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Surface electrical degradation of helium implanted SiO_2

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Abstract

KS-4V quartz glass is a candidate material to be used in ITER diagnostic systems where it will play an important role as optical components and possibly as electrical insulation. In addition to neutron and gamma radiation, the material will be subjected to bombardment by low energy ions and neutral particles. Possible material damage has been examined by implanting He into KS-4V at different temperatures to simulate ion bombardment. The results are comparable with previous H implantation observations with severe reduction of both optical transmission and surface electrical resistivity. The electrical conductivity over the SiO_2 implanted zone increases by more than seven orders of magnitude. Such surface electrical and optical degradation is due to the loss of oxygen from the implanted surface, with the degradation depending strongly on implantation temperature.

1. Introduction

Oxide materials will be extensively used in ITER in heating and current drive, and diagnostic systems, where they will play important roles as electrical insulators, and RF and optical transmission components. These materials will be subjected to neutron and gamma radiation, and additionally to bombardment by low energy ions and neutral particles of energies between eV and keV as a consequence of neutron reactions and related sputtering at vacuum surfaces, as well as ionization and acceleration of the residual gas due to local electric fields. Low energy particles deposit most of their energy at or very near the surface, producing high levels of ionization, atomic displacement, and sputtering. Hence, the resulting local damage and consequent degradation of the physical properties at the vacuum surface could be high. In particular, optical transmission and electrical resistivity are important properties for heating and current drive, and diagnostic system applications. Such potential degradation needs to be assessed in detail.

One candidate material for use in ITER is a low OH silica known as KS-4V quartz glass. Severe electrical and optical degradation for proton (H) implanted KS-4V has been reported [1]. It was suggested that such degradation was possibly due to chemical oxygen reduction by implanted hydrogen. In the work presented here, the optical and electrical degradation of this material due to He implantation has been addressed as well as the oxygen loss. Optical absorption and surface electrical conductivity measurements have been performed before and after 54 keV He implantation at different temperatures, indicating that implantation temperature

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plays an important role. He and H implanted samples exhibit similar electrical and optical degradation showing that the process is not related to oxygen reduction by hydrogen.

2. Experimental procedure

The KS-4V quartz glass used in these experiments was kindly provided by the Russian Federation within the ITER programme. Three samples of approximately $10 \times 10 \times 0.9$ mm^3 were cut and optically polished, and implanted at 50, 250, and 450 °C with 54 keV He ions (He^+), 0.5 μA/cm^2, up to a dose of 10^{17} ions/cm^2, at the CIEMAT Danfysik 60 kV ion implanter. The average penetration depth for 54 keV He^+ in SiO_2 is about 0.44 μm with maximum penetration of 0.65 μm. Optical absorption from 195 to 3000 nm was measured before and after implantation in a Varian Cary 5 spectrophotometer. For the electrical properties, unimplanted and implanted samples were mounted in a system which permitted one to measure the surface electrical conductance in high vacuum (10^{-6} mbar) at temperatures between 15 and 450 °C, with a current sensitivity of 10^{-11} A. A 5 mm diameter aperture stainless steel collimator was placed in front of the surface sample to be implanted. To measure the surface conductivity, two platinum electrodes separated by 1.5 mm were sputtered onto the sample face to be implanted. In this way DC electrical surface conductivity for and implanted area of approximately 5×1.5 mm^2 was measured by applying 100 V between the electrodes. After implantation X-ray element analysis (depth profile about 2 μm) was performed with an Hitachi S-2500 scanning electron microscope.

3. Results

Fig. 1 shows the surface electrical current as a function of 54 keV He dose for different implantation temperatures. Up to a dose of 3×10^{15} ions/cm^2, the current measured at 50 and 250 °C is at or below the measurement limit (10^{-11} A). The higher current at 450 °C is probably due to ionic conductivity, as may be seen in Fig. 2 for the unimplanted sample. Above about 3×10^{15} ions/cm^2, a nearly exponential current increase with dose is observed for all three samples. By about 10^{17} ions/cm^2, the current increase begins to saturate for implantation at 50 and 250 °C, however, at 450 °C, saturation is not observed. In Fig. 2 electrical current as function of temperature for the unimplanted and implanted KS-4V samples is shown. The electrical conductivity for the unimplanted sample observed above about 280 °C is due to ionic conductivity corresponding to high activation energy (high slope). Surface electrical conductivity for the implanted samples is orders of magnitude higher and the activation energy far less (lower slope) consistent with electronic conductivity. Furthermore, the degradation is higher for higher temperature implantation.

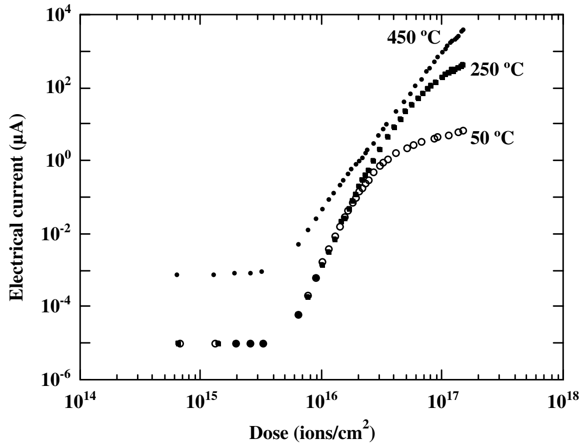

Fig. 1. Surface electrical current as a function of ion dose for different implantation temperatures.

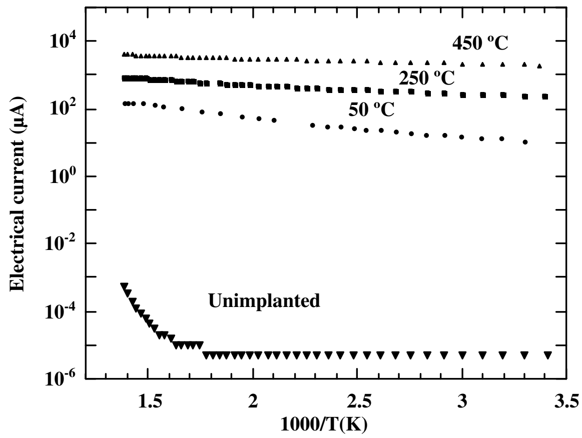

Fig. 2. Surface electrical current as a function of temperature for an unimplanted sample and implanted samples up to 1.5×10^{17} ions/cm^2 at 450 °C.

The induced optical absorption for three different implantation temperatures is given in Fig. 3. The absorption increase is monotonic for decreasing wavelength for all three temperatures, but the optical degradation markedly decreases with increasing

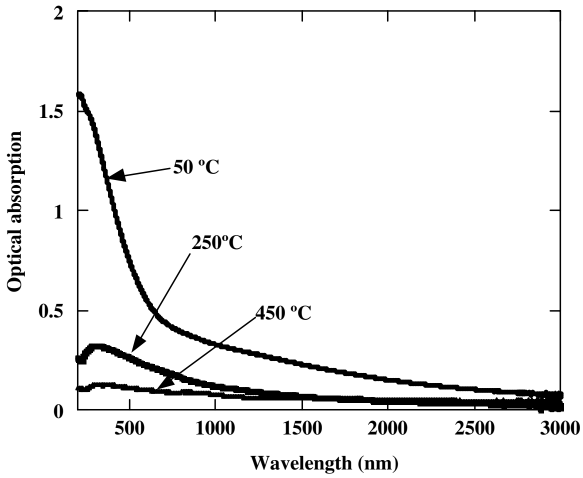

Fig. 3. Optical absorption spectra for samples implanted at different temperatures up to a dose of 1.5×10^{17} ions/cm^2.

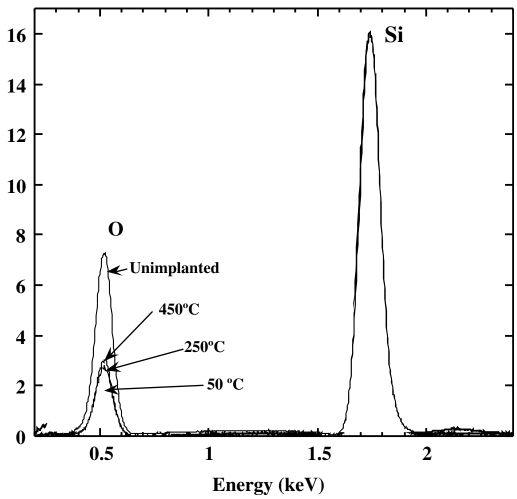

Fig. 4. SEM X-ray analysis for both the unimplanted and implanted zone of a sample implanted at 50 °C, 250 °C and 450 °C.

implantation temperature. Fig. 4 shows the SEM X-ray analyses for unimplanted and implanted KS-4V samples at 50, 250, and 450 °C. The oxygen and silicon peaks are clearly identified. The proportion of oxygen is reduced by 60% or more after implantation and depends on implantation temperature, being more marked at lower temperature.

4. Discussion

KS-4V surface degradation after He implantation is similar to that observed after H implantation, characterized by high electrical conductivity, enhanced optical absorption, and severe loss of oxygen [1]. Such similarity between He and H implantations indicates that this type of degradation is not due to any chemical process associated with the strong reductive power of hydrogen. The most likely mechanism inducing this type of surface degradation is preferential radiolytic sputtering of oxygen giving rise to oxygen vacancies in the surface region [2].

The He bombardment produces primary damage/modification to a depth of about 0.5 μm. It is clear from Figs. 3 and 4 that the observed optical degradation and oxygen reduction is largest for implantation at 50 °C. The large oxygen reduction (about 75%) implies that the surface region must contain substantial amounts of SiO and Si rich zones due to such a high concentration of oxygen vacancies. The optical absorption is in good agreement with that measured for μm thin SiO films [3], with a diffuse non-crystalline band edge smaller than the band gap energy of 9 eV for SiO_2 [4] . This marked reduction in the band gap helps to explain the fact that although the optical degradation and oxygen loss is largest at 50 °C, the electrical degradation is less and tends to saturation. The energy available in radiolytic processes is close to the material band gap energy [5] hence band gap reduction implies sputtering yield reduction. The situation at 250 and 450 °C is more complex because oxygen vacancies produced in the surface region can more easily thermally migrate into the bulk, and hence cause less structural modification. At higher implantation temperatures the density of oxygen vacancies in the near surface region ($\leqslant$1 μm) will be less, giving rise to less SiO and hence the band gap reduction and related sputtering yield will be less. This is in agreement with the X-ray SEM analysis where the apparent oxygen reduction is less for higher temperature, and the optical absorption at 250 and 450 °C shows no clear SiO absorption. The electrical degradation does not correlate with the optical degradation nor with the apparent oxygen reduction, it must therefore be related to the number of isolated oxygen vacancies which at higher temperatures are produced at a higher rate and are distributed over a larger surface volume.

The surface electrical and optical degradation reported here is an issue for both ITER and future fusion reactors where electrical insulators will be subjected to bombardment by low energy ions and neutral particles of energies between eV and keV as a consequence of neutron reactions and related sputtering at vacuum surfaces, and ionization and acceleration of the residual gas due to local electric fields. The flux and energy of the residual gas ions will be function of pressure, electric field, and gamma radiation levels. Work is under way in order to evaluate and study surface electrical degradation for other possible fusion reactor insulators, as well as to assess the realistic particle spectrum and flux due to residual gas ionization.

5. Conclusions

Severe electrical and optical surface degradation occurs when KS-4V silica is He implanted. The degradation is due to loss of oxygen from the surface caused by radiolytic sputtering by energetic ions. Electrical degradation is higher for the higher implantation temperature while the optical degradation is lower. This is foreseen as a serious technological issue for ITER and future fusion reactors where insulator vacuum surfaces will be bombarded by ions produced by gamma radiation and accelerated by local electric fields.

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