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

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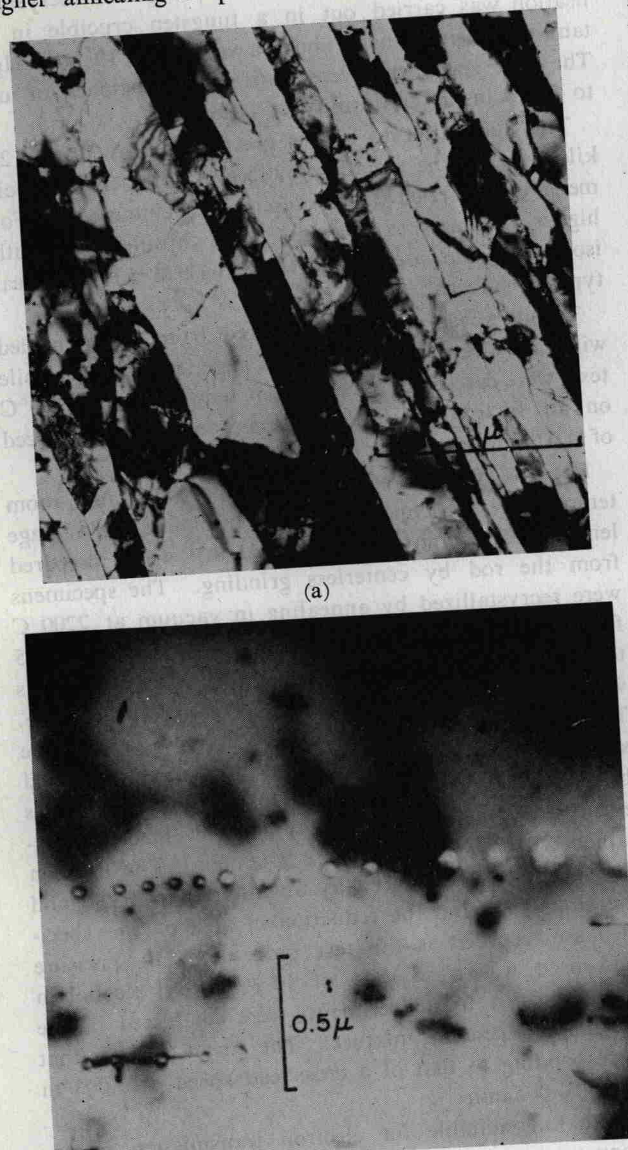
to be in good agreement with those reported previously for temperatures up to 1600°C[13]—see Fig. 1(a). At higher annealing temperatures, migration of certain of



(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

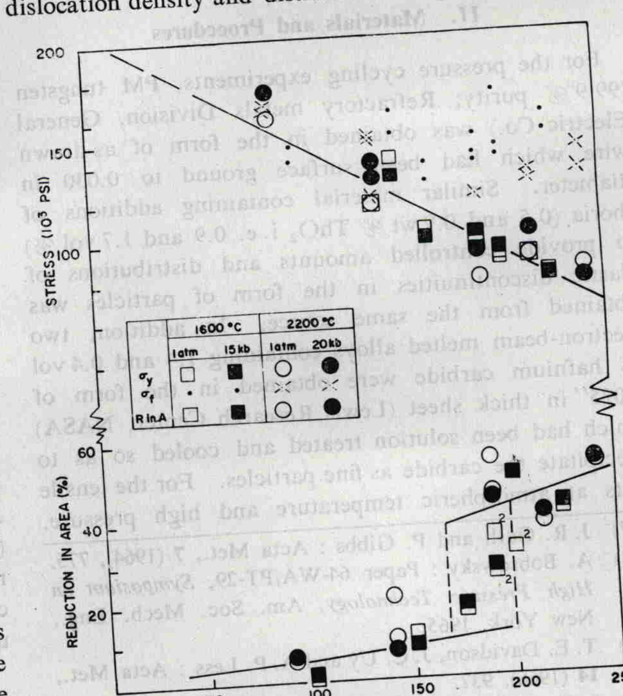


Fig. 2 Temperature dependence of yield stress, fracture stress and reduction in area for PM tungsten annealed at indicated temperatures and cycled to indicated pressures. All tests conducted at atmospheric pressure.

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