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RHEED and REM study of Si(111) surface degradation under Ar bombardment

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Résumé. — L'effet d'un bombardement d'ions argon de 10 à 40 keV sur des surfaces Si(111) à température ambiante est étudié par RHEED et REM en utilisant un microscope électronique. Les mesures d'intensités RHEED et les images obtenues en REM montrent que le désordre dans les premières couches atomiques est d'autant plus faible que l'énergie des ions incidents est élevée. La décroissance de l'intensité de l'onde électronique « spéculaire » en cours de bombardement peut être décrite en utilisant un modèle simple dans lequel le paramètre dépendant de l'énergie est une « aire moyenne endommagée » par ion frappant la surface. De 10 à 40 keV, ce paramètre varie de $4,7 \times 10^{-14}$ à $1,4 \times 10^{-14}$ cm² alors que la dose nécessaire à la disparition complète des tâches de diffraction, D_c , varie de 5×10^{13} à $1,8 \times 10^{14}$ ion/cm². Le calcul de l'énergie déposée, en collisions nucléaires, au voisinage de la surface, montre que celle-ci ne varie que de 15 % dans la gamme d'énergie étudiée et ne peut donc pas rendre compte des fortes variations de D_c . Une estimation grossière de la concentration d'argon dans les premières couches atomiques, en fonction de l'énergie montre une meilleure concordance et suggère que la décroissance de l'intensité RHEED est liée à l'augmentation des distorsions du réseau cristallin au voisinage de la surface, dues à l'incorporation de l'argon.

Abstract. — The effect of 10 to 40 keV argon ion bombardment on Si(111) surfaces, at room temperature, has been investigated by RHEED and REM imaging using an electron microscope. Both RHEED intensity measurements and REM images show that the disorder of the first top-layers proceeds more slowly when increasing ion energy. The decrease of the specularly reflected beam intensity for increasing fluence can be fitted using a simple model in which the energy-dependent parameter is a « mean damaged area » per incident ion incoming on the surface. From 10 to 40 keV, this parameter varies from 4.7×10^{-14} to 1.4×10^{-14} cm² while the dose needed for the complete disordering of the surface, D_c , varies from 5×10^{13} to 1.8×10^{14} ion/cm². Damage energy depositions on the near-surface region were calculated and did not show a variation of more than 15 % over the whole energy range. This variation cannot account for the more than 3-time variation of D_c . A rough estimate of argon concentration in the first layers with increasing energy shows better agreement, and suggests that the RHEED intensity decrease is related to increasing distortions of the surface network due to argon entrapment.

1. Introduction.

Since argon ion bombardment is one of the most standard techniques for preparing clean, well-ordered single crystal surfaces of semiconductors, a variety of studies of the damage process for low ion energy on Si and Ge surfaces have been previously reported [1, 2]. The change which occurs in the near-surface region when energetic ions (10 to 40 keV) penetrate the crystal has been less studied.

Reflection High Energy Electron Diffraction (RHEED) provides a powerful experimental tool for investigating ion implantation effects on solid surfaces. Since RHEED is very sensitive to the structural arrangement of atoms in a few atomic top-layers, any disturbance from the perfectly flat surface can be observed in the RHEED response.

On the other hand, because of the low penetration

depth of even 100 keV electron beam at glancing angle on perfectly plane surfaces [3], RHEED intensities do not sense the effect of ion bombardment under these few layers.

Here we report the results of the implantation 10-40 keV argon ions on very flat Si(111) surfaces, at room temperature, under different ion energies. The particularity of this experimental study consists in the use of an electron microscope for RHEED experiment during *in situ* bombardment of the sample. This enables us to image the surface before and under irradiation using the Reflection Electron Microscopy (REM) technique described elsewhere [4-6], and to ensure by use of micro-diffraction patterns that the surface under investigation is really flat and clean, with a low step density.

In this paper we give results concerning the degradation of RHEED intensities *versus* ion beam

energy and its relation to surface disordering induced by implantation. First REM images from the surface before and during bombardment will be presented and briefly discussed. An attempt will be made to compare these results with theoretical calculations, damage energy deposition and projected range for Ar, based on LSS theory [14, 20].

2. Experimental details.

2.1 CONNECTION ION IMPLANTER-MICROSCOPE. — The electron microscope used for these experiments is a Phillips EM-300 with a 100 keV accelerating voltage. The specimen is set on a modified double tilt specimen holder able to heat the Si surface up to its melting point by feeding it with DC current [7]. The illuminating beam is bent towards the surface using the beam-tilt system with an incident angle of about 1 degree, so that a RHEED pattern can be obtained in the back focal plane of the objective lens.

The ion-implanter includes a universal ion source (Bernas Nier type) with a maximum accelerating voltage of 60 keV [8]. This source is associated with a magnetic sector so that isotopes up to 200 amu can be easily separated according to the ratio q/m . Then the ion beam scans the specimen surface in order to implant uniform ion doses. The implanter and the microscope are connected with the ion beam axis at 25° off the normal surface and far from a low index crystallographic axis. In this case, we assume that the target is seen as « amorphous » by the ion beam and theoretical calculations will be made under this assumption.

This connection allows us to implant almost any kind of ions on Si surfaces during REM and RHEED experiments.

The vacuum in the implanter is about

2×10^{-6} torr and less than 5×10^{-5} torr in the specimen chamber, taking into account a liquid nitrogen anticontamination device.

2.2 Si(111) SURFACE PREPARATION. — P-type silicon single crystals wafers, (2-4 ohms $\times$ cm) oriented nearly parallel to 111 planes (misorientation less than 1°), mirror-like polished, were used for these experiments. After vacuum cleaning and deoxidizing, gold was deposited onto the wafers then annealed for 20 min at 500 $^\circ$ C under a 5 % H_2 and 95 % N_2 atmosphere to produce ohmic contacts. Wafers were finally cut into samples ($2.7 \times 0.5 \times 0.3$ mm³) and then set on our specimen holder which is described elsewhere [7].

The experimental cleaning process we performed for cleaning and flattening the sample is also described elsewhere [6]. By *in situ* heating the sample near its melting point by Joule effect, oxides and carbides epitaxially grown can be removed or sublimated. One obtains a flat Si(111) surface with a low step density (~ 1 step/micrometer), as can be directly measured by REM imaging of the surface (see first micrograph, Fig. 1a).

2.3 RHEED RECORDING AND REM IMAGING OF Si SURFACES. — When a flat and clean Si(111) surface is obtained, the incident electron beam and the surface are oriented so that the 444 specularly reflected beam is fully excited for the $\langle 112 \rangle$ azimuth. Then, by use of the beam-tilt system, this diffracted beam is aligned on the optical axis of the microscope column. RHEED patterns can then be recorded by the standard camera equipment for spot profile analysing, while integrated intensity can be measured by means of a special Faraday cage set near the phosphorescent screen. For these measurements, diffuse scattering was subtracted from the total reflected intensity by recording the current out of

Fig. 1 — a) (444) REM image from Si (111) surface. Arrows indicate the sense of the steps with respect to incident beam direction. Note the reverse of characteristic contrasts (black and white fringes) due to the reverse of sense of the steps. b) corresponding RHEED pattern.

the spot, under the same « exchange angle », since we assume that only the scattered intensity is related to the ordering of the surface.

As it is usual in TEM, a dark field image can be obtained by using the specularly reflected beam. This image is shown in figure 1a : the (111) planes of Si are imaged using the 444 reflection at an azimuth close to the $\langle 112 \rangle$ direction, as can be seen in the corresponding RHEED pattern from figure 1b. Then the image, which exactly satisfies the Bragg condition, is foreshortened in the beam direction by a factor of about 40, due to the projection of the surface. The wavy lines are atomic steps on the surface whose image contrast is mainly produced by Fresnel contrast and is seen as dark and white fringes. Contrast rules [3] allow the sense of the steps to be determined as indicated by arrows in figure 1a. In spite of a more « classical » vacuum, this image is similar to those obtained by Yagi *et al.* [3, 4] under UHV conditions, although a thick amorphous layer of contamination must cover the flat surface and is responsible for the 1×1 structure [9].

A micro-diffraction pattern, from a selected surface area, can then be recorded to ensure that the RHEED degradation characteristics under ion bombardment are really those of a perfectly flat Si(111) surface. All RHEED intensity measurements were done under this precaution.

2.4 IMPLANTATION PROCEDURE AND CHARACTERISTICS. — Ion dose measurements are made with two Faraday cages, the first one, before scanning the surface, gives the total ion beam intensity, and the second one, just behind the sample, measures the ion flux on the surface. Both measurements were carried out before and after experiments to test flux stability.

For each intensity measurement, the ion beam is stopped at the dose required by an electric window and the electronic current is measured after a few seconds when the sample is stabilized. After each measurement the electron beam is deviated from the surface, since we noticed that the degradation of the reflected beam, due to the contamination of the surface, is negligibly small under this precaution.

Usually the ion beam current density is about $0.4 \mu\text{A}/\text{cm}^2$ or 2.5×10^{12} ions/ $\text{cm}^2 \times \text{s}$, which is 1 ion for 300 target surface atoms per second, considering a single 111 plane. Since, in our case, the beam power dissipated in the sample is in the 4-16 mW range, according to the surface temperature measurements from Te-Chang [10] on Si under 75 mW Ar irradiation, the surface temperature is not noticeably enhanced. All the experiments were done at room temperature.

On the other hand, according to Sielanko [11], the sputtering yield for Ar on Si in the 10 to 40 keV energy range does not change significantly and is estimated both from experimental measurements and theoretical calculations to be about 1.5 at/ion incoming on the surface. Now considering our data, a final dose of about 10^{14} ion/ cm^2 , one can roughly

estimate that less than one monolayer is sputtered during the total experiment.

3. Results and discussion.

3.1 REM IMAGES. — Figure 2 depicts the Si(111) surface topography before (a), under (b), and after (c) bombardment with a 10 keV ion beam. The influence of Ar bombardment on the crystal structure is shown through these 3 images. The terrace intensity is reduced, as the fluence increases, by the same ratio as the specular reflected beam while a granulation of the image becomes visible. This granulation is not yet completely understood, although it was suggested by Uchida *et al.* [12] that it could be related to the presence of ion etch-pits in the case of 2 keV Ar bombardment on Au(111) surfaces.

Fig. 2. — a) REM image before bombardment. b) During 10 keV Ar^+ bombardment. Note the increasing granulation from the terraces. Steps contrast remains constant. c) After bombardment with a fluence of about 5×10^{13} ion/ cm^2 . Steps are no more visible while the granulation is really high.

First micro-densitometric measurements of the step contrast evolution on increasing fluence showed that this contrast remains constant until a critical dose is reached, at which the steps disappear rapidly. For any energy, this dose is always slightly lower than that required for the complete extinction of the

specularly reflected beam. Since, according to Cowley [13], the step contrast is mainly due to interference phenomena between electronic waves leaving the surface from the top and the bottom of the 2 terraces, the vanishing step contrast might be related to the disordering of the first (111) plane.

Anyway, at the dose needed for the complete disappearance of the reflected diffracted beam, the step structure completely disappears and the granulation is really high (see Fig. 2c). We did not notice any faceting of the surface nor any special influence due to the presence of atomic steps.

3.2 RHEED PATTERN EVOLUTION. — A typical set of RHEED patterns is shown in figure 3 for increasing fluence of a 10 keV Ar ion beam. By use of the selected area technique, a good pattern, with low background intensity and only one strong excited reflected beam, can be obtained if the « coherence width » is reasonably large. This can be easily achieved by defocussing the condenser lenses of the microscope.

Fig. 3. — RHEED pattern evolution during 10 keV Ar⁺ bombardment. Electron energy = 100 keV ; azimuth close to $\langle 112 \rangle$.

With increasing ion fluence, this beam tends to disappear as the disorder of the surface increases during bombardment. From micro-densitometric measurements of the spot profile, no variation of the half-width of the Bragg peak could be clearly distinguished within the experimental resolution, which is high. For doses as high as 10 times the dose needed for the complete disappearance of the spot, no extra reflection appears by rotating the sample. Thus, we conclude that there is no faceting of the surface under irradiation.

3.3 DEGRADATION CHARACTERISTICS. — In the degradation experiments, the flat and ordered Si(111) surface was subjected to increasing doses and the effect was noted in the RHEED specular intensity measurements. The RHEED intensity from the perfect surface (prebombardment state)

was first recorded, then following implantations were carried out, recording the intensity reflected by the surface under irradiation, at a given dose ($I = f(D)$), showing degradation characteristics. A typical $I/I_0 = f(D)$ curve is plotted in figure 4 for a 10 keV argon bombardment on Si(111) for increasing doses. A highly ordered surface corresponds to $I/I_0 = 1$, while a heavily disordered (near amorphous?) surface gives $I/I_0 = 0$. Since it is difficult to estimate the dose corresponding to $I/I_0 = 0$, we define as the critical dose the dose for which $\ln(I/I_0) = -5$, taking background intensity correction into account.

Fig. 4. — Degradation of the specularly reflected beam (444) under 10 keV bombardment versus the number of Ar ions/cm² bombarding the surface. Background intensity correction was made.

The surface disordering caused by the impact of an ion of energy E can be estimated using the simple model of Jacobson *et al.* [2] and the degradation characteristics from figure 4.

Assuming that each ion incoming on the surface disorder, as seen by RHEED, a mean surface area $\bar{A}$, and taking a damage overlap into account, if T is the ratio of the surface area damaged at a given damage state, then

$$dT = (1 - T) \times \bar{A} \times dD.$$

Since RHEED intensity, under Bragg condition, is related to T , assuming that the reflected wave amplitude is proportional to the undamaged area, we have

$$I/I_0 = \exp(-2\bar{A}D)$$

where I is the diffracted reflected intensity at the given dose D .

According to this equation, a plot of $\ln(I/I_0)$ versus D should give a straight line with a slope equal to $-2\bar{A}$.

— We must notice that the mean damaged area $\bar{A}$ not only includes the heavily disordered region near the impact point, but also the region where

Fig. 5. — Dependence of the degradation characteristics upon bombardment energy. Straight lines were drawn after averaging all experiments, and represents the theoretical curve $I/I_0 = \exp(-2AD)$.

lattice distortions are noticeable as may be the case when argon ions are entrapped near the surface layer.

A set of $\ln(I/I_0) = f(D)$ versus energy is shown in figure 5, where a straight line is drawn to fit the slope. Within the experimental errors, a good fit was obtained for each experiment in the 10 to 40 keV range. The fit is always better for lower than for higher dose measurements. The first reason for this discrepancy is that it is more difficult to extract the diffuse components of the total intensity when they are of about the same order of magnitude. A second reasonable question arises considering the model used here which does not distinguish between structural defects and lattice distortions. However, except in the region of heavier damage, agreement was rather good. Values of $\bar{A}$ are plotted in figure 6 for various ion energies indicating the experimental dispersion of the results. The corresponding equivalent number of surface atoms displaced (out of phase or vacancies) is derived assuming that only a double layer 111 (i.e. the « rough » 111 plane) is responsible for the recorded reflected intensity. It is clear that this assumption is not really valid and should be improved by integrating a « depth contribution » from the first top layers. This point will be discussed later.

In table I we also give the critical dose D_c , for which $\ln(I/I_0) = -5$; as $\bar{A}$ decreases for increa-

Table I. — Values of $\bar{A}$, the mean damaged area; N , the equivalent number of atoms in a rough layer (111) within $\bar{A}$; and D_c , the fluence for which $\ln(I/I_0) = -5$, as functions of the ion energy E_0 .

E_0 (keV)	10	20	30	40
$\bar{A} \times 10^{-14} \text{ cm}^2$	4.7	3.8	2.3	1.4
N eq. number surf. atoms	75	60	35	20
$D_c \times 10^{14} \text{ ion/cm}^2$	0.5	0.7	1.2	1.8

sing ion energy, D_c increases in the same way from 0.5 to $1.8 \times 10^{14} \text{ ion/cm}^2$ for a complete disordering of the surface. Since the surface density of Si(111) is about $16 \times 10^{14} \text{ at/cm}^2$, the D_c correspond to doses equivalent to 1 incoming ion for 30 surface atoms at 10 keV to 1 for 9 at 40 keV.

3.4 PROJECTED RANGE AND ENERGY DEPOSITION. — As the ion penetrates the target, it loses its energy by nuclear and electronic interactions and finally comes to rest at some position on the depth axis. The initial energy E_0 can be partitioned between the amount E_i lost by electronic excitations, and the remaining amount which results in displacements of target atoms, the so-called damage energy E_d [14, 15]. According to LSS theory [16], the probability of coming to arrest at a final position Z_0 is described to a first approximation by a Gaussian distribution, in which Z_0 as a mean value $R_p(0)$ and a standard deviation $S_d(0)$. Values of $R_p(0)$ and $S_d(0)$ were calculated for the Ar/Si system using the Schiott formulation [17] and are listed in table II; the range distributions for 10 to 40 keV ions are plotted in figure 7. Damage energy deposition calculations were performed on a micro-computer IBM-PC, using the formulation of Gibbons [18] and Robinson [19, 20]. The E_d/E_0 values listed in table II represent the « damage efficiency » for one average ion incoming the target.

Fig. 6. — Variation of $\bar{A}$, the mean damaged area, versus bombardment energy. Bars indicate the experimental dispersion of results.

Table II. — *Implantation characteristics for the Ar/Si system as a function of the ion energy E_0 . (The parameters and the methods of the calculations are described in the text.)*

E_0 (keV)	10	20	30	40
R_p (Å)	130	210	290	400
S_d (Å)	70	110	150	200
E_d/E_0	0.67	0.64	0.62	0.60
$E_{\text{surf.}}$ (eV/Å)	60	63	59	54
N_d^*	5	5	5	5

Fig. 7. — Ion range distributions of argon implanted into silicon from LSS theory.

As we are only interested in the disordering of the near surface region, mean damage energies deposited in the first top layers, $E_{\text{surf.}}$, were calculated and values are listed in table II.

The most important particularity of the Ar/Si system in the 10 to 40 keV can now be understood. As the nuclear stopping power S_n is maximum for 20 keV, its value does not significantly change from 10 to 40 keV; the calculated damage peak is always very close to the surface. Then the damage energy deposited through the first layers varies only of 15 % over the whole energy range and cannot directly explain the strong decrease with energy of the mean area damaged by ion impact. Anyway, as we assume that only a double layer (111) is responsible for the RHEED recorded intensity, it is worthwhile to estimate the number of surface atoms displaced by nuclear interaction in this rough layer. According to Kichin and Pease [21], the average number of displaced atoms can be estimated to be

$$N_d = k E_d / 2 E_s$$

where k the so-called displacement efficiency is usually taken as equal to 0.8. E_s is the displacement energy which is about 15 eV for bulk silicon since no surface displacement energy was considered. Then taking into account only the first rough layer, one can estimate the number of displaced atoms N_d^* , by one average incoming ion. Values are listed in table II.

These values are considerably smaller than those obtained for $\bar{A}$ from RHEED experiments, but we

know that $\bar{A}$ is not only representative of the heavily damaged region where target atom displacements should occur but also takes into account distorted areas due to vacancy agglomerates or entrapped argon atoms.

The specific damage energy density for crystalline to amorphous transition has been determined to be about 6×10^{23} eV/cm³ or 12 eV/at at 4 K [22] and 80 K [23] both from ESR measurements and TEM imaging. Since the dose needed for amorphizing a complete layer, at room temperature is known to be 4 times higher than at 4 K [24], one can roughly estimate the damage energy density for crystalline to amorphous transition, in the bulk at room temperature to be about 50 eV/at. In table III, we give the final damage energies deposited, in the near-surface

Table III. — *Deposited damage energies and argon concentrations in the near-surface region at D_c , versus the ion energy E_0 as predicted by LSS theory.*

E_0 (keV)	10	20	30	40
E_{sur} (eV/at)	6.6	8.2	13.6	19
$\frac{N(\text{Ar})}{N(\text{Si})} \times 10^{-4}$	1.1	0.8	1.1	1

region, at D_c which correspond to the complete disordering of the surface as seen by RHEED and REM. It is then noticeable that, even for the 40 keV bombardment, the damage energy density deposited in the surface layers is at least 2-3 time lower than that needed for continuous amorphous layer formation, as measured by Dennis [25]. Even if a lower value of the damage energy density needed for amorphising the surface is assumed, which is reasonable, this cannot be consistent with the observed bombardment energy dependence. Then, it is reasonable to think that our « critical dose » D_c is more representative of a distorted surface network than of an amorphous state.

As it is clear that the variation of the mean damaged area $\bar{A}$ cannot be explained in terms of damage energy surface deposition, one can be interested in the argon concentration in the near surface region. From the curves plotted in figure 7 and known as range distributions, we can at least estimate the relative Ar concentrations in this near surface region. Results are given in table III; the relative atomic concentrations $N(\text{Ar})/N(\text{Si})$ at D_c , averaged over the first ten angstroms and estimated from the range distributions, are seen to remain constant, within the experimental errors.

Then, the decrease of $\bar{A}$ must be correlated to the amount of argon atoms entrapped close below the surface, which is responsible for strong distortions in the surface network. Our first experiments on annealing effects on bombarded surfaces show that for doses up to our critical dose the initial surface topography and RHEED intensity can be restored by a rapid heating (a few seconds) at about 500 °C, which is in the range for argon desorption [26].

The effect of a 1 nm SiO₂ surface layer, which is a reasonable assumption [27], on both the range profiling and the energy deposition, was estimated by computing methods. In spite of a strong overestimate, the number of O recoil atoms was shown to be small, in the near-surface region, with respect to the Ar concentrations, and, strongly dependent on the damage energy deposited in this layer. This and the annealing experiments suggest that the oxygen implantation was not the driving mechanism of the observed degradations.

The model from Jacobson [2] in spite of no « depth contribution » from the (111) planes below the surface is sufficient for relative comparisons. As it was pointed out by Yagi *et al.* [3], the depth at which the incident electron beam intensity has fallen to 1/e from its initial value is about 20 Å for the 444 reflection on Si(111) at 100 keV. Anyway, ion range and energy deposition calculations show that these parameters do not vary strongly through these few layers. Then, a depth contribution correction would not affect the above results, at least when used for comparative study.

4. Conclusion.

RHEED intensity measurements and REM imaging of Si(111) surface under argon bombardment in the 10 to 40 keV range show that :

— With increasing energy the dose required for the complete disordering increases.

— This critical dose is lower than that required for amorphizing a complete amorphous bulk layer.

— No facetting occurs for doses up to 1×10^{15} ion/cm².

— From REM images of Si during irradiation, step contrast analysing did not show any specific effect on the very first layer nor due to atomic steps, although an increasing granulation of the terraces is observable.

— The RHEED degradation characteristics are consistent with the idea of « mean damaged area » including displaced atoms (amorphous zones and

induced relaxations) and network distortions (ion entrapment), both responsible for an out-of-phase setting of the surface network.

— The variations of the damage energy deposited in the near surface region, over the whole energy range, cannot account for the strong variations of $\bar{A}$.

— A rough estimate of the relative concentrations of argon in the near surface region shows better agreement.

These remarks lead us to conclude that RHEED intensities do not distinguish between localized defects such as divacancies from distortion effects resulting from the embedment in the lattice of the bombardment atoms. In the case of the 10-40 keV Ar/Si system, the RHEED and REM imaging degradation characteristics are mainly governed by the amount of embedded argon atoms. The same idea is of course valid for LEED (Low Energy Electron Diffraction) since it is often used to check the surface structure when cleaning it by ion bombardments and annealing cycles. While the degradation of diffracted intensities constitutes an index of surface damage, it does not provide by itself much insight into the type of introduced defects.

Anyway, it should be worth studying now an ion/target combination for which the damage energy deposition rate would strongly vary through the considered energy range.

The chemical composition of the first layers when increasing fluence should be experimentally carried out in order to compare the results with values predicted by LSS theory. The effect of the target temperature on the surface disordering characteristics will be soon investigated since one expects that increasing temperature will affect both the argon desorption rate and the critical dose required for the complete amorphization of the implanted layer.

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