

Brittle-Ductile Transitions in Vanadium and Iron-Chromium

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Abstract

We report ongoing experimental work on fracture and brittle ductile transitions in vanadium, iron and iron - 9% chromium single crystals and polycrystals. Tests were carried out by four-point bending of pre-cracked specimens in the temperature range 77K - 300K, at a variety of strain rates. For a given material, variation of the brittle-ductile transition temperature with strain rate allows us to estimate an activation energy for the controlling process; for vanadium this was found to be ~0.37eV, for iron ~0.21eV and for iron - 9% chromium ~0.11eV.

Introduction

Materials currently anticipated for pressure-vessel structural components of the proposed DEMO prototype fusion power plant include reduced activation ferritic-martensitic (RAFM) steels based on iron ~9% chromium [1,2], though vanadium alloys [2] and tungsten [3] have also been suggested for some specialist applications. These materials may offer adequate strength and toughness and minimal sensitivity to fast neutron / alpha particle irradiation, effects such as swelling and increase in brittle-ductile transition temperature. However, it has been noted that for RAFM steels, “Further reduction of brittle-ductile transition temperatures should be pursued.” [4]. It is difficult, expensive and time-consuming to perform experiments on irradiated materials. Modelling of effects of irradiation on flow properties is now becoming

more frequent (e.g. [5, 6]). While it is “obvious” that easier plastic flow implies a less brittle material¹, explicit links between flow behaviour and fracture are less easy to model, except, at present, for relatively simple, generally single crystal, materials.

Many crystalline solids fail by brittle cleavage at low temperatures, and by plastic processes at high temperatures. For most materials, the fracture stress intensity factor (K_{Ic}) increases from “lower shelf” values associated with brittle fracture over a brittle-ductile- transition temperature range of 100 K or more: single crystal BCC metals [7,8,9], intermetallics [10, 11, 12], MgO [13], germanium [14] and most other materials exhibit this type of transition (as of course do polycrystalline ferritic steels [15]). In some cases, e.g. for Si [16,17] and Al_2O_3 (sapphire) single crystals [18], the transition is very sharp, occurring over a temperature range of <10 K. The transitions are strain-rate dependent. For BCC metals the strain-rate dependence is small; for Si single crystals, increasing the strain-rate by a factor of ten increases the temperature of the transition typically by 100 K. The strain rate variation of the transition temperature T_c allows an activation energy E_a to be calculated:

$$\frac{d\varepsilon}{dt} = A \exp \left[\frac{-E_a}{kT_c} \right] \quad (1)$$

¹ “Obvious”, but not always true, as in materials with limited plasticity, stresses resulting from dislocation pile-ups at grain boundaries, second-phase particles or locked dislocations can initiate cleavage.

Where comparisons have been possible, this has been found to be equal to the activation energy for dislocation glide [19].

The increase in fracture toughness over the transition range is associated with an increase in the dislocation mobility resulting in an increasing amount of plasticity around the crack tip. A plastic zone is formed, which reduces the high stresses in front of the crack tip through elastic interactions, and which also blunts the crack.

Models have been developed in which the developing crack-tip plastic zone is modelled as a self-organising array of dislocations emitted from one or more sources near to the crack tip; the dislocations move according to an experimentally-derived velocity / stress / temperature law [19,20,21,22,23]. The elastic (“shielding”) interactions of the dislocations with the crack tip are calculated at each stage; when the total stress intensity at the crack tip reaches the low-temperature cleavage fracture toughness, fracture is assumed to occur. With increasing temperature, increasing dislocation activity gives increasing shielding of the crack tip, and thus increasing applied stress intensity for fracture.

Hence, for modelling brittle ductile transitions in such materials, essential input data are: (a) a nil-ductility fracture toughness, and (b) dislocation velocity as a function of stress and temperature. The models’ predictions then must be compared against (i) experimental data for fracture toughness as a function of temperature and strain rate, and, if possible, (ii) the sizes of plastic zones.

We describe here results of some initial fracture experiments for single crystal vanadium and polycrystalline iron and iron-9%chromium alloys, representing the major constituents of proposed structural alloys for fusion materials applications. They form a “snapshot” of work presently in progress.

Experimental methods

Specimen materials- single crystal vanadium and polycrystalline iron and iron 9% chromium - were supplied by Metal Crystal and Oxides, Button End, Harston, Cambs, UK. Specimens were cut using a Buehler isomet saw and polished using a succession of diamond pastes, to a final 1µm paste finish. Final specimen sizes were typically 10mm long, with 800µm square cross-section. Single crystal specimens were aligned so that each face was of {100} type. Polycrystals were etched to examine the grain structure; for all materials used, equiaxed grains of ~100-150µm diameter were found. Notches, with associated fine precracks, were made in the tensile faces of test specimens by electro-discharge machining. Specimens were tested to fracture over the temperature range 77K-293K, using four-point bending, with outer and inner contact spacings of 8mm and 4mm.

Results

Single crystal vanadium

Figure 1 shows bend test results for single crystal vanadium tested at a strain rates of $4.4 \times 10^{-5} \text{s}^{-1}$ and $4.1 \times 10^{-4} \text{s}^{-1}$. “Brittle” means that specimens failed immediately at a critical load, with no plastic strain, giving a planar fracture surface; “semi brittle” means that specimens failed in a brittle manner but with yield and some plastic strain

prior to fracture (again with a planar fracture surface), “ductile” means that specimens bent with no fracture. For ductile specimens the fracture toughness value shown is that at which bending initiated, assuming a crack depth equal to the mean value for fractured specimens (i.e. slightly greater than the notch depth); the real fracture toughness under these conditions will be higher than this value. For a surface strain rate of $4.1 \times 10^{-4} \text{ s}^{-1}$, the transition temperature, T_c , defined as the mean of the highest temperature at which brittle or semi-brittle fracture is observed and the lowest temperature at which ductile behaviour is observed, is 164K; for a strain rate of $4.3 \times 10^{-3} \text{ s}^{-1}$, $T_c = 174 \text{ K}$. For other strain rates tested, at $1.35 \times 10^{-2} \text{ s}^{-1}$, $T_c = 186 \text{ K}$, and at $4.4 \times 10^{-5} \text{ s}^{-1}$, $T_c = 138 \text{ K}$. If these results are interpreted using equation (1), the derived activation energy is 0.26eV, somewhat lower than the activation energy for screw dislocation motion in vanadium of 0.55eV found by Wang and Bainbridge [24].

For the brittle ductile transitions shown in figure 1, there appears to be no systematic variation in fracture stress intensity below the transition temperature; however, within a narrow transition range, fracture can occur at elevated stress intensity, associated with a non-zero plastic strain at fracture. This is very different from, for example, molybdenum [7] and tungsten [9], where the stress to fracture was found to rise steadily over the full range between 77K and T_c . Sharp transitions of the type observed here in vanadium have previously been associated only with dislocation-free covalently bonded materials [17,18], where dislocation activity at crack tip is triggered only just below the fracture load at T_c [20]. To determine the reasons for

this unusual behaviour in vanadium, dislocation activity around crack tips is presently being investigated by electron backscattered diffraction methods.

Iron and iron - 9%chromium

Experiments have to date been performed on polycrystalline materials only, as single crystals are currently unavailable with dimensions large enough to give the numbers of specimens required.

For polycrystalline iron, tests have been carried out at three strain rates: $4.46 \times 10^{-4} \text{ s}^{-1}$, $8.93 \times 10^{-4} \text{ s}^{-1}$ and $4.46 \times 10^{-3} \text{ s}^{-1}$. The 77K fracture toughness was found to be $20.6 \pm 4 \text{ MPa m}^{1/2}$, with no systematic variation with strain rate. Stress-strain curves for tests at 77K showed only elastic loading before fracture, as shown in Figure 2ai, and produced fracture surfaces showing only transgranular cleavage, as shown in figure 2b. For some tests at temperatures between 77K and the brittle-ductile transition, fracture could similarly be identified as brittle. In the transition temperature range, some stress-strain curves showed fracture after yield, as shown in figure 2aii; the serrations on the stress-strain curves after yield were accompanied by audible clicks, which we tentatively associate with crack progression from one grain to the next. The fracture surfaces in these cases showed mostly transgranular cleavage (Figure 2c), but with some evidence of local plastic flow near the crack tip (Figure 2d). Tests showing these characteristics were identified as “semi-brittle”. Above the brittle-ductile transition temperatures, specimens did not fracture, and stress-strain curves showed work-hardening after yield (Figure 2aiii).

Some overlap was normally found between the semi-brittle and ductile temperature regimes, due to the variability in test results as a consequence of the large grain size compared to the test specimen size. The brittle-ductile transition temperature (T_c) was defined as the mean of the highest temperature at which a test showed brittle or semi-brittle behaviour and the lowest temperature at which a test showed fully ductile behaviour. Tests at the highest strain rate gave a brittle-ductile transition temperature of 114K (± 3 K). Fracture toughness values measured in the semi-brittle range (100–116K) ranged from 22 to 31 MPa $m^{1/2}$. For the lower loading rates, transition temperatures were 103K (± 4 K) at $4.46 \times 10^{-4} \text{ s}^{-1}$, and 106K (± 2 K) at $8.93 \times 10^{-4} \text{ s}^{-1}$. Using eqn. 1, an activation energy for the BDT of $\sim 0.21 \text{ eV}$ is obtained, though given the temperature range and error margins of the T_c measurements, this can only be a tentative value.

For polycrystalline iron-9% chromium, tests have been carried out at the same three strain rates as for iron; see figure 3. The 77K fracture toughness was found to be $17.6 \pm 4 \text{ MPa m}^{1/2}$, with no systematic variation with strain rate. Iron - 9 % chromium, like iron and unlike vanadium, has a brittle-ductile transition in which semi-brittle behaviour is exhibited, where dislocation activity can raise the fracture toughness below T_c ; however, the data do not show any clear temperature dependence of semi-brittle fracture toughness. Tests carried out at $4.46 \times 10^{-3} \text{ s}^{-1}$ found a brittle-ductile transition between 127K (cleavage) and 122K (fully ductile), that is, about 10K higher than for iron. For the tests at $4.46 \times 10^{-4} \text{ s}^{-1}$, T_c was found to be 101K (± 1 K), and at

$8.93 \times 10^{-4} \text{ s}^{-1}$, T_c was found to be 107K (± 2 K). If these results are interpreted using equation (1), the derived activation energy is $\sim 0.11 \text{ eV}$.

Figure 4 shows the side faces near the notch root of iron - 9% chromium polycrystalline specimens tested at the highest strain rate used ($4.46 \times 10^{-3} \text{ s}^{-1}$), at temperatures just above T_c . In one specimen (figure 4a), a twin has propagated from the notch in one grain, and has triggered twinning in a second grain; in another (figure 4b) slip lines can be seen beneath the notch root, and also emanating from the tip of one of cracks in the notch root, which has become blunted. Though the specimen exhibiting slip lines was tested at a slightly higher temperature (129K) than the one exhibiting twinning (128K), the differences in behaviour seen are most likely due to differences in orientation of the grains present in the observed region of the specimens.

For one test on an iron 9% chromium specimen, at 119K and $4.46 \times 10^{-4} \text{ s}^{-1}$, a load drop characteristic of semi-brittle behaviour was noted at a tensile surface stress of 557 MPa ($\cong 8.77 \text{ MPa m}^{1/2}$ for short cracks in the notch root). The test was halted at a stress of 1250 MPa ($\cong 20 \text{ MPa m}^{1/2}$ for short cracks in the notch root), with the specimen remaining unfractured. SEM examination (figure 5a) showed the crack had propagated for several 100 μm below the notch base. After unloading, the specimen was re-tested at 77K (figure 5b). Fracture occurred from the extended cracks at 1291 MPa, well above the low temperature fracture stress and close to the maximum stress

used at 119K; that is, crack shielding and/or blunting at 119K has produced a “warm-pressing effect” [25,26].

Summary

Ten experiments so far have shown that miniature specimens can give useful data about the fracture of bcc metals. The activation energy for the brittle-ductile transition in vanadium ($\sim 0.26\text{eV}$) appears to be lower than that for dislocation motion (0.55eV), in contrast to findings for many other materials. In polycrystalline iron the activation energy was found to be $\sim 0.21\text{eV}$, and in polycrystalline iron - 9% chromium, $\sim 0.11\text{eV}$. The brittle-ductile transition behaviour of single crystal vanadium is unusual for metals, showing a transfer from purely brittle to ductile behaviour with no rise in fracture toughness except at temperatures very close to the transition. Iron and iron – 9% chromium show semi-brittle behaviour with elevated fracture toughness over a wider temperature range, but with no clear trend with temperature. Work continues on fracture and on dislocation behaviour, so that the fracture behaviour of these materials can be understood within the framework of existing dislocation-dynamic models. This is aimed at prediction of effects of radiation on fracture behaviour, via its effects on dislocation mobility.

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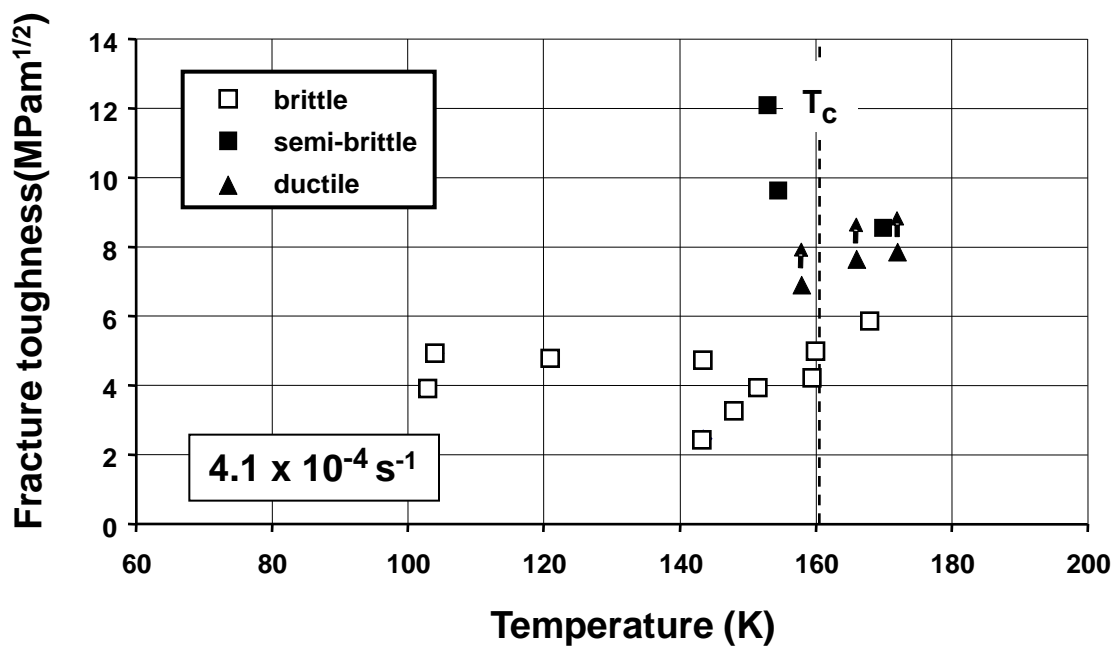
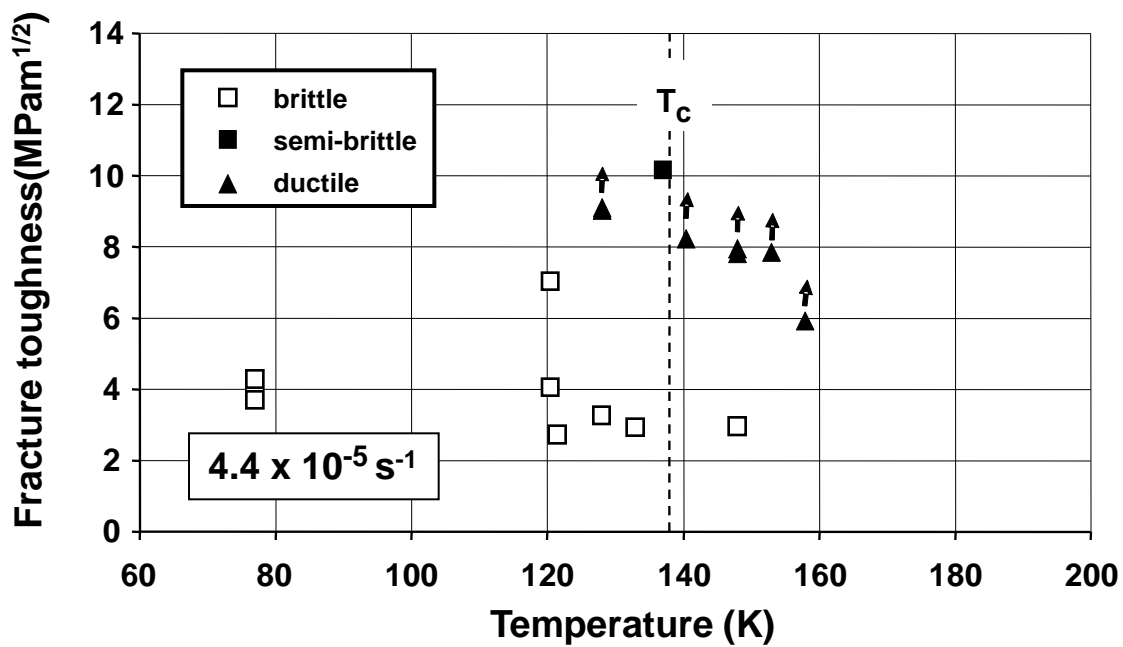


Figure 1: Test results for single crystal vanadium at two strain rates.

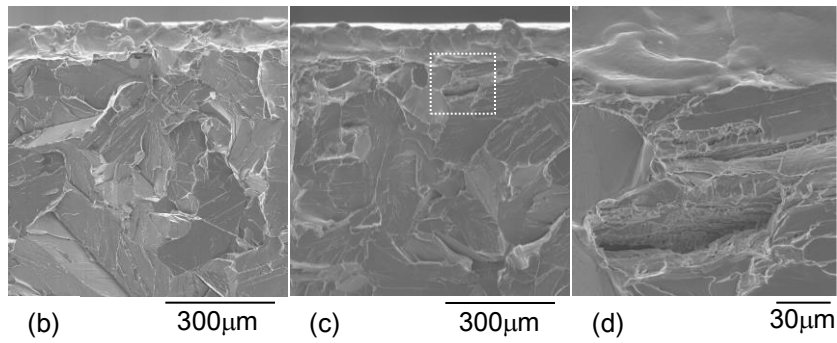
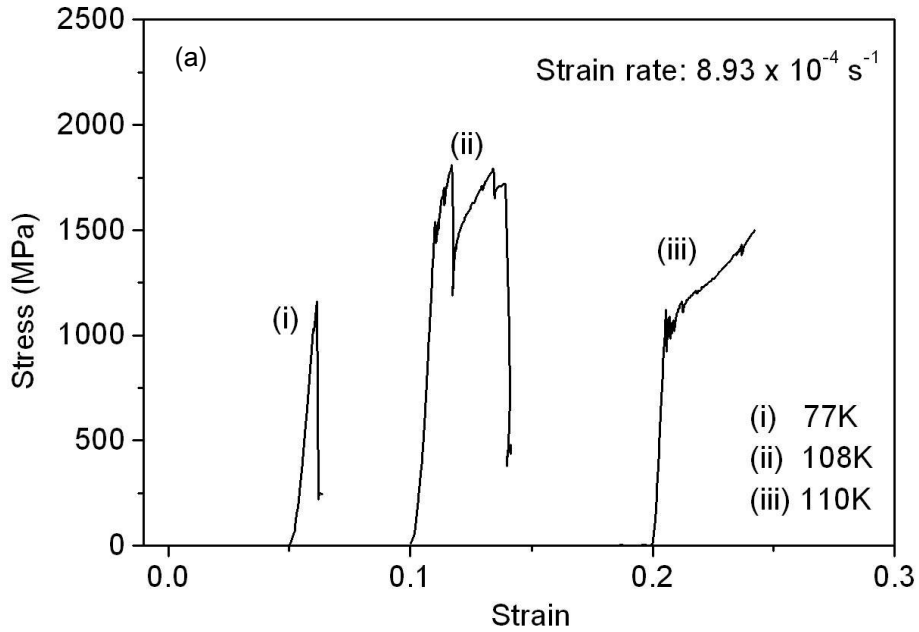


Figure 2: (a) Stress-strain curves from four-point bending tests of pure iron at a strain rate of $8.93 \times 10^{-4} \text{ s}^{-1}$: (i) 77K - brittle fracture, (ii) 108K - semi-brittle fracture, (iii) 110K - ductile bending; (b) Fracture surface for 77K test; (c) Fracture surface for 108K test; (d) Enlargement of the area indicated in (c), showing voids around part of the pre-crack.

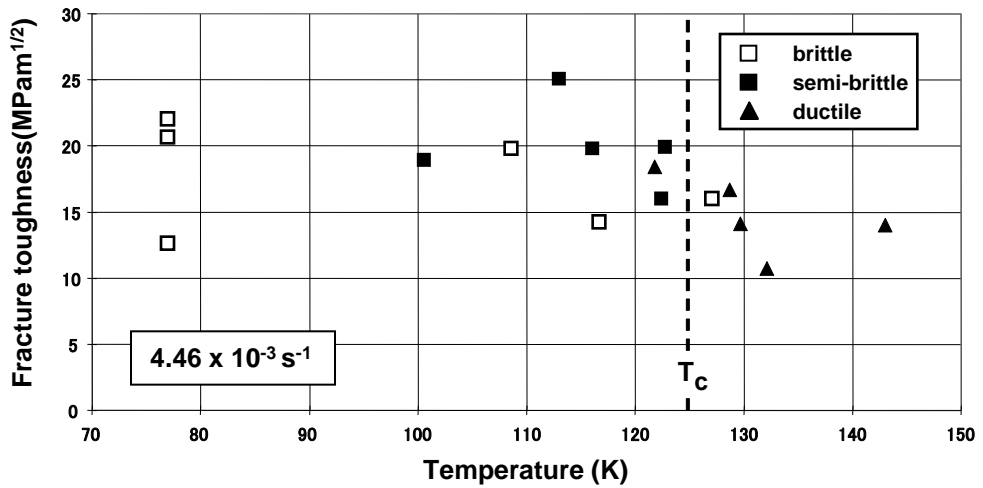
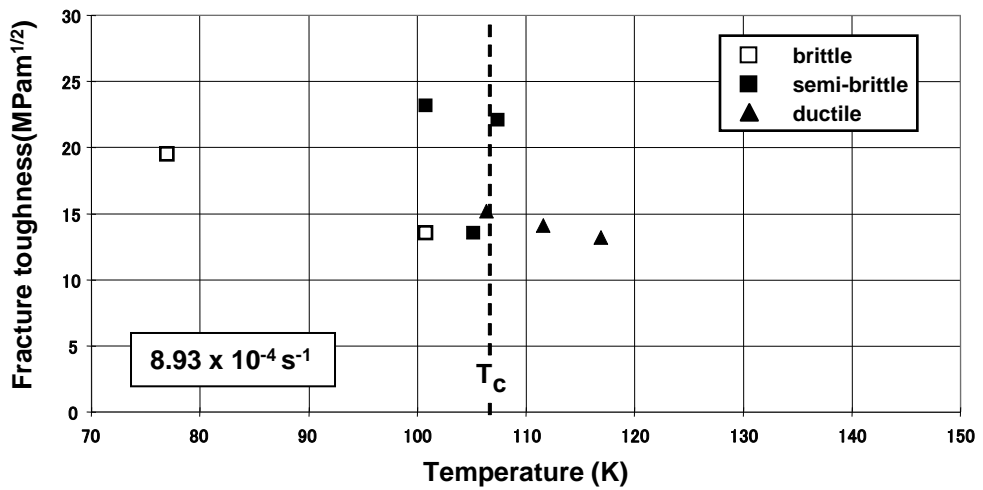
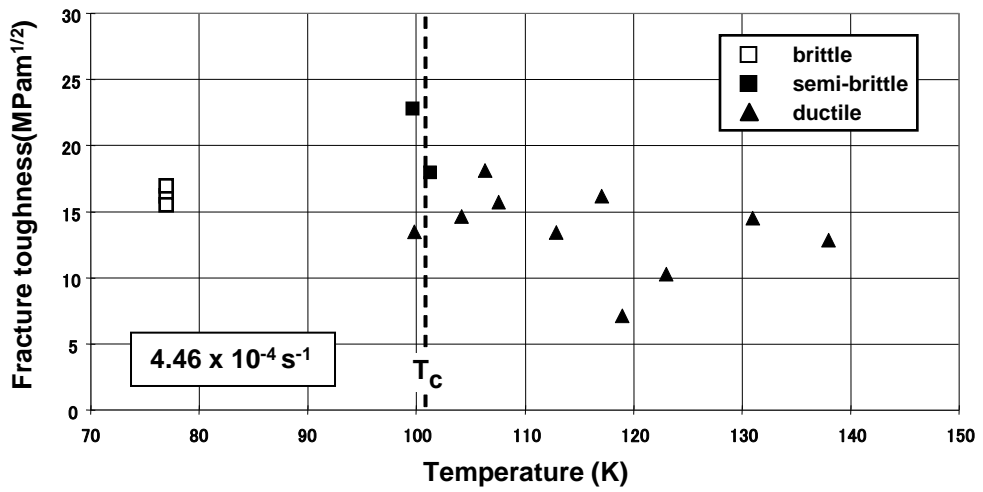


Figure 3: Test results for polycrystalline iron- 9% chromium at three strain rates.

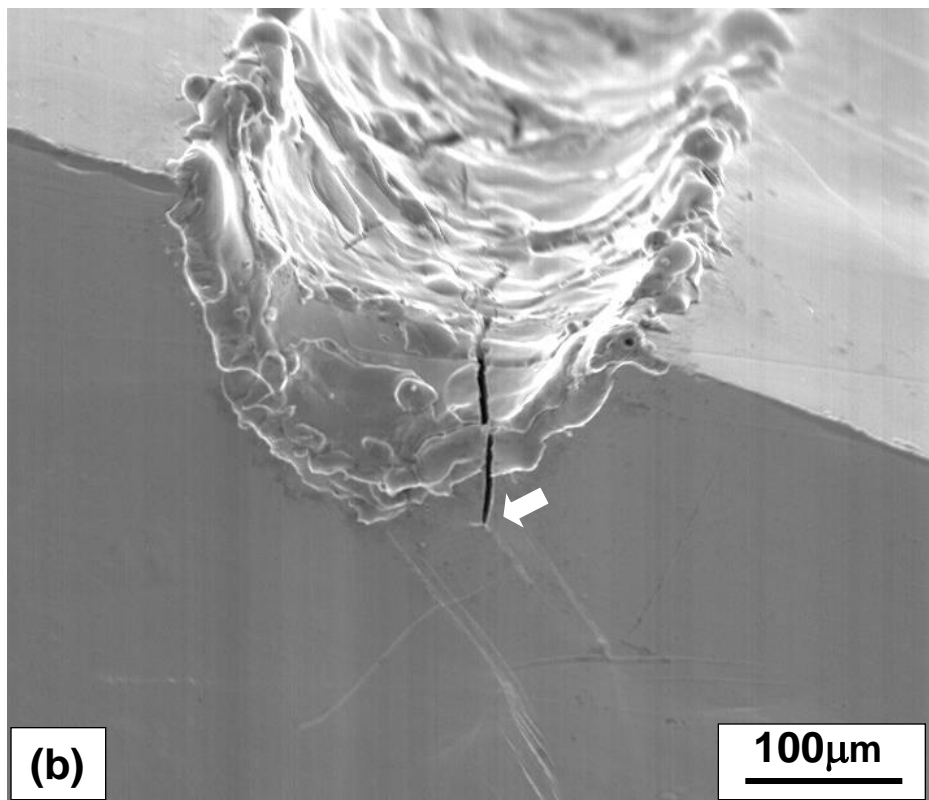
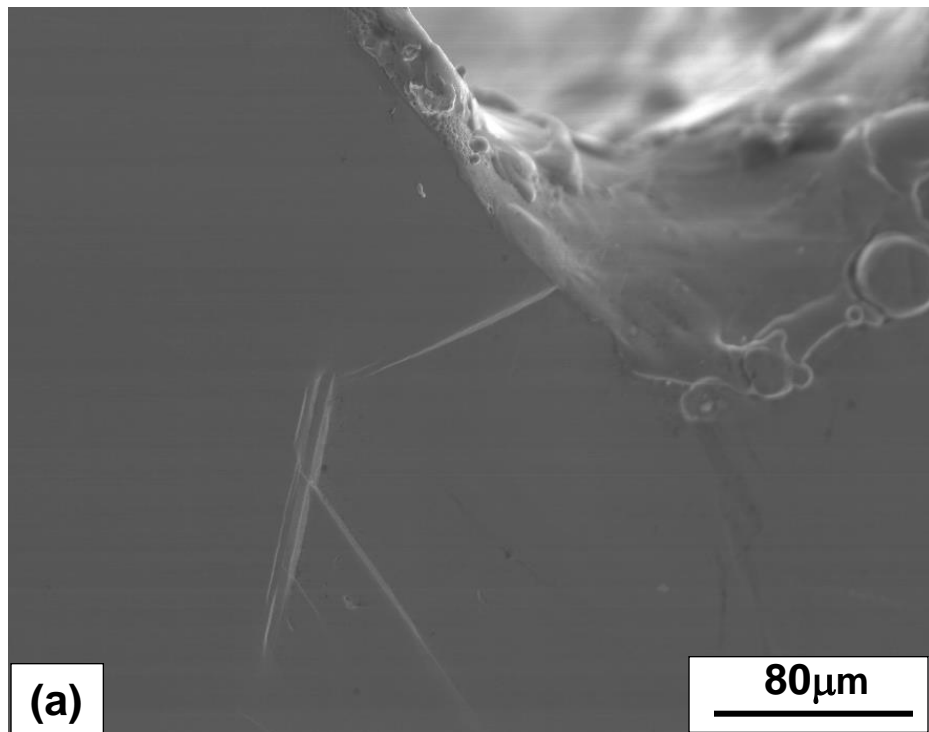


Figure 4: Iron 9% chromium polycrystalline specimens tested at a strain rate of $4.46 \times 10^{-3} \text{s}^{-1}$: (a) 129K (ductile) showing twins at the notch root; (b) 130K (ductile) showing slip bands emanating from the notch root and from a crack tip (arrowed).

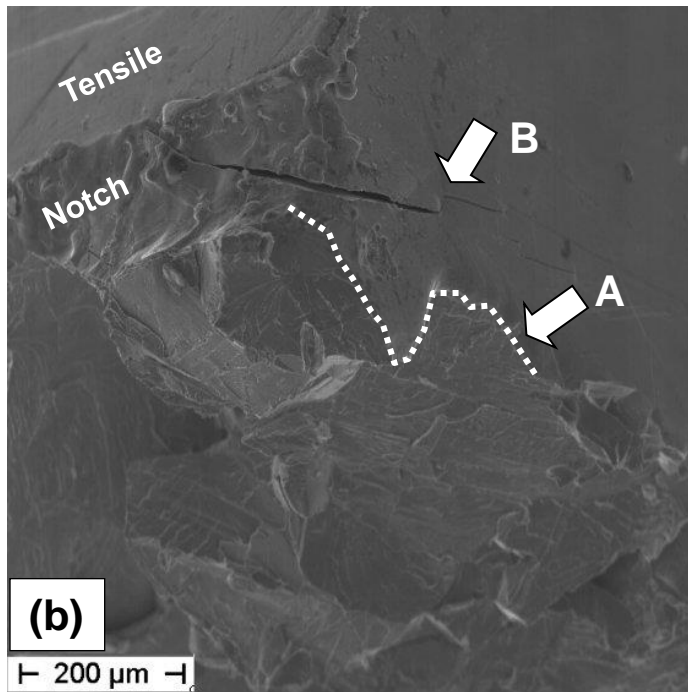
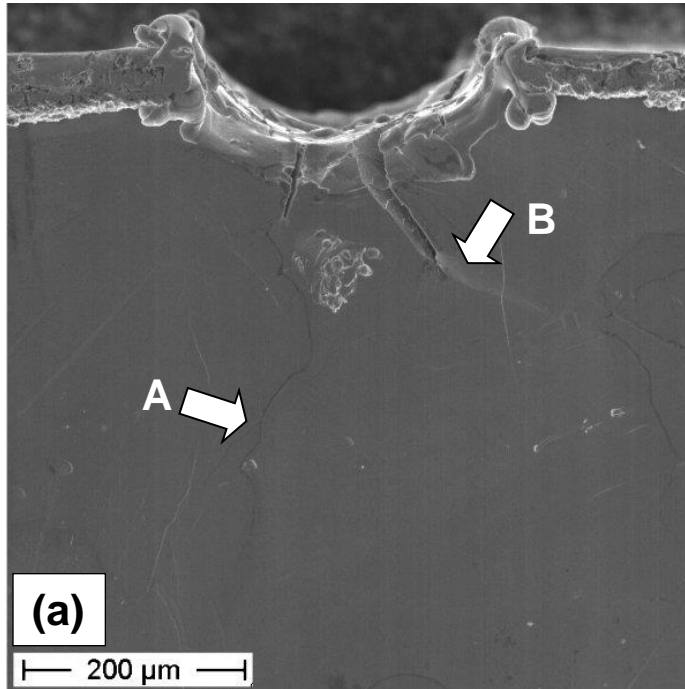


Figure 5: Iron 9% chromium polycrystal, tested at $4.46 \times 10^{-4} \text{ s}^{-1}$: (a) side face of specimen, after loading to 1250 MPa at 119K. Cracks (A and B) extend from the notch base; (b) oblique view of fracture surface of the same specimen, after fracture at 77K. Fracture has initiated from crack A.