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Transmission electron microscopy *in situ* investigation of dislocation mobility in semiconductors

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Abstract. TEM *in situ* straining experiments provide a unique way to investigate in real time the behaviour of individual dislocations under applied stress. The results obtained on a variety of semiconductors are presented: numerous dislocation sources are observed which makes it possible to measure the dislocation velocity as a function of different physical parameters (local shear stress, temperature, dislocation character, length of the moving dislocation, . . .). The experimental results are consistent with a dislocation glide governed by the Peierls mechanism, even for II–VI compounds which have a significant degree of ionic character.

For compounds, a linear dependence of the dislocation velocity on the length of the moving segment is noticed, whereas for elemental semiconductors a transition between a length-dependent and a length-independent velocity regime is observed. Analysed in the framework of the kink diffusion model (Hirth and Lothe theory), these results allow an estimation of the kink formation and migration energies.

For a variety of semiconductors, the dislocation behaviour is sensitive to electronic excitations. A strong increase of dislocation mobility with increasing electron beam intensity is observed (radiation-enhanced dislocation glide). It is attributed to a lowering of the lattice friction, due to non-radiative recombinations of electronic carriers at dislocation sites.

1. Introduction

In the low-to-medium temperature range, dislocation glide in solids with covalent or iono-covalent bonding is controlled by the lattice friction (the Peierls mechanism). This results in a thermally activated motion which, in the appropriate temperature range, could be observed in real time during *in situ* deformation experiments using a transmission electron microscope (TEM). By comparison with other techniques: double etching, x-ray topography [1, 2], where dislocation velocities are averaged over travel distances of a few tens of micrometres, a great advantage of the *in situ* TEM technique is that the dislocation behaviour in the early stages of source operation (short dislocation segments) can be analysed.

For a number of large-band-gap semiconductors, the behaviour of lattice defects is strongly influenced either by light illumination (the photoplastic effect) or electron beam irradiation (the cathodoplastic effect). For a review, see [3, 4]. These effects are related to the special properties of dislocations in these materials. Indeed they provide electronic levels in the band gap and act as active centres for electronic processes in crystals. Again, *in situ* straining experiments using a TEM provide a unique way to study the dislocation behaviour under electron irradiation at a microscopic scale and allow a determination of the physical parameters involved in this phenomenon. It is noteworthy that in addition to the fundamental aspects of this research, a growing interest in such investigations arises from the fact that this effect underlies the

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degradation of optoelectronic devices: it has been recently reported that degradation and aging phenomena are associated with propagation and/or multiplication of lattice defects due to carrier injection during the device operation.

The present paper focuses on the dislocation dynamics of bulk semiconductors, at the TEM scale (the behaviour of dislocations in heterostructures is not considered). For most compounds, experiments have been performed in a temperature range where electronic excitation has no influence on the dislocation behaviour. The experimental results will be presented and discussed. Then a report is given of a systematic study by the present authors on the radiation-enhanced dislocation glide (REDG) effect in a II–VI compound: ZnS. Thanks to TEM *in situ* experiments, the physical parameters controlling the REDG effect in this compound can be identified.

2. Properties of dislocations in semiconductors

In semiconductors, dislocation glide consists in two elementary steps: the formation of a kink pair of critical separation and the subsequent kink migration along the dislocation line. It is generally accepted that dislocation motion is well described by the kink diffusion model [5] in which kinks experience a Peierls potential of the second kind. Two regimes of dislocation velocities are predicted according to the conclusions of the model:

(i) When the mean free path of a kink pair (X) is lower than the length of the moving dislocation segment (L), the kink collides and annihilates with another kink of opposite sign generated on the same segment (kink-collision regime). The dislocation velocity (v) is proportional to X; one has:

$$v = 2\nu_D(\tau b^2h^2/kT)\exp\{-[(F_{kp}/2 + W_m)/kT]\} \tag{1}$$

where ν_D is the Debye frequency (it is assumed that the trial frequencies for both the kink formation and migration are equal to the Debye frequency), τ is the resolved shear stress, b is the dislocation Burgers vector, h is the distance between adjacent Peierls valleys (here $h = (b\sqrt{3})/2$), k is the Boltzmann constant, T is the temperature, W_m is the energy for kink migration and $F_{kp}(\tau)$ is the energy for nucleation of a kink pair, related to the formation energy of a single kink F_k by $F_{kp}(\tau) = 2F_k - \sqrt{(\mu\tau b^3h^3/2\pi)}$, μ being the shear modulus.

(ii) In the kink-collisionless regime, the diffusing kink reaches the end of the moving dislocation segment before being annihilated; v is proportional to L:

$$v = \nu_D(\tau bh^2/kT)L\exp\{-[(F_{kp} + W_m)/kT]\}. \tag{2}$$

Various experimental facts led some authors to the conclusion that the glide of long dislocations in crystals is not in the kink-collision regime. They suggest alternatively that the dislocation velocity is controlled by the presence of localized obstacles (the origin of which is still debated) on the dislocation line. For a review, see [4]. These obstacles impede the kink movement; they can be bypassed when a kink of opposite sign annihilates with the kink blocked at the obstacle location. In that case, the mean free path of kinks is the mean separation of obstacles along the dislocation line (D^*) and the dislocation velocity is given by a law similar to equation (2) in which L is replaced by D^*:

$$v = \nu_D(\tau bh^2/kT)D^*\exp\{-[(F_{kp} + W_m)/kT]\}. \tag{3}$$

That is, the velocity is length independent, as in the kink-collision regime; however, the activation energy is $F_{kp} + W_m$, as in the kink-collisionless regime.

3. Experimental procedure

A typical experiment proceeds as follows. First, monocrystalline samples, generally oriented for single slip, are prestrained up to a few per cent. Slices (typical size: $2 \times 1 \times 0.3$ mm^3) are then cut parallel to the primary slip planes and mechanically ground down to about 100 μm. They are then chemically thinned to electron transparency. *In situ* deformation experiments are performed using a high-temperature straining stage. The dislocation behaviour under applied stress is recorded in real time through a videocamera and an image intensifier. Dislocation sources are often observed. Quantitative measurements of dislocation velocity are achieved by a frame-by-frame analysis of dislocation motion. The local shear stress acting on a moving dislocation is evaluated from its curvature. In the study of the cathodoplastic effect, the electron beam intensity is varied by changing the excitation of the condenser lens. Further details on the experimental procedures used by different groups are given in the original papers [6–14].

Some important points have to be mentioned:

(i) For semiconductor compounds, the available temperature range is restricted to a few tens of °C, the convenient temperature being roughly equal to the activation energy divided by (25–30)k. Indeed, when the temperature is too low, the material is very brittle and when it is too high, the dislocations move so fast that reliable velocity measurements are hardly possible. This makes it impossible to determine the activation energies from an Arrhenius plot, although this has been done for silicon [9] where the available temperature range is higher, about 100 °C. When the dislocation velocity is sensitive to electron irradiation, combining the influence of temperature and electron beam intensity extends the available temperature range and makes it possible to determine the activation energies from an Arrhenius plot [12, 13, 15].

(ii) Care should be taken to consider only dislocations which are not close to the thin-foil surface: in addition to image forces, the presence of the free surface could induce a modification in both the kink nucleation rate and the kink kinetics. A drastic change in velocities is often observed when the moving segment approaches—or emerges at—the sample surface [6, 9].

(iii) Due to the presence of the polishing hole, the stress state in the sample is no longer uniaxial, so secondary-slip systems could be activated.

4. Dislocation mobilities in semiconductors, as studied by *in situ* TEM

In all the materials studied, both elemental semiconductors [9, 10] and the III–V compounds GaAs [6], InSb [8] and InP [14], dislocations are moving smoothly while lying along $\langle 110 \rangle$ rows of the glide plane, indicating that they are subjected to a high lattice friction (the Peierls mechanism).

Figure 1 shows an example of a dislocation source in GaAs (sample surface $(\bar{1}54)$, tension axis $[\bar{3}\bar{1}2]$, deformation temperature 350 °C) operating in a secondary-slip system. The local shear stress is found to be equal to 50 ± 15 MPa. Here, the screw direction is approximately parallel to the sample surface making it possible to compare the relative velocities of 60° α- and 60° β-dislocations. In these experimental conditions there is no significant difference between the mobilities of screw and 60° β-dislocations, which are about 350 times lower than the mobilities of α-dislocations [6]. For all of the III–V compounds studied, α-dislocations move much faster than β-dislocations and screw ones, which have similar velocities.

It is noteworthy that in III–V compounds, the velocities of 60° β- and screw dislocations are proportional to their length (the length-dependent regime) for a given stress, up to lengths

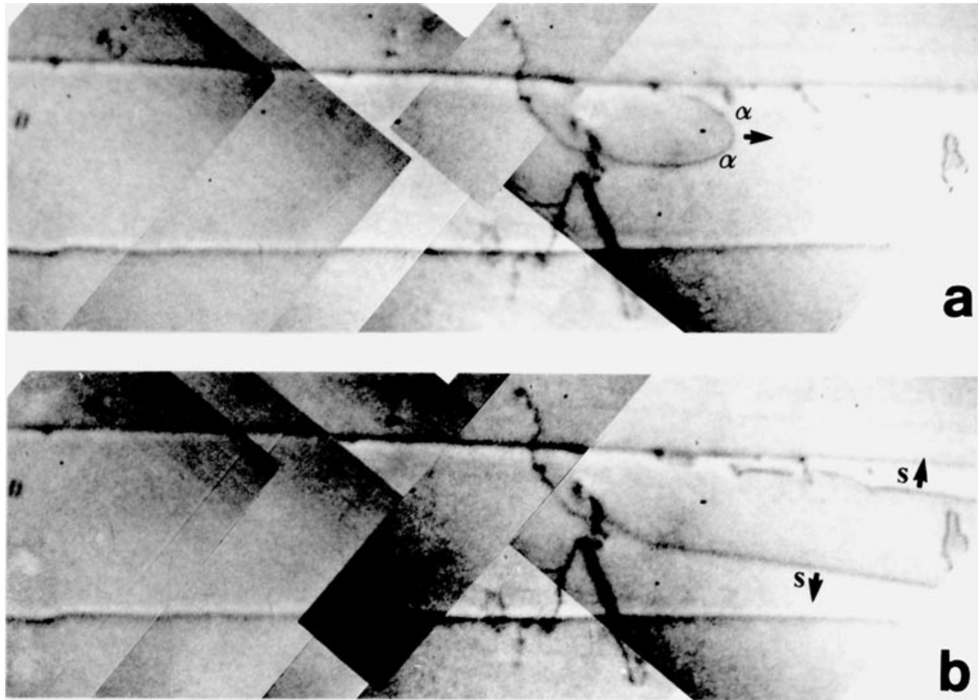

Figure 1. A dislocation source in GaAs; secondary-slip system. $T = 350$ °C; $\tau = 50 \pm 15$ MPa. (a) $t = 0$ s; (b) $t = 0.16$ s; (c) $t = 8$ s; (d) $t = 16$ s; (e) $t = 20$ s. Note the very large α/β asymmetry.

higher than a few micrometres (see table 1). On the other hand, a transition between the two velocity regimes has been evidenced in silicon and germanium.

In the framework of the Hirth and Lothe theory (the kink diffusion model), activation energies for dislocation movement can be estimated without using an Arrhenius plot. Indeed all the terms in the pre-exponential factors (equations (2) and (3)) are either known or determined experimentally. The activation energy in the length-dependent regime, $F_{kp} + W_m$, is estimated from the slope of the $v(L)$ curve. For Si and Ge, the activation energy in the length-independent regime, $F_{kp}/2 + W_m$, is determined from the 'saturation' velocity. (Note that for Si the activation energy for dislocation glide has also been directly estimated in the length-independent regime

Table 1. Activation parameters for dislocation glide as estimated from *in situ* straining experiments using a TEM. Experimental data are analysed in the framework of the kink diffusion model.

Material	Dislocation	Temperature (°C)	Stress (MPa)	X (μm)	$F_{kp}(\tau)$ (eV)	W_m (eV)	F_k (eV)
Si	60°, screw	520–615	240		1.2	1.3	0.9
		540	550	0.4	0.9	1.3	0.9
Ge	60°	430	40	0.55	0.8	0.85	0.5
	screw	405	35	0.7	0.8	0.9	0.5
InSb	Screw, 60° β	250	50	> 5	0.8–1.2	< 0.4	0.5–0.65
InP	Screw, 60° β	350	50	> 0.7	0.8–1.5	< 0.7	
GaAs	Screw, 60° β	350	50	> 3	1.1–1.6	< 0.5	0.65–0.9

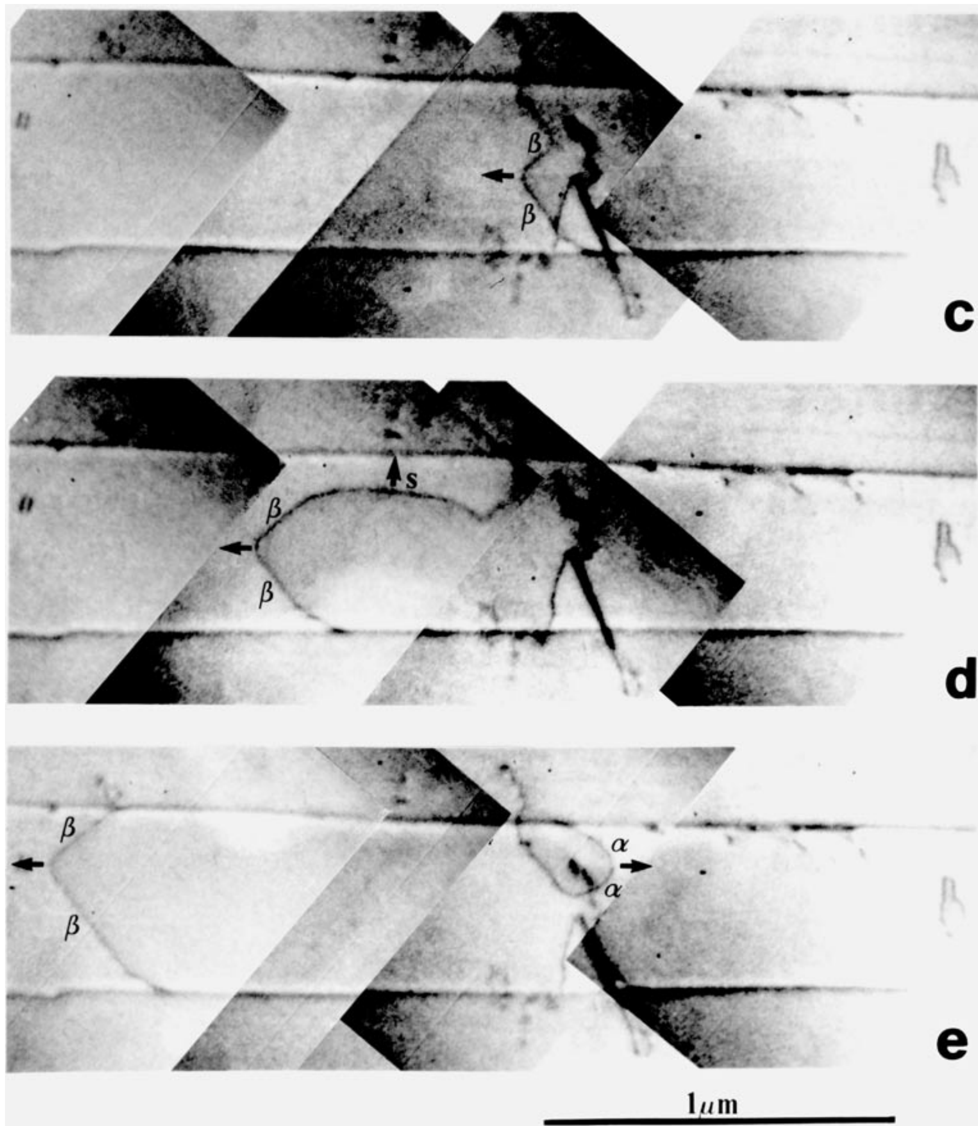

Figure 1. (Continued)

by means of an Arrhenius plot. The value obtained is in agreement with the Hirth–Lothe model since it is found to be equal to $F_{kp}/2 + W_m$.). For compounds, limiting values of $F_{kp}/2 + W_m$ are estimated by considering that the maximum observed velocity in experiments is lower than the saturation velocity. The results are summarized in table 1. Of course, for improving confidence in these estimates, confirmation should be obtained by use of an Arrhenius plot. Owing to the limited range of temperature, this has not been done for compounds.

The mean free path of a kink pair is much longer in compounds than in elemental semiconductors, since there is no evidence of a length-independent regime of dislocation velocity (see table 1). The larger value of X in compounds could be attributed to a more difficult reconstruction of atomic bonds in dislocation cores. Moreover, the analysis of the

experimental results leads to W_m-values that are much lower for compounds than for elemental semiconductors.

Again it should be pointed out that the values reported in table 1 refer to an analysis of the experimental results according to the kink diffusion model. In the authors' opinion we may have a good level of confidence in the determination of the activation energy in the length-dependent regime of dislocation velocity, which, according to theoretical models, is equal to $F_{kp} + W_m$. However, estimation of the exact values of the kink formation energy (F_k) and kink migration energy (W_m) requires further experiments to decide which mechanism—kink collisionless or with localized obstacles—controls the velocity of long dislocations.

5. Electron-beam-irradiation-enhanced dislocation glide

As evidenced by experiments performed at a 'semi-microscopic' scale, e.g. scanning electron microscopy in the cathodoluminescence mode or the double-etching technique, electronic excitation causes an enhancement of dislocation glide in a variety of semiconductors. The dislocation velocity can be expressed as [4]

$$v = v_{0d}(\tau)\exp\{-(E_d/kT)\} + \eta(\tau)I\exp\{-(E_i/kT)\} \qquad (4)$$

where E_d is the activation energy without excitation, i.e. beam intensity $(I) = 0$, and $E_i = E_d - \Delta E$ is the apparent activation energy under excitation. Above a critical temperature T_c, the first term dominates and no effect of excitation is observed. At lower temperature, the second term describes the dislocation motion. For Si [16, 17] and GaAs [18, 19], the main feature of excitation-enhanced glide does not depend on the nature of the excitation source, either electron beam or laser light.

The characterization of the microscopic processes affected by excitation requires knowledge of which condition of dislocation velocity (length-dependent or length-independent) is actually realized in the experiments performed at a semi-microscopic scale. It has been suggested [20] that the length-dependent condition is realized under experimental conditions, but this is only speculative. As described below, *in situ* deformation experiments using a TEM give further information on the behaviour of dislocations under excitation at a microscopical scale. A report on a systematic study of the REDG effect in the II–VI compound ZnS is given first; then a summary of the results obtained for a variety of semiconductors is presented.

The effect of electron beam irradiation on the dislocation behaviour in cubic (sphalerite) ZnS single crystals has been studied in the temperature range from room temperature to 230 °C [7, 15]. During *in situ* straining experiments (foil surface (011); tensile axis $[21\bar{1}]$) the dislocation motion is observed to be very smooth and viscous, regardless of the temperature and the electron beam intensity (in the range 35–5600 A m^{-2}). This strongly suggests that the dislocation glide is still controlled by the Peierls mechanism, even in the enhancement regime. Dislocation sources are frequently observed: in the example shown in figure 2 ($T = 120$ °C, $I = 5600$ A m^{-2}, local shear stress $\tau = 40 \pm 10$ MPa) the single-ended source rotates anticlockwise, the screw direction being almost parallel to the slip traces. As has been observed for III–V compounds, dislocation segments lie along the $\langle 110\rangle$ rows of the $\{111\}$ slip plane. However, dislocation half-loops have a less marked hexagonal shape than in III–V compounds; the length of the rectilinear segments is rather short ($L \leqslant 0.4$ μm). This suggests that the Peierls valleys are deeper in III–V compounds than in ZnS.

Throughout the temperature range and electron beam intensity range studied, the dislocation velocity varies linearly with its length (the maximum length measured being 0.4 μm). This suggests that no kink collision takes place before the initial kink reaches

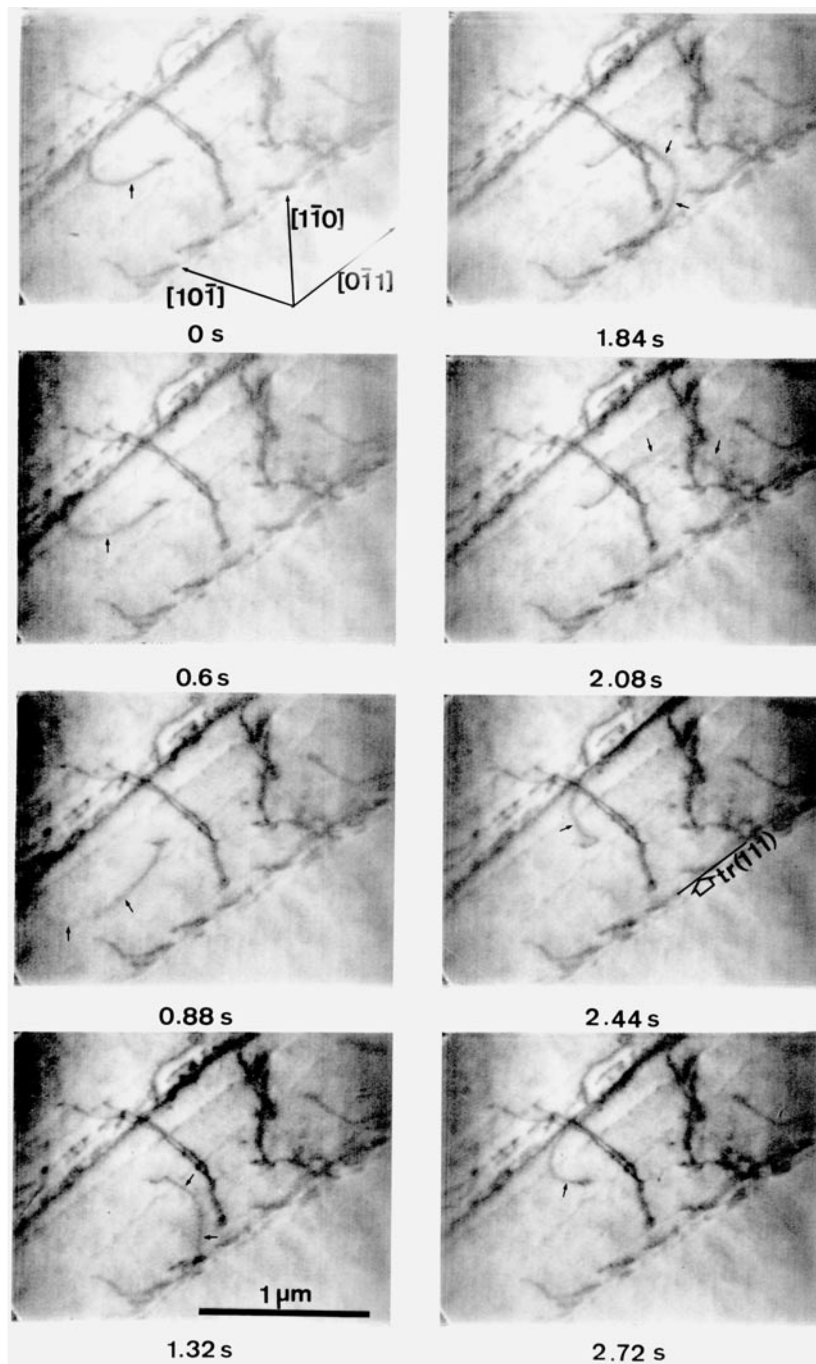

Figure 2. A dislocation source in ZnS; slip system: $(111)\frac{1}{2}[0\bar{1}1]$. $T = 120\,^\circ$C; $I = 5600$ A m^{-2}; $\tau = 40 \pm 10$ MPa.

the end of the moving segment. As indicated above, a strong enhancement of dislocation mobility under electronic excitation is evidenced. Figure 3 reports an example of the variation

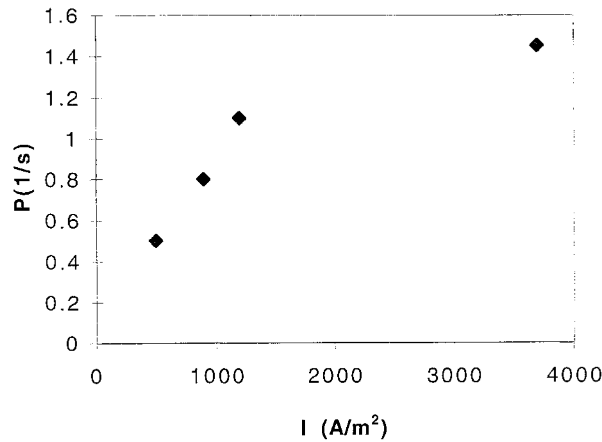

Figure 3. Screw dislocation velocity per unit length versus electron beam intensity for ZnS. Room temperature; $\tau = 25 \pm 5$ MPa.

of $P = \mathrm{d}v/\mathrm{d}L$ with I. At low intensity, P varies linearly with I, in agreement with the law deduced from experiments performed at a semi-microscopic scale (equation (4)). Note that the extrapolation of the curve to $I = 0$ gives an estimate of the dislocation mobility in the absence of electron beam excitation. As I increases, the curves $P(I)$ deviate from the linear relationship and P seems to come to a saturation level for electron beam intensities higher than about 1200 A m^{-2}.

Analysis of the experimental results to evaluate the activation energies for dislocation motion under electronic excitation (E_i) requires the use of an Arrhenius plot. Indeed, the pre-exponential factor depends on the efficiency of electronic capture on dislocation levels, which is still unknown. It is found [15] that $E_i = 0.3 \pm 0.1$ eV, to be compared with the activation energy in the absence of electronic excitation: $E_d = 1.15 \pm 0.1$ eV. (Contrary to what is observed for III–V compounds, the dislocation velocity depends only slightly on its character, α, β or screw.)

The observed excitation enhancement of dislocation glide can be interpreted in terms of the reduction of activation energy by non-radiative recombination of injected carriers at dislocations. The induced lattice vibrations contribute to the activation processes through a modification of the Peierls mechanism [4]. In the framework of the kink diffusion model, Maeda and Takeuchi [20] have analysed what changes are brought about by the elementary processes of dislocation motion, due to non-radiative capture on electronic levels associated with either straight-dislocation (SD) sites or kink (K) sites. Three cases have to be considered:

(i) Recombination enhancement assists only kink pair formation; i.e. the capture of carriers only occurs at straight-dislocation sites.
(ii) Recombination enhancement assists only kink migration; i.e. the capture of carriers only occurs at kink sites.
(iii) Recombination enhancement assists both kink pair formation and kink migration.

The results of this analysis are given in table 2.

Considering the linear dependence of the dislocation velocity on both the dislocation length and the electron beam intensity (for low intensities) leads to the conclusion that either case (i) or case (iii) is relevant for ZnS. Whichever the actual case is, the observed reduction in activation energy should correspond to assistance of kink formation, and is therefore interpreted as the

Table 2. Electron beam intensity dependence of dislocation velocities (at low injection level) and the expected reduction in activation energy for the REDG effect [20]. The dislocation motion is analysed in the framework of the kink diffusion model. SD: straight-dislocation site; K: kink site; ΔE_s: reduction in kink formation energy; ΔE_k: reduction in kink migration energy.

Glide regime	Length dependence	Site of recombination	Pre-exponential factor	Reduction in activation energy
With kink collisions	No	SD + K	I	$(1/2)(\Delta E_s + \Delta E_k)$
		SD	$I^{1/2}$	$(1/2)\Delta E_s$
		K	$I^{1/2}$	$(1/2)\Delta E_k$
Without kink collisions	L	SD + K	I	ΔE_s
		SD	I	ΔE_s
		K	No effect	No effect

difference between an electronic energy level associated with a straight portion of dislocation and a relevant band edge.

Finally the tendency to saturation of P at high electron beam intensities can be explained by the expected saturation of the recombination rate at high rates of generation of electron–hole pairs.

Few *in situ* TEM straining experiments have been performed so far to investigate the influence of electron beam irradiation on the dislocation motion in semiconductors. In GaAs [11], Ge [13] and polycrystalline Si [12, 21], an enhancement of the dislocation glide is observed and the corresponding reduction in activation energies has been estimated (see table 3). However, as the dislocation velocity regime (length dependence or not) is not established, no definite conclusion as regards the microscopic processes can be given. What is rather surprising in the case of Ge is that the estimated reduction in activation energy (1 eV) is larger than the band gap (0.67 eV); this cannot be explained by the recombination of thermalized carriers. (Note that no enhancement is evidenced during *in situ* straining experiments using a scanning electron microscope [17].) For the II–VI compound ZnSe, recent experiments performed at room temperature (200 A m^{-2} $\leqslant I \leqslant$ 8000 A m^{-2}) have revealed that the dislocation velocity under electronic excitation presents the same characteristic features as for ZnS [22]: length dependence even under excitation; linear variation with the electron beam intensity followed by a plateau (figure 4). The activation energy under electronic excitation has not been estimated due to the lack of data at different temperatures.

Table 3. Parameters describing the electron-irradiation-enhanced dislocation glide in semiconductors as estimated from *in situ* deformation experiments. E_g is the band gap.

Material (dislocation type)	E_d (eV)	E_i (eV)	ΔE (eV)	E_g (eV)	References†
Si (60°)	2.2	1.6	0.6	1.11	[21]
Ge (60°)	1.7	0.7	1.0	0.67	[13]
GaAs (α)	1.0	0.3	0.7	1.42	[4, 11]
GaAs (β, screw)	1.7	0.6	1.1	1.42	[4]
GaP (α)	1.5	0.4	1.1	2.26	[4]
InP (β)	1.6	0.7	0.9	1.35	[4]
ZnS (α, β, screw)	1.15	0.3	0.85	3.6	[15]

† [4]: *in situ* deformation observed using a scanning electron microscope; [15]: *in situ* deformation observed using a transmission electron microscope operated at 200 kV; [11]: *in situ* deformation observed using a transmission electron microscope operated at 350 kV; [13, 21]: *in situ* deformation observed using a high-voltage electron microscope operated at 1 MV.

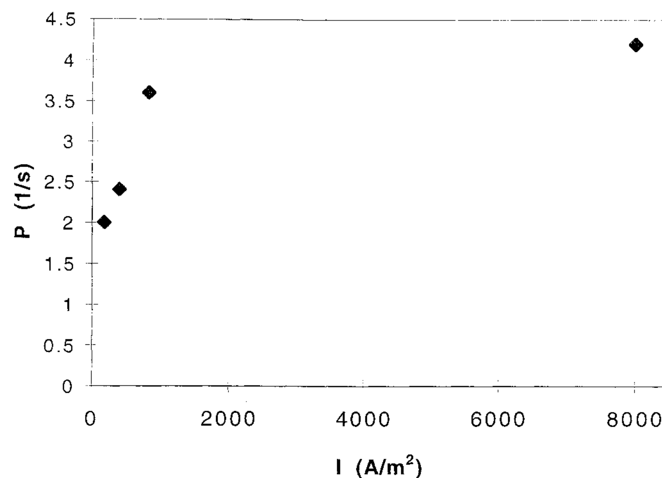

Figure 4. 60° dislocation velocity per unit length versus electron beam intensity for ZnSe. Room temperature; $\tau = 32 \pm 5$ MPa.

6. Conclusions

In situ straining experiments provide a unique way to investigate the influence of various parameters (temperature, electron beam intensity) on the mobility of dislocations in semiconductors. This technique makes it possible to estimate the physical parameters that control dislocation glide: kink formation and migration energies, mean free paths of kinks. For compounds, only limiting values could be given, because of the limited range of temperature available. Quantitative features of the electron-irradiation-enhanced dislocation glide have been surveyed. Moving dislocations experience a lattice friction which is lowered under excitation, due to a reduction in activation energy. This is in agreement with a theoretical model in which the microscopic mechanisms are assisted by non-radiative capture of carriers at electronic levels associated with dislocations.

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