

changes in structure and mechanical behavior. Similar tests were made on W-1 wt % ThO<sub>2</sub> 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<sub>d</sub>*, 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<sup>(3)</sup>. 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<sub>2</sub> alloy, again no new dislocations 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<sub>2</sub> 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<sub>3</sub>C alloys containing between 1 and 2 vol % of carbide<sup>(1)(3)</sup>. Although the morphology and distribution of the second phase, in addition to its volume proportion, has been shown to be an important factor<sup>(1)(3)</sup>, 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<sub>2</sub>, 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<sup>(15)</sup> of the effects of pressure cycling up to 15 kilobars on the ductile-brittle transition temperature, *T<sub>d</sub>*, in bend specimens of annealed commercial purity PM tungsten sheet and a tungsten -0.5 % hafnium -0.02 % carbon alloy sheet, decreases in *T<sub>d</sub>* of 25° and 50°C, respectively, were reported. While the 25°C

change is close to the accuracy with which changes in *T<sub>d</sub>* 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<sup>(12)(16)</sup> 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.

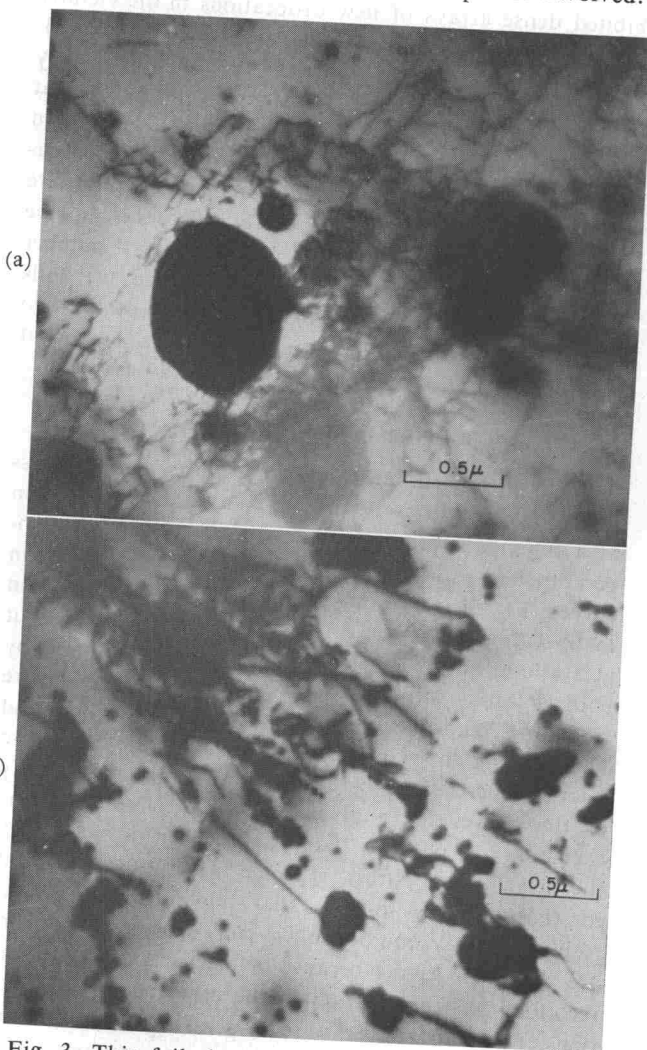


Fig. 3 Thin foil electron micrographs showing dislocation arrays induced in (a) 0.9 vol % ThO<sub>2</sub> alloy, and (b) 1.4 vol % HfC alloy after a pressure cycle to 40 kilobars.

(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<sub>2</sub>, 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<sub>2</sub> 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 similar to that observed previously for carbide particles in iron<sup>(11)</sup>. 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<sup>(5)(8)(11)</sup> 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

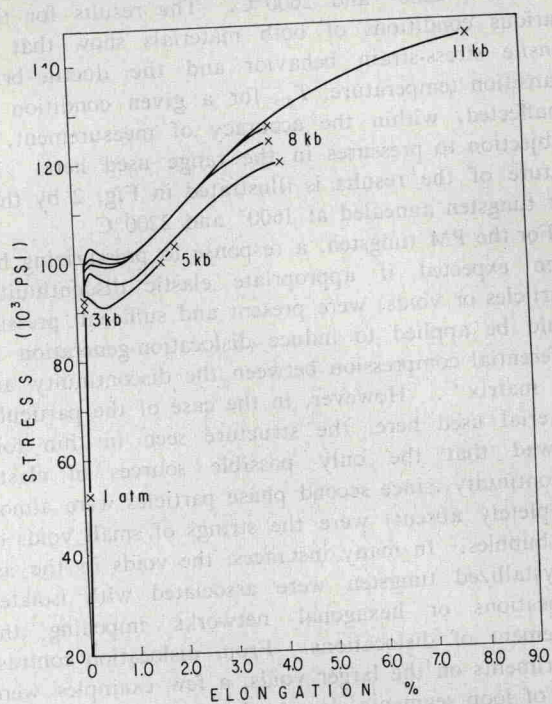


Fig. 4 Tensile stress-strain curves for recrystallized PM-tungsten as a function of environmental pressure at room temperature.

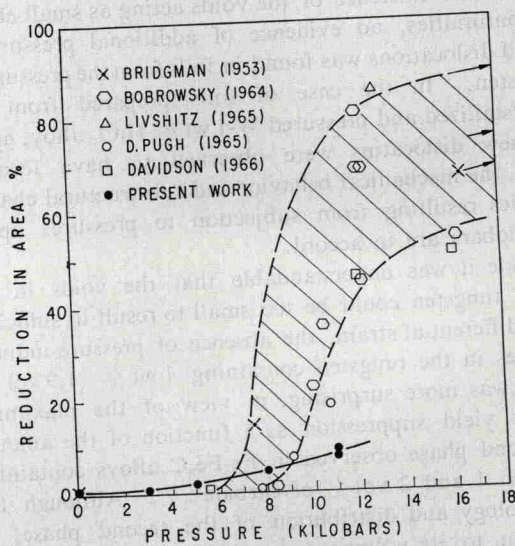


Fig. 5 Pressure dependence of the ductility (reduction in area) of recrystallized PM tungsten. The data reported previously for tungsten is collated in the figure and their range of values indicated by a band.

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<sup>(5)</sup>, 192000<sup>(11)</sup> and 210000<sup>(8)</sup> 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,