 5 are the data reported previously[5][8][11] for the ductility of 'tungsten' as a function of pressure. The ductile-brittle transition indicated by these various data in the pressure range from 7 to 9 kilobars suggests that, in an analogous manner to the ductile-brittle

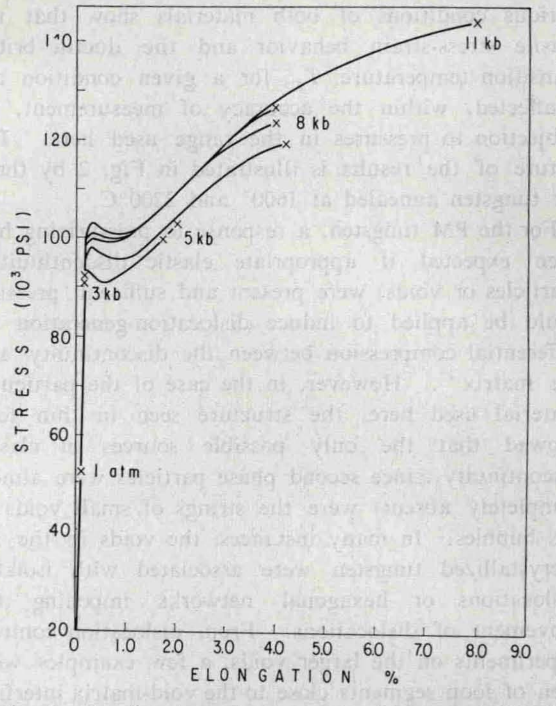


Fig. 4 Tensile stress-strain curves for recrystallised PM-tungsten as a function of environmental pressure at room temperature.

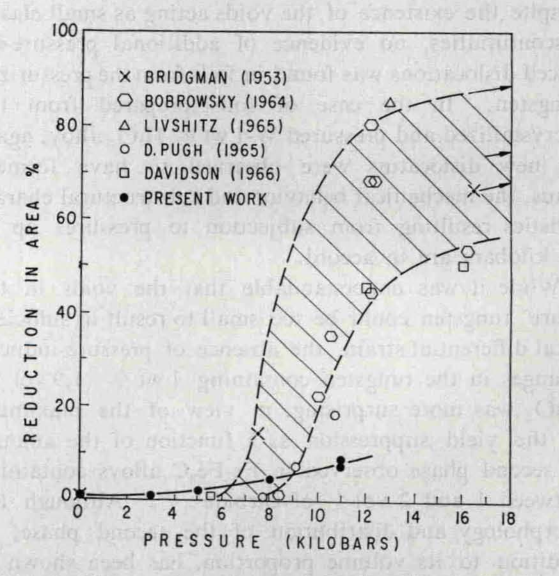


Fig. 5 Pressure dependence of the ductility (reduction in area) of recrystallised PM tungsten. The data reported previously for tungsten is collated in the figure and their range of values indicated by a band.

transition temperature for tungsten, the transition pressure is higher for the recrystallized material. The much lower yield stress measured in the present work some—96000 psi—compared with three previously reported values of 160000[5], 192000[11] and 210000[8] for 'tungsten' at similar pressures is a further indication of the probable unrecrystallized condition of the earlier material.

The observed variation of the lower yield stress with test pressure (Fig. 6) exhibits a slight upwards trend, although the data are represented here as lying within ±5000 psi of a mean constant value of 96000 psi. A small increase in yield stress with pressure for tungsten is qualitatively in keeping with the decrease in screw dislocation mobility with pressure reported recently[17] for lithium fluoride and shown to be consistent with jog control of the mobility. While the mechanism(s) of yielding in tungsten are currently uncertain—in particular as to the cause of the strong dependence of the macroscopic yield stress for single crystals on their orientation and the sign of the applied stress[18][19]—the jog-controlled mechanism proposed by Rose et al.[20] to account for the orientation dependence is also in accordance with the trend in yield stress with pressure observed here. Further clarification of this point is expected to result from the study currently in progress of the pressure dependence of the tensile behavior of higher purity, arc-melted tungsten in which the influence of the grain boundaries (as discussed later) should be less prominent.

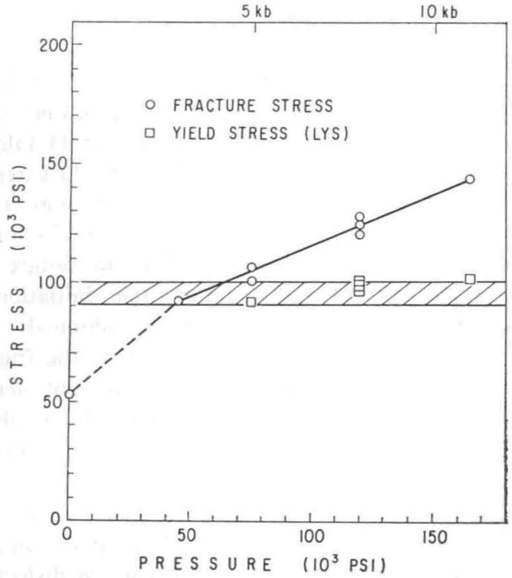


Fig. 6 Pressure dependence of yield and fracture stress in tension of recrystallised PM tungsten.

Although Fig. 6 indicates that the fracture stress initially increases rapidly with pressure to 3 kilobars by an amount similar to the increase in applied hydrostatic stress, the fact that brittle fracture in tungsten at atmospheric pressure is well known to occur over a wide range of stress values suggests that this simple pressure dependence is only apparent. Beyond 3 kilobars, i.e., beyond the onset of plastic strain, the fracture stress increases linearly with pressure and at a rate much larger than for the accompanying change in yield stress. As the increase in fracture stress is less than the corresponding increase in hydrostatic stress, the concept of a constant net stress (the difference between applied unaxial tensile stress and the hydrostatic compressive stress) as a general criterion for fracture[7][11] does not hold for PM tungsten.

The temperature dependence of the yield stress in tension cannot be measured in recrystallized PM tungsten below some 150°C at atmospheric pressure due to the onset of brittle fracture, but measurements have been reported for the compression yield stress down to −196°C. The various yield stress values which have been published[14][21]~[26] are plotted in Fig. 7. The smooth curve represents the mean of the scatter band (omitted for clarity) which encompasses the data points and it is seen that the temperature dependence appears continuous for both tension and compression yield data. The average tensile yield stress of 96000 psi obtained at high pressure and 25°C is also plotted on this figure and lies on the lower edge of the scatter band (which ranges from 96000 to 145000 at 25°C with a mean of 120000 psi). If the possible pressure dependence of the yield stress discussed above is assumed, the corresponding extrapolation to room pressure gives a value of some 83000 psi. However, although both these values of the tensile yield stress obtained from the

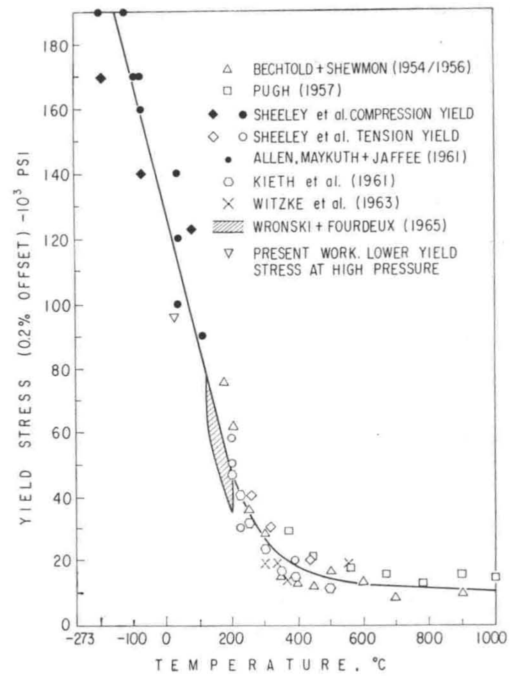


Fig. 7 Compilation of reported measurements of yield stress in tension and compression for recrystallised PM tungsten at atmospheric pressure.

(17) J.E. Hanafee and S.V. Radcliffe : J. Appl. Phys., In press.
(18) D. Hull, J.F. Byran and F.W. Noble : Can. J. Phys., **45** (1967), 1091.
(19) M. Garfinkle : Trans. AIME, **236** (1966), 1373.
(20) R.M. Rose, D.P. Ferriss and J. Wulff : Trans. AIME, **224** (1962), 981.
(21) J.H. Bechtold and P.G. Shewmon : Trans. ASM, **46** (1954), 397.
(22) J.W. Pugh : Proc. ASTM, **57** (1957), 906.
(23) C.R. McKinsey, A.L. Mincher, W.F. Sheeley and J.L. Wilson : ASD TR 61-3, July 1961.
(24) B.C. Allen, D.J. Maykuth and R.I. Jaffee : J. Inst. Metals, **90** (1961), 120.
(25) R.H. Schnitzel : J. Less-Common Metals, **8** (1965), 81.
(26) W.R. Witzke, E.C. Sutherland and G.K. Watson : Tech. Rept. TND-1707, National Aeronautics and Space Administration, 1963.

high pressure results are lower than those expected from the curve in Fig. 7, the scatter in the previously published data is unfortunately too large to conclude that there is a difference between the yield stress in tension and compression at room temperature.

In view of the well established fact that in most crystalline materials some plastic flow accompanies fracture and the several fracture theories which assume plastic strain as a pre-requisite for the initiation of fracture, it is of interest to compare the yield stress observed at high pressure with the fracture stress at atmospheric pressure. In Fig. 8, the reported data for

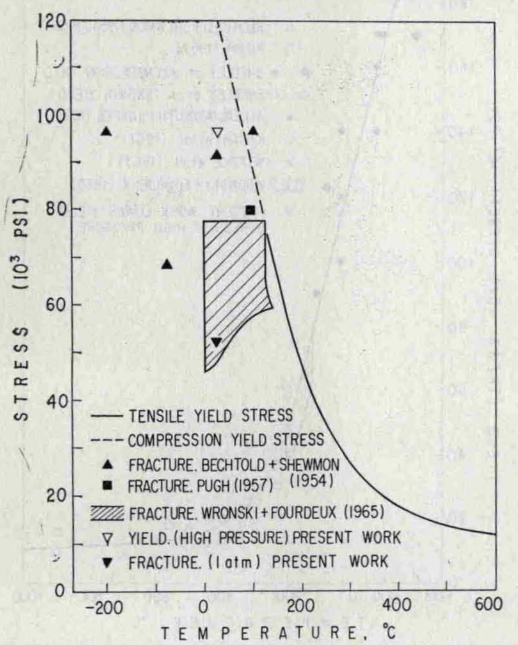


Fig. 8 Comparison of yield stress at high pressure with yield and fracture stresses reported for atmospheric pressure. Recrystallised PM tungsten.

the temperature dependence of the brittle fracture stress of recrystallized PM tungsten[14][21][22][24] are shown together with the yield stress curve from Fig. 7 and the average yield stress observed at high pressure. At 25°C, the latter is seen to be close to the upper limit of the values of the fracture stress measured at atmospheric pressure, as also is the yield stress of 83000 psi obtained by extrapolation from the high pressure data. Thus, these results are consistent with the initiation of fracture as a consequence of local plastic yielding at stress concentrations when the general stress level is below the macroscopic yield stress or, in the absence of stress concentrations, of general plastic yielding when that stress is reached i. e., it represents an upper limit for the fracture stress at room temperature. Examination of the prepolished surfaces of the fractured tensile specimens by optical microscopy and of the fracture surfaces by electron fractography established that the fractures occurred by a mixture of transgranular and intergranular cleavage over the complete range of pressure. For the specimens tested at all pressures up to 8 kilobars no evidence of cracking was found on the prepolished surfaces except for rare examples of grain boundary and transgranular cracks at the edge of the fracture surface. In contrast, the specimens fractured at 11 kilobars (8 % elongation) exhibited grain boundary cracks, occasionally with an associated transgranular crack, along the complete gage length. Most of the intergranular cracks were transverse to the tensile axis, but a number of instances of grain boundary separation in longitudinal and other directions were also noted. The transgranular cracks were not observed to propagate completely across the grains. In addition to the microcracks, occasional surface slip markings, usually associated with grain boundary junctions, were found. These various observations indicate that at pressures below 11 kilobars the principal effect of the imposed hydrostatic stress is to inhibit the formation of microcracks, but that once initiated—apparently by intergranular failure leading to transgranular cleavage—the initial crack propagates catastrophically. In contrast, at 11 kilobars the increased plastic deformation leads to the extensive development of intergranular separation and some transgranular cracks. However, the internal cracks formed in this way do not immediately lead to failure i. e. their rapid propagation and/or the initiation of catastrophic transgranular cleavage is inhibited. The fact that for a given increase in pressure the fracture stress does not increase by the same amount demonstrates that the effect of pressure cannot be attributed to a simple reduction in the applied stress normal to the crack.

The dislocation substructure observed by thin foil electron microscopy in the series of specimens strained to fracture showed a discontinuous change in dislocation density and distribution with increasing pressure. The structure of the as-recrystallized tungsten exhibited only large grains separated by high-angle boundaries and containing the low density of dislocations typical of a well annealed metal. No impurity particles were found either in the grains or at the boundaries, but voids of the type discussed earlier were present. Little change occurred in this structure after fracture at atmospheric pressure and 3 kilobars with the exception of isolated dislocations which appeared at boundaries more frequently at the higher pressure. Also at 3 kilobars, the development of internal elastic strains was evidenced by the presence of multiple diffraction contours. In contrast, the development of 2 % plastic strain before fracture at 5 kilobars (see Fig. 4) resulted in both increased numbers of dislocations at boundaries and a high dislocation density within the grains, as illustrated in Fig. 9(a). The dislocation arrays exhibit the dipoles, jogs and associated small loops characteristic of plastic deformation in the *bcc* transition metals. The nature of the dislocations at the grain boundaries is shown by the dark field micrograph in Fig. 9(b). The further strain (8 %) at 11 kilobars caused a considerable increase in the density of dislocations within the grains and the development of tangles—Fig. 10.

Occasional examples of grain boundary separation were seen in the foils and in one instance an associated transgranular cleavage crack penetrating partially across its grain was observed. The overall length of the crack lay in a single direction, but the detailed path was made up of short zig-zag segments parallel to

(a)
(b)

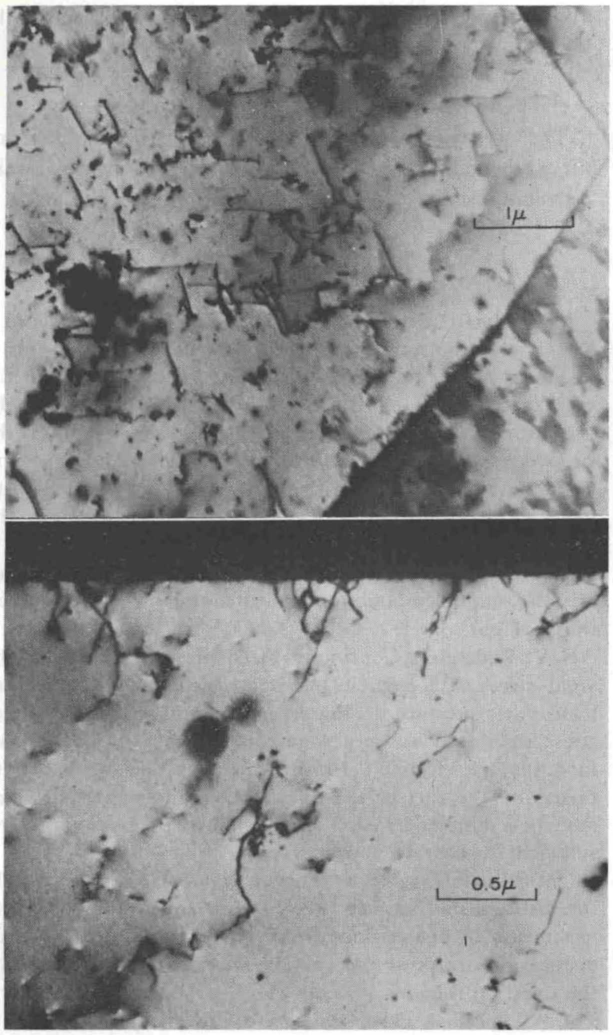


Fig. 9 Thin foil electron micrographs illustrating substructure of recrystallised PM tungsten strained to fracture at 5 kilobars and room temperature. 2 % elongation.

(a)
(b)

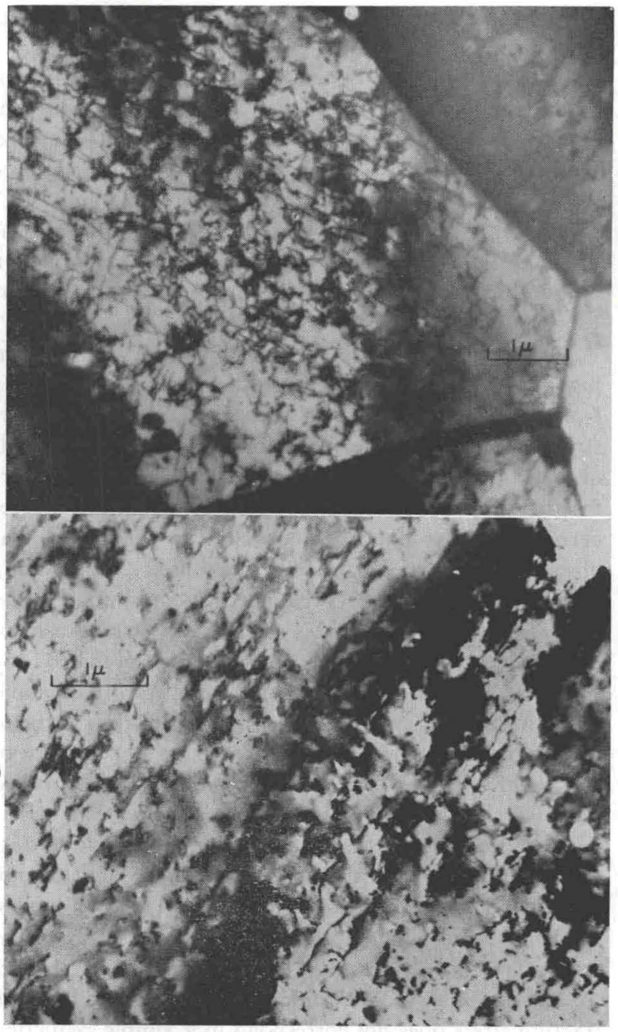


Fig. 10 Substructure of recrystallised PM tungsten strained to fracture at 11 kilobars and room temperature. 8 % elongation.

the two directions of straight dislocation segments visible in the foil. Although the thickness of this particular foil precluded diffraction analysis of the relevant crystallographic directions, this observation has been interpreted as direct evidence of the influence of the presence of the deformation substructure in impeding the propagation of transgranular cleavage. No twinning was observed to occur at any of the pressures investigated.

The substructure developed on plastic straining at high pressure differs from that reported[27] for recrystallized polycrystalline tungsten deformed similar amounts at atmospheric pressure and 200°C, i. e. in the region of the transition temperature, with respect to the density and distribution of dislocations. The density is consistently higher after deformation at room temperature and high pressure, and the "band structure" of alternating light and dark contrast (long parallel cells separated by dislocation walls) observed by Wronski and Fourdeux to be already well developed by 7 % strain does not occur. In this respect and in general appearance, the dislocation structure resembles more closely that observed recently[28] in [010] single crystals of high purity tungsten deformed in tension under ambient conditions. However, the density is considerably higher in the single crystal—for 8 % plastic strain (the largest at pressure) the dislocation density in the high pressure specimens is two to three times that reported for 200°C, whereas that for the single cystal at room temperature is approximately ten times.

In view of the similar high pressure study currently in progress on polycrystalline arc-melted tungsten, it is considered inappropriate to attempt a more detailed analysis of the present results here. In particular, the role of impurity and void effects on yielding and grain boundary rupture may become clearer since preliminary results for the purer material indicate that the lower yield stress is considerably smaller.

IV. Summary and Conclusions

The effects of hydrostatic pressure on the substructure and mechanical behavior of recrystallised PM tungsten have been investigated with respect both to

(27) A. Wronski and A. Fourdeux : Phil. Mag., **10** (1964), 969.

(28) B. Warlimont-Meier, P. Beardmore and D. Hull : Acta Met., **15** (1967), 1399.

changes during pressure application and to behavior at pressure. The results show that:

1. PM tungsten is essentially unaffected by the simple application of pressure, in keeping with the scarcity of impurity particles and absence of pressure-induced dislocations. Such dislocations can be induced in the presence of added particles of ThO_2 and HfC; the large magnitude of the required pressure (in the region of 40 kilobars) compared with that for iron and chromium is attributed to the higher flow stress of the tungsten matrix.

2. The tensile stress-strain behavior of tungsten at high pressure and room temperature differs substantially from that under ambient conditions. The fracture stress is raised and above 3 kilobars increasing amounts of plastic strain and work hardening occur before fracture. The reduction of area at fracture increases correspondingly but no ductile-brittle transition was observed up to the highest pressure used (11 kilobars).

3. Yielding always takes place discontinuously and the mean lower yield stress (96000 psi) is in agreement with that for atmospheric pressure obtained by extrapolation from published high temperature data and with the highest fracture stresses reported for ambient conditions. A possible small pressure dependence of the yield stress is in keeping with a jog-controlled model for yielding.

4. Below 11 kilobars, the imposed hydrostatic stress inhibits only the initiation of microcracks but cannot prevent catastrophic propagation. At 11 kilobars, numerous intergranular cracks and some transgranular cracks are formed but their rapid propagation appears to be inhibited. The influence of pressure on the fracture stress does not correspond to a simple reduction in the applied stress normal to the crack.

5. While the dislocation structure developed by plastic straining at high pressure exhibits several features characteristic of the *bcc* transition metals, the "band structure" observed after comparable plastic strain in recrystallized PM tungsten in the region of the transition temperature at atmospheric pressure is absent and the dislocation density is substantially higher.

Acknowledgment

The authors wish to acknowledge the support of this research by the Materials Division, National Aeronautics and Space Administration.

DISCUSSION

M. Yajima (Central Research Laboratory, Hitachi Ltd.): The stress concentration at the surface of second phase under pressure is too small to nucleate dislocations. In the case of iron, a simple calculation shows that, when the pressure applied is 10000 kg/cm^2, the shear stress at the surface of particle of the second phase is about several kg/mm^2, which is about one hundredth of the stress to nucleate dislocations, but which is enough to unpin the old dislocations and multiplicate them.

That is, our view is that the pressure-induced dislocations are the unpinned and multiplicated ones. How do you think of our view?

S. V. Radcliffe: Continuum mechanics calculations of the yield stress at a particle in a matrix subjected to external hydrostatic pressure do indeed indicate that the maximum stress developed is only some one hundredth of the shear modulus i. e. G/100. However if steps acting as stress raisers on the surface of the particle are taken into account (see for example, Friedel) the computed stress can become sufficient to operate new sources.

Unfortunately, it has as yet been impossible experimentally to distinguish between new source operation and the generation of dislocations from pre-existing sources such as dislocation arrays at the particle/matrix interface.——thus, the question remains unresolved.

J. W. Spretnak (The Ohio State University): There is a possibility that dislocations pre-existing at incoherent interfaces may be drawn off under pressure. Has this type of pressurization work be done both on coherent and incoherent interfaces?

S. V. Radcliffe: To the best of my knowledge, the various studies of pressure-induced dislocations have been concerned solely with incoherent particles.