JOURNAL OF MATERIALS SCIENCE 17 (1982) 301–307

Surface temperature evaluation in multi-scan electron-beam-irradiated silicon by TEM observations and optical pyrometry measurements

M. SERVIDORI, I. VECCHI
CNR, Istituto LAMEL, Via Castagnoli 1, 40126 Bologna, Italy

E. OLZI, G. TURISINI
CNR, Istituto LTM, Via Induno 10, 20092 Cinisello Balsamo, Milano, Italy

Shrinkage of prismatic dislocation loops, generated in intrinsic silicon single crystals by neon implantation and furnace annealing, was observed to take place after multi-scan electron-beam irradiation by transmission electron microscopy (TEM). The decrease in loop radius by climb motion was found to increase with increasing electron current density, until complete loop annealing and successive surface melting of samples was observed. By applying the classical theory of loop shrinkage by climb, the surface temperature of electron-beam-bombarded silicon was evaluated in the range from 1300 to 1700 K, for the particular current density values and irradiation conditions used. Optical pyrometry measurements were also made on silicon wafers irradiated in the same range of current densities; the temperature values obtained after correction for absorption by the electron gun glass window and silicon emissivity were found to be consistent with those derived from TEM observations. This result suggests that multi-scan electron-beam irradiation can be considered as a very fast annealing process.

1. Introduction

Furnace thermal annealing is the technique commonly used to remove lattice damage and to restore the electrical properties of ion-implanted silicon devices. However, new methods, such as the use of pulsed lasers and pulsed high-power electron beams, have been extensively employed during the last three years [1–6]. Both these methods involve the melting of a shallow surface layer and a very rapid crystallization of the melted region, leading to a very good crystal quality, at least from the point of view of extended defects. Annealing of implanted layers by scanning continuous-wave laser beams and, more recently, by scanning electron beams has also been reported [4, 5, 7, 8]. In this case the physical process involved is a solid-phase recrystallization of the damaged region. The reports published up to now indicate that scanned electron beams have some advantages over pulsed beams, due to the possibility of annealing both large areas and very defined regions in microelectronics devices and to the ability of controlling the annealing depth by a proper choice of beam energy.

Because of the short times involved, the determination of the heating effects of an electron beam incident on a silicon sample is generally performed by a numerical solution of the heat conduction equation in the one-dimensional case under appropriate boundary conditions [9], such as no heat dissipation from the lower and the upper surface (i.e. sample on a thermally insulated substrate with negligible surface radiation), or back surface of the irradiated specimen having good thermal contact with a substrate kept at constant temperature and the absence of a lateral tempera-

0022–2461/82/010301–07$02.70/0

ture gradient. But, when the experimental set-up used for electron-beam irradiations does not allow for these boundary conditions, as in the case of non-adiabatic specimens and of electron guns generating two-dimensional thermal gradients (along surface and depth) in the target, the results obtained by computer calculation may be a long way from simulating the actual experimental situation.

The aim of this work is to show how, in these cases, it is sometimes possible to obtain information on the heating effects produced by electron-beam irradiation. In particular, the temperature rise, as a function of the current density of an elliptically scanned electron beam, has been evaluated by applying the classical theory of climb motion to the shrinkage of prismatic dislocation loops observed by TEM as an indirect method, and optical pyrometry as a direct method.

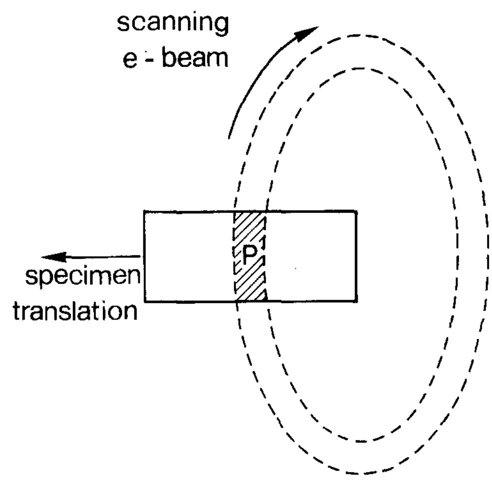

Figure 1 Sketch of the experimental set-up used for the electron-beam irradiation. P represents the region where the pyrometric measurements were made.

2. Experimental procedure

(100) silicon wafers, floating zone (FZ) grown, about 1000 Ω cm, p-type (B-doped) were used in our experiments. The dislocation loops were formed by 950° C, 30 min furnace annealing in an N_2 atmosphere of wafers previously implanted with a dose of 2.5×10^{15} Ne ions cm^{-2} at 20 KeV energy. After several TEM examinations, made to check the uniformity of both size and density of the loops, the wafers were subjected to multi-scanning electron-beam irraditations in an electron-beam melting furnace (E.S. 1/3/20, Leybold Heraeus). The beam, 20 KeV monochromatic energy and 0.7 cm diameter, was electrostatically scanned at a frequency of 50 Hz along an elliptical pattern, with major and minor axes of 9 cm and 5 cm, respectively. The wafers, 5 cm x 2 cm and 300 μm thick, spring-clamped on to a water-cooled specimen holder (poor thermal contact), were mechanically moved at a speed of 1 cm sec^{-1} at right angles through the major axis of the elliptical beam (Fig. 1). Our experimental set-up is substantially different from the others reported up to now (see, for example, [8]), where either a quick x–y electron-beam raster was used to irradiate a frame of a few mm^2 several times for about 5×10^{-3} sec, or slower, singly interlaced scans were employed to obtain selective annealing. In our case in fact, the fast electron sweep of the beam and the mechanical movement of the carriage generate a hot front running across the whole area of the wafers, thus producing a two-dimensional thermal gradient.

The specimens containing the dislocation loops were irradiated at different current densities (one wafer at one value of current density) in the range 0.26 to 1.04 A cm^{-2}. After this treatment, 3 mm diameter discs for TEM were ultrasonically cut in the same position for all the samples, i.e. along a line halfway through their long side, to avoid difficulties due to a possible inhomogeneous heating.

The temperature values were taken on another set of silicon wafers by a disappearing filament, optical pyrometer at a wavelength of 0.65 μm. Due to the rather high speed of the carriage, the data were obtained by successive approximations until a maximum spread of 5° C was measured for each value of current density. The temperature values were then corrected to account for the optical absorption (7%) of the lead glass window through which the measurements were made and for the silicon emissivity, as will be discussed later.

3. Results

3.1. Shrinkage of prismatic dislocation loops by climb

The study of the annealing kinetics of secondary defects, like dislocation loops, has been successfully applied to self-diffusion studies in metals [10, 11] and in silicon [12]. In particular, Sanders and Dobson [12] have carried out a detailed analysis of self-diffusion in silicon over a wide range of doping concentrations and have derived a value for the self-diffusion coefficient in intrinsic material, which is in good agreement with those obtained by other authors [13–16] in different ranges of temperature and by different experimental techniques.

To overcome the difficulty due to the presence of a high content of impurities in silicon, we started from a high-resistivity material and generated dislocation loops by neon implantation and subsequent thermal annealing, so as to utilize the values of the intrinsic self-diffusion coefficients available from literature, and then correctly apply the equations describing the loop shrinkage by absorption of point defects from the specimen surface.

Whenever self-diffusion studies are performed by this technique, successive TEM observations are generally made on the same foil by following the decrease in diameter of the same dislocation loops after a few heat treatments, so that the climb process is not appreciably influenced by chemical stresses resulting from point defect supersaturation [17]. These in fact can be neglected in a thin foil, where the two surfaces act as very effective sources or sinks of point defects.

In our case, due to the impossibility of irradiating the specimens already thinned for TEM observations, we tried to solve the problem related to the effects of chemical stresses, normally present in bulk materials, by generating a thin layer of dislocation loops very near to the wafer surface by low-energy ion implantation.

In fact, as discussed in detail by Silcox and Whelan [17], when the effects due to local supersaturation of point defects are expected to be important, as in the case of bulk materials, the large loops grow at the expense of the small ones, which therefore disappear, giving rise to a lower concentration of larger loops. While the final state produced by annealing bulk material for a sufficiently long time should be the same as that of the annealed thin foils (i.e. no loops), the detailed behaviour of loops, as they anneal out, will be different and will depend markedly on their proximity to point defect sources or sinks. Therefore, loops very near to the surface are expected to shrink in the same way as in a thin foil.

Our TEM observations have shown that chemical stresses could indeed be neglected, so that our attention was confined to larger loops for convenience.

The prismatic loops studied in this investigation have been found to be extrinsic in character, so that they shrink either by diffusion of interstitial atoms from the loop to the surface or by diffusion of vacancies from the surface to the loop, the nature of the diffusing species being governed by the self-diffusion mechanism. This mechanism in silicon is still not well understood, despite several studies of the subject. However, a simple monovacancy mechanism is generally assumed [18–21], even if a divacancy mechanism is also considered as being very probable [22].

If vacancies are the mobile species, then the rate of shrinkage of a prismatic loop of radius r is given by

$$\frac{\mathrm{d}r}{\mathrm{d}t} = -\frac{\pi D_\mathrm{s}}{f_\mathrm{v} b \ln (L/b)}\left(1 - \frac{C_\mathrm{L}}{C_0}\right), \qquad (1)$$

where

$$D_\mathrm{s} = D_{0\mathrm{s}} \exp(-E/kT) \qquad (2)$$

is the coefficient of self-diffusion in intrinsic material, f_v is the correlation factor (equal to 0.5 for monovacancy diffusion in the diamond cubic lattice [23]), b is the modulus of the Burgers vector of the loop, L is the distance of the loop from the surface, C_L is the vacancy concentration in equilibrium with the loop and C_0 is the equilibrium vacancy concentration.

Following the procedures of Silcox and Whelan [17] and Sanders and Dobson [12] and integrating Equation 1, we obtain the expression

$$(r_0^2 - r^2) + 2\alpha b(r_0 - r) = \frac{2\pi\alpha D_\mathrm{s}}{f_\mathrm{v} \ln (L/b)} t, \qquad (3)$$

where r_0 is the initial radius of the loop, r is the loop radius at time t, α is a temperature-dependent parameter, which for silicon we have calculated as $8 \times 10^5\, T^{-1.27}$, and t is the time during which shrinkage occurs.

From TEM observations the mean values of r_0 ($5.15 \pm 0.55 \times 10^{-6}$ cm), r and L ($\simeq 6 \times 10^{-6}$ cm) have been determined, together with the value of b, which for perfect dislocations in silicon is 3.84×10^{-8} cm.

Fig. 2 and Table I show the decrease of loop radius with increasing current density, I, of the electron beam. It can be observed that r remains constant up to $0.52\,\mathrm{A\,cm^{-2}}$, after which it experiences an abrupt shrinkage and at $0.91\,\mathrm{A\,cm^{-2}}$ no loop has been detected by TEM. It is important to point out, as will be seen later on, that in correspondence with the current density value of $1.04\,\mathrm{A\,cm^{-2}}$, incipient surface melting of the wafers was observed during irradiation.

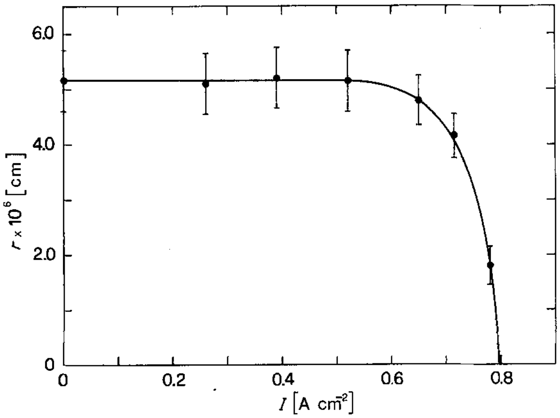

Figure 2 Decrease in the loop radius r as a function of the beam current density.

The decrease of the loop radius shown in Fig. 2 was found to be well represented by an expression of the type

$$r_0^2 - r^2 = A \exp(-B/I), \qquad (4)$$

where $A = 3.08 \times 10^{-7}$ cm^2 and $B = 7.41$ cm^2 A^{-1}. Assuming that Equation 4 is valid over all the range of current densities, it is possible to obtain, for each value of I, the value of $(r_0^2 - r^2)$ and then of $(r_0 - r)$.

Fig. 3 and Table II show the result of this fitting procedure in the range 0.52 A cm^{-2}, at which the loops start to decrease, to 1.04 A cm^{-2}, where incipient surface melting was observed. The dashed segment between 0.78 and 1.04 A cm^{-2} represents extrapolated negative values of r.

At this point, the solution of Equation 3, that is, the evaluation of the temperature T (present in Equation 2 and in the term α) as a function of the current density I, requires the determination of t. This can be done by taking the value of $I = 1.04$ A cm^{-2}, which gave rise to incipient surface melting, as a calibration point. Since the melting temperature of silicon is known ($T_m = 1683$ K), from the extrapolated values of

TABLE I Values of the loop radius observed after irradiation at different current densities

I (A cm^{-2})	$r \times 10^6$ (cm)
0.260	5.10 ± 55
0.390	5.20 ± 55
0.520	5.15 ± 55
0.650	4.80 ± 45
0.715	4.15 ± 42
0.780	1.80 ± 35
0.910	–
1.040	melting

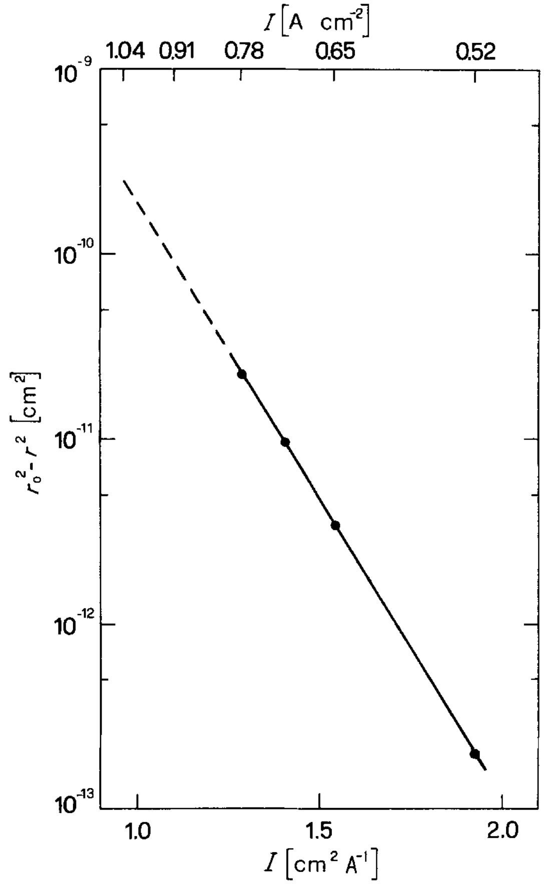

Figure 3 Calculated fit of the curve of Fig. 2. The broken segment of the straight line represents extrapolated values of negative r.

$(r_0^2 - r^2)$ and $(r_0 - r)$ it is possible to deduce the value of t, and, in the hypothesis that the electron-beam irradiations are isochronal heat treatments, the function T against I can finally be obtained.

As has already been pointed out, several authors [12–16] have experimentally determined the intrinsic self-diffusion coefficient of silicon in different ranges of temperature (Fig. 4) and a certain spread in the values of both the pre-exponential factor D_{0s} and the activation energy

TABLE II Values of $(r_0^2 - r^2)$ and $(r_0 - r)$ obtained from Equation 4 (see text)

I (A cm^{-2})	$r_0^2 - r^2$ (cm^2)	$r_0 - r$ (cm)
0.520	1.97×10^{-13}	1.92×10^{-8}
0.650	3.42×10^{-12}	3.43×10^{-7}
0.715	9.66×10^{-12}	1.04×10^{-6}
0.780	2.28×10^{-11}	3.22×10^{-6}
0.910	8.86×10^{-11}	1.30×10^{-5}
1.040	2.47×10^{-10}	2.00×10^{-5}

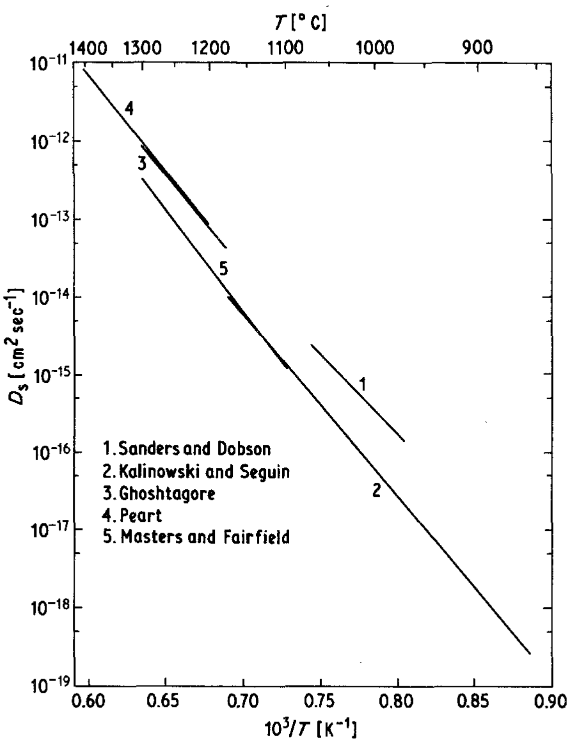

Figure 4 Temperature dependence of the self-diffusion coefficients of intrinsic silicon.

E results from their original papers. This spread will of course affect the value of t and consequently the trend of T against I. The complete outline is summarized in Table III, where a mean deviation in T of about 30 K between 1300 and 1700 K is evident in the range of current densities we have considered.

It should be emphasized that the temperature data of Table III have been obtained by extrapolating some of the self-diffusion coefficients at values of temperature different from those at which they have been determined. This can be done only in the case of no curvature of the Arrhenius plot of Fig. 4, as the experimental work by Kalinowski and Seguin [16] and the theoretical considerations by Bourgoin and Lannoo [21] seem to demonstrate.

3.2. Optical pyrometry measurements

Since, unlike the dislocation-loop annealing technique, optical pyrometry is routinely used for temperature determinations, here we only recall some of its fundamental aspects. As has already been pointed out, the pyrometric measurements were made on the wafers (Region P of Fig. 1), travelling under the scanning electron beam at a speed of 1 cm sec^{-1}, at the wavelength of 0.65 μm. The temperature data were corrected for the silicon emissivity and after that the absorption of the lead glass window of the electron gun was taken into account (7%). Due to the relatively low temperatures involved, it was possible to use, instead of the Planck equation, a very good approximation represented by the Wien expression

$$\frac{1}{T} - \frac{1}{T_p} = \frac{\lambda}{C_2} \ln \epsilon_\lambda, \qquad (5)$$

where T is the true temperature, T_p is the pyrometric temperature, λ is the wavelength at which the measurements are made, C_2 is the second radiation constant (= 1.438 cm degree) and ϵ_λ is the emissivity of the target.

Unfortunately, only extremely limited studies on the silicon emissivity at high temperature have been made [24] and these cannot be considered to be highly accurate due to difficulties of exper-

TABLE III Rise of the surface temperature of silicon with increasing current density deduced by inserting in Equation 3 the values of D_{0s} and E available from the literature. The t values corresponding to the different D_s are also reported

	Sanders, Dobson	Kalinowski, Seguin	Ghoshtagore	Peart	Masters, Fairfield
D_{0s} (cm^{-2} sec^{-1})	5.8	154	1200	1800	9000
E (eV)	4.1	4.65	4.73	4.77	5.13
t (sec)	0.70	1.17	0.26	0.23	0.55
I (A cm^{-2})	T (K)	T (K)	T (K)	T (K)	T (K)
0.520	1339	1373	1377	1379	1397
0.650	1459	1483	1486	1488	1500
0.715	1510	1529	1531	1533	1542
0.780	1559	1573	1574	1576	1583
0.910	1634	1640	1640	1641	1644
1.040	1683	1683	1683	1683	1683

TABLE IV Surface temperatures of silicon at increasing current densities, obtained by optical pyrometry. T are the values of T_p after correction for silicon emissivity

I (A cm^{-2})	T_p (K)	T (K)
0.520	1306	1362
0.650	1388	1451
0.715	1438	1506
0.780	1484	1556
0.910	1541	1619
1.040	1601	1685

imental nature. Therefore, according to other authors [8, 25], a value of 0.5 has been used for ϵ_λ which, on the other hand, is in agreement with the mean value which can be deduced from the results given by Allen [24]. The corrected temperature data, obtained for current densities of the electron beam between 0.52 and 1.04 A cm^{-2}, are reported in Table IV. The accuracy of these values is mainly related to the uncertainty in the silicon emissivity: it is significant, however, that Equation 5 shows a logarithmic dependence on ϵ_λ. So, an error of about ± 15% in $\epsilon_{0.65}$ (reasonable in the case of silicon) results in a corresponding error of about ± 15 K in the range 1300 to 1700 K. This mean deviation is comparable with that derived from the uncertainty in the silicon self-diffusion coefficient.

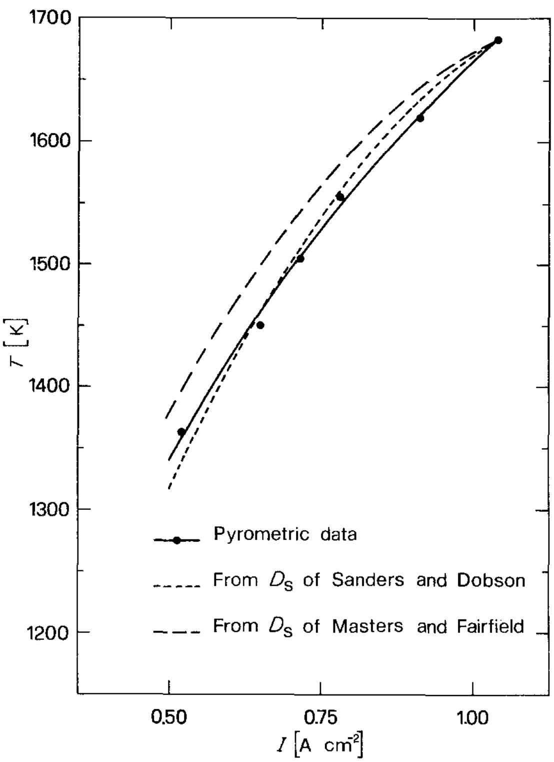

Figure 5 Surface temperature of silicon as a function of the current density of the incident electron beam.

4. Discussion and conclusions

The results reported in this paper suggest that it is possible to obtain information on heating effects produced by electron-beam irradiation using experimental techniques of investigation.

The temperature values as a function of the current density of the beam, obtained by applying the climb equations to the loop shrinkage and by optical pyrometry, are in fact in good agreement, as shown in Fig. 5 where only the curves relative to the most different D_s have been reported for clarity. This result suggests that electron-beam irradiation can be considered as a very fast furnace annealing process, since heat treatment at 1300° C for 0.26 sec gives rise to the same loop shrinkage as that produced by furnace annealing at 1000° C for 15 min.

Since the purpose of this paper was to evaluate the surface temperature of irradiated silicon, no particular attention was given to the t values resulting from TEM observations. Anyway, on account of the experimental conditions of irradiation, the range 0.2 to 1.2 sec seems to be reasonable and we do not feel able to indicate one value as being more reliable than another.

In addition, it is to be pointed out that the curves of Fig. 5 are valid only in the case of the particular conditions we used for our experiments (diameter, frequency and pattern of scanning of the electron beam; thermal contact between sample and holder; speed of mechanical movement). Somewhat different shapes in the plot T against I must in fact be expected for different irradiation conditions, even when the same electron gun is used.

Finally, as our gun was not specifically planned for the kind of application presented here, the results we have obtained can be considered as very satisfactory.

Acknowledgements

The authors wish to thank F. Cembali, E. Gabilli and R. Lotti for wafer implantations and technical assistance.

References

1. R. RIMINI (ED) "Laser Effects in Ion-Implanted Semiconductors" Proceedings of Workshop, Catania, Italy (1978).

2. R. BAKISH (ED) "Electron and Ion-Beam Science and Technology" (Electrochemical Society, New York, 1978).
3. S. D. FERRIS, H. J. LEAMY and J. M. POATE (EDS) "Laser-Solid Interactions and Laser Processing, 1978" (American Institute of Physics, New York, 1979).
4. C. W. WHITE and P. S. PERCY (EDS) "Laser and Electron Beam Processing of Materials" (Academic Press, New York, 1980).
5. J. F. GIBBONS, L. D. HESS and T. W. SIGMON (EDS) "Laser and Electron Beam Solid Interactions and Material Processing" Proceedings MRS, Boston (1980), in press.
6. A. R. KIRKPATRICK, J. A. MINNUCCI, A. C. GREENWALD and R. G. LITTLE, Proceedings of the 1st Conference on Ion-Beam Modification of Materials, Budapest, edited by J. Gyulai, T. Lohner, E. Pàsztor (Central Research Institute for Physics, Budapest, 1978) p. 629.
7. G. G. BENTINI, R. GALLONI and R. NIPOTI, *Appl. Phys. Lett.* **36** (1980) 661.
8. R. A. McMAHON, H. AHMED, R. A. DOBSON and J. D. SPEIGHT, *Elect. Lett.* **16** (1980) 295.
9. P. G. MERLI, *Optik* **56** (1980) 205.
10. P. S. DOBSON, P. J. GOODHEW and R. E. SMALLMAN, *Phil. Mag.* **16** (1967) 9.
11. T. E. VOLIN and R. W. BALLUFFI, *Phys. Stat. Solidi* **25** (1968) 163.
12. I. R. SANDERS and P. S. DOBSON, *J. Mater. Sci.* **9** (1974) 1987.
13. R. F. PEART, *Phys. Stat. Solidi* **15** (1966) K119.
14. R. N. GHOSHTAGORE, *Phys. Rev. Lett.* **16** (1966) 890.
15. B. J. MASTERS and J. M. FAIRFIELD, *Appl. Phys. Lett.* 8 (1966) 280.
16. L. KALINOWSKI and R. SEGUIN, *ibid.* **35** (1979) 211.
17. J. SILCOX and M. J. WHELAN, *Phil. Mag.* **5** (1960) 1.
18. D. SHAW, *Phys. Stat. Solidi (b)* **72** (1975) 11.
19. J. C. BOURGOIN and J. W. CORBETT, Centre d'Etudes Nucléaires de Saclay, 19th Colloque de Métallurgie (1976) Vol. 11, p. 695.
20. J. A. VAN VECHTEN, *Phys. Rev. B* **10** (1974) 1482.
21. J. C. BOURGOIN and M. LANNOO, *Rad. Effects* **46** (1980) 157.
22. H. C. CASEY and G. I. PEARSON, in "Diffusion in Semiconductors" edited by J. H. Crawford and I. M. Slifkin (Plenum, New York, 1975) p. 181.
23. D. L. KENDALL and D. B. DE VRIES, in "Semiconductor Silicon 1969" edited by R. R. Haberecht and E. L. Kern (The Electrochemical Soceity Inc., New York, 1969) p. 358.
24. F. H. ALLEN, *J. Appl. Phys.* **28** (1957) 1510.
25. K. NISHIYAMA, M. ARAI and N. WATANABE, *Jap. J. Appl. Phys.* **19** (1980) L563.

Received 3 June
and accepted 29 June 1981