



On the argon and oxygen incorporation into SiO_x through ion implantation during reactive plasma magnetron sputter deposition

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ABSTRACT

The incorporation of argon in SiO_x ($0 \leq x \leq 2$) during reactive plasma magnetron sputter deposition using a O_2/Ar plasma and a silicon sputter cathode has been investigated and related to the flux of argon ions created in the plasma afterglow region on the growth surface. The argon concentration in the grown films appears to be mainly a function of the x -value, independent of the extent of ion bombardment on the growing surface, and only slightly dependent on the substrate temperature during the growth ($<400^\circ\text{C}$). The argon concentration amounts to 4.0 at.% for the $x = 0$ film. It shows a maximum above 5 at.% for an x -value around 0.3, and decreases monotonously for increasing x -values until it is essentially 0 for $x = 2.0$. It is proposed that the measured concentration of argon is a result of implantation and subsequent desorption. The latter process is suggested to be dependent on the x -value. The independence of the concentration of incorporated argon on the relative ion flux is explained by a quasi-saturation state of the process. The incorporation of oxygen as a result of oxygen ion implantation, similar to the Ar incorporation, becomes apparent since values for $x > 2$ are reached.

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1. Introduction

In magnetron sputter deposition of thin films the growing film is continuously bombarded with inert gas ions and neutrals [1,2], and, to a lesser extent, with reactive gas ions and ionized sputtered atoms and molecules [3]. The energy of the impinging ions depends on the pressure and power, and is closely related to the plasma potential, which in our unbalanced RF magnetron setup, where the anode is grounded, amounts to several tens of volts [4,5]. These positive ions are created at the substrate side of the plasma potential maximum in the so-called afterglow region. The positive ions created at the cathode side of the potential maximum cannot overcome the maximum, and they therefore do not reach the substrate. Negative ions created in the cathode region of the plasma or in the sputtering process do have a much higher energy [6], but their flux on the growing film surface is often much lower than the positive ion flux.

The role of this ion bombardment has been extensively investigated, since it is one of the important ingredients in plasma sputter deposition to control the mechanical, thermal and other properties of the growing film [7–9]. Much less attention has been paid to the ions implanted and incorporated in the growing film. For an argon plasma the main constituents of the bombarding ion flux are Ar^+ ions. The concentration of argon incorporated in the growing film can be appreciable [10–12] and may depend on the negative bias of the growing film [12,13]. For an O_2 containing plasma O_2^+ , O^+ and SiO^+ ion bombardment may contribute to the oxygen incorporation in the growing film [3], and is expected to be especially noticeable in those cases where the incorporation probability of neutral molecular oxygen is low.

In the present paper we describe and discuss measurements of the Ar concentration and its depth distribution in O_2/Ar reactive magnetron plasma sputter deposited SiO_x films for $0 \leq x \leq 2$, as a function of several deposition parameters: oxygen partial pressure, total pressure, substrate temperature, applied RF power and growth surface position relative to the cathode erosion region. Evidence for the oxygen incorporation as a result of oxygen ion bombardment in the SiO_x films is also reported.

Since oxygen incorporated through the reaction of molecular oxygen with the growing film cannot be distinguished from oxygen incorporated by other mechanisms, the role of the oxygen bombardment can only be studied for those deposition conditions

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where films with $x \approx 2$ are grown, thus at relatively large oxygen partial pressures. Under these conditions the O_2 decomposition probability on the growth surface is lower than at lower x -values, so that the relative contribution of O_2^+ ion bombardment to the total oxygen growth rate is larger than at lower O_2 partial pressures. Under these conditions the contribution of the ion bombardment to the oxygen incorporation might become visible in contrast to the conditions where films with $x < 2$ are deposited [3].

The results of the measurements of the oxygen and argon concentration in the grown films are related to results of the study of the extent of ion bombardment, as carried out with an energy-resolved mass spectrometer, and, to a lesser extent, using a movable Langmuir probe.

The deposited SiO_x films are amorphous and show a tendency to separate in nanoscale oxygen-poor and oxygen-rich regions as a result of spinodal decomposition [11,14].

2. Experimental

The experimental setup has previously been described [3], and therefore only the most important features of the experimental setup and methodology are mentioned here.

The process under investigation is the reactive RF magnetron plasma sputter deposition of SiO_x films from a silicon sputter target in a gas mixture of Ar and O_2 . The deposition system has a base pressure below 10^{-5} Pa. The water cooled cathode of polycrystalline silicon is circular and has a diameter of 10 cm. The cathode is powered with a 13.56 MHz RF voltage, and the total area of the circular cathode erosion area (the “racetrack”) amounts to 10–15 cm².

The entire cathode can be moved up and down over a distance of 30 cm in a direction parallel to the deposition surface plane. The plane of the cathode surface and the plane of the deposition surface are both vertical. The distance between the two planes amounts to 8.5 cm. The position “target height = 0” corresponds to the situation that the point on the circle describing the circular racetrack, which is also on the horizontal plane through the center of the racetrack, is in front of the substrate position. Unless indicated otherwise, this is the position where most depositions have been carried out.

In the present experiments, the input power was varied between 50 and 250 W, corresponding to a power density on the racetrack between about 5 and 25 W/cm² assuming that all applied power is dissipated on the racetrack, and thus neglecting other loss terms. The total gas pressure was varied between 0.2 and 4 Pa. The O_2 partial pressure was varied between 0 and 5×10^{-3} Pa. These conditions enable the deposition of SiO_x films within the entire composition range $0 \leq x \leq 2$. Mainly due to the relatively large ratio of the deposition surface area and the area of the racetrack, it is possible to deposit SiO_2 films while the cathode is in the unpoisoned mode [3].

Sample substrates have an area of 1 cm², and are made of crystalline silicon with a (1 0 0) surface, or Sigradur[®] G glassy carbon with a diamond polished surface. The samples can be heated by two filaments positioned behind the substrate. The temperature was measured using a thermocouple inserted into the sample holder assembly. Depositions were carried out for values of the substrate temperature ranging from room temperature up to 400 °C. During deposition without additional heating the measured temperature slowly rises to about 70 °C.

The deposition system is connected to one of the beam lines of a 6.5 MV EN Tandem Van de Graaff accelerator, enabling *in situ* and *online* elastic recoil detection (ERD) analyses of the growing films [3,15]. However, the Ar concentration in the film appears to

decrease under irradiation with the swift heavy ions, therefore, the depth resolved argon and oxygen concentrations have been determined using *ex situ* Rutherford backscattering spectrometry (RBS) using light projectile ions. The RBS measurements have been made using a 2 MeV He⁺ beam. In these RBS analyses the argon concentration remained constant in the films during the measurements. The high-energy ion beam techniques yield the areal density of the distinct elements Si, O and Ar, and thus the x -value, with a relative accuracy of several tenths of a percent.

An energy-resolved mass spectrometer (Hiden EQP-1000) is mounted with the entrance opening in the plane of the deposition surface. The geometry in our system is such that the deposition area and the mass spectrometer entrance have equivalent positions with respect to the racetrack, independent of the vertical position of the cathode. In this work the extraction voltage on the mass spectrometer was set at 0 V, the ionizer behind the aperture was switched off, and the mass spectrometer orifice (300 μm in diameter), was grounded, just like the substrate.

3. Results and discussion

3.1. Argon

The binding energy of argon on silicon amounts to only ~0.02 eV [16]. Therefore the steady state coverage of adsorbed argon under our conditions is very low ($<10^{-7}$ monolayer), and so we neglect the argon incorporation via knock-on implantation and/or burial of physisorbed argon. In view of the low particle reflectivity for argon atoms/ions impinging perpendicularly on silicon [17], the flux of energetic Ar neutrals from the cathode is negligibly low. Therefore incorporation of argon in the growing layer as a result of energetic neutral bombardment is a less plausible mechanism in our experiments. This is in contrast to the well-known mechanism of argon incorporation by neutral argon (with energies in excess of 100 eV) reflected from heavy atomic mass sputter targets [18]. The only other relevant mechanism for incorporation of argon is therefore the implantation of Ar⁺, ionized in the plasma afterglow region. These ions have a kinetic energy in the range 30–50 eV [2], resulting in an implantation depth of 0.5–1 nm according to SRIM simulations [19].

Thus, a straightforward hypothesis concerning the argon incorporation is that the concentration of incorporated argon is predominantly established in the interplay between the growth rate and the Ar⁺ ion flux on the growing surface. However, it is not straightforward to vary the growth rate at a certain O_2 partial pressure, while keeping the x -value constant [3]. A power variation does not affect the x -value only in extreme cases, i.e., without oxygen in the gas phase and in the case with such a high oxygen partial pressure that at all powers considered material with $x = 2$ is grown. We firstly focus on the case $x = 0$, and later we discuss the case $x = 2$.

Several silicon films ($x = 0$) were deposited on a carbon substrate for various values of the power, each with a deposition time of 20 min at 0.66 Pa and without oxygen in the plasma. In Fig. 1 the thickness, representative of the growth rate, and the argon concentrations in these films are shown. The argon concentration in the film amounts to ~4.0 at.%, independent of the applied power, and appears therefore independent of the growth rate. With a variation in the applied RF power also the Ar⁺ ion flux varies, but only to a limited extent [4]. Fig. 1 presents a measure for the ratio of the Ar⁺ ion flux and the growth rate. It is clear that this ratio decreases with a factor of about 5 as a result of increase in the power from 50 to 175 W. At the same time the ion energy increases only slightly from about 33 up to 41 eV (not shown). It is clear that the resulting argon concentration is not determined by the relative Ar⁺ flux in these experiments.

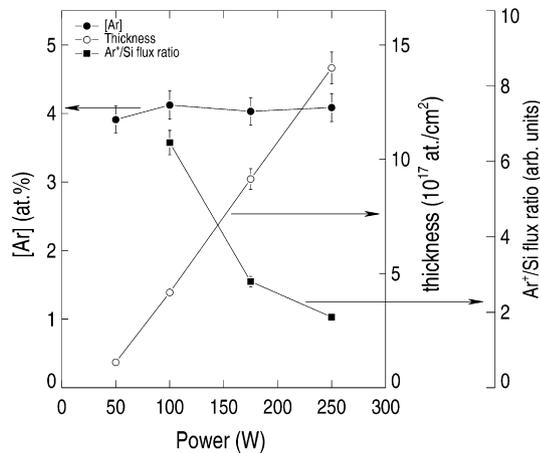


Fig. 1. Concentration of argon in silicon films and the thickness of these films, as a function of the applied RF power. The growth period amounted to 20 min. The argon pressure amounted to 0.66 Pa. Although no oxygen has been admixed in the sputter gas, the chemical composition of the films corresponds to that of $\sim\text{SiO}_{0.03}$. The figure also presents a measure for the ratio of the flux of bombarding Ar^+ ions and the silicon growth rate.

Another possibility to vary the Ar^+ ion flux is to vary the position of the growth surface/mass spectrometer entrance relative to the racetrack position. However, by doing so also the silicon growth rate varies, due to the non-uniform flux distribution of the sputtered silicon atoms. Since the oxygen growth rate does not depend on the position, as long as the x -value is below 1.7 [3], the x -value varies with the position of the cathode (in the case of non-zero O_2 partial pressure). Fig. 2 presents the measured values of the silicon growth rate (a), the ion flux relative to the silicon growth rate (a), the Ar concentration (b) and the x -value (b) as a function of the position. Both the Si growth rate and the Ar^+ ion flux decrease for increasing position values, but the decrease of the ion flux is faster: a factor of 3 decrease in the ratio of the ion flux and the

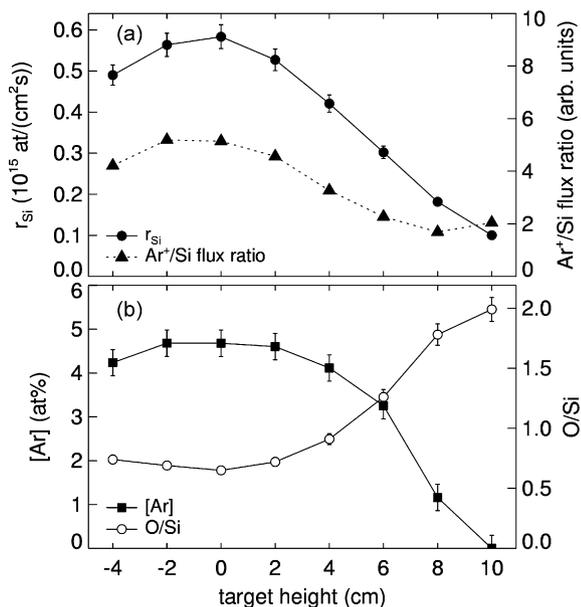


Fig. 2. (a) The silicon growth rate and a measure for the ratio of the Ar^+ flux and the silicon growth rate and (b) the Ar concentration and the x -value as a function of the position of the growth surface relative to that of the sputter cathode. The zero on the position scale indicates the situation that the analyzed growth surface and the spectrometer entrance are (symmetrically) positioned in front of the circular region of most intense sputtering on the cathode. The total pressure amounted to 0.66 Pa. The O_2 partial pressure was adjusted at 1.2×10^{-3} Pa and the power at 175 W.

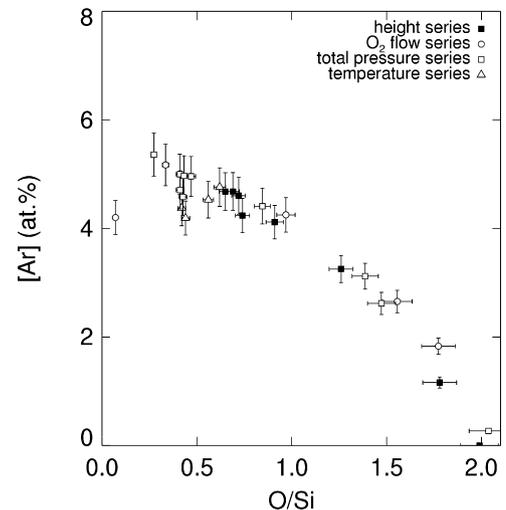


Fig. 3. The Ar concentration in the rf sputtered SiO_x films as a function of x , for the measurement series as indicated.

growth rate is observed. At relatively large position values the x -value increases. It has the lowest value near position zero, as expected.

Assuming that also for the oxygen containing films, the Ar concentration is independent of the ion flux, we plot the measured concentration of argon in the films, considered in Fig. 2, as a function of the x -value in Fig. 3. This figure also presents the measured values of the concentration of Ar in films, in which the x -value was varied by varying the O_2 partial pressure. In the latter case the ion flux and the silicon growth rate do not significantly vary. Strikingly, the argon concentrations for both experiments show the same variation with the x -value.

In addition, Fig. 3 also contains the results of experiments in which the x -value was varied by a decrease in the silicon growth rate at higher total (Ar) pressures [20]. An increase of the total pressure results in a decrease in the ion flux and a small decrease in the average ion kinetic energy [2]. Despite these variations, also this series shows the same variation with x -value as the other experiments do.

Experiments in which the x -value was varied by variation of the substrate temperature during deposition show a somewhat different behavior: both the Ar concentration and the resulting x -value decrease with increasing substrate temperature [3]. For intermediate x -values the Ar concentration in the 400 °C deposited sample appears only slightly smaller than the room temperature deposited sample. This observation is in line with published results: the stability of argon implanted in silicon appears to be such that desorption only takes place at temperature above 500 °C [21].

It is concluded that the concentration of incorporated argon depends on the x -value, and to a smaller extent on the substrate temperature. For $x = 0$ this concentration amounts to 4.0 at.%, and then increases up to 5.3 at.% for $x = 0.3$, and for increasing x -values it decreases smoothly to very low values for $x = 2$.

The Ar concentration in the samples is constant over the full depth of the films, taking into account that the depth resolution in the RBS measurements amounts to about 5 nm, with the exception of the films with $x = 2$: see Fig. 4. The RBS spectrum of Fig. 4 clearly shows an increased Ar concentration close to the interface with the substrate in a total amount of $\sim 10^{15}/\text{cm}^2$ (see Section 3.2).

With the projected range of argon ions of 0.5–1 nm, with a growth rate of the order of 0.3 nm/s and an estimated ion flux of $10^{16}/(\text{cm}^2 \text{ s})$ (see references [3,4]), the surface layer of the growing film experiences a total ion fluence of $1.5 \times 10^{18}/\text{cm}^2$ before it is

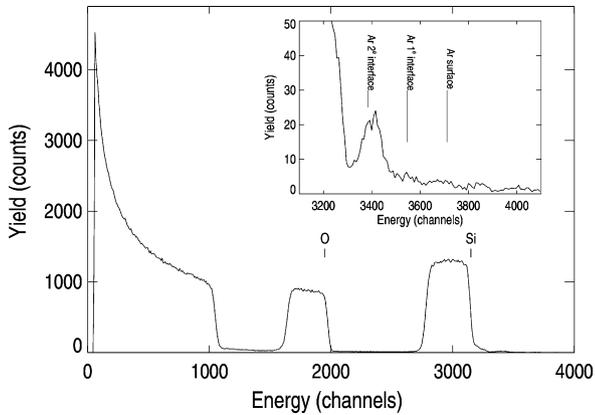


Fig. 4. RBS spectrum of a SiO_2 film deposited on glassy carbon in two consecutive, distinct deposition steps. The conditions during these steps are identical: applied power 175 W, 80 sccm Ar flow (0.66 Pa), and 1.6 sccm O_2 flow. The RBS surface position of argon and that the boundary between the two deposited films is indicated in the inset.

buried and has become unreachable for the ions. The measured concentration of argon in the silicon film ($x \approx 0.03$) amounts to 4 at.%, so the corresponding areal density of argon in the 1–2 nm top layer amounts to $(2\text{--}4) \times 10^{14} \text{ cm}^{-2}$. This simplified analysis indicates that the overall incorporation probability of the impinging argon ions is very low, in the order of 0.01. This low value is in agreement with the results of the study of argon implantation in silicon at similar low kinetic energies [21]. The same study indicates that at the argon areal density in the surface region of silicon of the order of 10^{14} cm^{-2} , the system is in a quasi-saturation, tentatively ascribed to an equilibrium between implantation and release, both under the influence of the same ion impingement. The release is supposed to be efficient due to the action of collision cascades and defect interactions [21]. This quasi-saturation explains satisfactorily why the incorporated argon concentration is independent of the power or growth rate in the silicon films.

The lack of dependence of the measured argon concentration on the ratio of the ion flux to the growth rate for $x > 0$ suggests that the quasi-saturation is reached for all x -values. We tend to ascribe the variation in argon concentration for varying values of x in SiO_x to a shift in the equilibrium between implantation and desorption [12]. It is conceivable that the SiO_2 -type of network, or the boundaries between the SiO_2 rich and O-poor regions, existing because of the spinodal decomposition in the SiO_x films [11], offer fast diffusion paths for argon stimulating a more readily desorption of subsurface implanted argon, resulting in a lower built-in argon concentration. Another explanation is the possible participation of silicon-rich regions in the stabilization of the implanted argon, as suggested by recent theoretical work on SiBCN materials [22]: the contribution of Si-rich regions to the suboxide network decreases with increasing x -value, and there are simply no silicon rich regions for $x = 2$.

Finally, a possible influence of relatively high energetic O^- ions on the argon incorporation may not *a priori* be excluded. Such ions, produced in the cathode fall region and arriving on the growth surface with a kinetic energy corresponding to the potential just in front of the cathode (maximal 250–300 V in our case [3]), appear to play a role in the formation of the structure of growing (oxide) films [23–27]. A hypothesis could be that with increasing O_2 partial pressure, and thus with increasing x -value, the flux of the energetic O^- increases [27,5]. The result is an increased rate of desorption of implanted Ar and hence a lower final Ar concentration with increasing x -value. The impact of the energetic negative ions on the

structural aspects of the growing film has been found to be considerable for conditions in which the sputter target is in the oxidic mode of operation [23]. For the reactive sputtering of titanium oxide in an O_2/Ar atmosphere the O^- yield in the metallic of operation appears 1–2 orders of magnitude lower than in the oxidic mode of operation [27]. In the present work the sputter cathode is always in the metallic mode, and therefore the relative flux of negative oxygen ions on the growth surface is assumed to be low. The O^- flux appears to decrease with increasing total pressure at constant product of pressure and distance between the cathode and the growth surface [5]. In the present work the total pressure has been varied between 0.1 and 4 Pa. Despite this large variation, the measured values for the Ar concentration and their variation with the x -value are equal to those measured at constant pressure, with a variation of one of the other deposition parameters. Therefore, it is concluded that O^- bombardment on the growth surface does not contribute measurably to the desorption of (low energy) implanted argon atoms and is therefore not a determining factor for the final Ar concentration. Furthermore, the lower Ar concentration at higher deposition temperature (Fig. 3) for the same x -value is consistent with an equilibrium between implantation and desorption from the surface layer, assuming that the latter occurs with a higher rate at higher temperature.

3.2. Interfacial argon in SiO_2 films

This section reports and discusses the non-uniform Ar concentration profile in samples with x -values near 2. In these samples, argon appears to be incorporated predominantly in the first few nm of the oxide near the interface with the substrate, in an areal density of $\sim 10^{15} \text{ cm}^{-2}$, as evidenced by RUMP simulation [28] (see Fig. 4).

The amount of (interfacial) argon and its depth distribution depend on the O_2 flow (see Fig. 5) also for x -values larger than 2. Fig. 6a shows that the argon areal density decreases with increasing oxygen flow down to $2 \times 10^{14} \text{ cm}^{-2}$. In this range of oxygen flows the x -value is more or less constant (see next section). At the same time the argon incorporation is limited to the interfacial region, from an argon containing interfacial region of.

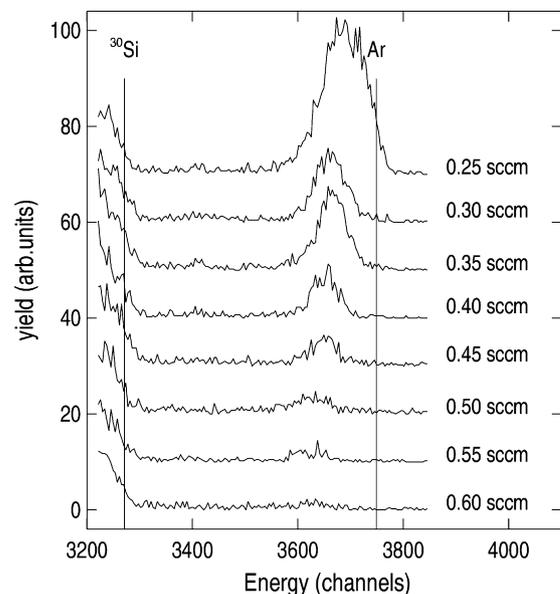


Fig. 5. The argon region in the RBS spectra of an $\text{SiO}_{1.62}$ and several SiO_2 -type films deposited on glassy carbon at various O_2 flows as indicated. The argon surface position is indicated by the vertical line. The deposition conditions are: applied power 75 W, total pressure 0.66 Pa. See also Fig. 6.

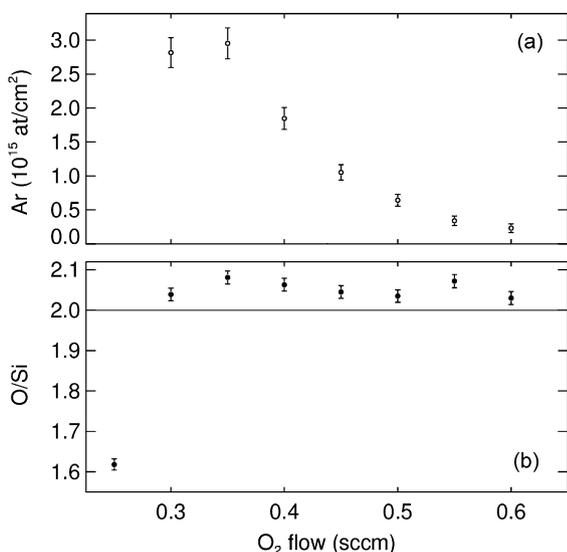


Fig. 6. (a) The argon areal density as a function of the O₂ flow, as deduced from the spectra given in Fig. 5. (b) The x -value of the same samples considered in Fig. 5.

40 nm at an O₂ flow of 0.30 sccm down to a layer of 20 nm at an O₂ flow of 0.60 sccm.

It seems straightforward to attribute this interfacial Ar incorporation to the growth of material with lower x -values in the first few nm of the substrate [29]. The interfacial region, where the x -value deviates from $x = 2$, is expected to be smaller for larger O₂ flows. But there are other possible explanations. To explain such a concentration profile one could hypothesize that this is due to a rise in temperature during deposition of SiO₂-type material. After a few minutes of deposition the temperature could have increased such that the desorption of implanted argon prevails over incorporation when depositing SiO₂. Therefore the following experiment was carried out: an SiO₂ layer was grown both on silicon and glassy carbon. After 135 nm had been grown, the process of deposition was stopped, and the sample was allowed to cool down overnight to room temperature. Next, exactly the same deposition was carried out. Fig. 4 presents the RBS spectrum of such a film, deposited on glassy carbon. It is seen that there is no increased argon concentration in the middle of the film, and therefore the explanation of the profile based on varying temperature of the growing film is not applicable. It must be remarked that a similar double deposition on a native oxide covered, crystalline silicon wafer gives the same result. This double deposition experiment also rules out an explanation based on the possibly present transients in the plasma conditions at the beginning of the deposition.

3.3. Oxygen excess in SiO_x

To assess whether oxygen implantation contributes measurably to the total oxygen incorporation we adopted the experimental approach to grow films under conditions in which the oxygen partial pressure was such that in principle SiO₂ films are deposited. These conditions result in an O₂⁺ flux, which is about 2 orders of magnitude smaller than that of Ar⁺ but about 1 order of magnitude larger than that of O⁺ [3]. At the same time, however, the effective molecular oxygen sticking probability is low [3]. Under the assumption that the incorporation of oxygen via dissociation of molecularly adsorbed O₂ effectively stops on a surface which has the SiO₂ stoichiometry, the growth of films be indicative for the contribution of oxygen implantation, either directly or through a knock-on process induced by bombarding

ions, i.e., mainly Ar⁺ ions. It must be remarked that under these conditions the SiO⁺ flux is of the same order of magnitude as the O₂⁺ flux [3], but this ionic species also carries a silicon atom and is therefore not expected to be responsible for an increase of the x -value.

To get the desired accuracy in the RBS measurements we made sure that desorption of oxygen during the He⁺ irradiation [30] did not influence the results measurably and, furthermore subtracted from the spectrum the contribution of oxygen present in the substrate surface region before the deposition, which amounts to an areal density of about 4×10^{15} cm⁻². Fig. 6b shows that the x -value can be significantly larger than 2.00. The maximum observed x -value amounts to 2.05 ± 0.015 , independent of the O₂ flow once this flow is large enough to form SiO₂. The value obtained must be considered as an x -value averaged over the entire thickness of the film. The quoted accuracy is derived from the statistics of the measurements and assuming that the ratio of the O and Si backscattering cross sections is given by the Rutherford scattering formalism. The lack of dependence on the O₂ flow is indicative for a similar saturation effect as has been suggested for the argon incorporation. Alternatively, the independence on the O₂ flow may be attributed to the O₂ flow independent on induced knock-on.

4. Conclusion

We have found that the concentration of Ar in reactive magnetrons sputter deposited SiO_x is in the range 0–5.3 at.% and depends in first order only on the x -value. For x -values higher than 0.3 the concentration of Ar decreases monotonically to almost zero for $x = 2$. In second-order, the concentration of incorporated Ar decreases slightly with increasing substrate temperature ($T < 400$ °C) during the deposition. Under the conditions of the present study, the observed Ar concentration is independent of the Ar⁺ ion bombardment on the growth surface. This observation can be understood by assuming a saturation in the equilibrium between implantation and desorption in and from the immediate surface region during deposition. For $x = 2$, i.e., in SiO₂ samples, argon is almost exclusively found in the deposited material in the interfacial region with the substrate in amounts in the order of 10^{15} cm⁻² decreasing with O₂ flow.

It is possible to grow SiO_x layers with x -values up to 2.05 and it is suggested that this oxygen excess is caused by O₂⁺ implantation or by knock-on implantation by ions bombarding the growth surface.

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