E-8 SOME EFFECTS OF HYDROSTATIC PRESSURE ON YIELDING AND BRITTLENESS IN IRON AND CHROMIUM

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ABSTRACT

Brittle recrystallized chromium becomes ductile and the yield point in Armco iron disappears at atmospheric pressure after these materials have been subjected to hydrostatic pressures of ~10kbars. Free dislocations are formed at inclusions of different compressibility to the matrix when the pressure is applied and the friction stress prevents some of these dislocations from running back and disappearing on removal of the pressure. After pressurization, chromium specimens which would otherwise be brittle commonly elongate ~60% before tensile fracture. Pressurized chromium is brittle at higher strain rates, showing that the presence of free dislocations is not a sufficient condition for ductility. Conversely, the absence of free dislocations does not ensure brittleness, because strain-ageing after pressurization produces a yield point but does not remove the ductility.

The condition for the brittle/ductile transition in chromium is considered in the light of these observations and it is concluded that the specimens are ductile when their yield or flow stress is insufficient for crack-initiation.

1. INTRODUCTION

In general, three processes occur consecutively in the brittle fracture of b.c.c. metals;[1] the most difficult in the particular circumstances determines the critical condition for fracture. The processes and their inter-dependence may be simply described in the following way. It is first necessary to move dislocations; these dislocations must then aggregate to initiate a crack; finally, the crack must propagate through the specimen.

The conditions for the initial movement of dislocations, yielding,

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have been studied extensively[2]. The behaviour of propagating cracks is also reasonably well understood from fracture studies of materials containing known cracks[3] and from metallographic examinations of fracture surfaces[4,5]. However, little is known about the mechanism of crack-initiation and its dependence on testing conditions and metallurgical structure.

Metallographic observations show that the brittle/ductile transition with temperature in recrystallized b.c.c. metals occurs when cracks of microscopic dimensions are unable to propagate against the high plastic relaxation[6,7]. Under these conditions, propagation is considerably more difficult than either yielding or crack-initiation.

Below the transition, fracture occurs at yield and the fracture stress is governed by the difficulty of moving solute-looked dislocations. The yield stress is sufficient for both the initiation and propagation of cracks, so that there is no indication of how difficult it is for these processes to occur. In theory at least, it should be possible to eliminate the yield point arising from solute-locking and so reduce the yield stress to a value insufficient for either initiation or propagation of cracks, i.e. to obtain ductility. Previously, there has been no way of eliminating the yield point without drastically changing the metallurgical structure. Methods such as extreme purification, quenching from high temperature, and prestrain at elevated temperature cause structural changes of such significance that comparison with the original material is almost meaningless. Consequently, little is known about the initiation or propagation of cracks in impure b.c.c. metals of structure reasonably comparable to recrystallized material, under conditions where the recrystallized material itself is brittle.

The present authors and colleagues[8,9] have developed a technique for removing the yield point in recrystallized material by a structural change so slight that it is difficult to detect. Specimens are subjected, by immersion in a pressurized fluid, to a hydrostatic pressure of $\sim$10 kbars for a short time; this procedure will subsequently be called pressurization.* Because of the differential compressibility between the specimen and its contained inclusions, the hydrostatic pressure causes local plastic deformation around the inclusions. Prismatic dislocations are formed as in the case of differential contraction during cooling.[10] However, there are two important differences between the two cases. Firstly, the pressurization is applied at room temperature where solute cannot readily diffuse to lock the newly-formed dislocations; the pressure-induced dislocations can therefore affect the yield point. Secondly, the pressure is applied and then removed, so that the local deformation should tend to reverse itself, i.e. the prismatic dislocations should run back and disappear. That they do not is shown by the effect on the yield point at atmospheric pressure and by

* See Appendix for fuller experimental details.

thin-foil electron microscopy (fig.1 and reference 11). The reason for this behaviour will be discussed later.

Before considering the effects of pressurization on brittleness in chromium we will briefly discuss its effect on yielding in centre-annealed[12,13] iron specimens. This will establish the structural change induced by pressurization and explain the unusual behaviour of pressurized specimens. Dislocations induced by pressurization effect properties because - (a) they are mobile and, (b) they are located near inclusions. Examples of both types of behaviour will now be considered.

2. EFFECTS DUE TO THE MOBILITY OF THE PRESSURE-INDUCED DISLOCATIONS

Yield points are extremely sensitive to the presence of free dislocations[2,14,15]. Pressurization can entirely remove the yield point in material containing a high density of suitable inclusions[8] (figs. 2a & b); the same pressure has far less effect on material which contains few inclusions[8](figs.2c & d).

Three aspects must be considered in discussing the removal of a yield point -

(a) The upper yield arises from the necessity to form a yielded zone of some critical size[2]. Hence the upper yield can be removed by a localized deformation, such as surface-abrasion or bending[13].

(b) The lower yield represents the propagation of yielding from this zone into adjacent unyielded regions. Grain boundaries provide the major resistance to propagation[2,16], so that the lower yield disappears only when virtually all grains contain free dislocations.

(c) Even when all grains contain free dislocations a yield point still occurs if the total number of dislocations or their mobility is insufficient; this effect has been treated for single crystals by many workers[2,14,15].

In accordance with these considerations, tests on Armco iron[8] show (fig.3) that, as the pressure increases, the UYP is first affected (reduced); then the LYP becomes unstable and finally disappears leaving a rounded yield point which decreases in size until it too disappears. This behaviour occurs because the inclusions are not uniform in size, composition or compressibility and thus will not all generate dislocations at the same pressure. At low pressures dislocations will only form around the few most favourable inclusions; the few grains containing such inclusions can aid the formation of a yielded zone and thus reduce the upper yield stress. The lower yield remains until pressures are applied which are sufficient to activate less favourable, but more commonly occurring, inclusions. For a given dispersion of inclusions, the pressure at which the LYP disappears is determined by the necessity to activate inclusions in nearly all grains. This is consistent with the observation[8] that the LYP disappears at lower pressures in coarser-grained materials, since a coarse-grained material requires fewer active inclusions to fulfil the above condition. The instability of the LYP before it disappears is probably due to inhomogeneous dispersion of inclusions.

The rounding of the yield point before its final disappearance arises from the effects of limited dislocation mobility. This can be shown by lowering the temperature and thereby reducing the mobility; even pressurized specimens which show no yield points at room temperature exhibit characteristic rounded yield points at low temperature[8] (fig.4).

The occurrence of marked effects due to low dislocation mobility shows that the density of free dislocations induced by pressurization is extremely low, probably several orders of magnitude less than the dislocation density in the recrystallized material.

3. EFFECTS DUE TO THE PRESENCE OF DISLOCATIONS NEAR INCLUSIONS

The linear compressibility of metals of cubic structure is isotropic[17] and macroscopic yielding does not occur under hydrostatic pressure. The disappearance of the yield point shows that dislocations formed at inclusions on application of the pressure remain after the pressure is removed. Hence pressurization leaves residual stresses around such inclusions; these stresses will remain until the dislocations run back to their 'parent' inclusions and disappear or until the pressure is re-applied. Evidence for the existence of these residual stresses will be given in the following sections.

3.1 The 'Memory' Effect[8,18]

The free dislocations resulting from pressurization can of course be locked by solute-diffusion. The yield point of a pressurized and aged iron specimen does not differ appreciably in shape or magnitude from that of the initial recrystallized specimen (fig.5). However, locking the dislocations does not remove the residual stresses. On re-application of the same pressure after ageing, these residual stresses oppose the formation of new dislocations, i.e. the residual stresses are relieved elastically. Hence re-pressurization at the same pressure has little effect on the yield point of a pressurized and aged (at 150°C) specimen (fig.5).

This 'memory' of a pressurized iron specimen disappears progressively as the ageing temperature increases from 150°C to 650°C (fig.6). Thermal activation assists the dislocations to glide back and disappear, thus reducing the residual stress. As the residual stresses decrease, more dislocations form on re-pressurizing and the yield point is increasingly affected. The wide temperature range over which this phenomenon occurs can be explained if the dislocations are prevented from running back at room temperature (after removal of the pressure) by the friction stress. This is likely because the repulsive interactions[19] between successive like prismatic dislocations from the same inclusion can force the leading dislocations far from the inclusion. On removal of the pressure the restoring force on these dislocations will be less than the friction stress. The degree of thermal activation required to 'free' these dislocations varies with their distances from the inclusions, so that a wide range of effective activation energy will result.

On re-pressurization at increased pressure after ageing at 150°C, more inclusions are activated and the yield point disappears.

3.2 Effect on Yield by Twinning[20]

The residual stresses around inclusions after pressurization can be expected to interfere with any process which depends for initiation upon stress-concentration by inclusions. At low temperatures, Armco iron yields by twinning rather than by slip; it is usually considered that under these conditions the glide stress exceeds the stress for twin-initiation[21]. Since twins are initiated at stress-concentrators, it is not surprising that pressurization increases the twinning stress; fig.7 shows that the yield stress of pressurized specimens at low temperatures is greater than that of recrystallized specimens and that the twin density is decreased by an order of magnitude.

At low temperatures, pressurized and aged specimens also have higher yield stresses than recrystallized specimens (fig.7). Hence the effect does not depend on the mobility of the pressure-induced dislocations. Since twinning is suppressed by locked dislocations around inclusions, we conclude that most twins in Armco iron are initiated at inclusions.

4. EFFECT ON BRITTLENESS[9]

In view of the small plastic deformation induced by pressurization, the effect on brittleness is surprisingly large. After pressurization, polycrystalline chromium of suitable metallurgical structure elongates ~ 60% before tensile fracture; the original recrystallized material is brittle at room temperature. Pressurized chromium yields at stresses as low as half the brittle fracture stress of recrystallized chromium, and the close parallel between the yield characteristics of chromium and iron after pressurization shows that the same structural changes occur in both cases.

Since pressurized chromium is ductile the yield and flow stresses must be insufficient for either crack-initiation or crack-propagation. An extremely high degree of plastic relaxation around crack-tips would be required to prevent cracks from propagating under the high flow stresses

($\sim 6 \times 10^4$ p.s.i.) observed before fracture in pressurized specimens.[22] The effects of high plastic relaxation during crack-propagation are well-known and can readily be detected metallographically.[6] However, the microstructure of pressurized chromium after tensile fracture is not at all typical of a material in which crack-propagation is extremely difficult; non-propagating micro-cracks have not been detected and the fracture surfaces show almost complete cleavage (fig.8). We have concluded, therefore, that pressurized chromium is ductile because cracks are not initiated (we adopt a broad definition of initiation to include the stable growth of a sub-critical crack).

It follows that the ductile/brittle transition in pressurized chromium represents the limiting condition for crack-initiation. Brittleness can be induced in as-pressurized chromium by slight variations in temperature or strain rate which increase the yield stress because of the low dislocation mobility; considerably greater variations and much higher stresses are required to fracture specimens which have been pressurized and then deformed even a few per cent under our standard testing conditions (fig.9). Hence macroscopic deformation considerably increases the difficulty of initiating cracks, probably because plastic relaxation reduces the effectiveness of the operative stress-concentrators.

Ageing after plastic deformation locks the dislocations but does not remove the plastic relaxation which has already occurred. Hence it is not surprising that pressurized and deformed specimens can be aged without loss of ductility, although large yield points are observed and the yield stresses are far greater than the stress at which as-pressurized chromium becomes brittle.

The above discussion illustrates how brittleness in pressurized chromium can be considered in terms of a balance between the yield (or flow) stress and stress for crack-initiation. The yield stress is determined by the most weakly-locked dislocations, whilst the stress to initiate cracks probably depends on some different aspect of the metallurgical structure. Plastic flow occurs if the yield stress is less than the stress to initiate cracks at yield. Cracks will not form during the subsequent deformation if the stress for crack-initiation increases with increasing strain more rapidly than the flow stress; the large elongation to fracture of pressurized chromium shows that this condition is fulfilled up to large strains.

In the next section we use the approach outlined above to consider whether or not pressurization has a direct effect on the mechanism of crack-initiation.

5. THE EFFECT OF PRESSURIZATION ON CRACK-INITIATION

In principle, a treatment such as pressurization can induce ductility by reducing the yield stress, by increasing the stress for crack-initiation or by combining both effects. Obviously, pressurization decreases the yield stress; this alone may alter the balance in favour of ductility.

However, it is important to establish whether or not pressurization also affects the stress for crack-initiation and, if so, to what extent. Once this is known, tests on pressurized specimens can be used to establish the stresses for crack-initiation in a variety of metallurgical structures under a range of test conditions. Such data would greatly advance our understanding of crack-initiation.

The decrease in yield stress can be considered responsible for ductility when dislocations can propagate from the yielded regions around inclusions and relax stress-concentrations in other parts of the crystal at applied stresses which are not sufficient to initiate cracks. The previous section showed that cracks will not then initiate unless the flow stress increases at a sufficient rate with increasing strain to compensate for the increasing plastic relaxation.

An increase in the stress for crack-initiation could then result directly from the pressurization, only if -

(a) the free dislocations so change the process or mode of yielding that some particular condition which is conducive to cracking, such as highly-stressed regions at grain boundaries, no longer arises,

or

(b) cracks form at inclusions, either because of the stress-concentration or through some intermediary structural change such as twinning. The residual stresses around inclusions could then affect crack-initiation.

These alternatives fall into the categories adopted to discuss yielding earlier in this paper. Case (a) is an effect due to the mobility of the pressure-induced dislocations; case (b) depends on the presence of dislocations near inclusions. In principle, experimental methods similar to those used to study yielding are again applicable. Such tests are still in progress and firm conclusions cannot be reached at present. However, we will consider the experimental evidence obtained so far and try to establish the important trends.

5.1 Relation between Brittleness and Mode of Yielding

It seems unlikely that there is any close relationship between brittleness and mode of yielding, as in (a) above. Pressurized specimens fracture at yield when the strain rate is slightly increased or the temperature is slightly decreased. These specimens contain sufficient free dislocations to give only small rounded yields under our standard testing conditions.

Tests on iron suggest that the yield stresses of such specimens can be increased because of the low mobility of the dislocations, but that the mode of yielding is unlikely to change; note how the large yield points at low temperature in pressurized iron are distinctly different in shape from those in recrystallized iron (c.f. figs. 2 & 4).

Further evidence that brittleness is not directly related to the existence of a yield point is given by the behaviour of specimens prestrained at room temperature after pressurization. At approximately -20°C these specimens fracture with little or no plastic deformation, although they would be unlikely to show any yield point; after ageing, similar specimens tested at room temperature show large yield points but no loss of ductility.

We cannot conclude from these tests that the type of yield has no effect on crack-initiation, but it appears to have no decisive effect on the choice between brittleness and ductility.

5.2 Relation between Inclusions and Crack-Initiation

In our study of yielding, ageing was used to lock the pressure-induced dislocations in situ so as to decide which effects arose from the presence of dislocations at inclusions. In principle, we can use a similar method to determine whether cracks are initiated at inclusions, as in (b) above. However, the effect of ageing on the yield stress of pressurized chromium is more complex than in the case of pressurized iron. The yield stress of pressurized iron is independent of ageing temperature between 150°C and 650°C; the yield stress of pressurized chromium increases with increasing ageing temperature (Table).

Even after ageing at 400°C, the temperature at which pressurized chromium is embrittled, the brittle fracture stress is considerably less than that of recrystallized chromium. This disparity shows that the pressure-induced dislocations are more weakly-locked than the dislocations in the initial recrystallized state. The increase in yield stress with increasing ageing temperature could then result either from an increase in the segregation of solute or from the disappearance of pressure-induced dislocations during ageing. The slight decrease in the yield point on repressurizing after ageing (fig.10) indicates that some dislocations disappeared during ageing and were replaced during re-pressurization.[18] However, the re-application of the initial pressure after ageing does not restore ductility (fig.10). Hence the disappearance of dislocations is not the prime cause of the embrittlement and pressurized chromium by ageing.

Re-pressurization at slightly increased pressure[18] restores ductility (fig.10). Hence ageing does not permanently embrittle pressurized chromium, as might be expected if some structural change (e.g. precipitation on the dislocations surrounding inclusions) occurred during ageing.

A more detailed interpretation of these tests cannot be made because the

yield stress varies with each treatment. There is no gradation between brittleness and ductility, so that it is obviously unsound to base a comparison on the behaviour at different levels of stress. A more quantitative study is required and this is now being undertaken.

5.3 Relation between Yield Stress and Crack-Initiation

Although there is a possibility that pressurization has a direct effect on the stress for crack-initiation, the reduction of yield stress appears to be mainly responsible for the ductility of pressurized chromium. Whatever technique is used to increase the yield stress of pressurized chromium, there is at least a qualitative correlation between the magnitudes of the yield stresses at which brittleness returns; the critical magnitude does not appear to be greatly in excess of the yield stress under our standard testing conditions. At present, the variation in behaviour between different batches of material prevents more quantitative comparison of the results.

The one exception to the correlation is the behaviour of macroscopically-deformed specimens. As discussed earlier it is reasonable to attribute their behaviour to an increase in the stress for crack-initiation; the same pattern of behaviour should then apply, but with a different stress-level for crack-initiation.

6. CONCLUSION

We have concluded that pressurized chromium specimens are ductile because their yield stresses are insufficient for crack-initiation. The large elongations to fracture occur because the stress for crack-initiation increases more rapidly than the flow stress with increasing macroscopic deformation.

The present tests have not shown conclusively whether pressurization alters the stress for crack-initiation, but it appears probable that the reduction of yield stress is of greater importance with respect to ductility.

APPENDIX

EXPERIMENTAL DETAILS

Armco iron wires of 0.048 in. diameter were used for the tests on yielding. These wires were recrystallized over a short central length in vacuo at 700°C; 'centre-annealing' eliminates the effects of stress-concentrations at grips[12,13]. Compositions of the Armco and high purity irons have been given elsewhere[8].

Chromium specimens of 1 in. gauge length and 0.14 in. diameter were ground from swaged rod, electropolished and recrystallized in vacuo at 1250°C. Specimens for each series of tests were taken from a single rod; the results for different series are not quantitatively comparable because of slight differences in purity and structure. The main impurities were nitrogen (0.0008-0.005 wt.%) and oxygen (0.015-0.03 wt.%).

Specimens were held freely in either kerosene or petroleum ether in a steel container during pressurization. The pressure was increased slowly through a plunger, maintained for 5 mins. and slowly released. Pressurized specimens were either tested immediately or stored in dry ice to prevent ageing. Tensile tests were carried out in an Instron machine; a cross-head movement of 0.2 in/min was used for iron, and strain rates of $\sim 10^{-3}$/min were used for chromium.

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TABLE Tensile data on pressurized chromium (Batch 241) after ageing at various temperatures.[23]

Condition	Fracture or Upper Yield load (lbs.)	Lower Yield Load (lbs.)	Behaviour
Recrystallized 1250°C	400	-	Brittle
Pressurized 10 kbars	220	210	Ductile
Pressurized, aged 250°C, 16 hrs.	253	226	Ductile
Pressurized, aged 300°C, 16 hrs.	267	224	Ductile
Pressurized, aged 400°C, 16 hrs.	275	-	Brittle

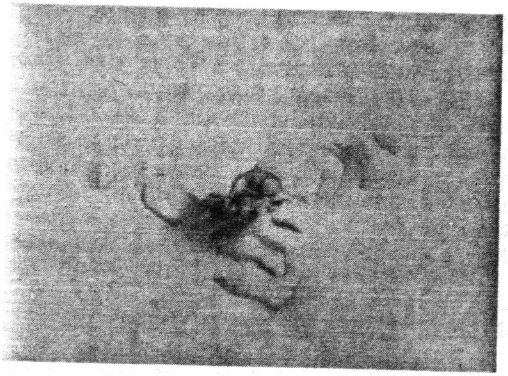

Fig.1 Transmission electron-micrograph of dislocations around an inclusion in pressurized chromium (24) 50,600X.

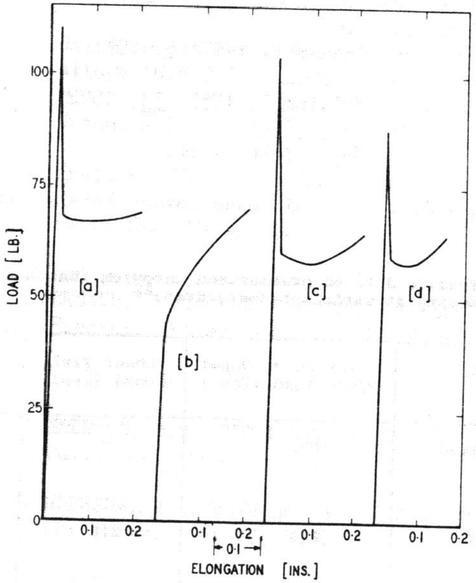

Fig.2 The effect of hydrostatic pressure on the yield point at atmospheric pressure.(8)

Armco iron -	(a) as recrystallized
	(b) after 10kbars pressure
High-Purity iron	(c) as recrystallized
	(d) after 10kbars pressure.

The Armco iron contains many more inclusions than the high-purity iron.

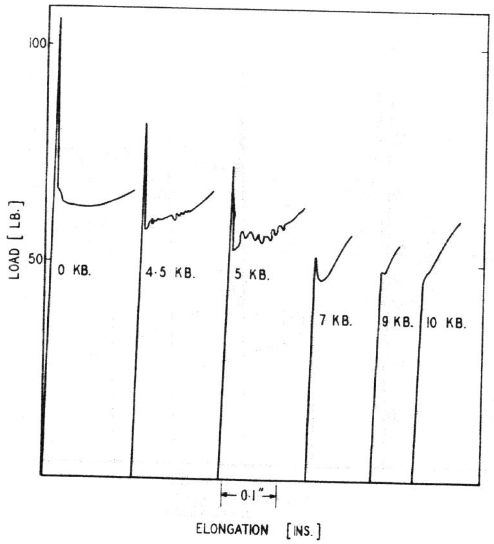

Fig.3 Stages in the removal of the yield point in Armco iron by pressurization;(8) pressures (in kbars) are shown.

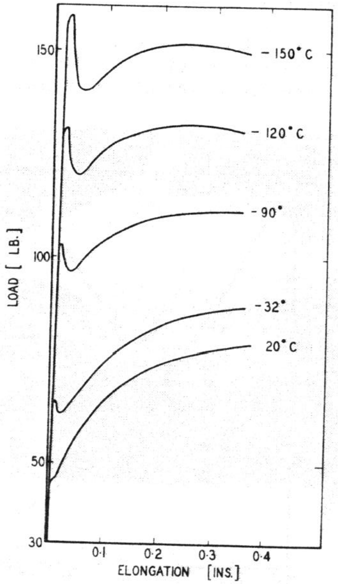

Fig.4 The effect of test temperature on the yield point in pressurized Armco iron.(8) Note that the yield point at low temperatures differs considerably in shape from that of recrystallized iron (Fig.2).

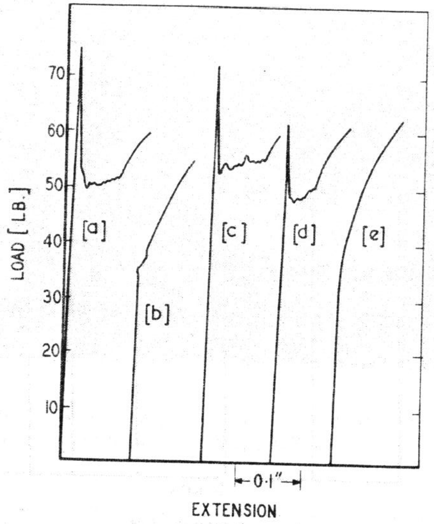

Fig.5 The effect of ageing and re-pressurizing on the yield in pressurized Armco iron.(18)

(a) as recrystallized
(b) as pressurized at 6.5 kbars
(c) after ageing at 150°C for 2 hours
(d) as (c) then re-pressurized at 6.5 kbars.
(e) as (c) then re-pressurized at 8.5 kbars.

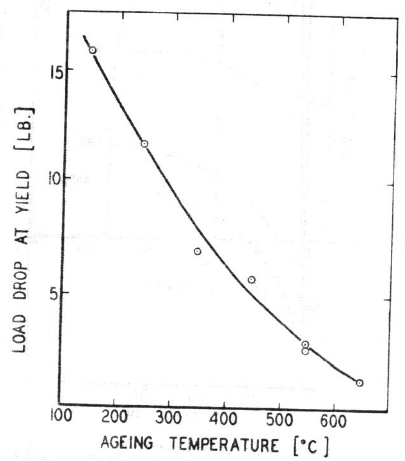

Fig.6 The load drop at yield of specimens which have been pressurized at 8 kbars, aged at various temperatures and then re-pressurized at 8 kbars.(18)

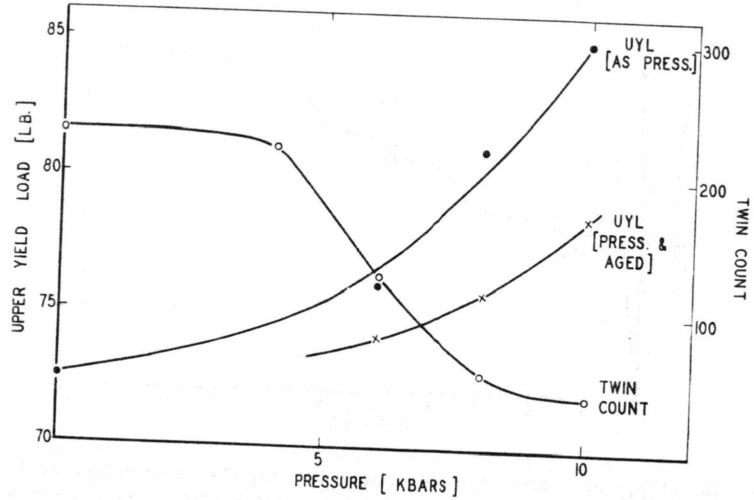

Fig.7 The yield stresses at 78°K of pressurized specimens before and after ageing at 150°C. The numbers of twins observed on a constant area of longitudinal section after yield are plotted for the pressurized specimens(20).

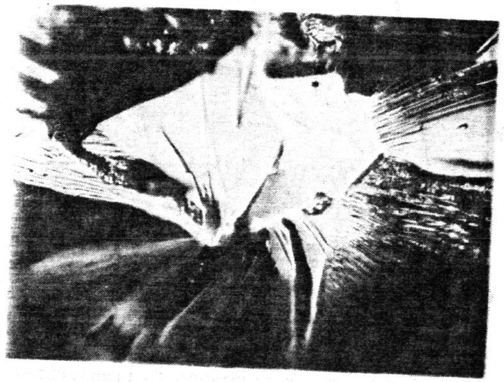

Fig.8 The fracture surfaces of chromium pressurized at 10 kbars and then fractured in tension after an elongation of 63%. 610X .

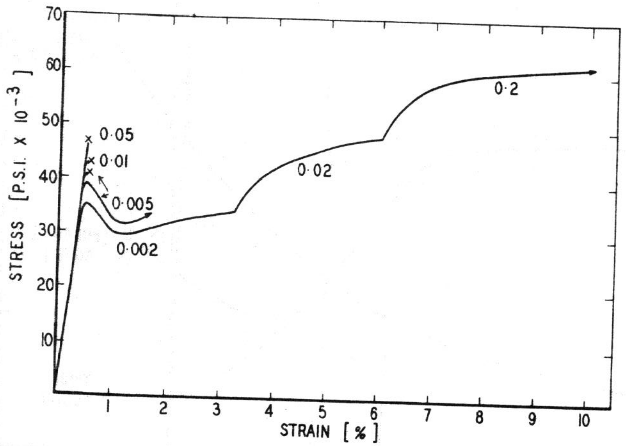

Fig.9 The effect of strain rate on fracture in chromium pressurized at 10 kbars.(9) Note that chromium deformed slowly even a few percent will then withstand far greater strain rates than those which cause fracture in pressurized chromium. The numbers shown are the strains/minute. Crosses denote fracture.

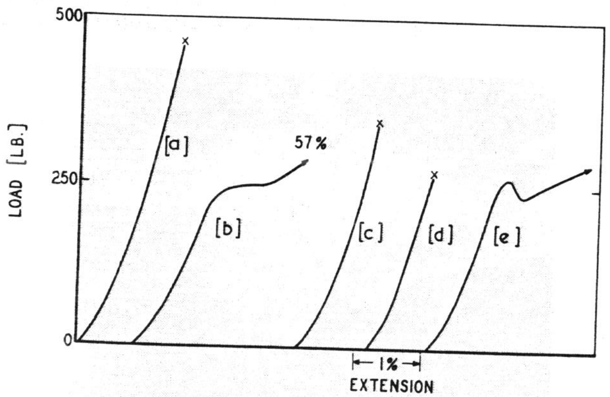

Fig.10 The effect of ageing and re-pressurizing on pressurized chromium.(18)

(a) as recrystallized
(b) as pressurized at 7 kbars
(c) after ageing at 400°C for 1 hr.
(d) as in (c) after re-pressurizing at 7 kbars
(e) as in (c) after re-pressurizing at 8 kbars.
Crosses denote brittle fractures.