A High-Temperature Deformation Stage for X-ray Synchrotron Topography. Applications to Dislocation Mechanisms in Silicon

By A. George and G. Michot

Laboratoire de Physique du Solide, LA CNRS No 155, ENSIM, Parc de Saurupt, 54042 Nancy CEDEX, France and LURE,* Batiment 209C, Université de Paris Sud, 91405 Orsay CEDEX, France

(Received 23 November 1981; accepted 1 February 1982)

Abstract

A high-temperature (~1070 K) tensile stage operating under controlled atmosphere convenient for X-ray synchrotron topography is described. Early results of in situ measurements of dislocation velocities in silicon and an observation of dislocations expanding from the tip of a crack under controlled loading conditions are briefly reported.

1. Introduction

The advent of very intense synchrotron radiation sources has renewed the interest of in situ observations of moving dislocations by X-ray topography (Tanner, 1977; Miltat & Bowen, 1979; Sauvage & Petroff, 1980). In the most simple settings, the geometrical resolution and image quality are equivalent to those of the conventional Lang technique (Lang, 1959) but the exposure times are considerably reduced allowing the quasi instantaneous positions of dislocations gliding under stress to be recorded on nuclear emulsion photographic plates provided that the dislocation velocity does not exceed about 0.1 μm s⁻¹. This is the case over a wide range of stress and temperature in semiconductors (Alexander & Haasen, 1968; Schaumburg, 1972; George, 1977, 1979; George & Champier, 1979) where the dislocation mobility is controlled by the lattice friction. As a drawback, following the motion of dislocations in these materials requires a high-temperature (T>920 K for Si) deformation stage in which, moreover, the specimen has to be protected against oxidation.

The purpose of this note is to describe such a stage designed to be operated at LURE (Orsay) using the radiation emitted by the storage ring DCL. This stage, which is markedly different from others described in the literature (Kume & Kato, 1974; Bowen & Miltat, 1976; George, 1977; Debrenne, Mathiot & De Tournemine, 1980; Baumgart, Markewitz & Hartmann, 1980), has been used to study elementary dislocation mechanisms in silicon. Two examples will be briefly given: measurements of dislocation velocities and punching of dislocations at a crack tip in a pre-cracked specimen when loaded at high temperature. The range of applied forces is different in these two experiments, so two stages have been built which differ only in the loading system.

In both cases the in situ observations aimed to complete previous experiments in which observations were performed at room temperature after unloading. Therefore it was possible to check that the results obtained with the high-temperature stage were in agreement with previous measurements.

2. Design criteria

The requirements for the stage to be efficient are the following:

(i) The order of magnitude of dislocation velocity in silicon shows that in situ observations are of interest only at temperatures higher than 870 K. Because of the high temperature sensitivity, reliable measurements are attainable only if the temperature variation during each experiment or from place to place in the gauge length does not exceed 5 K. (This introduces a scatter of about 15°o in the dislocation velocity.) It is interesting to be able to change the temperature quickly. However, this low thermal inertia is reached at the expense of temperature stability and uniformity.

(ii) Crystals must be prevented from oxidation which would lead to dislocation pinning close to the surface.

(iii) Fresh dislocation loops must be nucleated at the testing temperature. This gives the magnitude of the stress that must be applied. For temperatures ranging from 870 to 1070 K, nucleating isolated dislocation half-loops from scratches made by a diamond needle requires resolved shear stresses of about 25 to 30 MPa (George, Escaravage, Champier & Schröter, 1972), which, with the specimen used for velocity measurements (Fig. 1a), means nominal stresses of 50 to 60 MPa and external forces of 100 to 120 N. The
uniaxiality of the applied force must be carefully checked (George & Champier, 1979).

In pre-cracked specimens very strong local stress concentrations arise in the vicinity of the crack tip and the external load applied to the specimen is several newtons.

(iv) In order to fit onto the spectrometer equipping the topography port of the LURE DCI facility (Sauvage, 1978; Sauvage & Petroff, 1980), the stage must be neither too heavy (a few tens of newtons) nor too big. The crystal-to-photographic-plate distance must be as short as possible as the loss of the geometrical resolution is about 1 μm cm⁻¹. As the topography port is not at the end of the ray line, the whole setting cannot extend beyond the specimen by more than 5 cm in one direction in order not to collide with the continuing line.

The following solutions have been chosen:

(i) Heaters are notched graphite plates, fed with a low-voltage current. Since X-rays are weakly absorbed by graphite, the heaters totally wrap the specimen to ensure a uniform temperature. Also for that reason the design of the high-temperature part of the stage has the mid-point of the specimen as a centre of symmetry.

(ii) Rather than using complicated techniques to keep the specimens under a sufficiently high vacuum, we used a continuous flow of 90% N₂, 10% H₂. Yet, the stage still has to be leak-tight.

(iii) Load is applied using a screw and a calibrated string and measured by a small-size load cell when large stresses are needed, by hanging up marked weights with a wire and pulley system for small stresses. To achieve a fair axiality of the stress, great attention was paid to the alignment of grips which are not allowed to rotate in any way, the moving grip being accurately guided by a precise ball-slider.

3. Design details and performances

The stage is shown in Fig. 1. To ease the description, part A comprising the furnace and part B, on which is attached the loading system, can be viewed separately.

Part A consists of a rigid frame made of stainless steel plates of 3.5 mm thickness assembled by screws and leak-tightened with refractory cement Autostick.* On the frame are fixed two electrical feed-throughs, two nozzles for gas in- and outlets and a passage for the thermocouple. (We used a chromel–alumel thermocouple, Inconel covered. The diameter of the Inconel tube is 0.3 mm, small enough to prevent inconvenient heat loss by conduction along the thermocouple.) The fixed grip is insulated from the steel frame by a boron nitride plate to avoid heat loss. A hole has been made in the opposite side of the frame for the passage of the alumina rod (3 mm diameter) connecting the moving grip to the ball slider fixed on the support B. Metallic bellows give the necessary scanning freedom while preserving tightness. Bellows must be soft and give a negligible contribution to the

---

*Carlton Brown & Partners Ltd.

---

Fig. 1. High-temperature deformation stage for dislocation velocity measurements in Si. (a) Sample. (b) General view of the stage with covers on. A furnace, B support, k Kapton window, f feedthroughs, g gas inlet and outlet, b bellows, s slider, th thermocouple, c load cell, st string, sc screws. (c) General view of the stage with covers removed: g grips, h heater, sh graphite shield. (d) Details of specimen mounting.
applied force when moved, because any accurate calibration is very difficult due to fast temperature variations during heating and cooling periods. Calorstat bellows of thin electrodeposited nickel foil were used.

Heating graphite plates 0.3 mm thick are fixed on two steel blocks (parallel connection) on both sides of the specimen-grip assembly at a distance of 10 mm apart.

The furnace A is shut by two stainless steel plates 3.5 mm thick. In each plate a 45 mm diameter window has been opened which is covered by a foil of Kapton* (0.4 mm thick) stuck with Autostick. As Kapton would be destroyed above 670 K, each window is protected against excessive heat by a graphite shield 0.3 mm thick with a thin deposit of gold on the furnace side. This shield is fixed on the cover 3 mm from it by means of small alumina pieces. Tightness is achieved by Papyex seals 1 mm thick (Papyex is a kind of strong graphite paper). Sufficient tightening is obtained by ten M3 screws on the cover plate.

The shape of the specimen used for velocity measurements is shown in Fig. 1(a). The grips used with such a specimen adjust themselves by means of graphite rounded parts free to rotate in a steel housing. Graphite blocks keep the specimen in the mid-plane of the grips. Sheets of boron nitride screwed on the grips ensure mechanical cohesion of the whole assembly whatever the setting in front of the X-ray beam. These sheets are insulating, which allows the heaters to be very close to the grips without any risk of a short circuit. The load cell used was a Sensotec, series wafer-thin, of capacity 250 N. In that configuration the maximum temperature is ~1020 K (current ~ 15 A, 12 V). This temperature can be kept constant over more than 2 h without any damage to the stage. The windows and photographic plates are simply cooled by air blown during operation.

A general view of the stage used with pre-cracked specimens (the shape of which is shown in Fig. 2a) is shown in Fig. 2(b). The specimen is held in two slit alumina rods (8 mm diameter) by means of tungsten pins. The load must be corrected for the weight of the moving part of the slider if its axis is not horizontal. A temperature of 1070 K is easily obtained (the improvement is due to the smaller mass of grips reducing heat loss).

Testing temperature is reached and stabilized in less than 10 min. Cooling down is very fast ( > 5 K s⁻¹).

Changing the specimen can be done in less than 1 h, including cooling, adjustment of the new specimen in Bragg setting and heating. It is important to reduce this time since the synchrotron beam is usually available for periods of 24 h and one experiment to be described later lasts about 2 h.

* Kapton (Du Pont de Nemours) is a polyimide film.
The stage is positioned on a goniometer cradle providing a free rotation of ±30° around an axis normal to the specimens' large face. It can be set on the first or the second axis of the LURE-DCI spectrometer (Sauvage, 1978; Sauvage & Petroff, 1980) depending whether topographs are to be obtained with a white incident beam (Guinier & Tennevin, 1949; Tuomi, Naukkarinen & Rabe, 1974; Hart, 1975) or a monochromatized incident beam. Far better images have been obtained using the latter technique, doubtless because strong parasitic rays scattered by metallic parts of the stage are more easily avoided in that case. A tantalum shield, reducing the incident beam to just the size needed to image the gauge length of the specimen, is put in front of the stage.

The synchrotron beam delivered by the DCI facility is ~6 mm high and ~5 cm wide, allowing the specimen to be imaged without scanning it in the beam. A monochromatic beam is obtained with a 220 Bragg-case reflexion on a plane Ge single crystal. The incident wavelength on the specimen can be tuned by rotating the monochromator. A wavelength of 0.8 Å was chosen for our experiments. A (+, +) setting was used. Exposure times of ~30 s were necessary for DCI operating at 1.72 GeV, 200 mA, if the topographs were to be recorded on Ilford L4 50 μm nuclear plates. The 220 reflexion was used. No effort was made to avoid higher-order reflexions, the intensity of the synchrotron beam at wavelength $\frac{\lambda}{n}$ with $n > 2$ being relatively weak.

4. Dislocation velocity measurements in silicon

In situ measurements of the velocity of screw and 60° dislocations were performed and the results compared with those of George (George, 1977; George & Champier, 1979) in order to check the performance of the stage. In intrinsic silicon two testing temperatures $T=923, 983 \text{ K}$ and two resolved shear stresses $\tau=10 \text{ MPa}, 19 \text{ MPa}$ were used. Dislocations whose velocity could be measured at the given temperatures are shown in Fig. 3. The load was incremented step by step: from $t=0$, stress intensity factor $K_I=0.25 \text{ MPa m}^{1/2}$; from $t=43 \text{ min}$, $K_I=0.66 \text{ MPa m}^{1/2}$; from $t=105 \text{ min}$, $K_I=0.77 \text{ MPa m}^{1/2}$. 220 reflexion. Monochromatized beam, $\lambda \sim 0.8 \text{ Å}$. Exposure times 30 s. 1.72 GeV, 200 mA.
mobility was measured were either previously introduced dislocation half-loops or fresh ones. In the latter case, half-loops were first expanded from the scratch over about 100 μm at the testing temperature under $\tau = 25$ MPa, before their motion was recorded under reduced stress. In situ measurements were also done at $T = 883$ K, $\tau = 19$ MPa on fresh loops in n-doped silicon containing $1 \times 10^{19}$ phosphorus atoms cm$^{-3}$.

In all cases, the measured velocities agree within $\pm 30\%$ with the values of George, which was taken as good proof that both temperature and applied stress are fairly constant and correctly measured. However, the poor contrast of images on white-beam topographs (measurements were done on basic 220 topographs corresponding to $\lambda = 0.8$ Å) precluded accurate measurements, for example of the velocities of the two kinds of 60° dislocations (Wessel & Alexander, 1977). The experiments will be tried again using a monochromatized incident beam.

5. Observations of dislocations developed at crack tips in silicon

A sharp, unrelaxed crack is introduced at room temperature by limited cleavage in a single-crystal Si sample [details of the procedure are given by Michot, Badawi, Abdel Halim & George (1980)]. After characterization of the crack geometry, the sample is heated up at a temperature allowing silicon to be plastically deformed, i.e. $T \geq 923$ K, and loaded at time $t = 0$. A constant force is applied, giving a known stress field

$$\sigma(r,\theta) = \frac{K_f f(\theta)}{\sqrt{2\pi r}},$$

where $r,\theta$ are the polar coordinates of any point in a system centred at the crack tip and $K_f$ is the so-called stress intensity factor, the value of which is chosen smaller than the critical $K_{I_c}$ necessary for the fracture to occur. The strong stress concentrations that arise at the crack tip are released by dislocation punching in those slip planes that cut the crack tip. Fig. 3 shows a series of topographs taken at $T = 1073$ K at different time intervals. The plastically deformed volume grows very fast immediately after loading and the images of dislocations are poor, their displacement being several micrometres during the exposure. When the growth rate is lower, resolution compares with that of the Lang technique, taking into account the high observation temperature and the rather high dislocation density. Observations were done for periods up to 2 h, temperatures of 923–973 K, and stress intensity factors of 0.25, 0.55, 0.66 and 0.77 MPa m$^{-1/2}$ ($K_{I_c} \geq 1$ MPa m$^{1/2}$). Results are presented in detail elsewhere (Michot & George 1982).

We would like to thank Drs M. Sauvage and J. Miltat for constant help in operating the spectrometer of LURE. We are indebted to all members of LURE and of the Laboratoire de l’Accélérateur Linéaire d’Orsay, particularly to the group ‘Anneaux’ of this Laboratory.

This work was supported by the Délégation Générale à la Recherche Scientifique et Technique under contract No. 77.2425.

References