



Experiment title: Dislocation mobility in silicon: New Experiments for a challenging Problem	Experiment number: MA-100	
Beamline:	Date of experiment: from: 12/07/2006 to: 17/07/2006	Date of report: 31/08/2006
Shifts:	Local contact(s): Dr. Jürgen HÄRTWIG	<i>Received at ESRF:</i>

Names and affiliations of applicants (* indicates experimentalists):

Dr. Thomas SCHENK, LPM Nancy
Dr. Olivier FERRY, LPM Nancy
Dr. Amand GEORGE, LPM Nancy
Dr. Alain JACQUES, LPM Nancy
Philippe MASSCHELEIN, LPM Nancy

Report:

Those runs were entirely used for the first of the two experiments that were planned: the study of the reversibility of dislocation motion in Si, by comparing the forward velocity of dislocation segments during loop growth under sensible stress with their backward velocity when loops shrink under compressive stress. (The second experiment designed to reveal a possible non monotonous stress dependence of dislocation velocity when the dissociation width is changed by appropriate stress conditions is proposed one more time, without change).

Si samples containing dislocation loops in convenient density, distributed within the gauge length had been prepared in our laboratory, so that all the beam time was used for velocity measurements.

The dislocation velocity is obtained by dividing the distance travelled by the dislocation segment by the duration of the stress application. For several reasons, instantaneous snapshots are not feasible and the distance travelled is measured by comparing the positions of the loop before and after the stress application. Accurate recordings of the loop positions are not easily obtained because of the intrinsic width of the image of a dislocation by X-ray topography and of the important variability of the contrast when stress gradients are present, even at a rather low level.

Therefore dislocation positions were always recorded under residual stress (~ 2 MPa), necessary to keep the sample in Bragg condition, after cooling down at a temperature where dislocation motion can safely be neglected (~ 200°C). Further, to avoid errors due to changes of contrast when the stress was reversed (e.g. from tension to compression) a new topograph was taken after each stress reversal, under residual stress, prior to the stress application derived to move the dislocations.

Four samples could be tested, providing velocity data at three stress level τ (5, 10, 15 MPa) and three temperatures T (610, 650, 680°C). Each tensile test at given τ and T was followed by a compression test in the same conditions, but with a shorter time, since higher velocity was expected during backward motion.

Dislocation half-loops developing from the surface are semi hexagonal, with $\langle 110 \rangle$ oriented screw or 60° segments, one nearly parallel to the surface, two emerging under an angle of about 60° .

We use to measure the depth of half-loops and the distance between the emerging points, and this gives access separately to the displacement of the parallel segment and to the average displacement of the two emerging segments. The distances measured on the films are corrected, so that the velocity is expressed perpendicularly to the segment directions.

Results are as follows:

1. When loops grow, in tension, the knees or corners joining two segments are sharp.
The velocity we measured fairly agreed with published data.
2. After compression, loops have shrunk and their shape changed. They look more rounded, especially at those knees which are inside the sample.
3. For a majority of loops, velocities derived from depth and distance between emerging points were much larger during shrinkage (backward motion) than during growth (forward motion) as can be seen by the table. The ratio of backward over forward velocity was always larger than 4 and could reach more than one order of magnitude.
4. These data are still under examination, because it appears that:
 - a. backward velocities could depend on the loop diameter (forward velocities are not size dependent in the range of sizes used here).
 - b. the rounded loops are not symmetrical : one knee has a radius of curvature larger than the other. This has not yet been clearly established but could mean that a screw/ 60° corner and a $60^\circ/60^\circ$ corner have not the same efficiency as source of kinks, or that the migration rate of kinks is not the same along screw and 60° segments, which should be an important issue for our problem.

τ [MPa] \ / \ T[°C]	610	650	680
5			3.3
10	1.3	6	4*
15	0.5*	4.2	13.8

Tab. 1: dislocation velocity [10–6cm/s] : forward motion during tensile test. (*: to be verified)

τ [MPa] \ / \ T[°C]	610	650	680
5			30
10	15	22	55*
15	3.8*	30	68

Tab. 2: dislocation velocity [10–6cm/s] : backward motion during compression test.



(a) after first T - C cycle (b) T , 30 mn : 5 MPa @ 680°C (c) C, 3 mn : 5 MPa @ 680°C

Fig. 1: evolution of loop-shapes under traction (T) and compression (C) [1 μm : 1.5 pixel]