Cleavage strength and dislocation structure in polycrystalline chromium

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Summary

Brittle (cleavage) fracture can be induced in chromium in which the structure is varied widely from recrystallized to heavily deformed. The transition from ductility to brittleness is characterized by a stress which for chromium is the stress to initiate, rather than to propagate, a crack. The general trend is for the stress for crack initiation to increase as the scale of the dislocation sub-structure decreases; the rate of increase rises very sharply below $\sim 1\mu$. For the coarser sub-structures the stress increases in discrete steps with decreasing scale, and direct evidence is presented to show that each step is associated with crack initiation by a particular type of dislocation array.

Heavily drawn wires (99·7% reduction in area), which had the smallest sub-structure ($\sim 0{\cdot}1\mu$), fractured at $\sim$20% of the theoretical strength when tested at high strain rates (c. 20/min) and low temperatures (c. −100°C). Under these conditions, this material combined high strength with significant ductility ($\sim$1%) indicating that crack initiation is extremely difficult. Thus a degree of toughness is possible in a material which nevertheless eventually fails by cleavage and in which the resistance to crack propagation is probably not high.

Introduction

This paper is specifically concerned with crack-initiation and the influence upon it of initial structural state. Chromium is particularly suited for such an investigation since, as we shall show, cleavage can be induced in specimens of widely differing structural states, ranging from as-recrystallized to very heavily worked. In less brittle materials, cleavage is only common in the as-recrystallized or lightly worked conditions.

Our previous work [1, 2] led to the introduction of two rather novel concepts which are further discussed herein, viz (i) even heavily deformed metals can fail by cleavage, and (ii) the change from ductility to brittleness is characterized by a transition stress, the value of which is determined by the dislocation configuration at 'yield'. The latter concept is related to the former by the *a priori* principle that (cleavable) specimens are only ductile when cracks do not initiate and propagate at the flow stress. Thus if by some means the yield stress for a given structural state is progressively increased, the transition to brittleness occurs when the flow stress becomes sufficient for cracks to initiate and propagate; under our conditions, it has been possible to link the transition stress with that required for crack initiation.

We have established that the transition stress in chromium does not vary continuously with either increasing pressurization [3] or prestrain. Constant levels exist, and comparatively rapid changes from one level to another occur at critical values of pressure or prestrain. Each level is believed to be characteristic of a particular dislocation configuration which forms at yield and which gives a particular degree of stress-concentration.

The present paper describes further experiments which extend the scope of these observations considerably; in particular, the proposed interpretation of the various levels of transition stress is checked by the direct determination of dislocation structures. The pressurization range is extended from 10 to 25 kbars and the use of drawn wires has increased the prestrain range up to 99·7% reduction in area. For brevity, we only detail those observations which are directly relevant to our present arguments; fuller accounts will be given elsewhere.

Experimental procedure

The procedure in the previous work, and in the experiments to be discussed, was as follows. The flow stresses of a series of identically pressurized or prestrained specimens were progressively raised by increasing the strain rate and/or by light ageing, neither of which should significantly modify the initial structure, until cleavage occurred at yield. The yield stress at this ductile/brittle transition, the transition stress, is the important parameter for a quasi-elastic process such as cleavage; higher fracture stresses on the brittle side of the transition merely reflect the necessity for yield to precede fracture.

Variation of strain rate is a particularly apt method of determining transition stress since the cleavage process itself is unlikely to be seriously rate dependent and the high strain rates give little opportunity for gross structural modification before brittle fracture. Lightly aged specimens have always given the same transition stress as unaged specimens of the same initial structure.

Details of materials, specimen preparation and testing methods were given previously [1, 2].

Effect of pressurization

Experiments using an etch-pit technique [4] show conclusively (Fig. 1) that free dislocations are formed at second-phase particles by pressurization. Other direct evidence of this effect was obtained by electron microscopy [5]. These results leave little doubt regarding the validity of our proposal that pressurization effects occur because dislocations are generated by stresses arising from the differential in compressibility between particles and matrix.

Previous work [1, 2] showed a step in transition stress after pressurization below 10 kbars; the critical pressure decreased with increasing grain size. Further tests [5] on specimens pressurized between 10 and 25 kbars (by courtesy of the Case Institute, Ohio, U.S.A.) reveal a further step in transition stress between 15 and 20 kbars (Fig. 2). Transmission electron micrographs (Fig. 3) show a significant change in dislocation *distribution* in the same pressure range as the step in transition stress; the dislocation *density* increases throughout the entire pressure range. Below the step, the distribution is markedly inhomogeneous (Fig. 3 (a)); most dislocations are in groups, usually around particles, separated by clear regions of at least the order of size of the groups (see also Fig. 1). Above the step (Fig. 3 (b)), no large clear areas were found between the dense clusters of dislocations.

Relatively large arrays of dislocations can be expected to form at tensile yield in any existing clear regions. The elimination of such regions above the step will preclude the high stress-concentration associated with such arrays. Thus cracks will be initiated by some other array of lower stress-concentration with a resulting increase in fracture stress. The electron micrographs are therefore quite in accordance with our interpretation of the steps between levels of transition stress.

Effect of tensile prestrain

We have previously reported [2] that the stress required for cleavage in prestrained specimens shows a similar step-wise increase, between constant levels, with increasing prestrain. Specimens were pressurized at 10 kbars (to obtain ductility), prestrained given amounts at low strain rates and then tested to fracture at high rates. The cleavage stress did not vary with the extent of the (small) strain prior to fracture at the higher rate, nor with the rate itself. Specimens which were lightly aged after the prestrain underwent a ductile/brittle transition at the same stress.

Preliminary tests, combining stress measurements and electron microscopy on the same specimens, suggest that the step in cleavage stress is associated with a particular stage in the development of a dislocation cell structure. Strip specimens were prestrained given amounts at 150°C at slow strain rate (2×10^{-3}/min) and then fractured at room temperature and high rate (20/min). A typical step in fracture stress occurs after 5-8% prestrain (Fig. 4). Electron micrographs show the beginnings of a cell structure at as low as 2½% strain, but it is apparent (Fig. 5) that severe misorientations of the cells only occur commonly at strains beyond the step; at this stage, the cell walls also become sharper.

The increasing misorientation and increasing sharpness of the walls are consistent with a 'polygonization' of the cells which will decrease the long-range stresses from the dislocations in the cell walls. It would be surprising if this polygonization did not increase the cleavage stress, because either – (i) if the dislocations in the walls are directly responsible for crack-initiation, then a decrease in their long-range stresses would automatically increase the applied stress required for crack-initiation, or (ii) the polygonized cell walls would offer greater resistance to dislocations and thus would tend to restrict the length of dislocation arrays to a single cell width instead of multiples thereof. The stress required for crack-initiation by arrays of such restricted length could be higher than that required by some other mechanism; this would account for the constant level of fracture stress beyond the step.

Thus the micrographs again give clear evidence of significant changes in dislocation substructure which can be associated with the observed sudden increase in cleavage stress.

Heavily drawn wires

Wires were prepared by drawing from 0·190 in to 0·110 in diameter, after initial hot-swaging and surface grinding. The extent of work in the wires after drawing was varied by recrystallizing individual wires at 950°C at selected intermediate diameters; reductions in area subsequent to recrystallization ranged from 80% to 99·7%. The wires showed a marked [110] fibre texture.

As-drawn wire specimens of 0·010 in diameter and 1 in gauge length were fractured in tension at a variety of temperatures and strain rates. As with pressurized and prestrained wires, there appears to be a definite stress condition for fracture. This fracture stress has small rate and temperature dependences, each considerably less than that for the yield stress (Fig. 6). Three types of behaviour were observed and these are explained in terms of the relativity between yield stress and fracture stress as follows:

(i) at low temperatures (c. –100°C) and/or high strain rates (c. 20/min), the yield stress exceeds the fracture stress and brittle fracture occurs,

(ii) at intermediate temperatures and rates the fracture stress, although slightly beyond the yield stress, can be reached by work-hardening and thus specimens fracture suddenly after small plastic strain ($<\sim 5\%$),

(iii) at higher temperatures (c. room temperature) and/or lower rates (c. 2×10^{-3}/min), the fracture stress exceeds the ultimate tensile strength and the specimen necks and fractures suddenly.

In all cases, the stress/strain curve did not deviate measurably before fracture; the trace fell from (apparently) the normal stress/strain curve to zero in less than 10^{-3} secs. Under all conditions of test, the fractures were remarkably similar in appearance and quite characteristic in their unusual morphology which can best be described as a double cup and cone. The

complex nature of the fracture will be discussed elsewhere [6], but it is apparent from the radiating 'chevron' pattern of Fig. 7, the suddenness of fracture, the reasonable analogy with previous results and the characteristic appearance of the fractures, that all fractures are brittle, and presumably occur by cleavage.

Non-propagating cracks were not detected metallographically, even in sections of specimens unloaded during the necking stage immediately prior to fracture. Thus we conclude that the ductility at these high strength levels reflects the extreme difficulty of initiating cracks. Indeed, the strength and ductility of the wires are quite remarkable for pure chromium. After 99·7% reduction in area, elongations exceeding 1% were obtained under (tensile) conditions as adverse to ductility as –100°C and 20/min; the fracture stress was greater than one-fifth of the theoretical strength. Tests showed (Fig. 8) that the fracture stress increased rapidly with reduction of area up to 98% (equivalent to 50 times the initial length) but only increased slightly thereafter (up to 330 times). Possibly, this small rate of increase corresponds to one of our constant levels of fracture stress; these levels, however, are usually confined to very restricted ranges of strain or pressure and are thus only constant within a first order approximation. Alternatively, little further change in substructure may occur beyond 98%; this would be reflected by a fairly small change in fracture stress.

The latter alternative is perhaps to be favoured since the yield stress also shows little increase beyond 98% reduction; in most worked structures [7] the flow stress varies as (sub-grain diameter)$^{-1/2}$. Work is now in progress to determine directly the variation of sub-grain size with deformation by drawing. To date we have found that after 97% reduction the sub-grains are relatively clean with $\sim\frac{1}{4}$ micron spacing (in the direction transverse to the wire axis) (Fig. 9).

Discussion

Our observations show that, over an extremely wide range of sub-structural states, each state has a characteristic stress for crack initiation, just as it has a characteristic flow stress under given conditions of test. Brittle fracture results if this flow stress exceeds the crack-initiation stress. The variation of fracture stress with *scale* of sub-structure is summarized in Fig. 10. The outstanding features are (i) the step-wise increase in cleavage strength, at low strengths at least, and (ii) the rapid increase in fracture stress, tending towards the theoretical strength, as the scale decreases below ~ 1 micron.

We will not repeat our discussions [1, 2] of the constant levels of fracture stress, beyond pointing out that we have now shown that significant changes in the *type* of the sub-structure occur at approximately the same strains or pressures as changes in levels of fracture stress. Thus our direct determination of sub-structural variations entirely supports our previous interpretations. Our main interest here is to reconcile the existence of ranges in which the cleavage stress is constant, although the scale of the particular sub-structure must vary considerably, with the general trend (Fig. 10) towards increased fracture stress with decreasing scale.

This apparent anomaly probably arises because, at relatively large scales, the crack-nucleating array does not occupy the entire spacing of the sub-structure. For example, on the coarsest scale, the transition stress for recrystallized and lightly pressurized specimens does not vary significantly with grain size [2]; this is quite understandable if pile-ups (say) do not spread completely across grains. Similarly, in our study of the 15-20 kbar step, the fracture stress is constant during a large reduction in the size of the clear spaces between groups of dislocations, although it increases rapidly when the spaces are nearly eliminated.

It is implicit in our argument that the cleavage stress will begin to be affected when the scale of the sub-structure is reduced to that of the operative crack-initiating array. On further reducing the scale, the cleavage stress should increase continuously unless it exceeds the stress required for some other type of array (or mechanism) to initiate cracks; if, at this stage, this other array is smaller in scale than the sub-structure, another constant level can result. Thus it is not unreasonable, at large scales, for constant levels of fracture stress to be interspersed with rapid variations over small ranges of sub-structural scale.

For the heavily drawn wires, it seems improbable that the crack-nucleating array is of smaller scale than the sub-structure (c. ¼ micron). Judging from the slight increase in yield stress, the scale of sub-structure changes only slowly beyond 98% reduction in area and the slow increase in fracture strength probably shows a progressive restriction in the size of the crack-nucleating array by the sub-structure. The exact variation of fracture strength with scale of sub-structure, at these scales where the strength approaches the theoretical, is of great interest since it is at least possible that the same fracture strength results whatever means are adopted to reduce the scale. Thus this relationship could possibly be used to predict the fracture strengths of complex alloys, such as those in which extremely small scales are induced by complex thermo-mechanical treatments which produce finely dispersed second phases. These alloys are of greater practical significance than heavily worked pure metals, which are limited by the instability of their cold-worked state but which are easier to study.

Finally, it is of interest to contrast the behaviour of chromium in its various worked states. In recrystallized or lightly worked states, the yield and transition stresses are always close and ductility is confined to special

conditions of test. In the intermediate worked states, the difference between transition and flow stresses is large under normal test conditions, but this is of little practical advantage; the high rate and temperature dependences of the flow stress and the rapid work-hardening make it easy to bridge the gap and initiate cracks which can propagate catastrophically. However, the very heavily worked states are a different proposition because then the work-hardening rate and the rate and temperature dependences of the flow stress are all relatively low. Thus although this state appears capable of cleavage, and probably still lacks resistance to crack-propagation, conditions suitable for crack-initiation are unlikely under normal usage. Indeed the reasonable elongation to fracture coupled with the very high flow stress gives some semblance of toughness to the fracture characteristics. It is interesting to speculate whether other high-strength alloys, which depend on very fine substructures for their properties, also derive their fracture toughness from their resistance to crack-initiation, instead of from their resistance to propagation as is commonly assumed.

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Fig. 1. Etch-pits at the sites of dislocations induced at second-phase particles by pressurization at 10 kbars (×320).

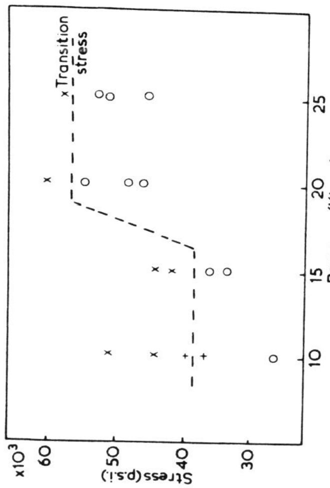


Fig. 2. Yield and brittle fracture stresses for specimens tested at various rates, at room temperature and pressure, after soaking at various pressures; open circles show yield stresses of ductile specimens, crosses show fracture stresses of brittle specimens (Ref. 5).

(a)

(b)

Fig. 3. Electron micrographs showing dislocation structures of specimens after pressurization (a) at 15 kbars, (b) at 20 kbars.

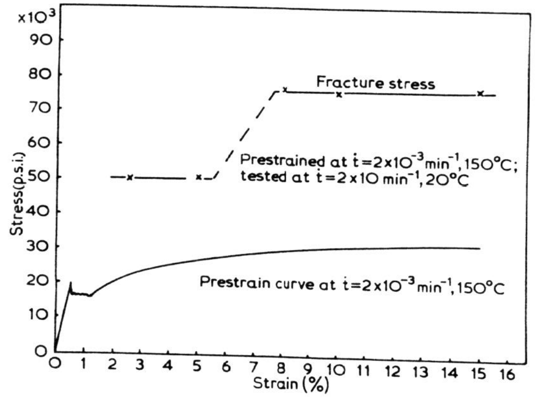
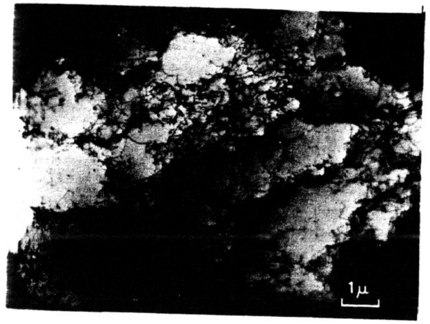
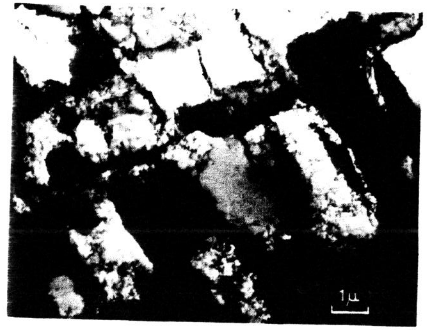


Fig. 4. Fracture stresses, at room temperature and high strain rate, of specimens prestrained given amounts at 150°C and low rate. No significant deformation occurred prior to fracture at high strain rate.



(a)



(b)

Fig. 5. Electron micrographs showing dislocation structures of fractured specimens initially prestrained at 150°C; same specimens as for Fig. 4. (a) after 5% prestrain, (b) after 10% prestrain. Note the increased sharpness of walls and the increased misorientations of subgrains in (b).

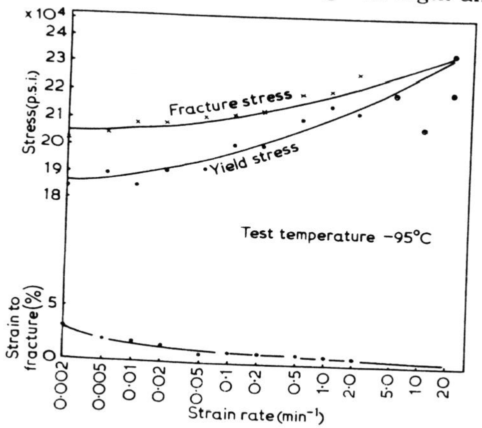
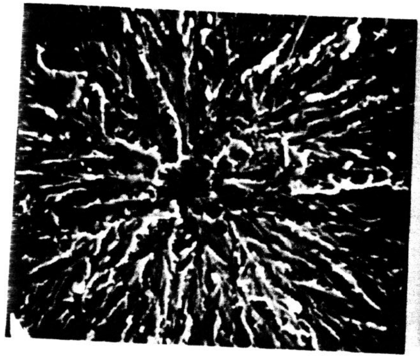


Fig. 6. Yield and fracture stresses of heavily drawn (97% reduction in area) wires tested at −95° C at various strain rates; elongations to fracture are also given.

Fig. 7. Fracture surface, taken by scanning electron microscopy, of heavily drawn (97% RA) wire fractured at −110° C, 20/min with no measurable elongation (× 1100).



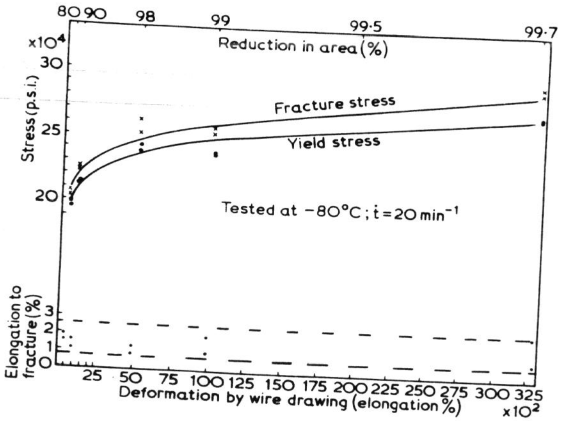


Fig. 8. Yield and fracture stresses and elongations to fracture of drawn wires tested at 20/min, −80°C. The highest strength exceeds one fifth of the theoretical strength.

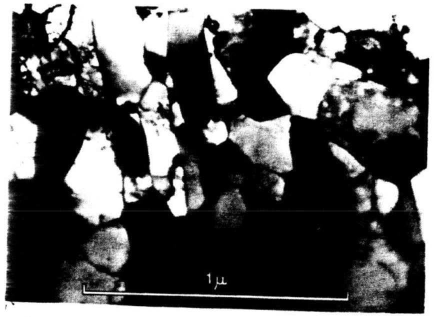


Fig. 9. Electron micrograph of a transverse section of heavily drawn (97% RA) wire.

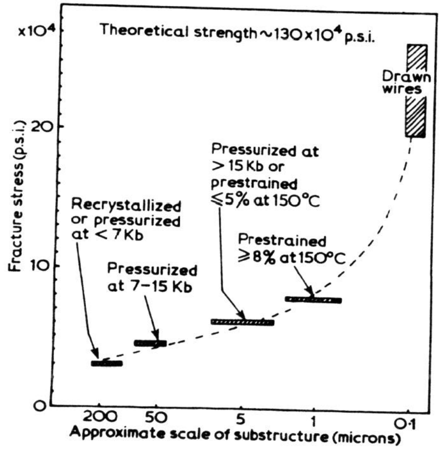


Fig. 10. Schematic variation of the fracture strength, determined by the crack-initiation stress, with scale of substructure. Note the rapid increase in strength with scale below ~1 micron.