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Carbon deposition in Si as a consequence of H and He irradiations: A systematic study

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In the present work we have investigated the influence of different parameters that determine the C deposition on a Si target. Among them we have studied the pressure of the irradiation chamber, the implantation fluence, the current density, the target temperature, the energy of the beam, the charge state of the ion, the ion species and the molecular state of the irradiation beam. In order to determine the quantity of C deposited on the Si substrate we have used the Rutherford backscattering/channeling technique together with the resonant $^{12}\text{C}(\alpha,\alpha)^{12}\text{C}$ reaction. After careful analysis we arrived at the conclusion that the real independent parameters are the pressure of the irradiation chamber, the temperature of the target, the irradiation time and the electronic stopping power of the ion–target combination. © 2002 American Institute of Physics. [DOI: 10.1063/1.1467960]

I. INTRODUCTION

It is known that ion irradiation or implantation performed at pressures higher than 10^{-8} Torr bring as a consequence the deposition of impurities on the surface of the sample.¹ It is also well established that mostly C and a lesser quantity of O and H are the primary elements that stick to the surface of the sample as a result of the combined action of the irradiation beam and emission of secondary electrons from the sample under analysis. Consequently the deposition of a C film on the surface of the sample has very detrimental effects on near surface analysis. The depth scale obtained from Rutherford backscattering spectroscopy (RBS) experiments is altered due to the loss of energy in the C film. In channeling experiments dechanneling, depending on the thickness of the film, occurs. Charge-exchange experiments are strongly hampered. Finally, results of very narrow nuclear resonant reactions like the $^{18}\text{O}(p,\alpha)^{15}\text{N}$ one (with $\Gamma = 50$ eV) are strongly distorted by the C deposition.

On the other hand, implantation performed under insufficient vacuum conditions can lead to wrong conclusions. As an example we can cite the results of Refs. 2 and 3 in which the authors implanted B and N into stainless steel and attributed the improvement of the tribological properties to action of the implanted ions. However, a few years later Faussemagne and Benyagoub⁴ showed that the tribological improvement was due to the carbon recoil implantation from the C film deposited on the surface of those stainless steel samples.

Recently, Healy,¹ using the $^{12}\text{C}(d,p_0)^{13}\text{C}$ reaction, performed a systematic study of the parameters that can influence C deposition on Si samples. He concluded that the pressure of the analyzing chamber does not play a relevant role in the C deposition rate of the sample. This is a very surprising result that only can be understood if the irradiation time is short enough.

In view of this and of other conclusions, we decided to perform another systematic study of the beam and vacuum parameters that can influence the formation of a C film on Si. The parameters under study were (a) the chamber pressure, (b) the irradiation fluence, (c) the current density, (d) the temperature of the target, (e) the energy of the projectile, (f) the charge state of the ion, (g) the ion species, and (h) the molecular state of the irradiation beam. With this in mind, we used He and H beams on Si targets. We have chosen these ion–target combinations because H and He are the most widely used beams in material analysis and Si is one of the most important materials in technological applications.

II. EXPERIMENT

The carbon films were produced by He or H irradiation on polished silicon $\langle 100 \rangle$ single crystal specimens of approximately 4 cm^2 in area. The irradiations were performed with samples misaligned by 7° off the beam direction in order to avoid channeling implantation. The samples were cleaned following microelectronic standards and immediately thereafter were mounted in the irradiation chamber. The latter is equipped with a drug-turbomolecular pump backed by a membrane pump. In addition a LN_2 trap was used in order to improve the vacuum of the system. The irradiations were done at two different accelerators: a 500 kV ion implanter and a 3 MV Tandetron accelerator.

The carbon contamination produced by the ion irradiation was assessed in two different ways. The first was by using the $^{12}\text{C}(\alpha,\alpha)^{12}\text{C}$ resonant reaction in $E_r = 4265$ keV with a width of $\Gamma = 27$ keV.⁵ The second was via the Rutherford backscattering technique in the Si $\langle 100 \rangle$ channeling direction (RBS/C). This was done in order to reduce the Si background with respect to the superimposed C signal. In addition, in this last case, in order to improve the depth resolution, the Si barrier detector was placed at 70° with respect to the sample normal direction. The energy of the analyzing He beam was 1.2 MeV and the overall detector plus elec-

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FIG. 1. Partial pressures of the residual gas components of the irradiation chamber: total pressure (a) $p = 10^{-5}$ and (b) 4×10^{-7} Torr.

tronic resolution was better than 12 keV full width at half maximum (FWHM). As a carbon standard we used a $10 \mu\text{g}/\text{cm}^2$ film evaporated on a Si wafer.

III. RESULTS

As mentioned above we studied the C deposition on the Si films as a function of the irradiation fluence, pressure of the chamber, current density, ion energy, type of ion (He or H), temperature of the sample charge state of the ion and molecular state of the impinging ion. In all cases the results obtained were corrected by the deposited C induced by the He analyzing beam. This was done by measuring the C deposition before and after each analysis run.

A. He irradiation

1. Vacuum pressure

The dependence of carbon deposition on the pressure as a function of the irradiation fluence was investigated by establishing equilibrium between the pumping rate and air deliberately leaked into the chamber. The pressures investigated were 10^{-5} , 4×10^{-6} and 4×10^{-7} Torr. In Figs. 1(a) and 1(b) are the partial pressures of the residual gas components of the irradiation chamber at 10^{-5} and 4×10^{-7} Torr.

For each pressure the irradiation fluence was varied between 2×10^{16} and 2×10^{17} at./cm². A typical RBS/C spectrum is shown in Fig. 2(a) while the corresponding $^{12}\text{C}(\alpha, \alpha)^{12}\text{C}$ resonant spectrum taken at 4265 keV is displayed in Fig. 2(b). The irradiation parameters of the example were pressure in the chamber of around 10^{-5} Torr, irradiation fluence of 5×10^{16} He/cm², He⁺ energy of 400 keV and current density of $1 \mu\text{A}/\text{cm}^2$. A direct comparison with the corresponding spectra of the C standard film taken under identical conditions allowed us to calculate the surface

FIG. 2. (a) Typical RBS/C spectra of an irradiated Si sample. The Si barrier detector is at a 70° to the direction of the sample. (b) The same as in (a) but obtained through the $^{12}\text{C}(\alpha, \alpha)^{12}\text{C}$ reaction.

concentration C_s in C at.cm⁻². The results obtained are shown in Fig. 3(a) where the carbon concentrations for each chamber pressure are displayed as a function of the irradiation fluence. It is difficult to convert the real measured C quantity (C/cm²) into the C thickness, since the actual chemical and physical status of the C deposited is not known. However, Faussemagne and Benyagoub⁴ claimed that in similar experiments the C film deposited was diamond-like carbon type. Then, and just to give an order of magnitude of the thickness of the deposited C film, we assumed that the C density is that corresponding to diamond ($\rho = 1.76 \times 10^{23}$ at.cm⁻³). Consequently, on the right side of Fig. 3(a) the equivalent thickness of the C film is correspondingly displayed. As can be observed, the higher the pressure, the higher the C concentration of the C films. Even at $p = 4 \times 10^{-7}$ Torr irradiation of 10^{17} He/cm² leads to deposition of a C film equivalent to 20 Å.

On the Tandatron accelerator we performed a similar experiment, but changed some of the parameters. The pressures investigated were $p = 4 \times 10^{-6}$ and 4×10^{-7} Torr. We used He⁺ beam at 1.2 MeV with a current density of $1.6 \mu\text{A}/\text{cm}^2$. The results are displayed in Fig. 3(b). Again it can be observed that the pressure plays an important role in C deposition, the latter one being higher at higher pressure. At these

FIG. 3. C concentration as a function of the irradiation fluence for different chamber pressures: (a) obtained at the ion implanter and (b) the same as (a) but obtained at the Tandetron.

current densities it typically takes 20 min to irradiate 10^{16} at/cm².

2. Temperature of the sample

The dependence of C deposition on the sample temperature was investigated under the following conditions: the He beam energy was 400 keV, the current density was $1 \mu\text{A}/\text{cm}^2$, the temperature of the target was varied between -45 and 350°C , the pressure of the chamber was maintained at 4×10^{-7} Torr, and the fluence was varied between 2×10^{16} and 2×10^{17} He/cm². The results are shown in Fig.

FIG. 4. C concentration as a function of the irradiation fluence for different target temperatures.

FIG. 5. C concentration as a function of the irradiation fluence obtained at different He bombardment energies.

4. It can be clearly observed that the implantation temperature plays an important role in C film formation. For the lowest temperature and for an irradiation fluence of 10^{17} He/cm² we observe the formation of a $x \approx 30$ Å C film. This thickness diminishes with an increase in temperature and for 350°C it remains almost constant, independent of the irradiation fluence at a level of $x \approx 5$ Å.

3. Energy of the He ion beam

In order to study the effect of beam energy on the formation of the C film we performed the irradiations at both the ion implanter and the Tandetron accelerator. At the ion implanter the conditions were the following: pressure of 4×10^{-7} Torr, current density of $1 \mu\text{A}/\text{cm}^2$, and He⁺ energies of 100 and 400 keV, respectively. For comparison, at the Tandetron the He⁺ beam energies were 1.2 and 3.5 MeV, respectively, while both the current density and the chamber pressure were maintained at the same values as those above, i.e., $1 \mu\text{A}/\text{cm}^2$ and 4×10^{-6} Torr, respectively. The results are displayed in Fig. 5. It can be observed that the C formation is a function of the beam energy. However the dependence is not a monotonic one. For lower energies the thickness of the film increases with the energy but for higher ones it decreases with an increase in energy.

4. He current density

In this case we have irradiated the Si samples under two different density current conditions. In the first case the current density was $j = 0.7 \mu\text{A}/\text{cm}^2$ and in the second it was $1.6 \mu\text{A}/\text{cm}^2$. All other conditions were similar, that is, a 1.2 MeV He⁺ beam incident on the Si target with chamber pressure of 4×10^{-6} Torr. The results are displayed in Fig. 6. It can clearly be observed that for a given irradiation fluence the smaller the current density, the higher the thickness of the C film deposited.

5. Charge state of the He beam

Here, we have investigated whether the charge state of the He beam has any influence on the formation of the C film on the Si substrate. To this end we have used He⁺ and He⁺⁺

FIG. 6. C concentration as a function of the irradiation fluence obtained for different irradiation current densities.

3.5 MeV beams with a current density of $j=0.75 \mu\text{A/cm}^2$ and an irradiation chamber pressure of $p=4 \times 10^{-6}$ Torr.

The results obtained are shown in Fig. 7. Surprisingly, it can be observed that the charge state of the ion plays an important role in the C deposition on the Si target. For the same irradiation fluence, the higher the charge state, the greater the thickness of the deposited C film, the difference being of the order of 40%.

B. H irradiation

In the same way as for He irradiation, we also investigated the influence of the chamber pressure and of the energy of the H beam on the formation of the C layer on the Si target.

1. Pressure of the chamber irradiation

In this set of experiments we performed measurements using a 100 keV H beam with current density of $1 \mu\text{A/cm}^2$ under two different pressures in the irradiation chamber. In the first, the pressure was $p=10^{-5}$ Torr and in the second $p=4 \times 10^{-7}$ Torr. The results, displayed in Fig. 8 show, like in the He case, that the higher the pressure, the larger the thickness of the C film.

FIG. 7. C concentration as a function of the irradiation fluence obtained for different charge states of the bombarding He ion.

FIG. 8. C concentration as a function of the irradiation fluence for different chamber pressures. The bombardments were done with H ions.

2. H energy beam and molecular effects

We changed the H energy beam from 100 to 400 keV while keeping all the other irradiation conditions similar, that is, $p=4 \times 10^{-7}$ Torr and current density $j=1 \mu\text{A/cm}^2$. In addition we performed an irradiation experiment using a 200 keV H_2 molecular beam with the same particle density current, $j=1 \mu\text{A/cm}^2$. The results are displayed in Fig. 9. An inspection of Fig. 9 shows two interesting features, namely, for the highest beam energy we have deposited less C than for the lowest one and for the same energy per amu, that is, 100 keV/amu, the molecular beam deposits onto the Si substrate a C film whose thickness is almost twice with respect to the one created by the atomic beam.

IV. DISCUSSION

From the description of the results given in Sec. III it is easy to see that some of the parameters that we have investigated are really independent while others seems to be related to each other. Among the former certainly we can mention the pressure of the irradiation chamber and the temperature of the target. Others, like the irradiation fluence and the current density, on one hand, and energy, charge state and kind of ion, on the other hand, are related to each other as we will show in what follows.

A. Pressure of the chamber and temperature of the target

The pressure of the irradiation chamber plays an important role in C deposition on the sample as demonstrated by inspection of Fig. 3. This result is in contradiction with the findings of Healy¹ who found exactly the contrary. The only explanation for these opposite results is the irradiation time. While in Ref. 1 a typical irradiation time was of the order of few minutes, in the present one is of the order of hours.

With regard to the temperature our findings agrees with those of Ref. 1. The higher the temperature the lower the C deposition. This behavior can be understood by assuming that at higher temperature, the desorption C rate of the sample is greater.

FIG. 9. C concentration as a function of the irradiation fluence obtained at different H bombardment energies.

B. Irradiation fluence and current density

It is easily recognized that the current density and irradiation fluence both depend on a third parameter, which is the irradiation time t . Therefore if we combine the variables n , Φ and j according to $t = ne\Phi/j$, with n being the charge state of the ion beam, e the electron charge, j the current density and Φ the implantation fluence, the data can be described as a function of the irradiation time only.

The above procedure was only applied to irradiations performed at the Tandetron, since the ones done at the ion implanter were performed at the same irradiation current density ($j = 1 \mu\text{A}/\text{cm}^2$). In Fig. 10(a) we show the results of the He^+ irradiations performed at 1.2 MeV with different current densities ($j = 0.7, 1$, and $1.6 \mu\text{A}/\text{cm}^2$, respectively) for a chamber pressure of 4×10^{-6} Torr. In Fig. 10(b) shown is the same kind of results obtained at the same pressure, but with a 3.5 MeV He^+ beam with current densities of $j = 0.75, 1$, and $1.5 \mu\text{A}/\text{cm}^2$, respectively. As can be observed in both cases a linear relationship can be established between the thickness of the carbon film and the irradiation time.

Therefore it can be concluded that the irradiation time rather than the implantation fluence or the current density is the independent parameter in C deposition onto the sample.

C. Electronic stopping power

At this point it is necessary to develop a qualitative model on the mechanism which led to the deposit of impurities on the sample due to ion bombardment.

It is known that ion bombardment induces the emission of secondary electrons (SEs) from the target.⁶⁻⁹ SEs can be emitted from the solid by two mechanisms: for low ion velocities ($v < 10^7$ cm/s) ejection of electrons will occur primarily by the potential or Auger mechanism provided that neutralization of the positive ions exceeds twice the work function of the solid.⁹ Ions with higher velocities eject electrons by the so-called "kinetic" mechanism in which electrons may be accelerated as a result of close or distant collisions between the incident ion and the lattice atoms. Another possibility is that the electron ejection stems direct "binary"

FIG. 10. C concentration as a function of the irradiation time.

collisions between the projectile and nearly free valence electrons.

At velocities larger than the Fermi velocity of the target electrons, another process becomes important. The projectile will then be able to excite surface and bulk plasmons which decay mainly through the creation of electron-hole pairs. The electrons may then be ejected out of the sample with a maxi-

FIG. 11. C concentration as a function of the irradiation time times the electronic stopping power. Shown are all the previous data with the exception of those that correspond to the temperature of the target.

TABLE I. Electronic stopping powers for H and He ions in Si.

Energy	100 keV ^a	400 keV ^a	1.2 MeV ^b	3.5 MeV ^b
$S_{\text{He}^+ \rightarrow \text{Si}}^+ [\text{eV}/(10^{15} \text{ at./cm}^2)]$	40.0	71.4	32.2	21.2
$S_{\text{He}^{++} \rightarrow \text{Si}}^+ [\text{eV}/(10^{15} \text{ at./cm}^2)]$	...	...	56.7	35.4
$S_{\text{He}^+ \rightarrow \text{Si}}^+ [\text{eV}/(10^{15} \text{ at./cm}^2)]$	26.3	14.2	...	...

^aValues taken from subroutine Rstop of the TRIM program.^bValues taken from the CasP program (see Ref. 11).

num energy that is equal to the plasmon energy minus the work function.⁹

It was also demonstrated that the “escape zone” of the electron emission is confined to a few atomic layers of the target⁶ and also that the yield of SEs is proportional to electronic stopping of the incident ion on the target, that is,

$$N_{\text{SE}} \approx dE/dx|_e.$$

Taking this background into account, one can assume two different mechanisms for C deposition on the target to be the following.

- (1) Model 1: The ion beam ionizes hydrocarbons of the residual gases that are near the sample. These are attracted by the electric field of the sample created by the SEs produced by the incident beam on the target.
- (2) Model 2: The SEs created by the incident beam ionize nearby hydrocarbons which are attracted to the sample by the electric field of the target created by the same secondary electron emission process.

We have no means by which to decide which of the above qualitative models is the correct one. However, in both cases the secondary electron emission plays a crucial role: either by only attracting the ionized hydrocarbons or by being responsible for both the ionization process together with the attraction of the hydrocarbons to the sample.

Based on the above discussion we realize that the electronic stopping power of the incident ion should be an important parameter in the process of C deposition on the Si sample.

In order to test this hypothesis and assuming that the hydrocarbon ionization cross section is constant, we have plotted in Fig. 11 the thickness of the C film as a function of the product of the irradiation time times the electronic stopping power of the irradiating ion. For the stopping powers we have used the values given in Table I. For the low energy implantation we have taken the values given by the subroutine Rstop of the TRIM program.¹⁰ However for higher energies we have to take the pre-equilibrium stopping values given by the program CasP,¹¹ since what is important is the stopping in the few first layers of the target. Finally for the H₂ molecular stopping we have taken the value given in a review by Arista.¹²

As can be observed all the experimental data nicely follow two straight lines, one corresponding to the experiments done at $p = 4 \times 10^{-6}$ Torr, and the second corresponding to $p = 4 \times 10^{-7}$ Torr. Therefore we can conclude that what really matters is the electronic stopping power of the incident ions rather than the charge state, ion energy or type of ion

used in the experiments. This dominance of the electronic stopping power also explains why the highest energy in Fig. 4 does not yield the highest C deposition yield, because in that case the ion stopping power has already exceeded its maximum.

This is a very important point for planning any experiment. Whenever one has to make a RBS, RBS/C or elastic recoil detection analysis (ERDA) measurement, one should use the irradiation beam with minimum stopping power in order to minimize C deposition on the target. That means, for example, that, if possible, one should use a He⁺ beam rather than a He⁺⁺ one. Also if possible, one should avoid using ions with energies that correspond to the maximum of their stopping power.

V. CONCLUSIONS

In this work we have studied the influence of different parameters that determine C deposition on a Si target. Among them we have studied the pressure of the irradiation chamber, the implantation fluence, the current density, the target temperature, the energy of the beam, the charge state of the irradiating ion, the kind of ion and the molecular state of the irradiation beam.

After careful analyses we arrived at the conclusion that the real independent parameters are the pressure of the irradiation chamber, the temperature of the target, the irradiation time and the electronic stopping power.

This last parameter is a direct consequence of the relation between the number of secondary electrons emitted from the target and electronic stopping of the incident ion. This conclusion is not only valid for atomic ions but also for molecular ones. What is more surprising in this last case, is that, even though the molecular stopping power is different from the atomic one due to the *vicinage effect*, the direct relationship between C deposition and the molecular electronic stopping power is still maintained.

One can therefore draw the following general conclusions. In order to minimize C deposition on the target one has to keep the pressure as low as possible, the sample temperature as high as possible and use the ion beam under conditions in which the electronic stopping power is as small as possible.

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