

EFFECTS OF HYDROSTATIC PRESSURE  
ON THE MECHANICAL BEHAVIOR OF  
BODY CENTERED CUBIC REFRACTORY METALS AND ALLOYS  
(NASA Research Grant No. Nsg-654)

INTERIM TECHNICAL REPORT

December 1966

prepared by

S. V. Radcliffe (Principal Investigator),  
G. Das and P. Trester

Submitted to:

Office of Grants and Research Contracts  
Attention Code SC  
National Aeronautics and Space Administration  
Washington, D. C. 20546

FACILITY FORM 602	N 67-19033	
	(ACCESSION NUMBER)	(THRU)
	32	1
	(PAGES)	(CODE)
CR-82477	17	
(NASA CR OR TMX OR AD NUMBER)	(CATEGORY)	

DEPARTMENT OF METALLURGY  
CASE INSTITUTE OF TECHNOLOGY  
CLEVELAND, OHIO

### ABSTRACT

The magnitude and form of the decrease in yield stress in the model system Fe-Fe<sub>3</sub>C has been investigated as a function of applied pressure and the volume proportion and morphology of the second phase. The results show that the critical pressure for the onset of yield decrease depends on volume fraction, morphology and the purity of the matrix. The existence of a 'saturation' pressure is indicated, beyond which there is no further change. Measurements of the microyield stress  $\tau_A$  for specimens subjected to 20 kilobars indicate that the stress to move the pressure-induced dislocations is independent of the volume fraction of second phase within the range examined (up to 1.5 vol%). In contrast, the microyield stress for specimens which were prestrained plastically in tension increased progressively with increasing volume fraction. This result is in keeping with the hypothesis that the introduction of pressure-induced dislocations does not modify the main ferrite matrix.

A comparative transmission electron-microscopy study of different types of tungsten has demonstrated that the presence of doping additives in powder metallurgy tungsten can lead to the formation of small internal voids ('gas bubbles') on annealing and recrystallisation; such voids are a factor influencing brittle crack nucleation and growth. Thin foil studies are in progress on dislocation-generation at ThO<sub>2</sub> and HfC particles in a tungsten matrix. The investigation of the tensile flow and fracture behavior of tungsten at high pressure has been initiated. Of the three types of tungsten selected, one batch has been delivered and characterisation and preparation of recrystallised tensile specimens is in progress. The grip design of the high pressure tensile apparatus has been modified to accommodate the buttonhead specimen design.

## CONTENTS

	<u>Page</u>
I. INTRODUCTION	1
II. IRON-CARBON ALLOYS	2
(a) Macroyield Phenomena	2
(b) Microyield Phenomena	6
III. TUNGSTEN AND TUNGSTEN ALLOYS	8
(a) Internal Void Formation	9
(b) Influence of Proportion and Type of Second Phase	12
(c) Tensile Behavior of Tungsten at High Pressure	14
IV. FUTURE WORK	19
V. REFERENCES	20
VI. FIGURES	

## I. INTRODUCTION

During the 12-month period of the present research program (beginning 1 June, 1966), the principal objectives are: (a) to continue the study of pressure effects on iron and iron-carbon alloys as being a model system for the investigation of the mechanism of dislocation generation under hydrostatic pressure and as providing a new means of examining the initiation of plastic flow in iron; and (b) to complete the investigation of pressure effects on tungsten and two-phase tungsten alloys and to examine the influence of environmental pressure on the flow and fracture characteristics of tungsten during tensile straining at pressure. The studies of the iron-carbon system are being conducted by Mr. P. Trester and those on tungsten by Mr. G. Das.

The present report describes the research carried out during the first six-month period from 1 June 1966 - 30 November 1966. During this period, the research effort on the model system has been directed to establishing quantitative relationships between second-phase volume and morphology and changes in macroyield and microyield behavior. For alloys exhibiting the maximum pressure effects ( 1 vol.%  $\text{Fe}_3\text{C}$ ), electron-microscopy of thin foils is being used to examine the nature of the initiation and propagation of plastic flow i.e., the conditions for free dislocation movement in a ferrite matrix of high purity. In the case of tungsten and two-phase tungsten alloys, the investigation of pressure cycling on substructure has been directed to a lower volume fraction thoria alloy and a tungsten-hafnium-carbon alloy, and the study of the tensile behavior of tungsten at constant high pressure has been initiated. The latter is concerned both with the mechanisms

of flow at room temperature and with the relationship between brittle-ductile transition behavior as a function of temperature to that as a function of pressure.

## II. IRON-CARBON ALLOYS

In the case of this model system of second-phase particles dispersed in a matrix of isotropic linear compressibility, the influence of the volume proportion of second-phase on the decrease in flow stress for a series of annealed high-purity iron-carbon alloys after subjection to a hydrostatic pressure of 20 kilobars was established earlier<sup>(1)</sup>. The maximum change occurs for alloys containing 1 to 2 vol.% for this particular carbide morphology. Examination of microyield characteristics demonstrated that the introduction of mobile dislocations by pressurisation results in a substantial decrease in the microyield stress  $\sigma_A$  compared with the values obtained by the normal method of uniaxial plastic prestraining at atmospheric pressure. The current work has been concerned with establishing accurately the magnitude and form of the decrease in yield stress in relation to the applied pressure and the volume proportion and morphology of the second phase, using both subcritically annealed and quenched and tempered alloys, and with studying microyielding in these high purity alloys. The methods of specimen preparation, vacuum annealing, application of high hydrostatic pressures, and tensile stress-strain measurement in the axial loading rig were as described previously<sup>(1)</sup>.

### (a) Macroyield Phenomena

The observations reported earlier<sup>(1)</sup> on the changes occurring

in the macroyield region of the tensile stress-strain curves for a series of annealed high purity iron-carbon alloys as the result of subjection to a hydrostatic pressure cycle to 20 kilobars have been extended to examine the nature of the progressive changes as a function of pressure. Appropriate measurements have been made at 5, 10 and 15 kilobars for the alloy series up to 0.3 wt% C (4.7 vol.%  $\text{Fe}_3\text{C}$ ). The results are presented in Figure 1\*. For all the alloys, a continuous decrease in the lower yield stress is observed once a critical pressure is exceeded. This critical pressure increases with increasing volume fraction of carbide - it is less than 5 kilobars for the 0.065 wt% C (1.0 vol.%  $\text{Fe}_3\text{C}$ ) and lower carbon alloys, and between 5 and 10 kilobars for the 0.3 wt% C (4.7 vol.%  $\text{Fe}_3\text{C}$ ) alloy. The fact that the critical pressure reported earlier<sup>(1)</sup> for a similarly annealed plain-carbon steel (AISI 1018) containing only 2.8 vol.%  $\text{Fe}_3\text{C}$  is higher (some 15 kilobars) than that for the high purity alloy containing 4.7 vol.%  $\text{Fe}_3\text{C}$  indicates that the critical pressure is dependent not only on the volume fraction of second phase, but also on the impurity content of the matrix. This is in keeping with the interpretation that the higher strength of the more impure ferrite matrix in the plain-carbon steel requires a higher critical pressure to induce new dislocations by differential compression between particle and matrix. Such a role of the inherent matrix strength is also a possible cause of the failure of annealed tungsten-thoria alloys to exhibit

---

\* The fact that the yield stress levels do not increase regularly with increasing carbon content is associated primarily with the variation in grain size between the different alloys.

changes in their macroyield characteristics after subjection to a pressure as high as 25 kilobars<sup>(1)</sup>. A further point of interest is the existence of a 'saturation' effect indicated in the results for the lower carbon alloy (0.35 vol.% Fe<sub>3</sub>C) for which the decrease in yield stress is essentially unchanged for pressures beyond 15 kilobars. This corresponds to a limiting pressure beyond which either no further dislocation generation occurs because of interfering stress fields from adjacent sources or the introduction of further dislocations no longer drastically modifies the stress-strain relationship. Further attention is being directed to this point.

In order to examine the role of the morphology of the second phase in determining the nature and magnitude of the pressure effects on discontinuous yielding, different second phase morphologies were introduced in the 0.065 wt%C and 0.30 wt%C alloys by the quenching and tempering treatment developed previously<sup>(1)</sup> (austenitizing at 925°C for 30 minutes in a vacuum, quenching into ice water, and tempering at 700°C for one hour, followed by rough machining and tempering for an additional hour at 700°C before final grinding of the tensile specimens). This treatment results in a considerably more uniform size and dispersion of carbide, and also in a finer ferrite grain size - see Table I. The effects of pressure cycling on yield stress for these two alloys are compared in Figure 2. The missing data points correspond to specimens which deviated considerably in their response to pressure from the general trend of the data. The deviations were found to be associated with a different microstructure developed in these particular specimens which was

traced to a faulty control of temperature during heat treatment such that these specimens were not correctly austenitised. As further material was not available for the 0.065 wt%C alloy, which is the composition of greatest interest, repeat tests were conducted on a new alloy of similar composition (0.09 wt%C), using salt bath austenitising in place of the vacuum furnace. The results are included in Figure 2. The results for all three alloys differ from those obtained with particle morphology developed by sub-critical annealing principally in that the discontinuous yield drop is not eliminated even at the highest pressure used, 20 kilobars, although both the upper and lower yield stresses are reduced substantially. Figure 3 illustrates these different trends of the tensile stress-strain curves for the 0.30 and 0.004 wt%C alloys.

Attention is now being directed to examining the changes in substructure by thin-foil electron microscopy for the various conditions discussed above. Improved techniques to facilitate foil preparation and observation have been developed. The characteristics of the pressurised alloys and also of the early stages of tensile deformation in specimens strained after pressure cycling will be discussed in the next report.

Although the current investigation of the influence of the several variables on the change in yield behavior induced by pressurisation is not yet completed, it is appropriate to note that the present data indicate that a progressive lowering of the level of the 'lower yield stress' region with increasing pressure is a



characteristic effect, whether or not the 'upper yield point' is eliminated. The variables include volume proportion of  $\text{Fe}_3\text{C}$ , its distribution and morphology, and the grain size and purity of the ferrite matrix. This characteristic is in keeping with the hypothesis of an increased density of mobile dislocations and sources leading to a lower stress for the propagation of the Luder's band(s) at the constant strain rate of the testing machine. Such an interpretation is in agreement with the attribution of irregularities (i.e., oscillation of the stress level) in the Luder's -strain or heterogeneous yield region to the nucleation of additional Luder's band(s) and, correspondingly, an increased number of mobile dislocations contributing to plastic strain<sup>(2)</sup>. Another characteristic of the pressurised alloys, that of the progressive shortening of the Luder's strain region with increasing pressure (see Figure 3) would be expected to arise from the increased density of dislocations induced by pressurisation leading to dislocation interaction and work-hardening at smaller plastic strain.

(b) Microyield Phenomena

The investigation of microyielding in pressurised iron alloys which was initiated previously on a plain-carbon steel has been applied to the 0.004, 0.019 and 0.09 wt%C alloys. Using the cyclic loading technique with resistance strain gages on the specimen<sup>(1)</sup>, the microyield stress,  $\sigma_A$ , (corresponding to an initial permanent strain of  $10^{-5}$  in/in) has been determined for these materials for the as-annealed condition and after subjection to 20 kilobars. The results, together with  $\sigma_A$  measured for annealed specimens using the

normal tensile prestraining technique are given in Table II. The earlier results for the plain-carbon steel (0.18 wt%C) are included for comparison.

The fact that the 0.004 and 0.09 wt%C alloys exhibit a similar  $\sigma_A$  (12.2 and  $12.5 \times 10^3$  psi, respectively) suggests that the stress required to initiate movement of pressure-induced "mobile" dislocations in the high-purity iron matrix is independent of the volume proportion of second phase within this range of 0.06 to 1.5 vol.%. The steel contains a larger volume proportion of carbide (2.8 vol%) and its  $\sigma_A$  value is larger ( $15.5 \times 10^3$  psi). However a contribution to  $\sigma_A$  would be expected from the impure ferrite matrix, and the higher critical pressure found for the steel (see p.4) points to the matrix contribution being the major factor rather than the second phase content.

In the case of the prestrained specimens, the value of  $\sigma_A$  for a given alloy was found to remain essentially constant for magnitudes of prestrain within the Luder's region in agreement with the observations of Brown and Kossowsky<sup>(3)</sup> for a high-purity iron. However, with increasing carbon content, the value of  $\sigma_A$  in the prestrained specimens increases progressively, which is in marked contrast with the absence of a carbon dependence of  $\sigma_A$  for the pressurised alloys. In the prestrained alloys, plastic strain is effected from the motion of dislocations generated as the result of the stress concentration at the front of the moving Luder's band(s) which itself represents a region of substantial plastic strain, i.e. of substantially changed substructure. In contrast, the pressurised alloys

contain a relatively homogeneous distribution of new dislocations and sources formed discretely at the second-phase particles without necessarily modifying the matrix substructure. On applying a tensile load the new dislocations move directly under the action of the resolved shear stress. Thus, in the pressurised alloys,  $\tau_A$  represents more closely the stress to move a dislocation in a matrix of constant substructure. Further information on the substructures, the mechanisms of generation and the types and densities of dislocations moving is being sought to clarify these observations.

### III. TUNGSTEN AND TUNGSTEN ALLOYS.

It was shown previously that the macroyield behavior and ductile-brittle transition temperatures in wire specimens of a recrystallised powder-metallurgy tungsten and a tungsten-1 wt% thoria (nominal) alloy, both from the same commercial source were essentially unaffected by pressure cycling up to 25 kilobars<sup>(1)</sup>. The results for the tungsten are in keeping with the scarcity of impurity particles observed by thin-foil electron microscopy and the corresponding absence of pressure-induced dislocations. During the annealing and recrystallisation of the tungsten, linear arrays of small internal voids parallel to the direction of working in processing were shown to develop. Although the voids are elastic discontinuities, they are apparently too small to be effective as dislocation sources. In the tungsten containing deliberate additions of second-phase ( $\text{ThO}_2$ ) particles, a similar absence of pressure-induced dislocations at particles was found. The failure to generate dislocations at the particles was attributed mainly

to their distribution and morphology, but it is also possible that the pressures used (up to 25 kilobars) may be insufficient for the compressibility difference between tungsten and thoria, which is unknown.

The current work has been concerned principally with three problems - the origin and nature of the voids in tungsten, examination of the influence of smaller proportions and different types of second phase and the initiation of the study of the tensile flow and fracture behavior of recrystallised tungsten at high hydrostatic pressures. In addition to these problems, an electron microscopy study has been made of thin foils prepared from the earlier specimens of tungsten wire which showed an 'anomalous' decrease of approximately 50°C in the ductile-brittle transition temperature after pressurising to only 13 kilobars<sup>(1)</sup>. It had been anticipated from this behavior that these particular specimens contained a larger amount of impurity particles. Extensive examination of the thin foils has confirmed that the structure was recrystallised, but has not revealed any marked differences in particle content or substructure from the other tungsten wires. Thus, the origin of the anomalous behavior remains unexplained.

#### (a) Internal Void Formation in Tungsten

In the powder metallurgy tungsten wires, it was observed earlier<sup>(1)</sup> that small internal voids or gas bubbles developed progressively with increasing annealing temperature. For low temperatures (1100°C) the voids appear as elongated shapes predominantly at the boundaries of the longitudinal cells or fibers characteristic of that stage of annealing. After full recrystallisation at high temperatures

(circa 2000°C), the voids appear at the grain boundaries and also within the grains as parallel rows of small rounded shapes. The fact that the rows are approximately the same distance apart (0.5 microns) as the original fiber width in the as-drawn structure, suggests that the voids originate from volatile impurities or dissolved gas at the original grain boundaries. Wronski and Fourdeux<sup>(4)</sup> have recently described the presence of small gas bubbles or voids of similar appearance in sintered tungsten rod and attributed them, from their absence in melted tungsten, to dissolved gases.

In order to determine the origin of the voids in the tungsten wire, three different types of tungsten from the same manufacturer have now been examined by transmission electron microscopy. The three are (i) the commercial doped wire (0.030 in.dia.) used for the main study; (ii) undoped wire (0.075 in.dia.); and (iii) melted material prepared in an electron-beam zone refiner from specimens of the main stock of doped powder metallurgy wire and from a 1/8 in.dia.rod of doped tungsten. The two powder metallurgy materials were recrystallised. Using the micro-jet electrothinning technique<sup>(1)</sup>, thin foils were prepared from the wire specimens and from slices spark cut from the melted rod materials and examined at 100 kV in a JEM-6A electron microscope. The structure of the electron-beam melted tungsten from both the wires and the rod is essentially a single crystal with a low density of dislocations and no internal voids or gas bubbles. The undoped tungsten (Fig. 4a) (annealed at 2200°C by the manufacturer) exhibits a recrystallised structure and again no voids are present. In contrast, the recrystallised doped material (Fig.4b) consistently shows a distribution of internal voids, usually having dislocations associated with them.

of the voids formed in the tungsten.

These results are interpreted as indicating that the voids are produced as a result of the evaporation of doping additives\* (such as  $K_2SiO_3$ ) which are located preferentially on the original grain boundaries in the as-sintered material. As the melting points of such compounds can be low (e.g.,  $1076^\circ C$  for  $K_2SiO_3$ ), dissociation would be expected to occur at the higher annealing temperatures, giving gaseous products which could lead to the formation of the observed voids. The size and shape of the voids will depend on the internal pressure and the surface tension of the tungsten. At the higher temperatures, diffusion of the products into the lattice could facilitate the formation of the smaller, spherical cavities.

The significance shown here of the presence of the doping additives provides clarification of the source of the 'gas bubbles' observed by Wronski and Fourdeux, since it is extremely likely that their material was doped also (a widely used practice for commercial powder metallurgy tungsten). However, dissolved gas (or possible microporosity due to incomplete densification on sintering) as a source of void formation in sintered material cannot be eliminated as there are some indications that gas bubbles can be present in sintered molybdenum<sup>(4)</sup> and platinum<sup>(5)</sup>, but are absent in the melted materials. In all three metals, the sintered condition has inferior mechanical properties<sup>(4,5)</sup> and the possible importance of internal voids as an additional factor (to grain boundaries and precipitates) influencing stress concentration and crack nucleation and growth is becoming

---

\* Specific details of the additives and metal processing in current use are not available in the literature or from manufacturers.

increasingly apparent. It is concluded from the present work that the substitution of a means of grain size control other than doping in powder metallurgy tungsten should result in the development of more reproducible mechanical properties in this material.

(b) Influence of Proportion and Type of Second Phase.

The distribution, volume proportion and nature of the second-phase particles in the matrix is now known to play an important part in determining the possibility of the pressure-induced generation of dislocations. Thus, for the model system of  $\text{Fe}_3\text{C}$  particles in a ferrite matrix, the lowering of the flow stress reaches a maximum for 1 - 2 vol.%  $\text{Fe}_3\text{C}$ , and subsequently returns to zero with further increase in the amount of carbide. Accordingly, to examine the pressure response of tungsten containing deliberate additions of second-phase, a nominally 1 wt.%  $\text{ThO}_2$ -W alloy (1.9 vol.%) was selected. Following the absence of pressure-induced dislocations in recrystallised specimens of this alloy after subsection to a pressure of 20 kilobars, a chemical analysis has been made of the thoria content. This established the composition as 0.9 wt%  $\text{ThO}_2$ , i.e. 1.7 vol.%. The fact that the volume fraction seen in thin foil specimens in the electron microscope appears to be substantially higher suggests that particles are remaining attached to the surfaces of the foil after polishing, leading to an apparently larger volume fraction than is appropriate to the foil thickness. In order to permit a more detailed examination of the matrix substructure adjacent to isolated particles of thoria and because of the possibility that the optimum volume proportion may be lower than for the model system, samples of tungsten

rod (0.075 in. diameter) containing 0.5 wt% ThO<sub>2</sub> (0.9 vol%) have been obtained. Experiments on this material are currently in progress.

Although thoria has been considered as a 'hard' particle relative to the tungsten matrix, data on the compressibility or hardness is not available. Accordingly, in order to examine the possibility that the properties of the thoria relative to tungsten may be inappropriate for dislocation generation within the range of hydrostatic pressure available, the effect of a different type of second phase particle is being examined. The compound selected is hafnium carbide, which is substantially harder (1940 - 2300 kg/mm<sup>2</sup><sup>(6)</sup>) than tungsten although its compressibility has not been reported. This particular compound is also of interest in that Schaffhauser<sup>(7)</sup> recently reported a reduction in the ductile-brittle transition temperature in bending of 50°C for annealed (2000°C) sheet specimens of a tungsten-0.5 wt% hafnium - 0.02 wt.% carbon alloy of commercial purity after subjection to 15 kilobars. The result was attributed to dislocation generation at particles of HfC, although a direct examination of the substructure was not made.

The alloy which was available initially for the present work contains 0.88 wt% Hf and 0.062 wt%C. It was obtained in the form of 0.025 in. thick sheet which had been solution treated at 2770°C for 15 minutes and cooled rapidly (by helium gas) to develop the optimum mechanical properties. This optimum is generally believed to be associated with the formation of a very fine dispersion of HfC particles. To prepare thin foils for electron microscopy, pieces of the sheet were reduced in thickness to 0.015 in. by electropolishing and then



0.125 in. diameter disc were trepanned from the sheet by high-voltage spark-cutting with a tubular tool. Several of these discs were subjected to a pressure of 20 kilobars in the modified piston-cylinder apparatus. Foils were made directly from the unpressurised and the pressurised discs by the electrolytic jet method. The electron micrograph shown in Figure 5 illustrates the general structure of the material and shows the relatively high density of precipitate particles observed. Comparative examination of the structure of the foils in the microscope did not reveal any clear evidence of pressure-induced changes in substructure. While the actual particle density is lower than observed in the thin-foils for the reason discussed earlier, the composition of this alloy corresponds to a higher proportion (1.4 vol.%) of HfC than the alloy used by Schaffhauser. As the proportion of second phase may be beyond the optimum for pressure effects, a second alloy having a lower carbide content has now been obtained for investigation. The new alloy contains 0.22 wt% Hf and 0.0135 wt% C (corresponding to 0.37 vol.% HfC) and is in a similar form and condition to the first alloy.

(c) Tensile Behavior of Tungsten at High Pressure.

While the general effect of a superimposed hydrostatic pressure in enabling a number of 'brittle' materials to undergo substantial plastic deformation before fracture was established some years ago by Bridgman<sup>(8)</sup>, detailed studies of specific metals have not been made until 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 metals zinc and bismuth by Pugh and co-workers<sup>(9)</sup> who proposed that this 'brittle-ductile transition' would be associated with metals of low coefficient of strain-hardening, whereas those with high coefficients would show only a steady increase in ductility with pressure.

In the case of tungsten, Bridgman<sup>(10)</sup> in 1953 reported an almost linear increase (based on 4 specimens) in true strain at fracture with increasing pressure up to 27.8 kilobars, in agreement with his general observations on changes in ductility with pressure. The data point for 7 kilobars, which lies well below the line drawn through the other 3 points, was discounted as a premature fracture, but taking it into account would indicate that a discontinuity exists in this pressure region - in keeping with later observations. Thus, Bobrowsky<sup>(11)</sup>, Davidson, Uy and Lee<sup>(12)</sup> and Livshitz, Ryabinin and Beresnev<sup>(13)</sup> have subsequently observed discontinuous increases in ductility (reduction of area at fracture) in the region from 6 to 12 kilobars in tensile tests at room temperature. These tests were conducted in apparatus utilising various forms of the Bridgman yoke design in which the tensile straining is effected by the motion of the main piston in the pressure chamber. Consequently, the pressure increases during the test by an amount which increases with the amount of strain before fracture and the pressure at which the piston contacts the tensile device. Measurements on tungsten have also been

reported by Pugh<sup>(14)</sup> using an apparatus of different design (also used in the work on Zn and Bi) in which the pressure is maintained constant during tensile straining. The apparatus is limited to maximum pressures of the order of 12 kilobars, but has the considerable advantage of direct and continuous measurement of load and specimen diameter within the pressure chamber during the test. The observations on tungsten showed that the metal was brittle up to some 7.6 kilobars, but exhibited increasing ductility with further increase in pressure. At the highest pressure used (10.7 kilobars) the reduction area increased to some 18%, compared with the value of approximately 48% reported by Bobrowsky<sup>(11)</sup> and Davidson<sup>(12)</sup> for a pressure of 12 kilobars at fracture. A further interesting feature of the continuous measurement on the tungsten was the indication of a possible yield drop for pressures above some 7 kilobars as shown in the load/extension curves of Bridgman<sup>(10)</sup> and the true stress/true strain ( $\ln A_0/A$ ) curves of Pugh<sup>(14)</sup>.

The above observations on tungsten together with the recently published measurements by Davidson et al<sup>(12)</sup> of the pressure and temperature dependence of the transition from brittle to ductile behavior in magnesium 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 is 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 of tungsten used. Thus, Bridgman<sup>(10)</sup>

used "annealed rod, 99.9% purity from Fansteel Co."; Bobrowsky<sup>(11)</sup> and Livshitz et al<sup>(13)</sup> used "tungsten"; Davidson et al<sup>(12)</sup> used a "99.9% purity pressed and sintered condition, grain size 0.09 mm". Pugh<sup>(14)</sup> provides more useful detail - "tungsten, commercially pure 0.002%C, 0.01% Mo., and 0.015% Fe, sintered, hot-swaged and vacuum annealed at 1200°C" - but even here, the annealing temperature is below the generally accepted range of recrystallisation temperatures.

In the present investigation, attention is being directed to working with recrystallised tungsten of known history and characterised structure, and to using a constant pressure tensile apparatus of the type developed by Pugh et al to examine the pressure dependence of the characteristics of flow and fracture in tungsten. Effort is being directed particularly to the nature of discontinuous yielding in recrystallised tungsten at room temperature, a phenomena which cannot be investigated under ambient conditions due to premature fracture.

Three different types of polycrystalline tungsten of commercial purity have been selected for the study:

- (i) Doped powder metallurgy rod.
- (ii) Undoped powder metallurgy rod.
- (iii) Arc melted and cast rod.

The use of these particular types was made with the objective of comparing and elucidating the influence of the different metallurgical characteristics. The doped rod has already been obtained and tensile specimens are being machined. Delivery of the other two types of tungsten is expected shortly.

The high pressure tensile tests are being conducted in a constant pressure tensile apparatus of the same basic design as that developed by Pugh and co-workers at The National Engineering Laboratory<sup>(9,14)</sup>. The general arrangement of the apparatus, which is essentially a constant strain-rate tensile machine contained within a high pressure chamber, is shown in Figure 6. The main pressure chamber is a thick-walled steel cylinder, eleven inches long and 9 inches outside diameter, with a stepped concentric bore of 1.5 in. diameter at the upper end and a bottom bore of 0.7 in. diameter. The tensile specimen is strained by lowering the bottom plunger, to which it is attached; the other end being fixed to the cylinder via an electrical resistance strain load cell and a support tube. Electrical leads to the load cell and a manganin-pressure gage are conducted into the chamber through seals in the upper plunger. The reduction of area of the specimen as the test proceeds is followed by direct photographic observation through thick glass windows mounted on the cross bore. Elongation is monitored by using a linear transducer to record the 'cross-head travel' of the lower plunger. Pressure is generated and maintained by the upper plunger which is forced downward by means of a 400-ton hydraulic ram to compress a suitable fluid in the chamber, while the bottom plunger is supported by a 60-ton upstroking ram.

Due to the brittleness of recrystallised tungsten, a button head specimen configuration is being used (Figure 7). A suitable split-collet grip has been designed to adapt to the seven-thread grips in the apparatus. The upper collets are threaded to screw into the lower grip which is in turn coupled with the bottom plunger. As the

result of preliminary tests of the grip operation in the apparatus using a hardened steel tensile specimen, some minor modifications are being made to facilitate the aligning of the specimen in the chamber before proceeding with tensile testing of the tungsten specimens which are now being machined.

#### IV. FUTURE WORK

During the next six-month period of the program, the effort on the model system will be directed mainly to a transmission electron microscopy study of the dislocation configurations induced by pressure and the dislocation structures which develop during the early stages of plastic flow during subsequent tensile straining. On the basis of these and earlier observations, an attempt will be made to develop a quantitative model to account for the pressure effects on the yield behavior. In the case of tungsten, measurements will be made of the plastic flow and fracture characteristics at room temperature as a function of pressure for the three types of tungsten. Attention will be given to direct observation of the nature of dislocation movement in material strained at the high pressures, at which extensive plastic deformation is anticipated before fracture.

V. REFERENCES

1. S. V. Radcliffe, G. Das and P. Trester: Interim Technical Report No. 4 (NASA Grant No. NSG-654) June, 1966, Case Institute of Technology.
2. J. F. Butler; Acta Met, 10, 1962, 258.
3. R. Brown and N. Kossowsky; Technical Report, Naval Research Contract NONR-551 (20), 1965.
4. A. Wronski and A. Fourdeux, J. Less-Common Metals 8 (1965), 149.
5. J. B. Hanley and W. I. Mitchell: J. Nat. Sci., 1 (1966), 412.
6. "Refractory Ceramics - A Materials Selection Handbook"; U. S. Air Force Rept. ASD-TDR-63-4102 (Oct. 1963).
7. A. C. Schaffhauser, Ann. Prog. Rept. ORNL-3870, Oak Ridge National Laboratory, U.S. Atomic Energy Commission, 1966.
8. P. W. Bridgman; Studies in Large Plastic Flow and Fracture, McGraw Hill Co., New York, 1952.
9. (a) H. L. D. Pugh and D. Green: "Behavior of Metals Under High Hydrostatic Pressure", MERL Plasticity Rept. No. 128, National Engineering Laboratory, 1956.  
(b) H. L. D. Pugh, J. Lees, K. Ashcroft and D. Gunn; Engineer (G.B), 212, (1961), 258.
10. P. W. Bridgman: J. Appl. Phys., 24 (1953), 560.
11. A. Bobrowsky: Paper 64-WA/PT-29, Symposium on High Pressure Technology, Am. Soc. Mech. Eng., New York, 1965.
12. T. E. Davidson, J. C. Uy and A. P. Lee; Acta Met., 14 (1966) 937.
13. L. D. Livshitz, Y. N. Ryabinin and B. I. Beresnev; Soviet Phys. - Tech. Phys., 10 (1965), 278.
14. H. L. D. Pugh; Bulleid Memorial Lectures, Nottingham University Press, 1965.

TABLE I.

## VARIATION OF FERRITE GRAIN SIZE WITH COMPOSITION AND HEAT TREATMENT

Wt.%C	Theoretical Vol.% Fe <sub>3</sub> C	Final Heat Treatment	Grains/mm <sup>2</sup> +	Grain Diam. (microns)
0.004	0.06	Anneal 667°C	50-60	150-160
0.019	0.35	Anneal 667	280-330	62-68
0.065	1.0	Anneal 667	500-600	45-52
	1.0	Temper 700	1100-2500	23-34
0.09	1.4	Temper 700		
0.18*	2.8	Anneal 667	600-1100	34-45
0.30	4.7	Anneal 667	++	
	4.7	Temper 700	4000-5500	16-18

+ Determined by averaging total grains on photomicrographs taken at 250X - Nital etch.

\* AISI 1018 steel

++ not calculable; pearlite colonies obscured grain boundaries



TABLE II.  
COMPARISON OF THE EFFECT OF TENSILE PRESTRAIN AND  
PRESSURISATION ON THE MAGNITUDE OF THE INITIAL FLOW STRESS  $\sigma_A$ .

<u>Wt. %C</u>	<u>Condition</u>	<u><math>\sigma_A</math> (psi)</u>
0.004	a) A*	< U.Y.S.
	b) A and prestrained 0.02%	$17.4 \times 10^3$ (ave. of 2)
	c) A and pressurised at 20kb	$12.5 \times 10^3$ (ave. of 2)
0.019	a) A	< U.Y.S.
	b) A and prestrained	
	0.016%	$22.8 \times 10^3$ )
	0.086%	$22.8 \times 10^3$ ) on same
	0.114%	$22.8 \times 10^3$ ) specimen
0.09	a) A	< U.Y.S.
	b) A and prestrained 0.15%	$19.8 \times 10^3$
	c) A and pressurised at 20Kb	$12.2 \times 10^3$
0.18 (AISI 1018)	a) A	~ U.Y.S.
	b) A and .53% prestrain	$25.4 \times 10^3$
	c) A and 20 kb	$15.5 \times 10^3$

\* Annealed at 667°C

$\sigma_A = 10^{-5}$  in/in plastic strain  
0.02 in/min. strain rate.

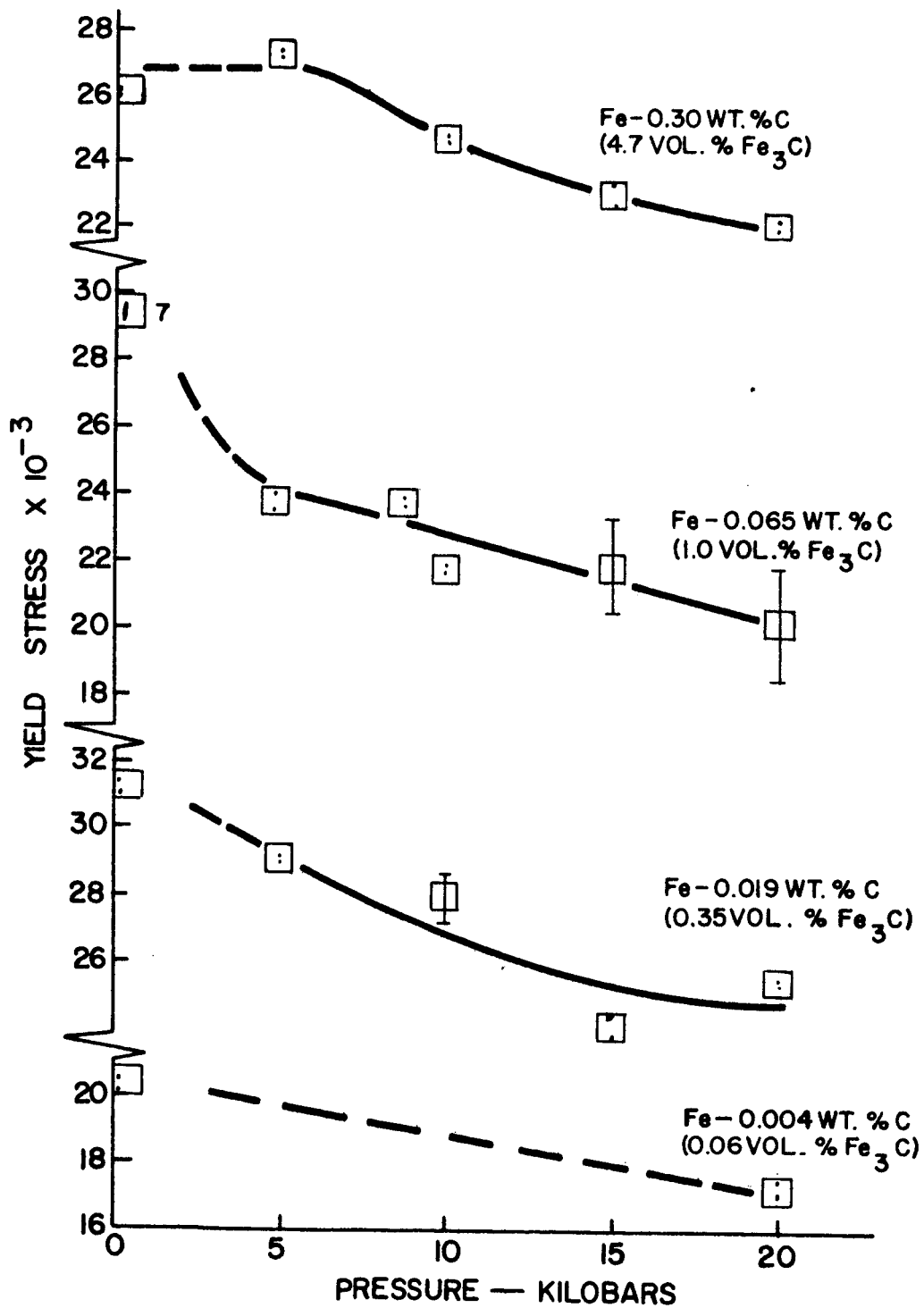


Fig. 1. Effect of volume proportion of second phase ( $Fe_3C$ ) on change in yield stress (LYS, or 0.2% offset) after subjection to indicated pressures. All alloys subcritically annealed at  $667^{\circ}C$ .

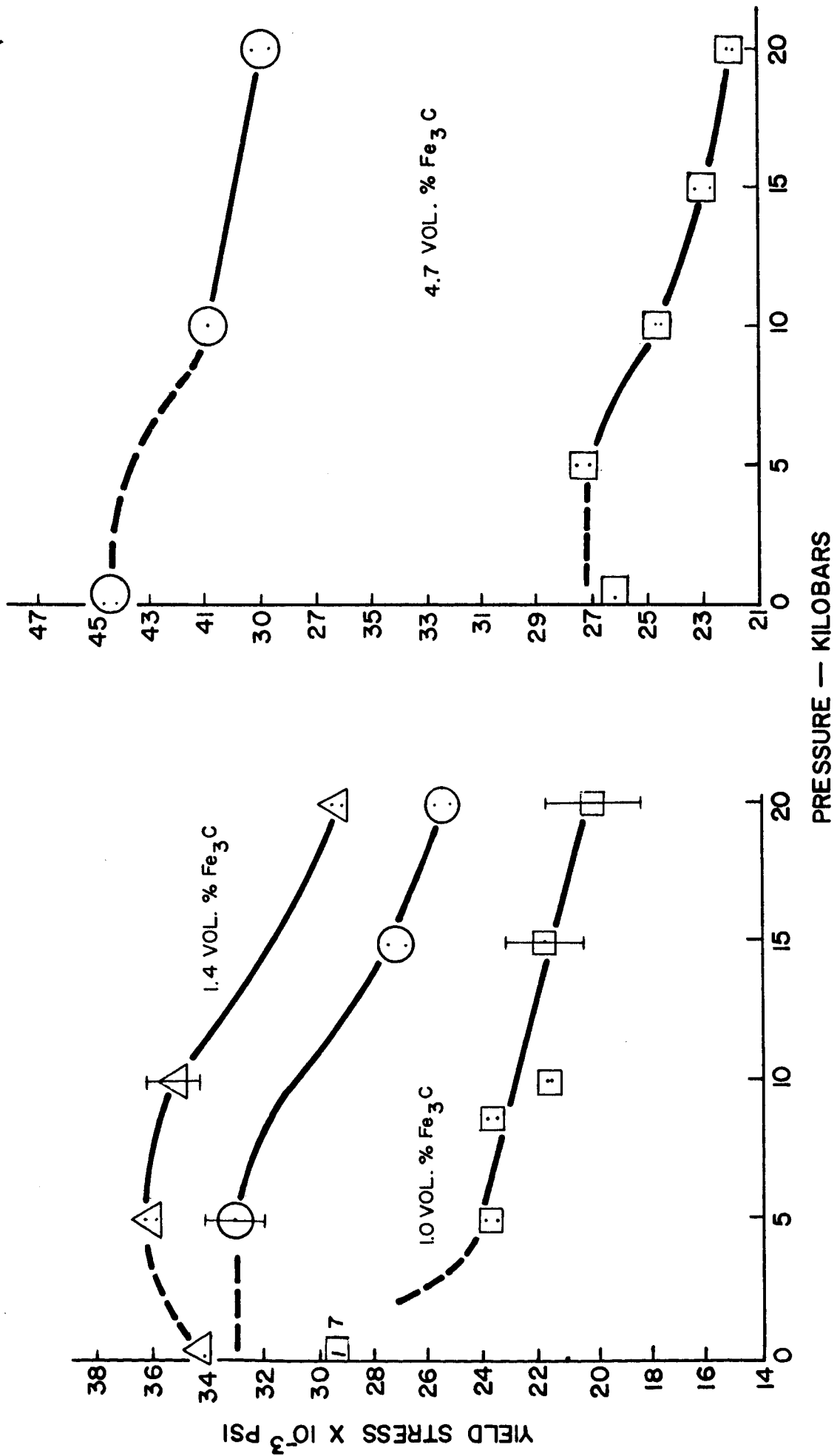


Fig. 2. Effects of second phase morphology on change in yield stress after subjection to indicated pressures. Iron carbon alloys - (a) quenched and tempered (b) subcritically annealed.

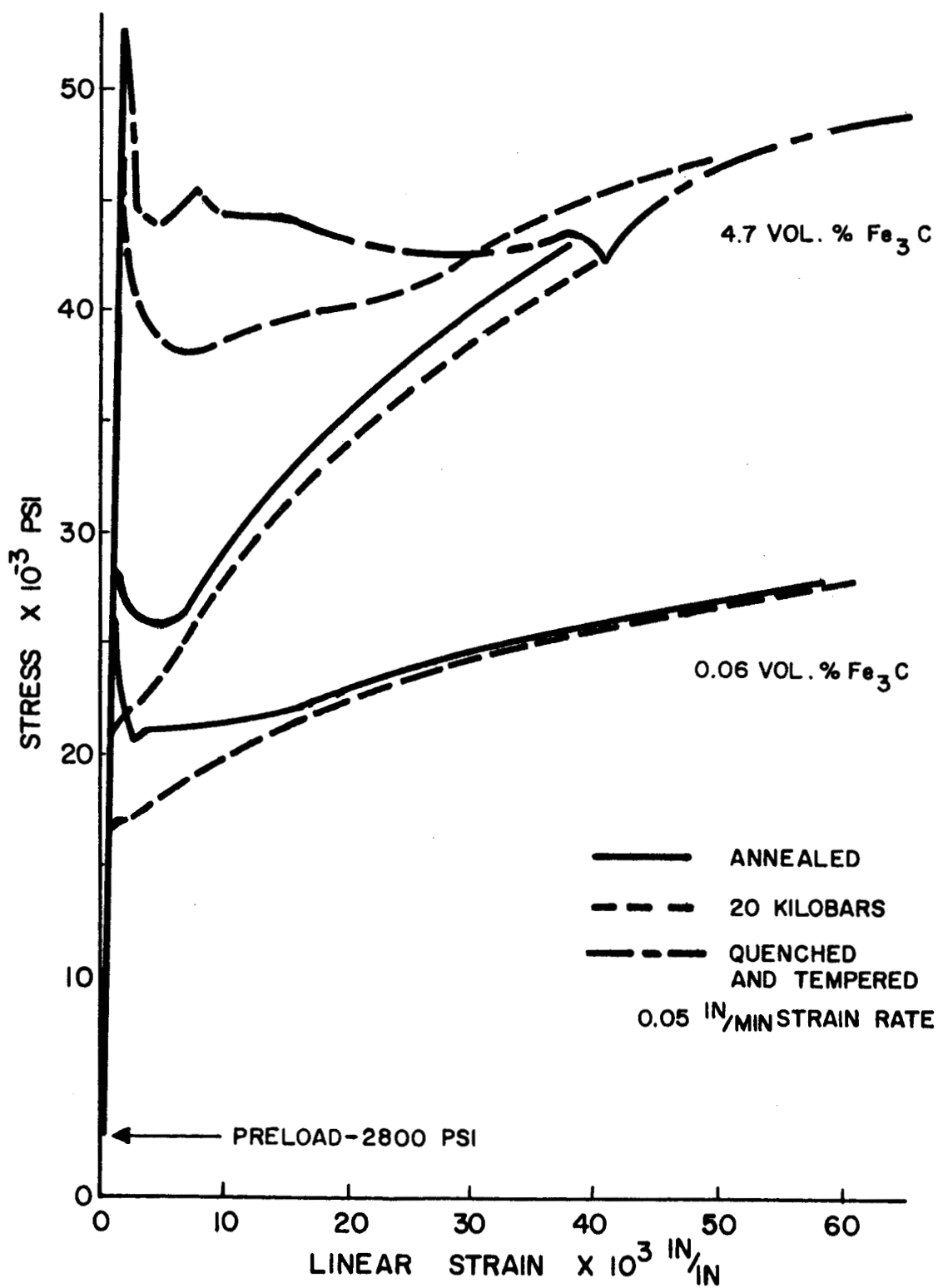
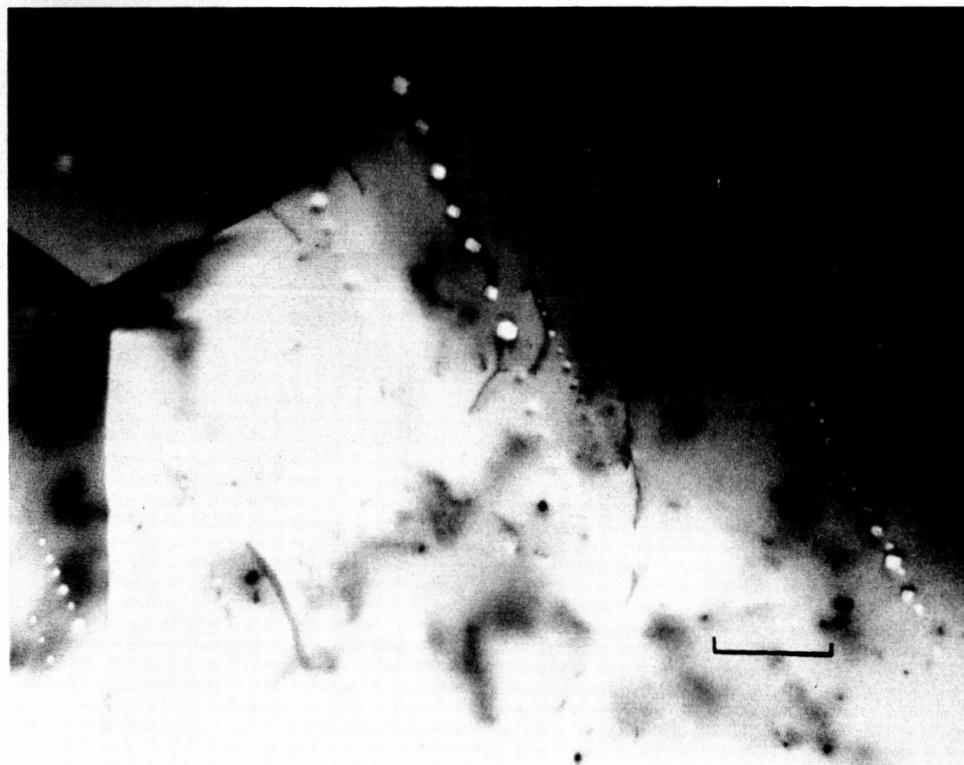


Fig. 3. Changes in the form of the tensile stress-strain curves following pressurisation to 20 kilobars for: (a) Fe-0.30 wt.%C alloy (two different Fe<sub>3</sub>C morphologies); and (b) Fe-0.004 wt.%C.



(a)



(b)

Fig. 4. Comparative thin-foil electron micrographs of annealed powder metallurgy tungsten.  
(a) undoped (X40K)  
(b) doped (X16K)  
Scale marker indicates 1 micron.

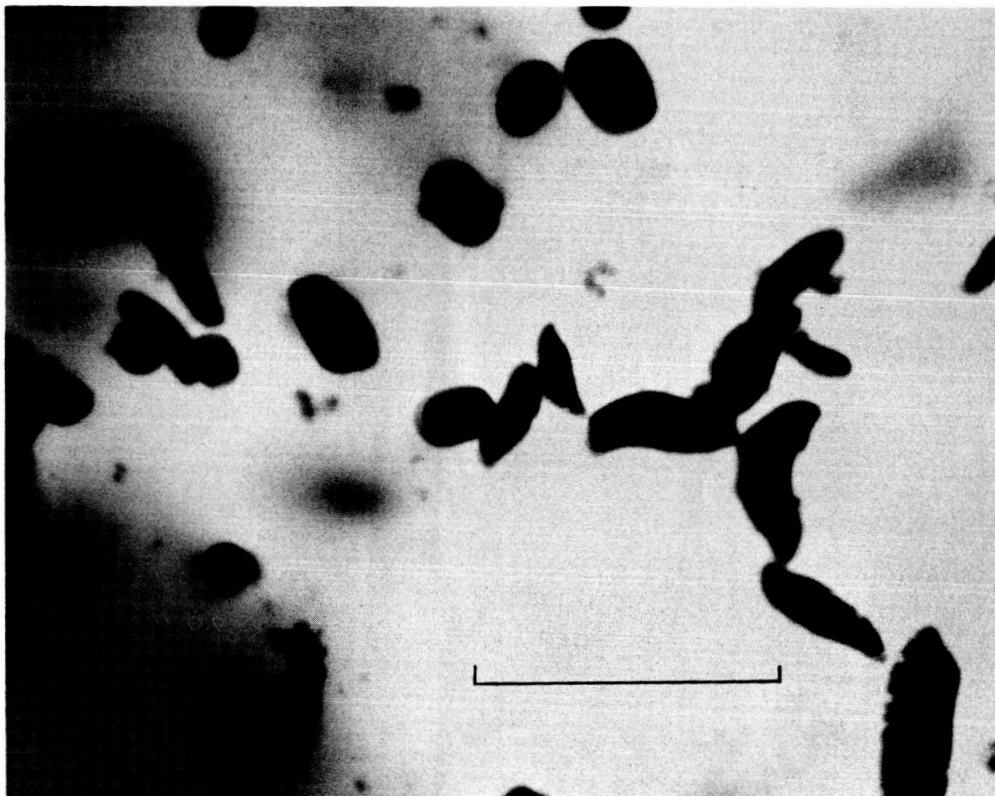


Fig. 5. Thin foil electron micrograph of W - 0.88 wt% Hf - 0.062 wt%C alloy solution treated at 2770°C for 15 minutes and cooled rapidly. X40K. Scale marker indicates 1 micron.

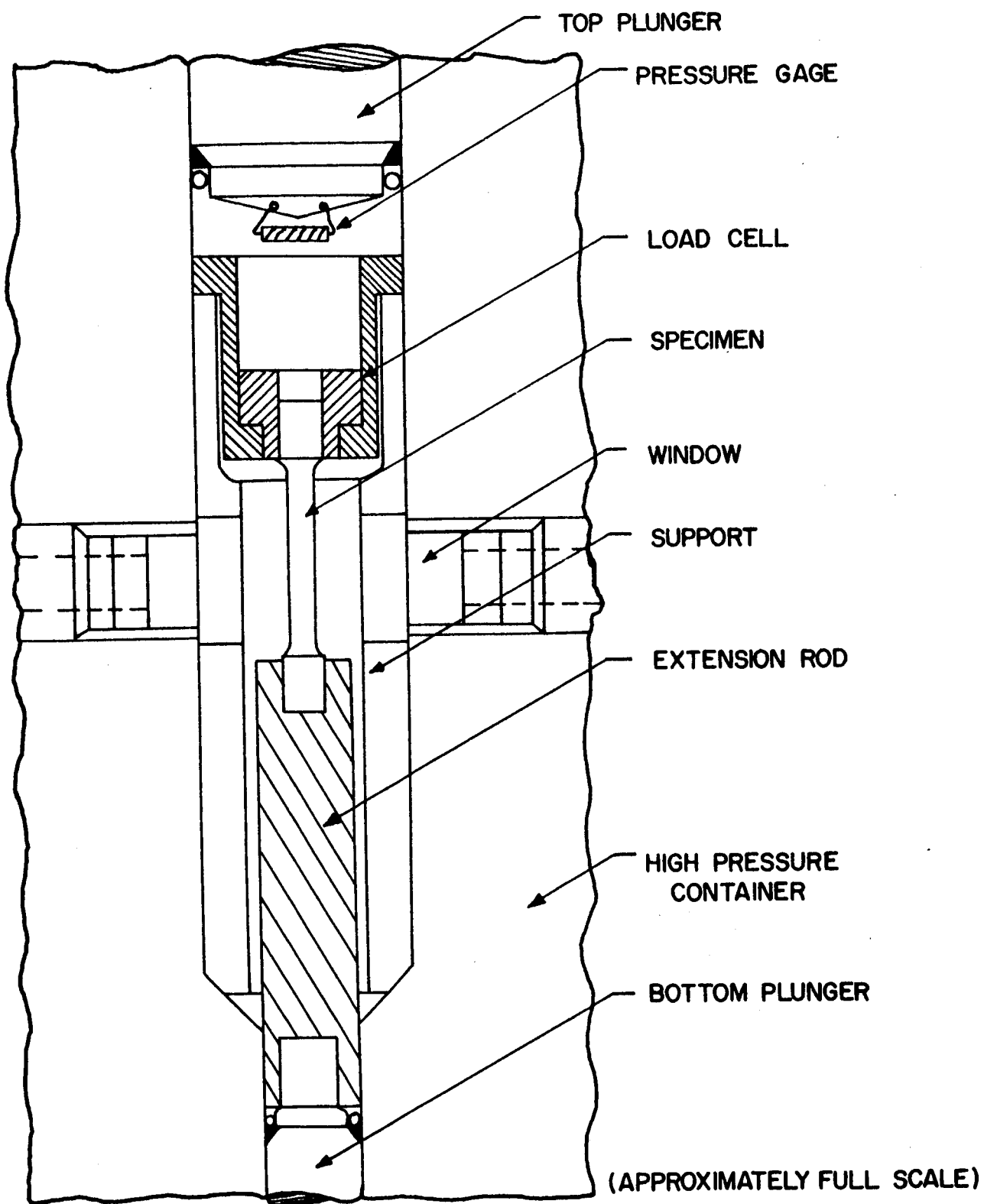
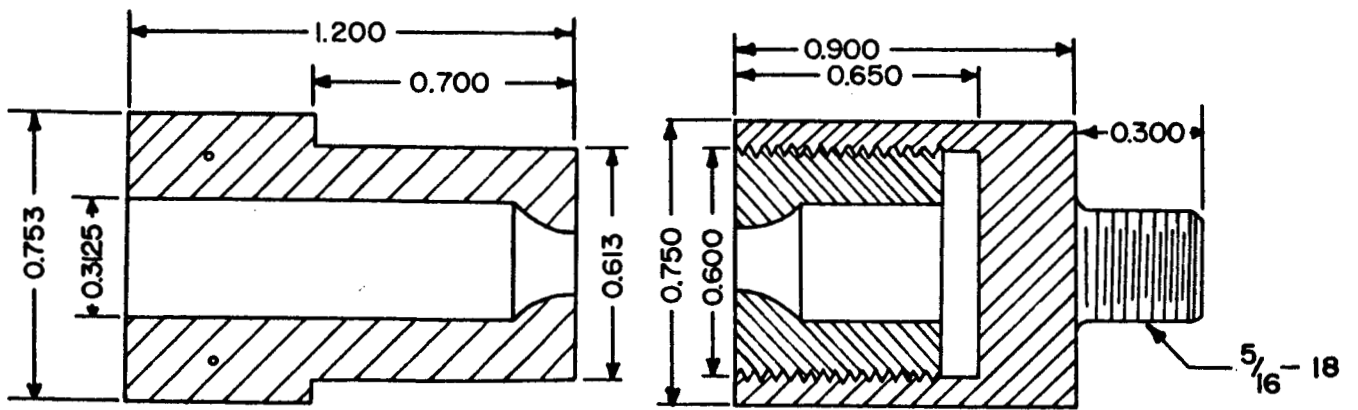
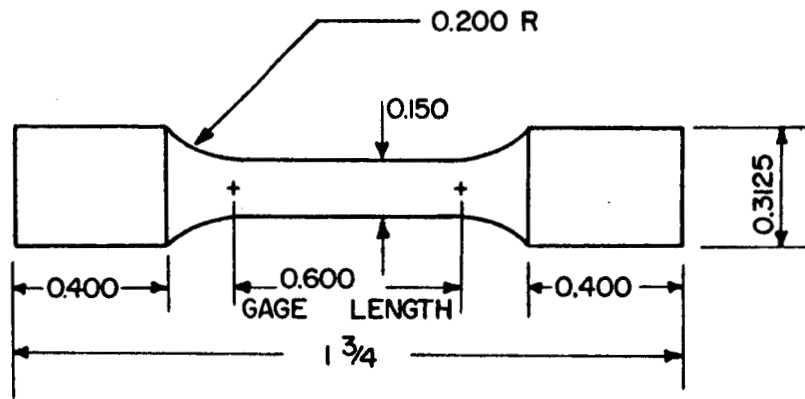


Fig. 6. General arrangement of constant pressure tensile apparatus.

TENSILE SPECIMEN



GRIPS FOR BUTTON-ENDED TENSILE SPECIMEN

Fig. 7. Details of tensile specimen (as-ground) and grips.