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Annealing effects on the migration of ion-implanted cadmium in glassy carbon

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ABSTRACT

The migration behaviour of cadmium (Cd) implanted into glassy carbon and the effects of annealing on radiation damage introduced by ion implantation were investigated. The glassy carbon substrates were implanted with Cd at a dose of 2×10^{16} ions/cm^2 and energy of 360 keV. The implantation was performed at room temperature (RT), 430 °C and 600 °C. The RT implanted samples were isochronally annealed in vacuum at 350, 500 and 600 °C for 1 h and isothermally annealed at 350 °C up to 4 h. The as-implanted and annealed samples were characterized by Raman spectroscopy and Rutherford backscattering spectrometry (RBS). Raman results revealed that implantation at room temperature amorphized the glassy carbon structure while high temperature implantations resulted in slightly less radiation damage. Isochronal annealing of the RT implanted samples resulted in some recrystallization as a function of increasing temperature. The original glassy carbon structure was not achieved at the highest annealing temperature of 600 °C. Diffusion of Cd in glassy carbon was already taking place during implantation at 430 °C. This diffusion of Cd was accompanied by significant loss from the surface during implantation at 600 °C. Isochronal annealing of the room temperature implanted samples at 350 °C for 1 h caused Cd to diffuse towards the bulk while isothermal annealing at 500 and 600 °C resulted in the migration of implanted Cd toward the surface accompanied by a loss of Cd from the surface. Isothermal annealing at 350 °C for 1 h caused Cd to diffuse towards the bulk while for annealing time >1 h Cd diffused towards the surface. These results were interpreted in terms of trapping and de-trapping of implanted Cd by radiation damage.

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1. Introduction

Glassy carbon (GC) is a micro-polycrystalline carbon material having combined glassy and ceramic properties with those of graphite. GC is a very stable material which does not transform to graphite at temperatures of up to 3000 °C [1]. Unlike graphite, glassy carbon has a fullerene-related microstructure [2]. It is an isotropic, continuous and non-porous material which is impervious [3]. GC exhibits attractive physical, chemical and mechanical properties. Its electrical resistivity is of the same order as that of regular carbon materials and its thermal conductivity is relatively high compared with that of common ceramic materials [4]. Its combined resistance to high temperature, corrosion and wear, together with low density, impermeability to gases and liquids makes glassy carbon more useful in many industrial applications such as vacuum evaporation sources, chemical containment, zone-refinement crucibles and more interestingly encapsulation of nuclear fuel elements [1,4]. GC has also been proposed to be used as a protective layer on the graphite reactor core surface in molten salt breeder reactors [5].

Cadmium (Cd) is used in reactor components such as control rods and neutron absorption shields, resulting in the formation of various radioactive isotopes including cadmium-113 m. Cd is a fission product which forms metallic precipitates whose fission yield is very low (about 0.0002%) [6]. This radioactive isotope is present in spent nuclear fuel and radioactive wastes associated with operating nuclear reactors and fuel reprocessing plants. Cadmium is a toxic heavy metal which can easily be dissolved by water and can damage the immune system, the central nervous system, and causes high blood pressure [7]. Therefore there is a need for proper containment of this fission product.

In the context of radioactive waste disposal related to the back end of the nuclear fuel cycle, we investigated whether glassy carbon can be a good nuclear waste containment material. For GC to be a good candidate for containment, it must be a good diffusion

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barrier for fission products such as Cd and its near-surface region structure must remain unchanged so that it retains its properties even under radiation conditions. In most of the above mentioned applications knowledge on Cd ion diffusion in GC is of crucial importance.

Previous work on ion species implanted in GC include Be [8], Cs [9], Sr [10], Co [11] K [12], Na [13], Ti [14], N [15], W [16] but after extensive literature research no data for diffusion studies of Cd implanted in GC was found. Most of these studies investigated the improvement of mechanical properties of GC surface layer after ion implantation [17].

In this study, the effectiveness of glassy carbon as a good storage material was investigated. Cd ions were implanted into GC substrates to a fluence of 2×10^{16} ions/cm^2 at room temperature and at higher temperatures (430 and 600 °C). Post ion implantation annealing experiments (isothermal and isochronal) of RT implanted samples were conducted to investigate behaviour of Cd in GC at a temperature range of 350–600 °C. The as-implanted samples and annealed samples were characterized by Raman spectroscopy and Rutherford backscattering spectrometry (RBS).

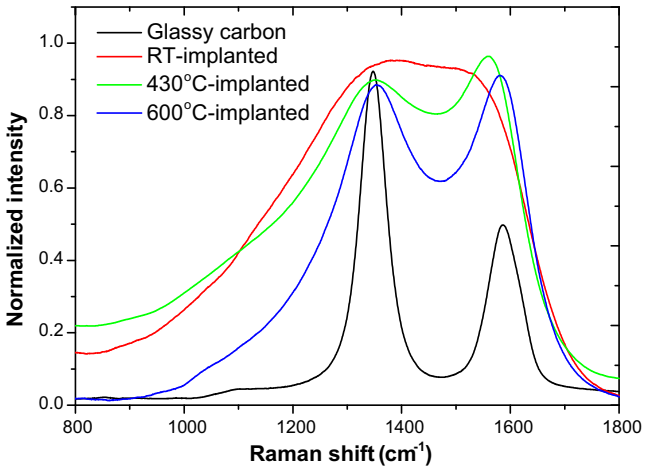

Fig. 1. Raman spectra of cadmium implanted into glassy carbon at room temperature (RT), 430 °C, and 600 °C, the un-implanted glassy carbon spectrum is included for comparison.

2. Experimental

The starting material was glassy carbon (Sigradur® G) which was mechanically polished to a mirror finish with a 1 μm diamond solution. Cadmium ions with energy of 360 keV were implanted into the polished glassy carbon surface, to fluence of 2×10^{16} ions/cm^2 at room temperature (RT), 430 and 600 °C. The flux was maintained at a rate of about 10^{13} cm^{-2} s^{-1} to minimize the increase in the substrate temperature. Some of the RT implanted samples were isochronally annealed in vacuum at temperatures of 350, 500, 600 °C for 1 h and some were isothermally annealed at 350, 500, 600 °C from 1 h to 4 h in step of 1 h.

The structure of the GC before implantation, the radiation damage retained after implantation and the effect of annealing were investigated by Raman spectroscopy. Raman spectra were recorded with a T64000 series II triple spectrometer system from HORIBA scientific, Jobin Yvon Technology, using the 514.3 nm laser line of a coherent Innova® 70C series Ar$^+$ laser (spot size ~2 μm) with a resolution of 2 cm^{-1} [18]. The samples were recorded in a backscattering configuration with an Olympus microscope attached to the instrument (using an LD 50× objective). The laser power was set at 1.5 mW. An integrated triple spectrometer was used in the double subtractive mode to reject Rayleigh scattering and dispersed the light onto a liquid nitrogen cooled Symphony CCD detector.

The distributions of cadmium implanted in glassy carbon before and after annealing were monitored by using Rutherford backscattering spectrometry (RBS) at RT using He$^+$ particles with energy of 1.6 MeV at a backscattering angle of 165°. The beam current was kept between 10 and 15 nA during the measurements. The Cd RBS profiles in energy channels were converted into depth profiles.

3. Results and discussion

Raman spectra of Cadium (Cd) ions implanted in glassy carbon at RT, 430 °C and 600 °C compared with the un-implanted glassy carbon in the 800–1800 cm^{-1} range are shown in Fig. 1. The Raman spectrum of the un-implanted sample shows the glassy carbon characteristic peaks of the D (at about 1350 cm^{-1}) and G (about 1590 cm^{-1}) bands representing the sp^3 and sp^2 bonds, respectively. After ion bombardment at RT, these two peaks merged into a broad single peak due to randomly distributed sp^3 and sp^2 bonds, indicating the amount of radiation damage retained after implantation. The randomly distribution of sp^3 and sp^2 bonds is termed disordered amorphous carbon. This implies that the RT Cd ions implantation induced amorphization in the near surface region of GC which has been previously observed [19,20]. Implantation at 430 °C resulted in broader D peak which became narrower after implantation at 600 °C. This indicates reordering of sp^3 bonds due high temperature implantations. The similar implantations (430 °C and 600 °C) also resulted into a shift of the G-peak from 1590 (of un-implanted GC) to 1564 and 1584 cm^{-1} respectively, indicating the recovery of radiation damage. These results indicate the presence of some sp^3 and sp^2 bonds after Cd implantation at these temperatures. The difference in the radiation damage retained after implantation with the same energy and fluence resulted in disordered amorphous carbon at room temperature and recrystallization of the GC due to self-healing/annealing occurring at high temperature implantations [9,20].

The RBS spectra of glassy carbon implanted with Cd ions at RT, 430 °C and 600 °C are shown in Fig. 2, surface positions of C and Cd are indicated by the arrows. Room temperature implantation resulted in the almost Gaussian distribution of implanted Cd. Implantation at 430 °C resulted in the broader profile that is skewed more towards the surface. This is due to Cd diffusion towards the GC surface during implantation at this temperature. More diffusion of Cd towards the surface accompanied by significant loss of Cd was

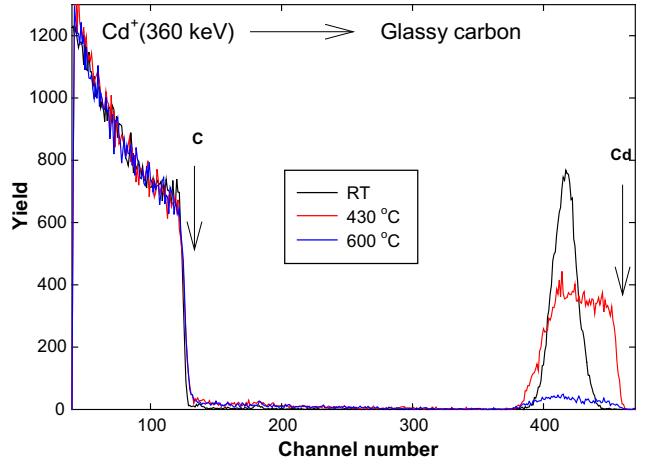

Fig. 2. Rutherford backscattering spectroscopy (RBS) spectra of cadmium implanted into glassy carbon at room temperature (RT), 430 °C, and 600 °C.

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