DISLOCATIONS DEVELOPED AROUND CRACK TIPS IN SILICON AND THEIR INFLUENCE ON FRACTURE TOUGHNESS

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In pre-cracked silicon specimens submitted to a static mode I loading at temperatures from 923 K to 1073 K, dislocations are generated around the crack tip when $K_I \gtrsim K_{I_{min}} = 0.25$ MPa$\sqrt{m}$, whatever the temperature.

The critical stress for fracture measured at room temperature in specimens previously plastically strained is noticeably higher than in dislocation-free silicon. This likely results from shielding of the crack tip by the observed dislocations.

INTRODUCTION

The purpose of this work is to investigate the effect of a plastically deformed region around a crack tip on fracture toughness in the very special case of silicon. In most experimental situations, crack propagation and plastic deformation occur simultaneously. In silicon, which is fully brittle at room temperature and can be plastically strained only $T \geq 0.5\ T_m$ ($T_m = 1690$ K), these two phenomena can be separated.

EXPERIMENTAL

Dislocation-free, n-type ($\rho \geq 5\Omega$.cm), float-zoned silicon single crystals were used. The geometry of the profiled specimens designed for mode I loading is shown in Fig.1, with the two cristallographic orientations chosen. To an applied load P corresponds a stress intensity factor $K_I = P x f(a,e)$ (1) -a crack length, e specimen thickness $\simeq$ 0.6 mm- where f(a,e) is a weakly varying function of a determined by Srawley and Gross (1).

The experiment consisted of three steps (for details see Michot et al. (2) or Michot (3)) :

(i) A crack was introduced by limited cleavage at room temperature using a technique first described by St John (4). As proved by X-ray topography, no plastic strain occured at this step.

(ii) The pre-cracked specimen was loaded at 650°C ≤ T ≤ 800°C. In that range, the dislocation velocity becomes large enough for plastic strain to occur.

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Two different tensile stages were used. In the first one, the specimen was heated up under vacuum and cooled down slowly (0.1 C/s) under a residual load. Then, the shape, slip plane, Burgers vector of dislocation loops were analyzed by conventional Lang technique of X-ray topography (2). In the following the results obtained with that stage are reported using open symbols. The second stage was built up for an 'in situ' observation of the growth of the plastically deformed region by X-ray topography using the synchrotron radiation of L.U.R.E.-D.C.I. (Michot and George (5), George and Michot (6)). In that stage, the specimen was heated up under a 10% H_2, 90% N_2 atmosphere and cooled down rapidly (5°C/s) under full load (results marked by full symbols).

(iii) The critical load for fracture, P_c^*, was then measured at room temperature -so that dislocations were no longer able to glide- on an Instron machine with a crosshead displacement rate of 5 µm/mn. X-ray observations were supplemented by etch pit counts on broken specimens.

RESULTS

The main features can be summarized as follows :

(i) at room temperature, the critical stress intensity factor leading to brittle fracture, K_{IC}, is equal to 0.93 MPa$\sqrt{m}$ in dislocation-free specimens, in agreement with (4).

(ii) at 650°C $\leq$ T $\leq$ 800°C, dislocations are generated around the crack tip when $K_I \geq K_{Imin}$ equal to 0.25 MPa$\sqrt{m}$, independent on temperature.

(iii) dislocations belong to those {111} slip planes that cut the crack tip (2). In orientation β (Fig.1), less than 10% of the total number of emitted dislocations belong to the planes parallel to the (ideal) orientation of the crack tip. A large fraction of the total number of dislocations -about 50% in orientation α, 90% in orientation β- have a Burgers vector parallel to the crack plane. Typical dislocation configurations are shown in Figure 2.

(iv) Figure 3 represents the average size of the plastically deformed region as a function of time at different temperatures and stress intensity factors. At T > 700°C, the curves exhibit two regimes : I, fast growth and II, stabilization. At a given K_I, the higher the temperature, the faster the growth but the size of the stabilized plastic region is temperature independent. (Stabilization was not reached at 650°C due to the smaller velocity of dislocations). At a given temperature, the size of the stabilized plastic region increases with the applied stress intensity factor (5).

(v) The maximum dislocation density close to the tip is $\sim 3.10^6 cm^{-2}$. The total number of emitted dislocation loops in one specimen is typically between 2000 and 5000. For a stabilized plastic region the number of dislocations no longer varies with time. It increases with the applied stress intensity factor.

(vi) Figure 4 shows the product, C x e, of the compliance, C, measured at room temperature, by the specimen thickness, e, as a function of the crack length, a. It can be seen that the compliance is higher in dislocation free silicon than in plastically strained specimens. There are significant differences between the two classes of specimens treated in the two tensile stages mentioned above.

(vii) In plastically strained specimens, we can define a 'critical stress intensity factor' K_C^* :

$$K_C^* = P_C^* \times f(a,e) \qquad (2)$$

where P_C^* is the critical load at fracture. On Fig. 5, we have reported the variation of the ratio K_C^*/K_{IC} as a function of K_I/K_{IC}, where K_I is the stress intensity factor applied at high temperature during the second step of the experiment. It is clear that previous plastic strain increases the critical fracture stress at room temperature. It must be noticed that the effect is less pronounced in specimens treated under vacuum and cooled slowly under residual load than in those treated under reducing atmosphere and quickly cooled down under load. (It was checked that both heat treatments without any applied load did not change the fracture stress in dislocation-free Si.

DISCUSSION

Discussion will be restricted to points (vi) and (vii). Features (i) to (v) were discussed previously in (2,3,5).

Plastic strain around the crack tip can modify the conditions for crack propagation either by blunting of the crack tip -dislocations having a Burgers vector with a component normal to the crack plane relax geometrically the sharpness of the tip (Friedel (7))- or by shielding- long range dislocation stresses oppose crack opening (Majumdar and Burns (8)).

There is good evidence that blunting is negligible here : first, most of the observed dislocations have their Burgers vector parallel to the crack plane and secondly, blunting by dislocation loops in slip planes intersecting the tip in very localized. Roughly, one loop increases the radius of curvature at crack tip from $\sim$ b to $\sim$ 2b over a length of $\sim$ 2b. Thus, 2000 loops with a suitable Burgers vector could achieve crack tip blunting over a tip length of ~ 1.5 µm only ! In the case of orientation β, blunting could be more efficient but very few dislocations belong to planes which may contain substantial lengths of the tip.

The increase of fracture toughness by crack tip shielding could be rationalized as follows : Following (8) we assume that dislocations exert compressive long range stresses opposing crack opening, so that the effective stress intensity factor is :

$$K_{Ieff} = K_I - K_D \qquad (3)$$

It appears reasonable to admit that K_{Ieff} cannot become smaller than K_{Imin}, then :

$$K_D \leq K_I - K_{Imin} \qquad (4)$$

At room temperature if no blunting took place previously the scaling function f(a,e) is not modified and K_D is still exerted by frozen-in dislocations :

$$K_{Ieff} = P \times f(a,e) - K_D \qquad (5)$$

Fracture occurs at the load $P_C^\star$ for $K_{Ieff} = K_{IC}$, so :

$$K_C^\star \leq K_{IC} + K_I - K_{Imin} \qquad (6)$$

As K_C cannot be lower than K_{IC}, with the reported values of K_{Imin} and K_{IC}, the ratio K_C/K_{IC} should be an increasing function of K_I/K_{IC} varying between the extreme values 1 ($K_I = K_{Imin}$) and 1.74 ($K_I = K_{IC}$). This fairly agrees with the results reported Fig.5.

The compressive stress due to dislocations also explains the decrease of the compliance shown in Fig.4.

The difference of toughness of specimens treated in the two tensile stages is probably due to the different cooling conditions. During slow cooling under a residual load, slip reversibility can take place, reducing the dislocation stress.

SYMBOLS USED

a = crack length

e = specimen thickness

K_I = applied stress intensity factor defined by equation (1) (MPa$\sqrt{m}$)

K_{Imin} = minimum value of K_I leading to plastic strain around the tip

K_{IC} = critical stress intensity factor for brittle fracture in dislocation-free specimens

K_C = critical "stress intensity factor" in pre-strained specimens

K_D = "stress intensity factor" due to the long range dislocation stress

K_{Ieff} = effective stress intensity factor in pre-strained specimens

P = applied load

P^*_c = load at fracture in pre-strained specimens

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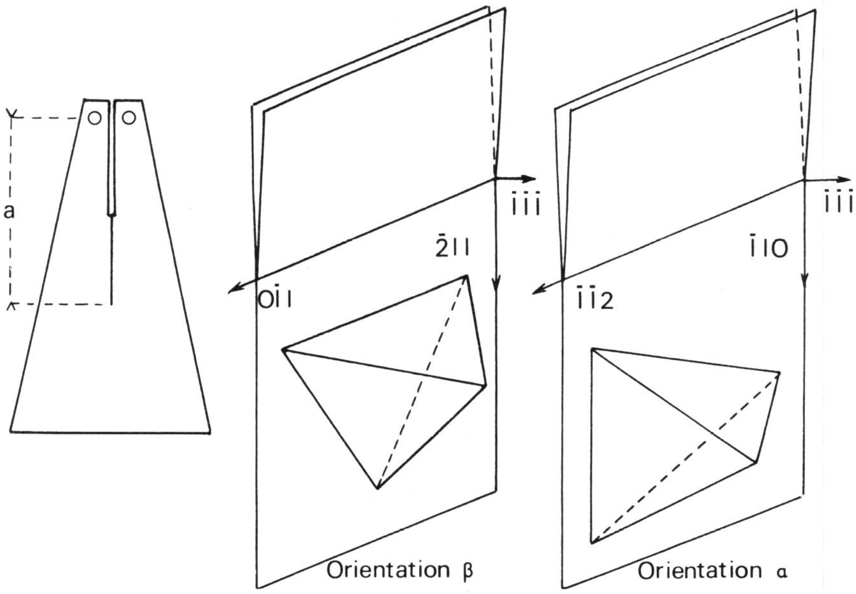

Figure 1 Specimen geometry and crystallographic orientations

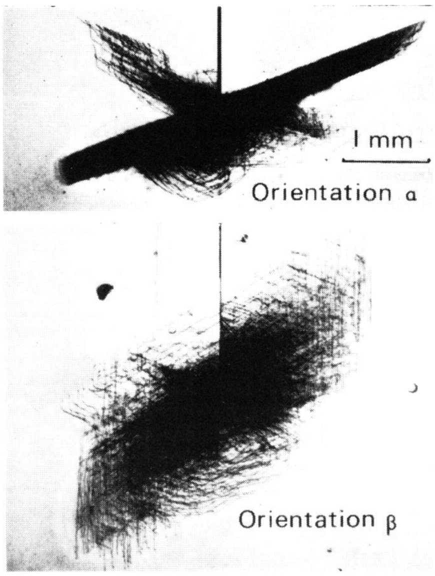

Figure 2 Typical dislocation configurations at crack tip after loading at 800°C.

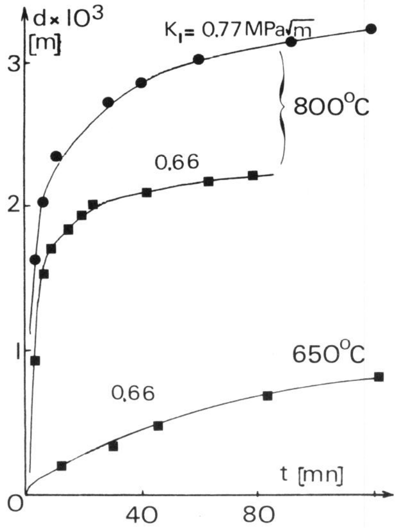

Figure 3 Size of the plastic zone vs time

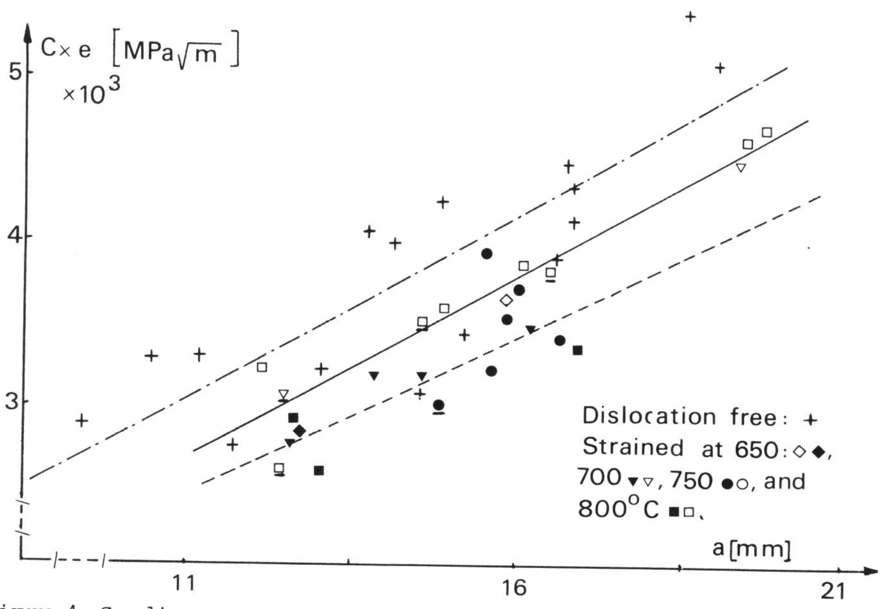

Figure 4 Compliances vs crack length for different pre-strain conditions (open and full symbols, see text ; underlined symbols : orientation β)

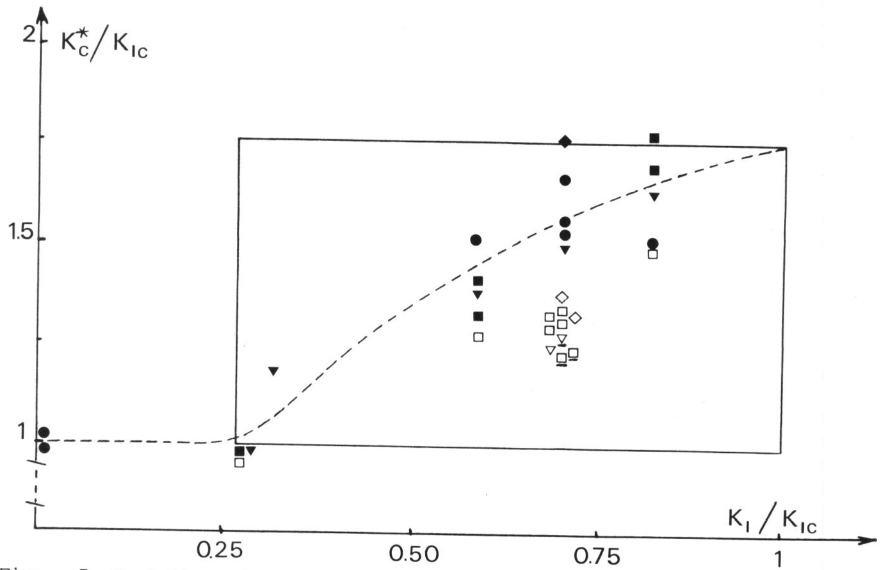

Figure 5 Variation of the critical "stress intensity factor" with pre-strain conditions (open and full symbols, see text ; pre-strain temperatures, as Fig.4)