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► **To cite this version:**

Steve G Roberts, Peter B Hirsch. Modelling the upper yield point and the brittle-ductile transition of silicon wafers in three-point bend tests. *Philosophical Magazine*, 2006, 86 (25-26), pp.4099-4116. 10.1080/14786430600643308 . hal-00513679

HAL Id: hal-00513679

<https://hal.science/hal-00513679>

Submitted on 1 Sep 2010

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Journal:	<i>Philosophical Magazine & Philosophical Magazine Letters</i>
Manuscript ID:	TPHM-05-Sep-0413.R1
Journal Selection:	Philosophical Magazine
Date Submitted by the Author:	08-Feb-2006
Complete List of Authors:	Roberts, Steve; University of Oxford, Materials Science Hirsch, Peter; University of Oxford, Materials
Keywords:	modelling, brittle-ductile transition, crystals, deformation, dislocations, fracture, dislocation dynamics, dislocation mechanics
Keywords (user supplied):	



Nabarro Festschrift
Submitted to Phil. Mag, Sept 2005
Revised version Feb. 2006

Modelling the upper yield point and the brittle-ductile transition of silicon wafers in three-point bend tests

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Abstract

Three point bend test carried out by Folk[1] on initially dislocation-free specimens of semiconductor-grade single crystals of Si showed that, for a given strain-rate, above a critical temperature, T_c , and at a certain stress, a large load drop occurs upon yielding and the sample deforms plastically. Below T_c specimens fail by brittle fracture after a small load drop, or at even lower temperatures without apparent prior plastic flow. This paper describes a simple dislocation model and computer simulation to explain the occurrence of a yield drop, the strain-rate dependence of T_c and Folk's etch pit observations of the plastic zone. Good agreement is obtained between the temperature dependence of the upper yield stress and the strain-rate dependence of T_c with the predictions of the model. These parameters are controlled by dislocation velocity.

Dedication

This paper is dedicated to Frank Nabarro in honour of his 90th birthday. He has made many outstanding and lasting contributions to dislocation theory,

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2
3 and has championed the application of quantitative analysis in crystal
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6 plasticity.
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10 **1. Introduction**

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15 The brittle-ductile transition (BDT) in silicon has been studied extensively
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17 over the last thirty years, with most experiments conducted on precracked
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19 specimens [2-7]. The observed transitions are sharp and the activation
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21 energy controlling the strain-rate dependence of the transition temperature
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23 (T_c) was found to be close to that controlling dislocation velocity (for
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25 reviews see [8,9]). The intercepts of the Arrhenius plots however vary
26
27 greatly depending on the test geometry, crystal growth technique,
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29 procedure of introducing the crack, and composition. This suggests that
30
31 although the temperature dependence in all cases is controlled by
32
33 dislocation velocity, T_c at any given strain-rate is structure sensitive. Two
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35 different models were suggested, applicable to different geometries, in
36
37 which the critical event in a constant strain-rate test is the nucleation of
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39 crack tip sources, which, in the high stress field near the crack tip, rapidly
40
41 emit avalanches of dislocations sufficient to shield the crack completely
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43 before the applied stress intensity factor reaches K_{IC} for brittle fracture
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45 [8,9]. In these models it is the nucleation of crack tip sources by gliding
46
47 dislocations which controls the strain-rate dependence of T_c . The structure
48
49 sensitivity of T_c is simply due to the different distances the dislocations
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51 have to move either to the crack or along the crack to generate these
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53 sources. Each intercept in the Arrhenius plots corresponds to a
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55 characteristic distance the dislocations have to move to nucleate the
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3 sources. These models are supported by experiment and computer
4 simulation [8,9]. The remarkable sharpness of the transition in crystals with
5 few initial dislocations is attributed to the sources, once nucleated, being
6 highly stressed for dislocation emission.
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14 Khantha, Pope and Vitek [10] put forward a theory for the BDT in which a
15 critical combination of stress and temperature results in the massive
16 generation of dislocations arising from a cooperative Kosterlitz-Thouless
17 type instability [11], permitting general yielding. No crack tip is necessary
18 for this transition. To test this hypothesis Folk [1] carried out three point
19 bend tests on initially dislocation-free specimens of single crystals of Si. No
20 precracks were intentionally introduced into the specimens, which were
21 produced from Si wafers using photolithography. The experiments revealed
22 that, for a given strain-rate, above a critical temperature (the BDT
23 temperature T_c) and at a certain stress, a large load drop occurs upon
24 yielding and the sample begins to flow. Below this temperature the sample
25 fails by brittle fracture, at a stress σ_f which is strongly temperature
26 dependent, either without apparent prior plastic flow, or, near the critical
27 temperature (within 10-50° of T_c), after some plastic flow which causes
28 deviation from linearity in the load-displacement curve, and/or a small yield
29 drop. Figure 1 shows the three types of behaviour. The lowest temperature
30 transition from type 2 to type 3 defines T_c . However some specimens
31 exhibit type 2 behaviour up to 30° above T_c . T_c is found to be strain-rate
32 dependent, with an activation energy (1.9 eV) close to that for dislocation
33 velocity. Etch pit observations carried out after unloading show the
34 development of a plastic zone under the knife edge loading probe before
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3 the critical stress at T_c is reached. No detailed interpretation of the nature
4 and development of the plastic zone, of the strain-rate dependence of T_c ,
5 and of the temperature dependence of the fracture stress σ_F have been
6 published, but it is claimed that the BDT is not controlled by dislocation
7 velocity [1,12] and that the behaviour is qualitatively consistent with the
8 Khantha, Pope and Vitek model [10].
9

10
11 The object of the present paper is to explain the observations of the plastic
12 zone, the occurrence of a yield drop, and the strain-rate dependence of T_c .
13
14 The following sections (2, 3, 4) give first a brief summary of the salient
15 features of Folk's results, secondly a description of a simple model to
16 explain the observations, and thirdly, a computer simulation to explain the
17 occurrence of a yield drop and to determine the temperature dependence of
18 the upper yield stress, the strain-rate dependence of T_c , and the etch pit
19 observations. Section 5 discusses the temperature dependence of σ_F , and
20 the final section compares the model with previous models for the BDT in
21 precracked specimens.
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45 **2. Summary of Folk's (2000) main results**

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49 Fig. 1 illustrates the three types of behaviour observed: brittle, brittle with
50 some prior plasticity, and fully plastic. Fig. 2 shows schematically the
51 experimental arrangement. Fig. 3 shows the maximum bending stress
52 against temperature for a strain-rate of $6 \times 10^{-5} \text{ s}^{-1}$. The results for different
53 strain-rates are similar. According to Folk (2000), between room
54 temperature and about 900K the fracture is purely brittle with no
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3 observable macroscopic deformation (type I behaviour) and the fracture
4 stress, σ_F , varies widely between specimens from different wafers, and less
5 widely between specimens from a given wafer. In this temperature range σ_F
6 varies between about 1.5GPa and 3.5GPa, indicated in fig. 3 by the dashed
7 horizontal lines. Above about 900K, the brittle fracture stress, σ_F , is
8 strongly temperature dependent. T_c is defined as the lowest temperature at
9 which the specimen becomes ductile. The strain-rates $\dot{\epsilon}$ used in the
10 experiments were $6 \times 10^{-6} \text{ s}^{-1}$, $3 \times 10^{-5} \text{ s}^{-1}$, $6 \times 10^{-5} \text{ s}^{-1}$, $1.2 \times 10^{-4} \text{ s}^{-1}$ and 6×10^{-4}
11 s^{-1} . A plot of $\ln \dot{\epsilon}$ against T_c^{-1} is consistent with an activation energy for T_c of
12 1.9eV. The applied maximum bending stress, i.e. the upper yield stress, at
13 which T_c occurs is found to vary slightly; the values are 802 MPa, 841 MPa,
14 843 MPa, 765 MPa for the first four strain-rates listed above. For the
15 highest strain-rate, $6 \times 10^{-4} \text{ s}^{-1}$, the specimens failed in a brittle manner at all
16 temperatures at which tests were made, up to 1246K.

17
18
19 Etch pit observations were made with the SEM on samples tested with
20 $\dot{\epsilon} = 6 \times 10^{-5} \text{ s}^{-1}$ at 967K and 979K, just below and above $T_c = 972\text{K}$, unloaded at
21 various stages. The images cover the inclined side walls of the beam as well
22 as part of the tensile surface. Fig. 4 shows the etch pit distributions for the
23 specimens tested at 979K, unloaded at a) 60%, b) 80% c) 100% of the
24 peak stress, d) after the load drop. The pictures corresponding to a, b, c for
25 the sample tested at 967K are qualitatively similar to those tested at 979K.
26 It should be noted that the etch pit distributions show the dislocation
27 arrangements after unloading, not in the loaded state (see section 4).

3. A model for the occurrence of a load drop

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6 The etch pit observations made by Folk [1] just above and below T_c show
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8 that there are essentially three types of dislocation activity:-
9

- 10 I) Shear bands parallel to (111) and ($\bar{1}\bar{1}1$) (the "primary" system)
11
12 generated by dislocation emission at the edges of the central loading
13
14 contact. Some of these dislocations travel across the specimen and
15
16 reach the tensile surface in the loading/unloading cycle at stresses as
17
18 low as 60% of the upper yield stress.
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21 II) Slip traces parallel to [110], corresponding to slip activity on the
22
23 ($\bar{1}\bar{1}1$), ($1\bar{1}1$) ("cross-slip") systems. The development of these slip
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25 lines lags behind those of the primary systems and they do not reach
26
27 the tensile surface (after unloading) till the stresses approach the
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29 upper yield point.
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32 III) Strong slip lines on the primary systems activated at the upper yield
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34 point, which are nucleated in the tensile section of the beam, or at the
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36 tensile surface.
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40 We suggest the following sequence of events (see figure 5):-
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- 42 1) The initial shear bands, I, on (111) and ($\bar{1}\bar{1}1$) are generated at the
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44 edges of the loading contact, which acts approximately as a flat
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46 punch. The shear stress on these systems is maximum at these
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48 locations [13]. The extra half planes of the dislocations lie above the
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50 slip planes as shown in fig. 5a. The signs of these dislocations are such
51
52 that the compressive bending stress, which acts on the loading probe
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54 side of the beam aids the generation of these dislocations, but the
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56 dislocations do not relieve the stresses on the tensile side of the bent
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58 beam. In fact they are repelled by the tensile stress (fiig. 5b) and
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3 increase the surface tensile stress (see section 5). The passage of
4 these dislocations across the neutral axis is primarily due to their
5 mutual interaction.
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10 2) The stress on system II from the loading knife edge, and close to it, is
11 largely hydrostatic compression with a smaller tensile component
12 parallel to the length of the punch [13]. The early development of
13 these slip systems will therefore lag behind that of system I. However
14 the dislocations on system I exert a compressive stress along [001]
15 (fig 5b: σ_{disln}), which aids the development of the dislocations on
16 system II, which will accelerate as the dislocations on I accumulate
17 (fig 5c).
18
19 3) Eventually these cross-slip dislocations will reach the tensile surface.
20 Since the dislocations in Si are dissociated into Shockley partials, for
21 any screw dislocation intersecting the surface these partials will either
22 constrict or expand depending on their sign [14]. Those which
23 constrict can cross-slip readily (onto the (111) and $(\bar{1}\bar{1}1)$ planes of
24 system I) (figs. 5c, 5d), forming single ended Frank-Read sources.
25 These will operate rapidly in the highly stressed surface regions of the
26 beam, producing avalanches of dislocations (system III) on slip planes
27 containing the sources (see fig. 5e), which produces a yield drop. The
28 fact that system III slip consists of a series of slip bands is consistent
29 with the operation of Frank-Read sources. The sign of these
30 dislocations is the same as for system I, but they are moving in the
31 opposite direction, from the tensile surface to the neutral axis.
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58 Fig. 6 shows a computer simulation for a strain-rate $\dot{\epsilon}=6 \times 10^{-5} \text{ s}^{-1}$ at
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60 $T_c=972\text{K}$ of the number of dislocations generated and the array length on a

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3 single type III slip plane, following nucleation of one source at the tensile
4 surface, at two stresses just below that at which T_c occurs. This emphasizes
5 the large number of dislocations produced very rapidly by one Frank-Read
6 source. Each slip band contributes a yield drop. With increasing numbers of
7 sources operating at the same time the yield drop increases resulting finally
8 in plastic flow at a low stress. No mechanism other than the operation of
9 Frank-Read sources is required.

10
11
12 The condition for obtaining a yield drop is that the dislocations on the cross-
13 slip plane reach the tensile surface and generate Frank-Read sources on
14 system III before fracture occurs. This means that the yield drop condition
15 and the upper yield stress are controlled by dislocation velocity. In the next
16 section we describe a model for this which gives the upper yield stress as a
17 function of temperature and strain-rate. Experimentally, T_c is found to occur
18 at an upper yield stress of about 800MPa at various strain-rates, and the
19 model for the upper yield stress is then used to determine the strain-rate
20 dependence of T_c .

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22
23 The change from elastic to fully plastic deformation in this case is
24 interesting. System I is activated first through the action of the loading
25 probe. Slip on this system reduces the bending stress in the compressive
26 part of the bent beam. But system I is repelled by the tensile part of the
27 beam which is still deforming elastically. When system III is activated, the
28 tensile stress decreases; this enables system I to develop further reducing
29 the compressive bending stress. Unless brittle fracture intervenes, this
30 process continues until most of the strain-rate can be accommodated by

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3 plastic flow both in the "tensile" and "compressive" parts of the beam.
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5 System I transports material away from the compressive side of the beam;
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8 system III extends the tensile part of the beam plastically. These two
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10 systems effectively act to produce a plastic hinge. System II acts as the
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12 agent which initiates slip on system III. In addition there is accommodating
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14 convergent slip under the finite width loading contact.
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19 **4. Modelling the upper yield stress and the strain-rate** 20 21 **dependence of T_c** 22 23 24 25

26 We assume that the loading contact acts as a long flat punch, subject to a
27
28 uniform pressure P . The punch stress field σ_p is given by Nadai [15] as
29
30

$$\begin{aligned}
 \sigma_{xx} &= -\frac{P}{2\pi} [2(\theta_1 - \theta_2) + \sin 2\theta_1 - \sin 2\theta_2] \\
 \sigma_{yy} &= -\frac{P}{2\pi} [2(\theta_1 - \theta_2) - \sin 2\theta_1 + \sin 2\theta_2] \\
 \tau_{xy} &= \frac{P}{2\pi} (\cos 2\theta_1 - \cos 2\theta_2) \\
 \sigma_{zz} &= \nu(\sigma_{xx} + \sigma_{yy})
 \end{aligned}
 \left. \vphantom{\begin{aligned} \sigma_{xx} \\ \sigma_{yy} \\ \tau_{xy} \\ \sigma_{zz} \end{aligned}} \right\} 1$$

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45 The coordinates and angles are shown in fig. 5f; ν is Poisson's ratio. The
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47 diverging slip lines on system I are generated at the edges of the punch
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49 where the shear stress is a maximum, as shown in fig. 5. These stresses,
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51 together with the applied bending stress σ_B , are used to calculate the
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53 resolved shear stresses on dislocations on the (111) and ($\bar{1}\bar{1}\bar{1}$) planes of
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55 system I. Each of the diverging shear bands are treated as slip on single
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57 planes. To simplify the calculation the two sets of dislocations on each
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59 plane (e.g. $\frac{1}{2}[\bar{1}01]$ and $\frac{1}{2}[0\bar{1}1]$ on (111)) are replaced by one edge
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3 dislocation (\underline{b} along $[\bar{1}\bar{1}2]$). The dislocations on the $(\bar{1}11)$, $(1\bar{1}1)$ planes of
4 system II are treated in the same way; i.e. dislocations with $b=\frac{1}{2}[0\bar{1}1]$ and
5 $b=\frac{1}{2}[101]$ on $(\bar{1}11)$ are replaced by pure edge dislocations with \underline{b} along
6 $[\bar{1}\bar{1}2]$. The stresses acting on these dislocations are σ_p and the interaction
7 stresses from system I. For simplicity the interaction between them is
8 calculated as that of an infinite straight dislocation on I on the midpoint of a
9 dislocation on II. (The reverse interaction is neglected because the number
10 of dislocations on a single slip plane on I is much greater than that on II).
11 Interaction between the dislocations moving on the same planes is fully
12 taken into account (assuming them to be infinitely long), as is that between
13 dislocations on the two shear bands on system I. Although the etch pit
14 distribution on the inclined face (see fig. 4) shows slip on system II to occur
15 on planes a few microns apart, this has been modelled as slip on a single
16 plane. A previous comparison between dislocations moving on a single slip
17 plane, and several parallel slip planes, in a crack tip stress field showed the
18 use of the single slip plane model to be a reasonable approximation [16].
19 Furthermore, the length of a single slip line representing the plastic zone at
20 a semi-infinite crack is found to be within about 5% of that predicted by the
21 finite element model [17]. The bend stresses are calculated for the
22 trapezoidal beam shown in fig. 2. P is calculated from the beam deflections
23 by assuming a punch width ω of $50\mu\text{m}$; in practice the etch pit observations
24 of the distance between the initiation locations of the shear bands show this
25 to be somewhat variable (see fig. 4 and [1] for other examples), but the
26 above value appears representative. (A simulation carried out for $\omega=70\mu\text{m}$,
27 gives essentially the same results for $\omega=50\mu\text{m}$). The dislocation velocity is
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$$v = v_0 \left| \frac{\tau}{\tau_0} \right|^m \exp\left(-\frac{U}{kT}\right) \quad (2)$$

For intrinsic Si, $v_0=1.8 \times 10^5 \text{ ms}^{-1}$, $\tau_0=19 \text{ MPa}$, $m=1.6$, $U=2.22 \text{ eV}$ [18]; τ is the shear stress acting on the dislocations. For the calculation of the interaction stresses the shear modulus $\mu=62 \text{ GPa}$, $b=0.381 \text{ nm}$, $\nu=0.218$.

In the calculation dislocation sources are assumed to be situated at b from the punch face along the slip plane. (The results of the simulations are insensitive to the distance of the source from the punch face for values ranging from b to $100b$). Each emitted dislocation moves away from the source according to equation (2), and when the total stress at the source becomes positive a further dislocation is emitted. The applied stress increases at a constant rate determined by the strain-rate. This dynamic simulation model is similar to that used previously in treating emission from loaded crack tips (e.g. [8]), except that the stress fields here are different.

Fig. 7 shows the results of the simulation for $\dot{\epsilon}=6 \times 10^{-5} \text{ s}^{-1}$ at 972K , the experimental T_c for this strain-rate. The figure shows the length of the slip lines and the total number of dislocations on systems I and II as a function of bend stress. System I develops initially more quickly than system II, reaching the neutral axis ($\sim 175 \mu\text{m}$ along the slip plane) at a much lower stress than system II. But then system II catches up and reaches the surface at 800MPa , while system I does not. The comparison with the etch pit results is not straightforward, since the latter observations are made on unloaded specimens above and below T_c (see fig. 4), while the predictions in fig. 7 correspond to the loaded state at T_c . Simulations in which the

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3 loaded specimens above and below T_c are unloaded at the temperature at
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5 which loading occurred, show that unloading allows the arrays to move
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7 further. The dislocations on system I, in particular, expand rapidly towards
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9 the tensile surface, since unloading reduces and then removes the bend
10
11 stress which opposes their motion near the tensile surface. Unfortunately
12
13 the thermal history of Folk's unloading experiments is not known, and so no
14
15 detailed comparisons are possible. Nevertheless, the etch pit results in fig.
16
17 4 and others in Folk's thesis are consistent with the proposed model that
18
19 nucleation of many new slip lines at the tensile surface occurs when the
20
21 system II dislocations reach the surface. On the other hand the etch pit
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23 results show that the primary dislocations seem to reach the tensile surface
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25 before the "cross-slip" dislocations, while this is not the case in the
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27 computer simulation. This may be partly due to unloading effects, but
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29 simulations carried out in which the back interaction from system II to
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31 system I is taken into account in an approximate way, show that this has
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33 the effect of speeding up the progress of the system I dislocations, but we
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35 cannot be certain whether they meet the surface before system II.
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45 The curves obtained at the other strain-rates and experimentally observed
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47 values of T_c are very similar to fig. 7. Fig. 8 plots the temperature at which
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49 system II reaches the surface (initiating nucleation of sources on system
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51 III) against the maximum bending stress (the upper yield stress) for
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53 $\dot{\epsilon}=6 \times 10^{-5} \text{ s}^{-1}$. The experimental values of the upper yield stress are also
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55 shown. The agreement between the model predictions and the
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57 experimental values of the upper yield stress is good, and the same is true
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59 for the other strain-rates, providing support for the dislocation velocity
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3 controlled nucleation model. At the stress at which T_c is observed
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5 experimentally (~ 800 MPa), the predicted T_c is 967K (compared with 972K
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7 observed experimentally). The predicted values of T_c for the other strain-
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9 rates are obtained in the same way, and are compared with experiments in
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11 table 1. Fig. 9 plots $\ln(\text{strain-rate})$ against T_c^{-1} for the model and for the
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13 experiment. The agreement is excellent. It should be noted that while the
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15 computer modelling includes several simplifications, there are no adjustable
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17 parameters in the calculation.
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24 It should be noted however that while this model explains the mechanism
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26 by which dislocation sources are nucleated to produce a yield drop, and
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28 while it predicts correctly the temperature dependence of the upper yield
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30 stress, and T_c at the experimentally observed stress, it does not explain
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32 why the BDT occurs at these particular stresses. To do this, we need to
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34 understand the origin of the fracture stress.
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40 **5. The temperature dependent brittle fracture stress**

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43 According to Folk [1], at low temperatures ($\sim 725\text{K} - 900\text{K}$) the fracture is
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45 purely brittle and σ_F varies between about 1.5GPa and 3.5GPa, similar to
46
47 the range observed at room temperature. Above this temperature the
48
49 maximum bending stress is strongly temperature dependent. Fig. 3 shows
50
51 the observed maximum bending stress at fracture σ_F against temperature
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53 for $\dot{\epsilon}=6 \times 10^{-5} \text{ s}^{-1}$. The fracture stress at this strain-rate drops in the high
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55 temperature regime from 1GPa at 936K to about 0.4GPa at 1004K. Such a
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57 rapid decrease with temperature is likely to be caused by dislocation
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3 activity. Furthermore, the scatter of σ_F above about 900K in Folk's results
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5 at different strain-rates is surprisingly low, suggesting that the controlling
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7 flaw above this temperature is generated by the deformation.
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11
12 The most likely site for crack initiation is probably at a stress concentrator
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14 at the tensile surface, such as a step or notch produced by slip lines on
15
16 system III nucleated where cross-slip dislocations meet the surface. The
17
18 fact that fracture takes place at stresses σ_F slightly lower than the upper
19
20 yield stress, suggests that for cracking to occur slip steps on system III
21
22 have to be formed, i.e. that σ_N is reached. The variation of σ_F with
23
24 temperature will then be similar to that of σ_N , but the stresses will be
25
26 slightly lower because of the load drop. This explains the observed variation
27
28 of σ_F with temperature. Fig. 8 shows the experimental values of σ_F
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30 (diamonds) as a function of temperature. Most of the points lie below σ_N ,
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32 and follow the same temperature variation.
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40 Slip on system III takes place on two planes, (111) and ($\bar{1}\bar{1}\bar{1}$). If slip on
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42 both planes occurs near the same location on the surface, and with the two
43
44 {111} slip steps facing each other, a V notch with semi angle 35° is
45
46 produced. Fig. 6 shows that if slip takes place on single slip planes at the
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48 fracture stress, σ_F , large steps, of the order of $1\mu\text{m}$ high can be generated
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50 in a short time. The tensile stress at the tip of the notch is $2\sigma_F(a/\rho)^{1/2}$ where
51
52 a is the height of the slip step and ρ is the radius of curvature at the root of
53
54 the notch. With $a \sim 1\mu\text{m}$ and ρ of nanometre dimensions, stresses $\sim 50\sigma_F$
55
56 can occur at the root of the notch, sufficient to nucleate cracking.
57
58
59

60 Alternatively the root may already be cracked if the slip steps occur at an

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3 existing flaw (see below). Treating the combined notch and microcrack as a
4 crack of length a , crack propagation could be expected at a stress of
5
6 approximately $K_{IC}/(\pi a)^{1/2}$, where K_{IC} is the fracture toughness of silicon
7
8 ($1\text{MPa m}^{1/2}$), i.e. about 560MPa for $a=1\mu\text{m}$, less than the observed σ_F at T_c .
9
10 Thus notches of this type (some bigger, some smaller) can cause fracture
11 of the specimen at the observed fracture stresses. We suggest therefore
12 that the flaws controlling fracture may be pairs of slip steps forming sharp
13 V notches. However, if the slip is diffuse, e.g. if the slip lines broaden by
14 double cross-slip, the resulting U notch will have a larger radius of
15 curvature, be less potent, and may not be able to cause cracking. It may
16 be noted that there are examples on Folk's micrographs of slip lines on the
17 (111) and ($\bar{1}\bar{1}1$) planes appearing, albeit on low resolution, to come from
18 the same location on the surface (see figs. 4c, d).
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36 Whether fracture or plastic flow occurs depends on the number of sources
37 nucleated at the same time and on the size of the slip steps. If only a few
38 sources are nucleated at a given time when the cross-slip dislocations meet
39 the surface and the slip lines do not broaden, the load drop will be too small
40 and fracture will be favoured, but when many sources are nucleated and
41 the slip lines broaden by double cross-slip, the load drop can be sufficient
42 (and the slip steps less potent) for fracture to be inhibited and plastic flow
43 to occur. Since the formation of a source by cross-slip is likely to involve
44 some thermally activated process, and the same will be true for broadening
45 of slip lines by double cross-slip, higher temperature is likely to favour a
46 large yield drop. Thus in fig. 8, within the range at which an upper yield
47 point is observed, below T_c only a few sources are nucleated, forming sharp
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3 slip steps, and above T_c the rate of nucleation and broadening by cross-slip
4 increases until no reversion to fracture occurs. To determine the bend
5 stress at which plastic flow, i.e. T_c , occurs, we need to model the load drop
6 in terms of the number of sources nucleated at σ_N , their rate of emission of
7 dislocations and the slip line broadening as the load decreases. This is not
8 included in the present model and remains to be done. In this paper we
9 therefore determine T_c by assuming the experimentally observed value of
10 σ_F at the transition (see section 4).
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24 At the highest strain-rate of $6 \times 10^{-4} \text{ s}^{-1}$ no transition occurs experimentally
25 and the specimens fail by brittle fracture even at the highest temperature
26 investigated (1246K). Fig. 10 shows the experimental values of the upper
27 yield stress and fracture stress as a function of temperature for this strain-
28 rate [1], and the curve of σ_N predicted by the model. The agreement in this
29 case is again good, particularly above 950K, and a transition might be
30 expected in this temperature range. The brittle behaviour may be due to a
31 paucity of sources and large slip steps at the high strain-rate, but this
32 requires further investigation by modelling the rate of nucleation and the
33 broadening of the slip lines.
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Fig. 10 also shows that at this strain-rate between $\sim 900\text{K}$ and 1000K ,
brittle fracture occurs without any observable macroscopic deformation at
values of σ_F which decrease sharply with increasing temperature, at a rate
similar to, but somewhat below, σ_N determined by the model. The fact that
plasticity cannot be observed on the stress-strain curves does not exclude
the possibility of some plastic flow occurring. The reason for the

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3 discrepancy between the predicted σ_F and observed values of σ_N is not
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5 clear. Below 900K the fracture is assumed to be controlled by pre-existing
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7 flaws in the specimen; at a mid-range stress of 2.5GPa the typical flaw size
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9 is $\sim 50\text{nm}$. At the lower strain-rates there are only 2 or 3 such datapoints at
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11 each strain-rate. At each strain-rate (except for the highest, see fig. 10)
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13 the fracture stresses, which fall within the same range as those at room
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15 temperature, are highest at the lowest temperature, down to about 725K
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17 (see e.g. fig. 3), but the data are too sparse to attempt any meaningful
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19 analysis.
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26 **6. Discussion**

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31 The conditions for the occurrence of a BDT in Folk's experiments have some
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33 similarities with those which control the BDT in precracked specimens. In
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35 Folk's experiments the plastic zone on system III in the tensile part of the
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37 beam has to be developed rapidly to cause a sufficiently large load drop to
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39 prevent brittle fracture. In the precracked specimen the crack has to be
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41 shielded before the applied stress reaches the value needed to cause brittle
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43 fracture. In both cases the plasticity is brought about by dislocation
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45 avalanches produced by new sources nucleated by the motion of
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47 dislocations either to the surface (present experiments) or to or along the
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49 precrack [8,9]. As in the case of precracked specimens the nucleation of
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51 the sources is controlled by dislocation velocity, and in both cases the
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53 dislocations move a distance characteristic of the experimental and
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55 specimen conditions, resulting in "structure sensitivity" of the BDT. In both
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3 cases it is this process which causes the strain-rate dependence of T_c to be
4 controlled by dislocation velocity.
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10 There are however some important differences. Firstly, in the present
11 experiments the brittle fracture stress σ_F is strongly temperature
12 dependent, whereas for the precracked specimens brittle fracture occurs at
13 the precrack at K_{IC} which is relatively temperature independent. The reason
14 for this strong temperature dependence is that in this case the critical
15 surface flaws are nucleated by the dislocation activity itself, and the
16 temperature dependence of σ_F follows that for nucleation of surface sources
17 by cross-slip, i.e. σ_N . Unlike the case of bend tests on precracked
18 specimens, in most cases fracture actually occurs after a small load drop,
19 due to the formation of stress relieving slip lines. Whether fracture or
20 plastic hinging occurs after the sources are nucleated on system III
21 depends critically on whether only a few dislocation sources are nucleated
22 which provide insufficient slip to relieve the tensile stress before fracture
23 occurs, and which form sharp slip steps acting as potent notches, or
24 whether many sources are nucleated, the slip steps are gradual and the
25 notches are less potent, in which case plastic flow occurs. The attainment of
26 this latter condition determines T_c . Secondly, the range of temperatures in
27 which it is uncertain whether flow or fracture occurs can be as much as
28 30° , compared to less than 5° for precracked undeformed Si (e.g. [4]).
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30 Thirdly, at a high strain-rate brittle fracture occurs at all temperatures
31 investigated, including temperatures much higher than that at which T_c
32 might be expected. This is quite different from the situation for precracked
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3 specimens, where a BDT is observed and expected at all the strain-rates
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5 used in the experiments.
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10 **Acknowledgements**

11
12
13 We are grateful to Professor David Pope for sending us a copy of Folk's
14
15 thesis, and to Professor Vaček Vitek for emailing copies of the micrographs.
16
17 We would like to pay tribute to Dr. Folk for his excellent experimental
18
19 study. Our thanks are also due to Professor G. D. W. Smith for providing
20
21 laboratory facilities.
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Nabarro Festschrift
Submitted to Phil. Mag, Sept 2005
Revised version Feb. 2006

Table 1

Experimental and modelled brittle – ductile transition temperatures

Strain rate (s^{-1})	Brittle – ductile transition temperature (K)	
	Experiment	Model
6.0×10^{-6}	885	890
3.0×10^{-5}	938	943
6.0×10^{-3}	972	967
1.2×10^{-4}	1006	994
6.0×10^{-4}	No Transition	1066

Figure Captions

Figure 1: Typical stress –strain curves for 3-point bending tests on silicon beams at temperatures around the brittle-ductile transition: type1 - brittle (727K); type 2 - brittle with prior plasticity (1003K); type 3 - fully plastic (1006K).

Figure 2: Geometry and crystallographic orientation of silicon three-point bend specimens used by Folk (2000) for brittle-ductile transition experiments.

Figure 3: Typical experimental results (Folk 2000); fracture, upper yield and flow stresses as a function of test temperature for a strain rate of $6 \times 10^{-5} \text{ s}^{-1}$. The dashed lines indicate the upper and lower limits of low temperature brittle fracture stresses.

Figure 4: SEM micrographs of the etched inclined side and tensile faces of specimens loaded at $6 \times 10^{-5} \text{ s}^{-1}$ and 979K to (a) 60%, (b) 80% and (c) 100% of the peak stress, and (d) after the load drop. The central loading contact is on the bottom edge in each case.

Figure 5: Proposed sequence of dislocation motion in the tests (see text): a) dislocations on type I slip systems nucleate at the contact edges and glide towards the tensile side of the specimen; b) secondary slip systems (type II) operate under the stresses from the contact and from dislocations on the primary systems; c) primary and secondary dislocations approach the tensile surface; d) secondary dislocation emerges at the tensile surface leaving a screw segment; e) screw segment cross-slips onto primary slip system forming a source near the tensile surface which can rapidly generate dislocations of type III; f) Dislocation types used in the model; co-ordinates used in equations (1) are also shown. For clarity, in (a)-(e), only one geometric variant of each slip system is shown, and the junctions between dislocations on type II and type I systems which terminate the type II dislocations are not shown.

Figure 6: Modelling of type III dislocation arrays, nucleated at the tensile surface at two levels of bend stress (750 and 790 MPa). Strain rate = $6 \times 10^{-5} \text{ s}^{-1}$, temperature = T_c for this strain rate = 972K. Fracture stress at the BDT is ~ 800 MPa (vertical dotted line).

Figure 7: Modelling of types I and II dislocation arrays. Strain rate = $6 \times 10^{-5} \text{ s}^{-1}$, temperature = T_c for this strain rate = 972K. Fracture stress at the BDT is ~ 800 MPa (vertical dotted line). Horizontal dotted lines indicate the dislocation array lengths corresponding to the specimen's neutral axis and tensile surface.

Figure 8: Temperature dependence of experimental fracture stress and experimental and modelled yield stress at a strain rate of $6 \times 10^{-5} \text{ s}^{-1}$. The horizontal dotted line indicates the average experimental fracture stress at the brittle-ductile transition (800MPa); vertical lines indicate the experimental T_c (972 K) and the modelled T_c , where the modelled yield stress equals 800 MPa (967K).

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4 **Figure 9:** Arrhenius plots of strain-rate dependence of experimental and
5 modelled brittle ductile transition temperatures (T_c). Activation energies
6 corresponding to the best-fit lines are 1.9 eV for the experimental data
7 and 2.2 eV for the model.

8
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10 $6 \times 10^{-4} \text{ s}^{-1}$; fracture and upper yield as a function of test temperature.
11 The results of the model for upper yield stress (σ_N) for the same
12 conditions are also shown..
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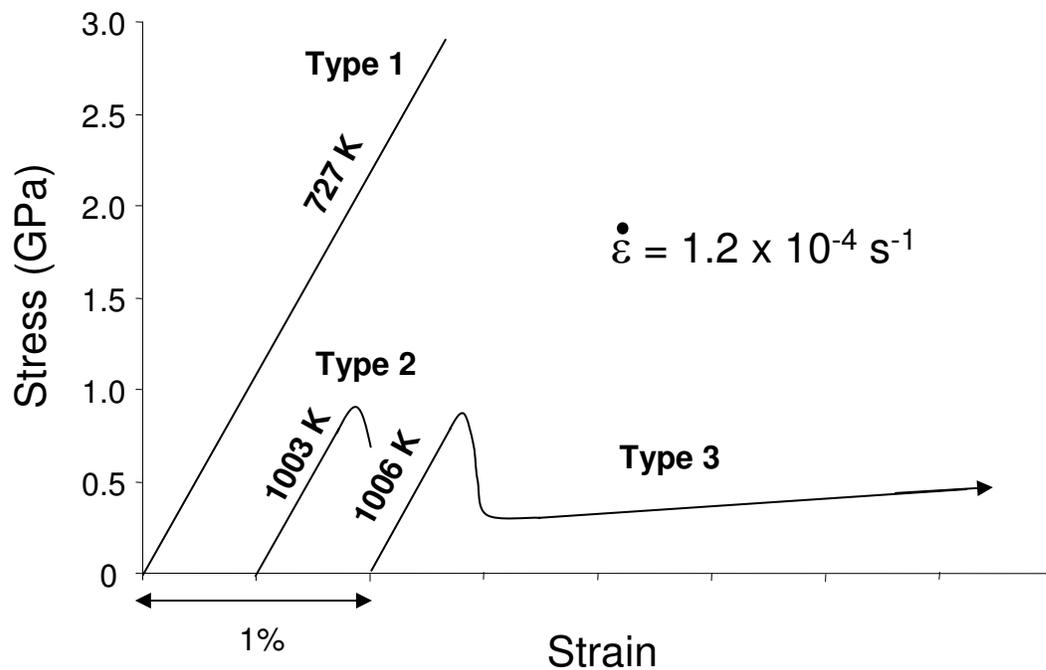


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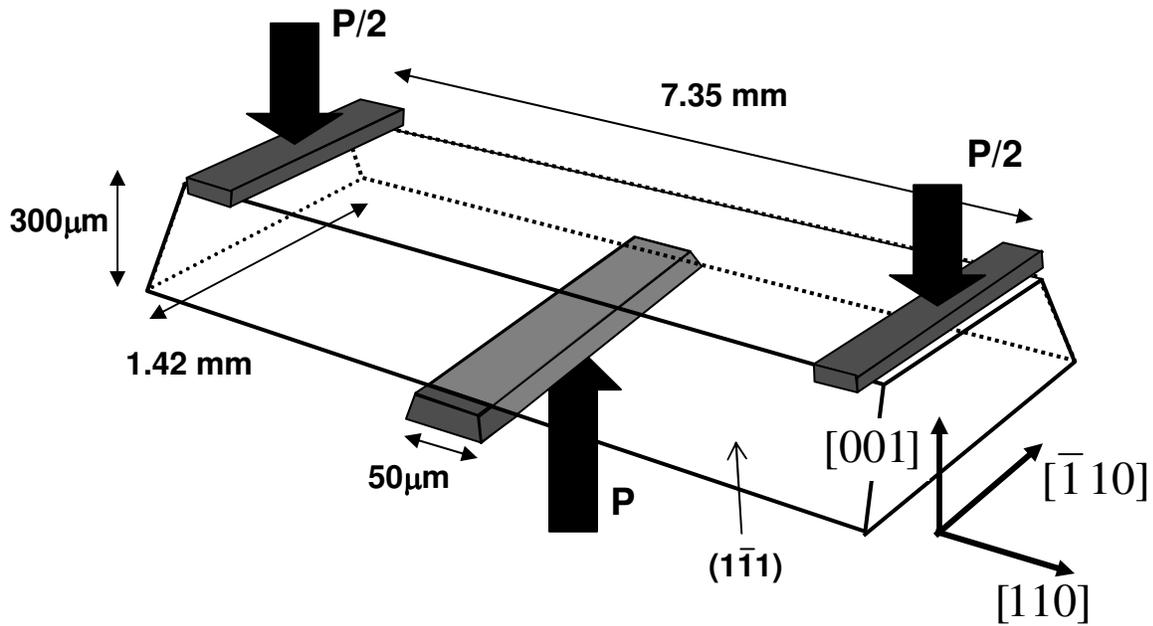


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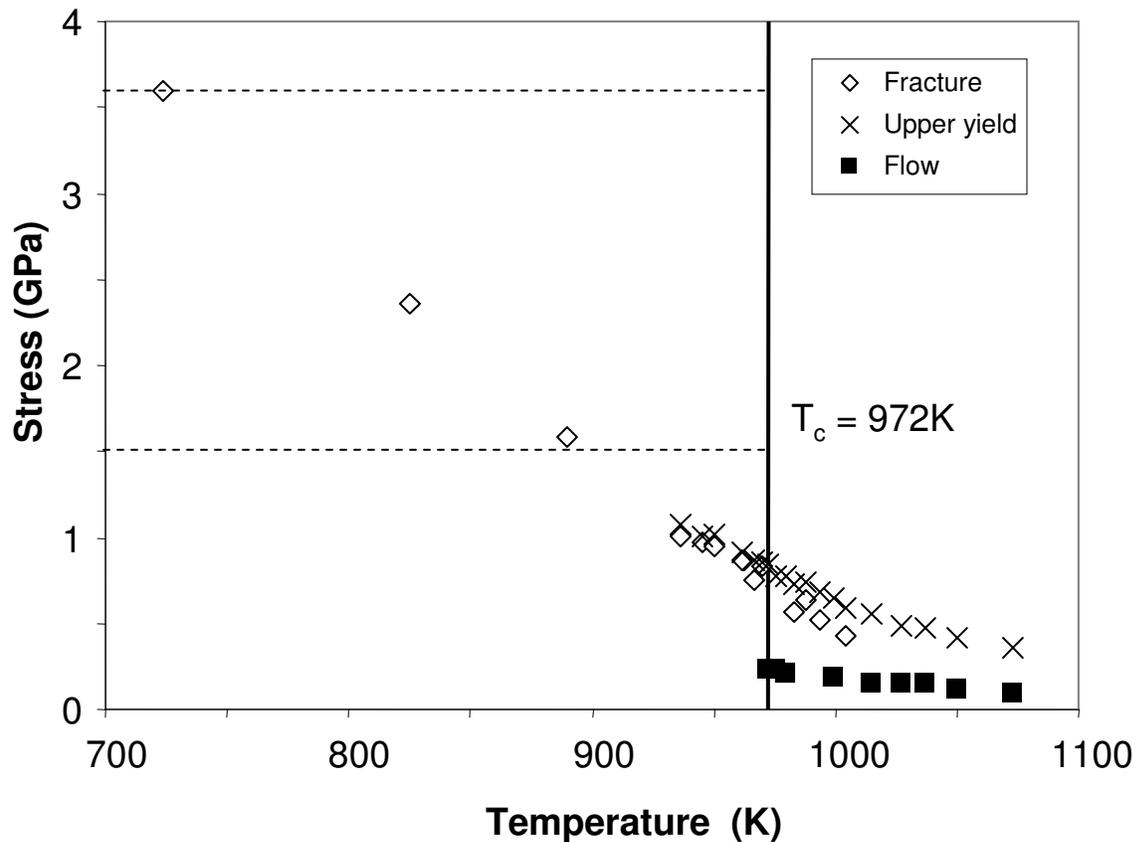


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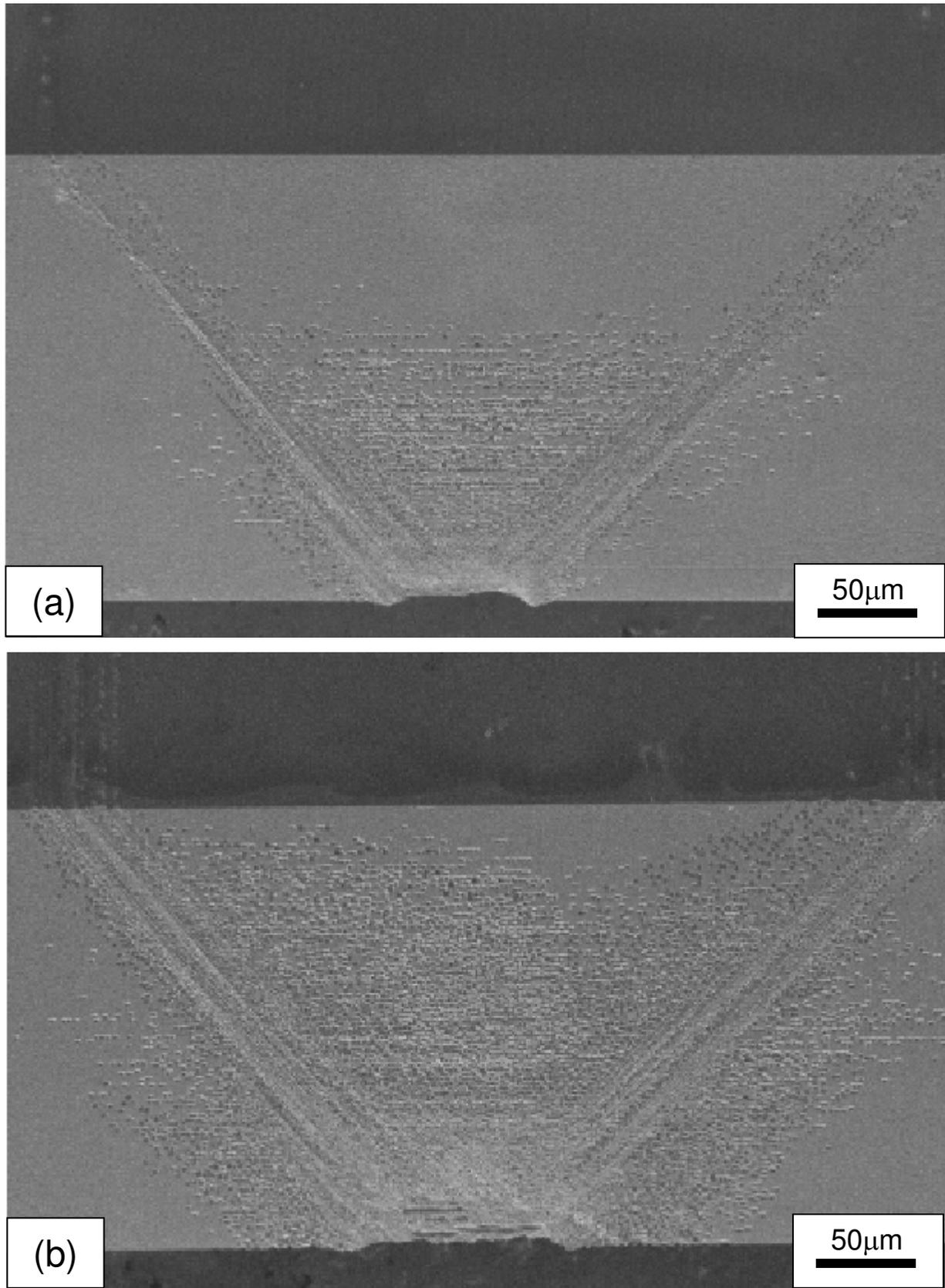
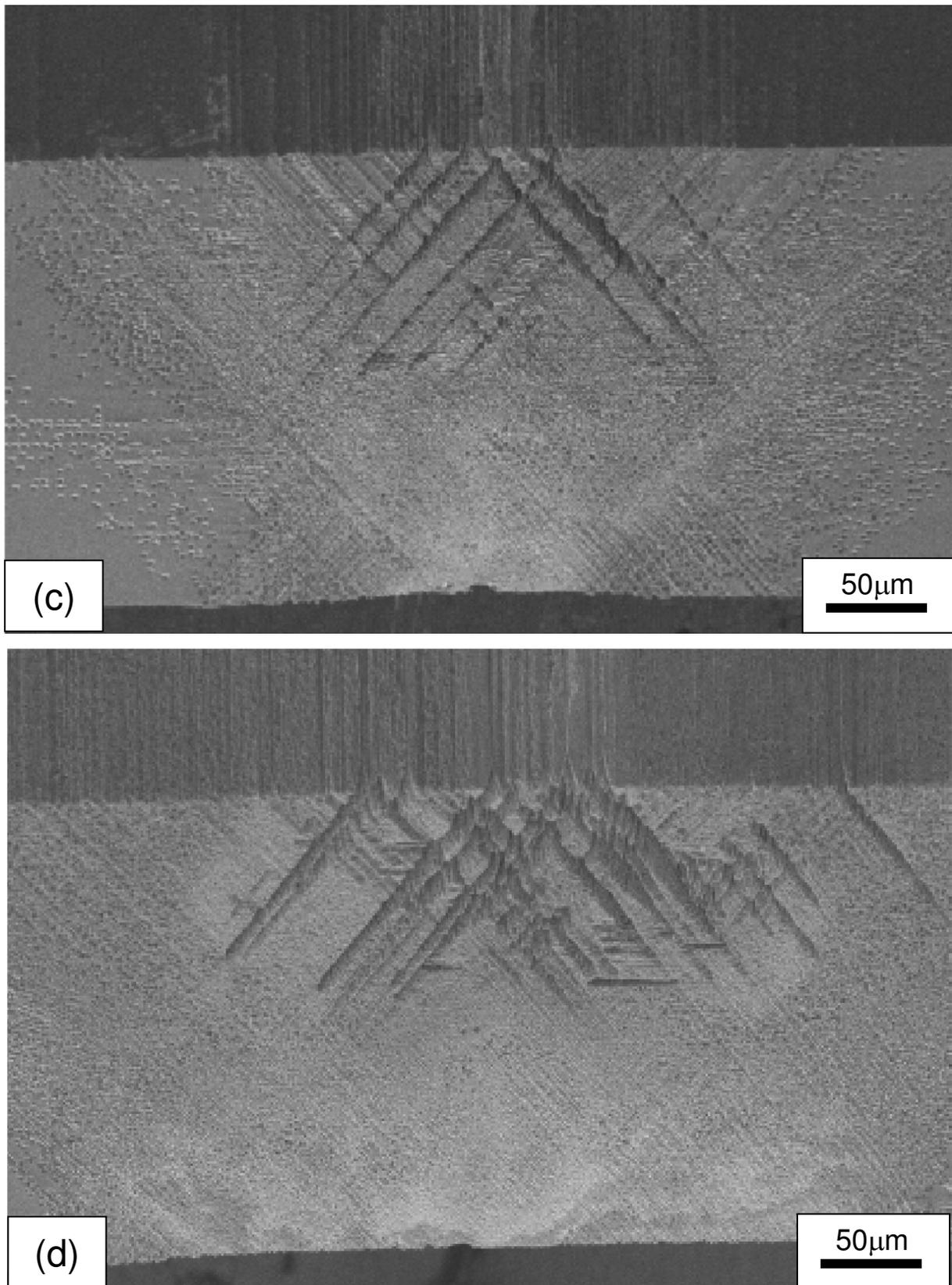


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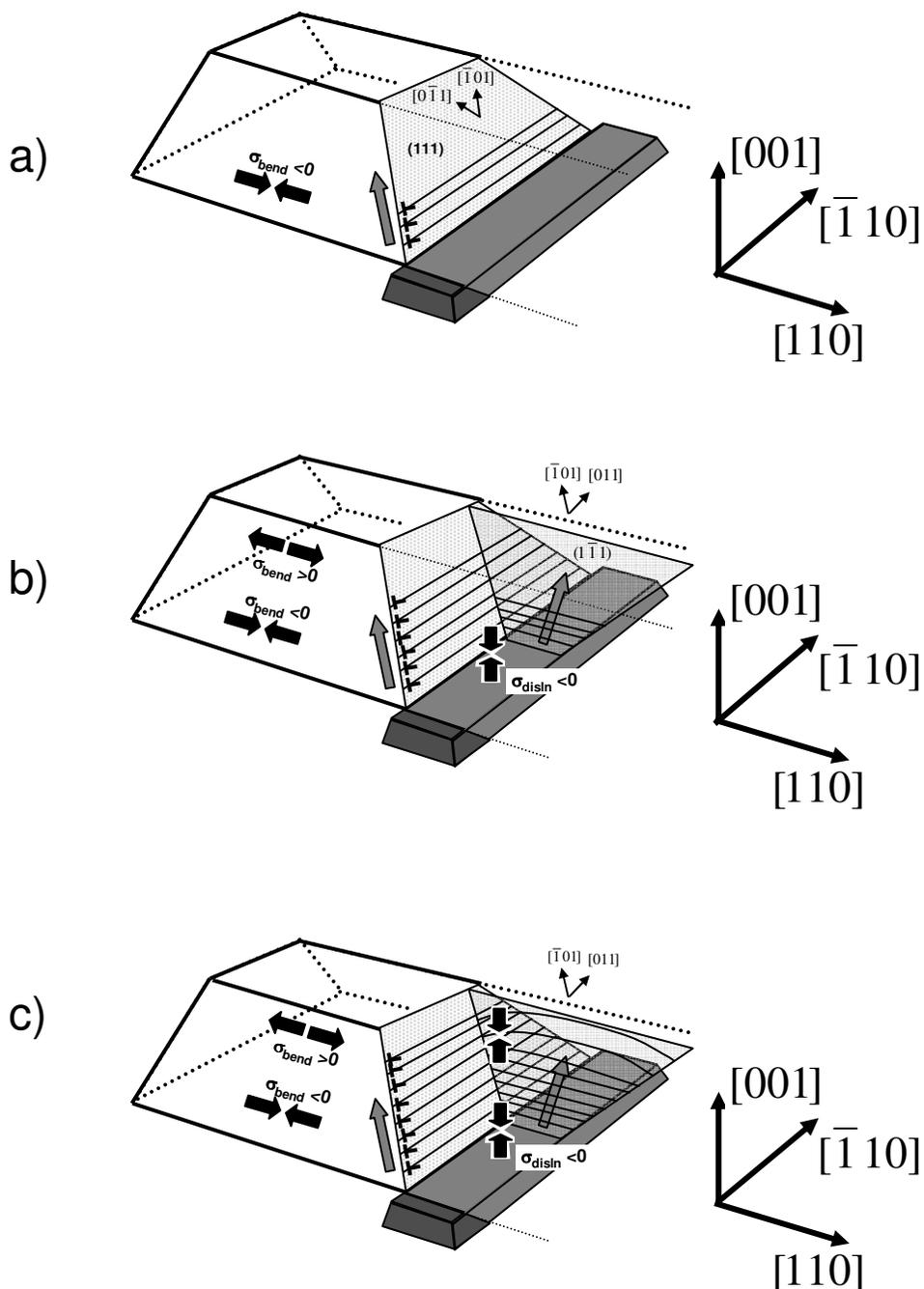


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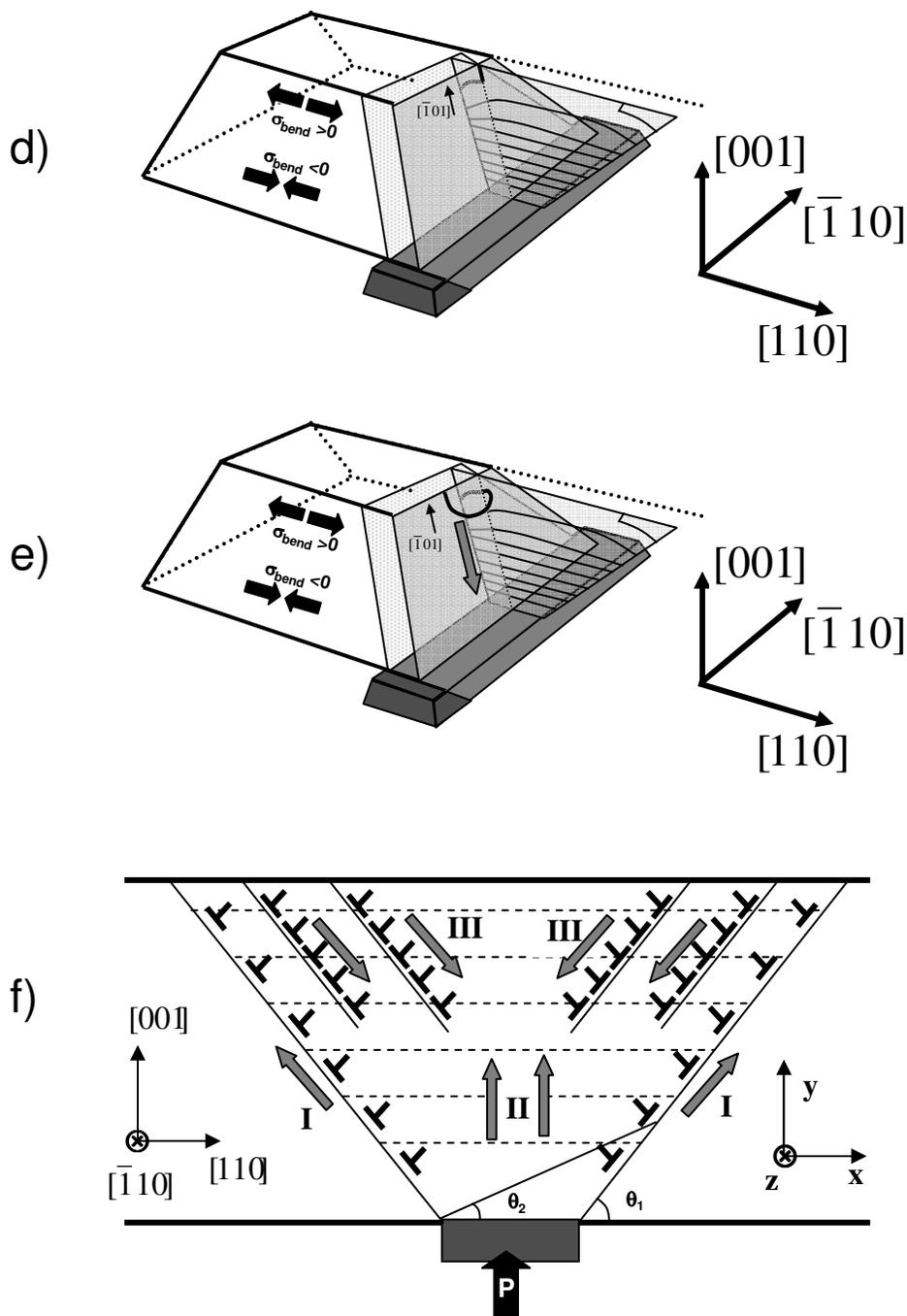


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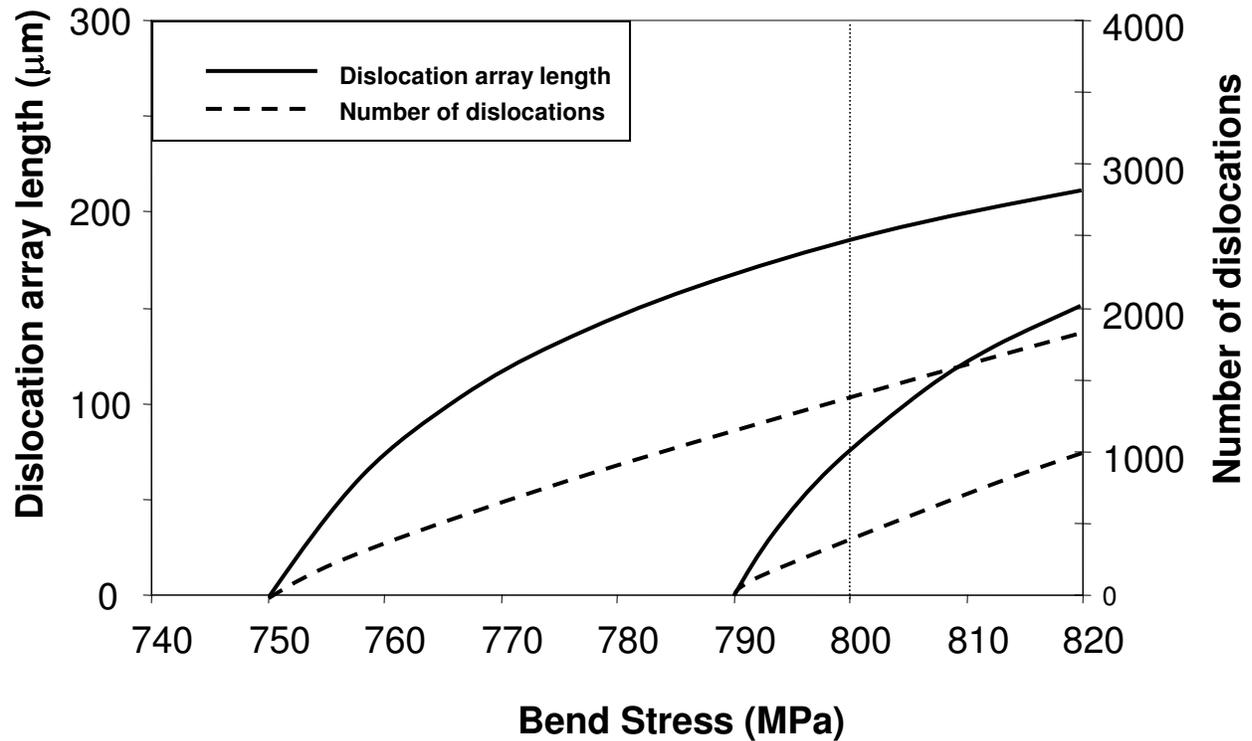


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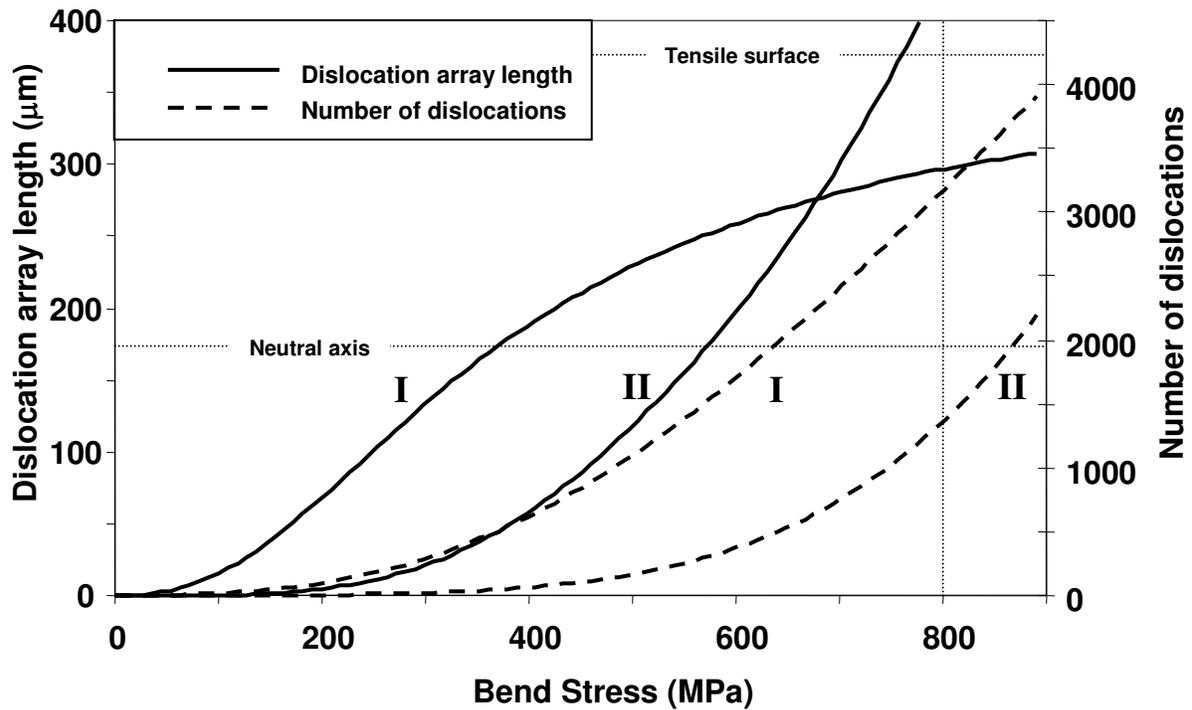


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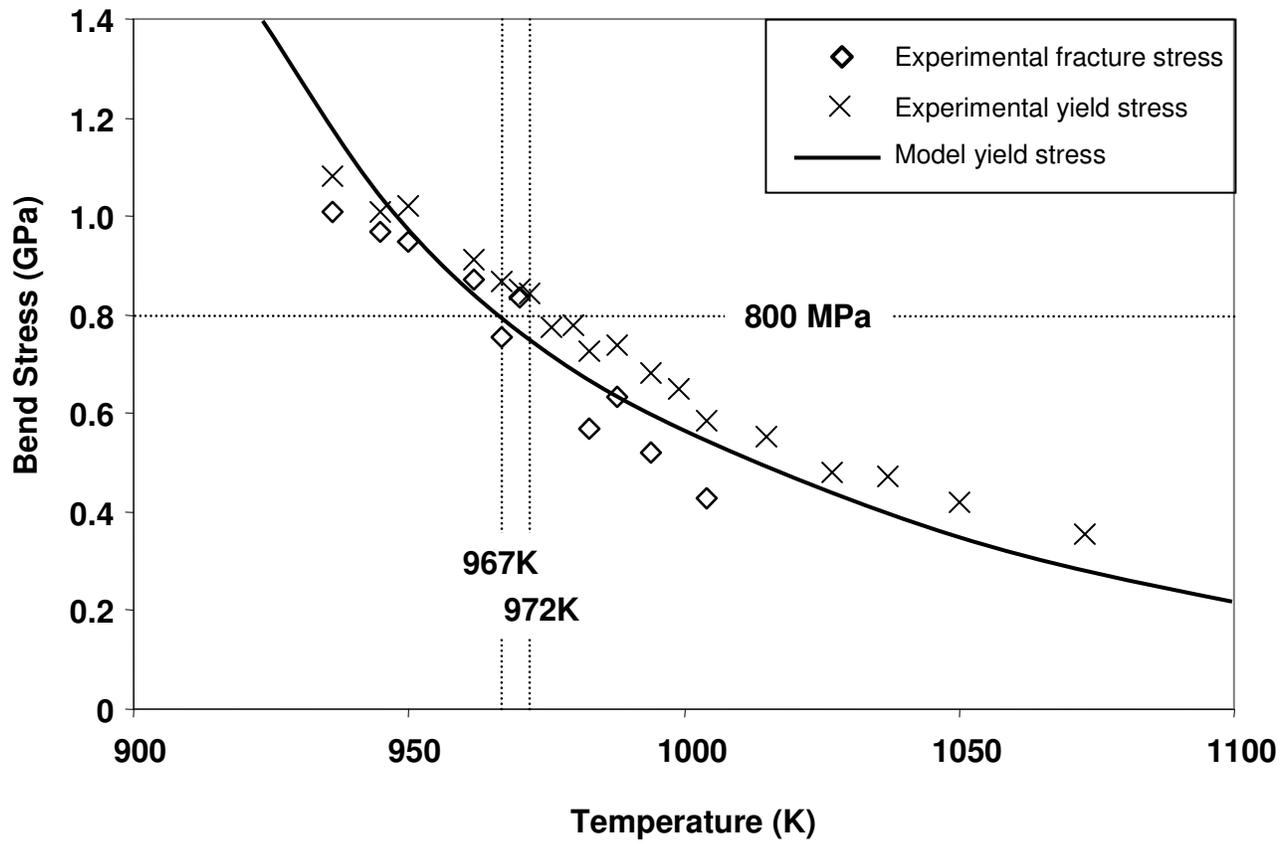


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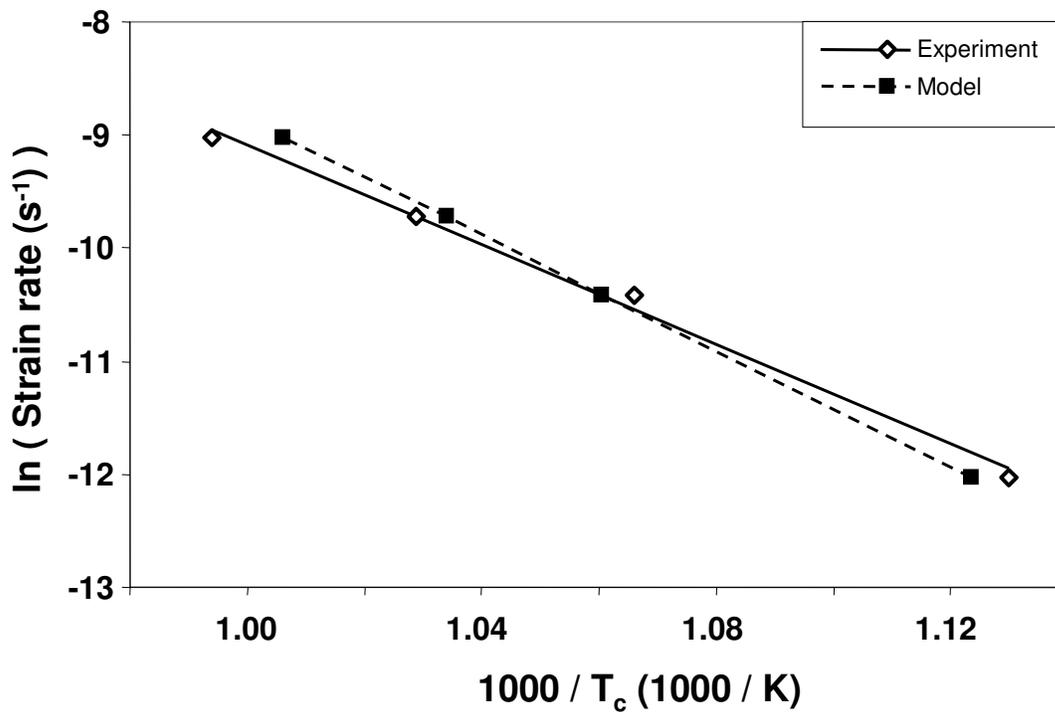


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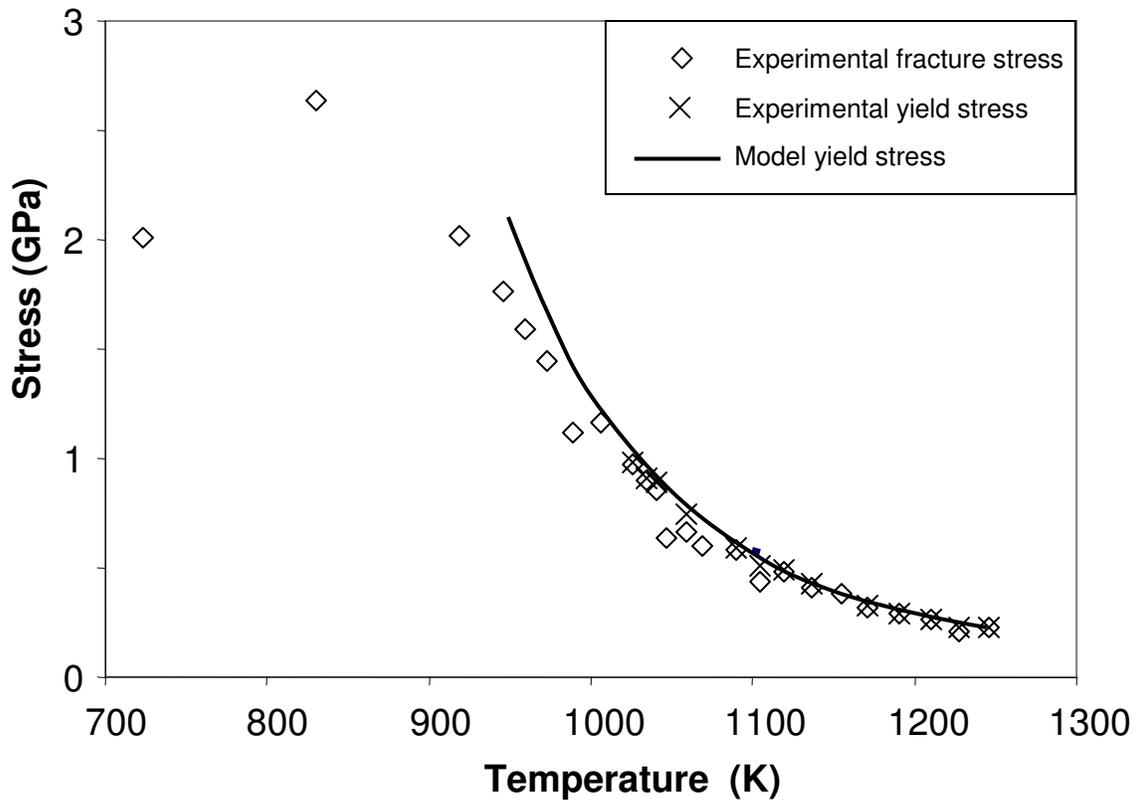


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