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In-situ RBS studies of strontium implanted glassy carbon

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ABSTRACT

The diffusion behaviour of strontium in glassy carbon was investigated using *in-situ* real time Rutherford backscattering spectrometry. The sample was annealed in vacuum from room temperature to 650 °C. Diffusion of the implanted strontium towards the bulk was observed after annealing at temperatures ranging from 450 °C–560 °C. The diffusion depth was limited to the end-of-ion-range region where there was still some radiation damage present. No diffusion into the pristine glassy carbon was observed suggesting that diffusion of Sr in glassy carbon can only occur in regions with radiation damage. Annealing the sample at temperatures higher than 560 °C resulted in migration of the implanted strontium towards the surface of the glassy carbon substrate. The amount of the accumulated strontium at the surface increased as the annealing temperature is increased. The RBS spectra obtained after annealing the sample isothermally at 650 °C for 2 h show that there was no further diffusion and accumulation of the strontium during this period.

1. Introduction

The environmental implications of burning fossil fuels for power generation have been well documented. Due to these environmental problems, there has been a need for a cleaner and more environmentally friendly source of power. Wind, solar and nuclear energy are cleaner ways of power generation [1]. Nuclear energy is perhaps the least favored source of clean power generation out of the trio due to the problem of nuclear waste storage, management and safety [2]. Due to the increase in energy demand over the years, there has been a resurgence of nuclear energy around the world including Africa.

The storage of nuclear waste especially the high level waste (HLW) including ^{239}Pu, ^{90}Sr, ^{93}Zr and ^{107}Pd has been a major concern for researchers [3]. Nuclear waste is typically stored by putting it in pools that are at least 20 feet deep or by keeping them in dry cask storages [4]. The dry cask storage systems are typically made from materials such as copper, stainless steel and titanium alloys. The drawbacks of using these materials include brittleness, corrosion and cost.

In this study, our aim is to propose a new and more suitable material namely glassy carbon for constructing the dry cask storage. Glassy carbon (GC) is an advanced allotrope of carbon which combines the properties of glass, ceramic and graphite. It has been described to be a purely sp^2 artificial carbon with superior features when compared to graphite [5]. Glassy carbon has been proposed as a favorable material for making nuclear storage cask because of the following properties: High temperature resistance, good corrosion resistance, hardness, strength and extremely high resistance to thermal shock [6,7].

In order for glassy carbon to be a good storage material, it must also be a good diffusion barrier for fission products and its structure must remain unchanged so that it retains its properties even in extreme conditions.

This paper aims to investigate the diffusion behavior of strontium in glassy carbon using *in-situ* and real time Rutherford backscattering spectrometry (RBS) during the heating process. Ion implantation was used to introduce strontium ions into the glassy carbon substrate. It was a preferred method in this study since the strontium depth profile and dosage could be controlled and monitored. Furthermore, it introduces radiation damage in the glassy carbon. For this project it is also important to investigate the diffusion of strontium in radiation damaged glassy carbon.

2. Experimental procedure

Commercial glassy carbon substrates were mechanically successively polished with 1 μm and 0.5 μm diamond solutions. The

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samples were cleaned and implanted with strontium ions by a 400 keV ion implanter at room temperature and at energy of 200 keV to a fluence of 1×10^{16} ions/cm^2. The maximum concentration of strontium present in the glassy carbon substrate was calculated to be about 5%. Because Sr forms a compound (SrC_2) with carbon, this concentration is below the solubility limit of Sr in glassy carbon.

The flux during implantation was kept at about 10^{13} cm^{-2} s^{-1} in order to keep the temperature during implantation low because of our interest in studying the effect of radiation damage on the diffusion of the implanted strontium.

Real-time RBS analysis was carried out *in-situ* in a vacuum chamber fitted with a copper heating stage. A thermocouple was attached to the vacuum system from the rear through a narrow hole in the copper heating stage so that the thermocouple tip was located exactly underneath the copper surface on which the sample was mounted. The thermocouple was calibrated by carrying out *in-situ* resistivity measurements. This was done by constructing a four-point probe made out of Ta wires. Resistance measurements were then carried out on Au/Si samples. The surface temperature of the Au/Si sample was measured as a function of the thermocouple temperature and the relationship between the measured temperature and the sample surface temperature was established. The temperature measured by the thermocouple was estimated to be less than 4 °C higher than that of the Au/Si sample surface temperature.

The setup allowed for the sample to be annealed and analyzed simultaneously over a wide temperature range. The RBS spectra were acquired with 1.6 MeV He^+ particles at a backscattering angle of 165° and the sample was tilted 10° towards the detector. The depth resolution for a step-function profile was about 6 nm.

The temperature profile illustrated in Fig. 1 shows the annealing regime used in this study. Previous studies have shown that diffusion of the strontium implanted in the glassy carbon substrates only starts occurring at 300 °C [8,9]. Therefore, the temperature of the sample was ramped quickly from room temperature to 300 °C; this took about 20 min. After the quick ramp, the temperature was then increased by 2 °C/min with the resulting RBS spectrum collected after every 2 min until the temperature rose to 650 °C. Isothermal annealing was also carried out by keeping the temperature constant at 650 °C for about 2 h. The resulting RBS spectra are shown in Fig. 2(a) and (b) respectively. The selected spectra of interest were also fitted using a Gaussian equation fit. The fitted spectra heights, projected range (R_p) and the straggling (ΔR_p) were plotted as a function of temperature.

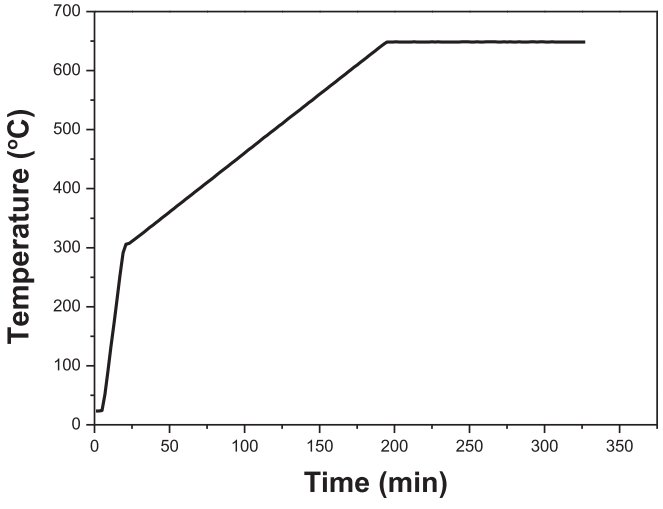

Fig. 1. Graph showing the temperature profile during in-situ RBS analysis.

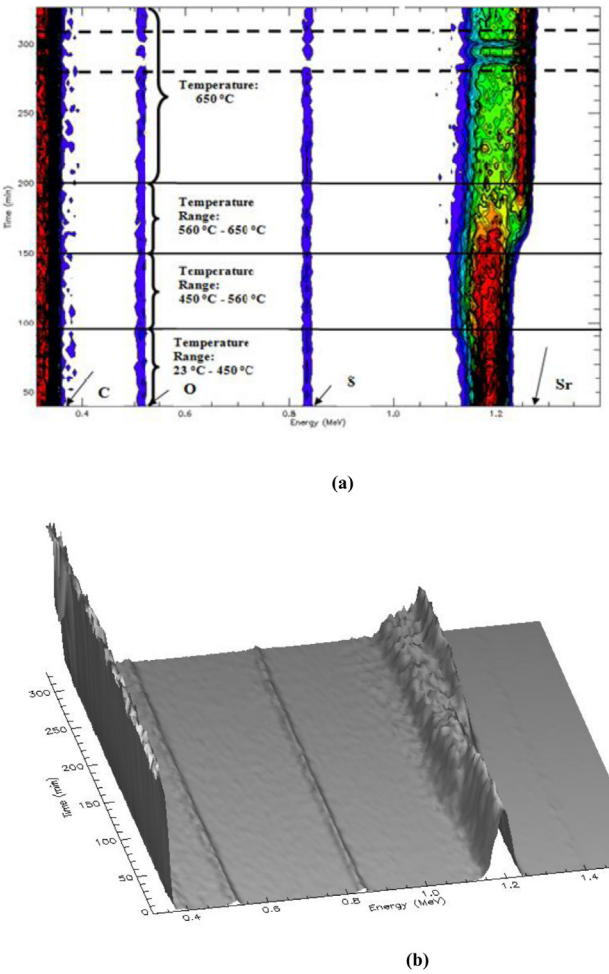

Fig. 2. (a) Contour plot representation obtained during real-time RBS analysis. The vacuum annealing was carried out in-situ. The arrows indicate the surface channels for all the atomic species present. (b) 3-D overlay of spectra obtained during in-situ real time RBS analysis.

3. Results and discussion

The resulting real-time RBS spectra obtained throughout the experiment are shown as a contour plot and 3-dimensional plot in Fig. 2(a) and (b) respectively. The contour plot gives a clear view on the diffusion behaviour of the implanted strontium. In the figure, four distinct bands which serve as representatives of the different ions in the system can be seen. The oxygen and sulphur lines that appear in the contour plots were also observed in a RBS measurement done on another virgin glassy carbon sample from the same batch obtained from the manufacturer. This suggests that they were present during the fabrication of the glassy carbon. The difference in the peak height is represented by different colours in the contour plot with red representing the areas with the highest counts and the green representing the area with the lowest counts. The yellow line is the intermediary height between the red and green lines. The contour plot also shows two dash lines which runs through the plot at 290 and 300 min respectively. At these times, the analysing charge was low, hence, the intensity of the RBS spectra recorded were low when compared to the other spectra.

The as-implanted depth profile was fitted with a Gaussian