# MODIFICATION OF GLASSY CARBON UNDER STRONTIUM ION IMPLANTATION

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**Abstract**.

***The effect of annealing on the surface morphology and on the diffusion of strontium ions implanted into glassy carbon is reported. Glassy carbon (GC) was implanted with strontium ions at an energy of 360 keV and a fluence of $2\times10^{16}$ ions/cm$^2$at room temperature. The sample was isochronally annealed in vacuum for 1h at 200 $^o$C, 300 $^o$C and 400 $^o$C respectively. Rutherford backscattering spectroscopy, Raman spectroscopy and scanning electron microscopy (SEM) were used to investigate and monitor changes in the sample. There was no visible diffusion of strontium after the sample was annealed at 200 $^o$C for 1h but after annealing at 300 $^o$C and 400 $^o$C respectively, the diffusion of Sr towards the surface of the GC was observed with a distinct Sr peak forming on the surface of the GC at 400 $^o$C. The Raman spectra showed that implantation of strontium damaged the structure of the glassy carbon but some of the damage was annealed out after heat treatment. SEM images of the sample showed that there is little change in the surface structure of the GC both after implantation and annealing.***

## 1. INTRODUCTION

The difficulty of radiation leakage from nuclear reactors during accident conditions, waste storage and management has made nuclear power unpopular around the world. One of the major features of the Pebble Bed Modular Reactor (PBMR) is its ability to contain radioactive fission products within the Tristructural-Isotropic (TRISO) fuel particles [1]. However, there is still a need to store spent fuels i.e. fission products. These fission products are extremely

dangerous if they escape into the environment, therefore it is important that they are stored in containers or storage made of a suitable material

Glassy carbon (GC) is a nuclear material with very distinct characteristics. It is a disordered form of carbon with high temperature resistance, high hardness and strength and chemical stability even in extreme environments [2]. Glassy carbon is also unaffected by nearly all acids and cannot be graphitized even at very high temperature [2,3]. Because of these characteristics, there is a need to investigate the modification of the structure and diffusion behavior of fission products in glassy carbon. This is to determine if glassy carbon will be a good diffusion barrier and storage material for nuclear fission products.

In this research, Strontium was implanted in glassy carbon (Sigradur® G) and the diffusion mechanism and structural modification was studied.

## 2. EXPERIMENTAL PROCEDURES

The glassy carbon was polished with 1μm diamond solution and then implanted with $^{84}Sr^{+}$ at energy of 360 keV and a fluence of $2\times10^{16}$ ions/cm$^2$ at room temperature.

The sample was then cut into small pieces of about 5 mm × 5 mm. A piece was vacuum annealed isochronally at 200 $^{o}$C, 300 $^{o}$C and 400 $^{o}$C for 1h.The diffusion pattern and distribution was determined by Rutherford Backscattering spectroscopy (RBS), using helium particles with energies between 1.4 and 1.6 Mev. Raman spectroscopy and the Scanning electron microscope (SEM) were then employed to investigate the effect of implantation and annealing on the microstructure of the Glassy Carbon.

## 3. RESULTS AND DISCUSSIONS

The Raman (D and G) peaks of the polished Glassy Carbon were very distinct and clearly resolved- see figure 1. However, after implantation, merging of the two peaks was noticed. This indicates that the glassy carbon was damaged due to the implantation of strontium at room temperature. The peaks reappeared after the sample was annealed; this implies that some of the damage was annealed out. the Although still merged, the peaks became slightly more prominent after the sample was annealed out; this implies that only some of the damage was annealed out.

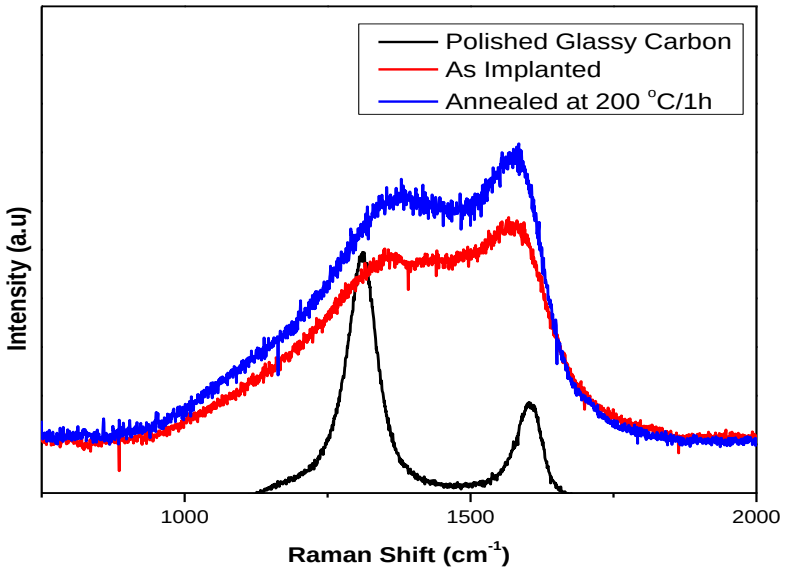
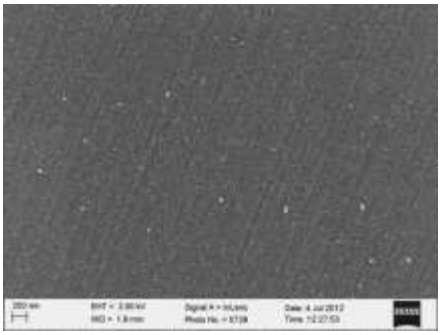
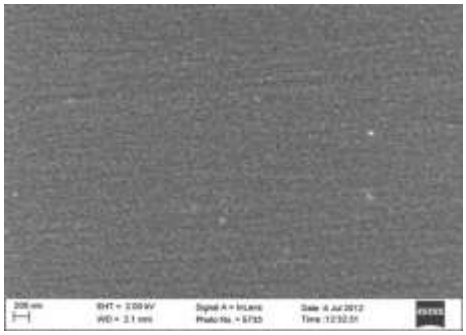



*Fig 1: Non- normalized spectra of glassy carbon after different heat treatment.*

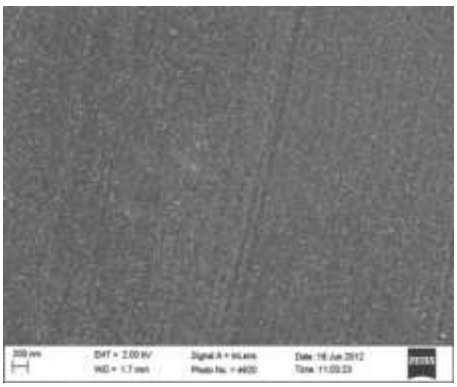
Because surface topography can affect RBS spectra, especially where broadening of peaks are of interest, we investigated the topography of our samples before analysis using SEM. Figure 2 show that the SEM images of the polished GC, as-implanted GC and the vacuum annealed GC. The polished GC has lots of mechanical scratches due to polishing. These scratches were reduced and the surface appeared smoother after implantation. However, after annealing the samples, the scratch marks reappeared and became more prominent compared to the as-implanted images. This was perhaps due to the sputtering of the lose carbons sitting on the surface of the GC during polishing. The polishing marks were still small enough to affect the RBS spectra.

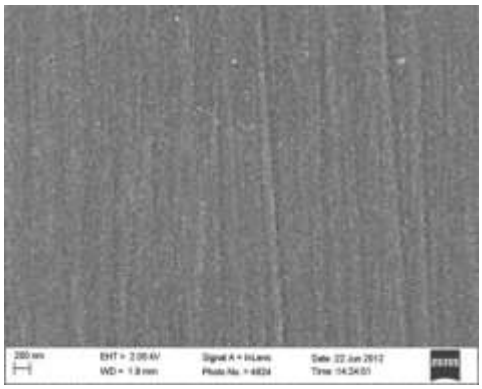




(a)

(b)





(C)

(d)

*Fig 2: SEM images of strontium implanted GC. (a) Polished. (b) As-implanted. (c) Implanted and annealed at 300 $^{o}$C for 1h. (d) Implanted and annealed at 400 $^{o}$C for 1h.*

Figure 3 illustrates the experimental profile and SRIM simulation [4] of GC implanted with strontium ions at room temperature. Also shown in Fig.2 is an edgeworth distribution fit to the experimental data. The experimental projected range of 231 nm was slightly lower than the theoretical estimate by SRIM of 271 nm. This discrepancy might be due to the inaccurate energy loss data used for the conversion of the RBS channels to depth and/or due to inaccurate interatomic potentials used in SRIM for glassy carbon.

Due to lack of suitable experimental values, data for graphite was used instead of glassy carbon. The projected range straggling was about 22 % higher than it was predicted by SRIM. The kurtosis (β) and skewness (γ) of the implanted distribution was almost Gaussian (β=3;γ = 0.036) for the SRIM and (β=2.5; γ = -0.5) for the experimental values. The negative gamma value derived from the experimental data showed that the implanted strontium peak was skewed to the left. This shows that even during implantation, there was already diffusion of strontium towards the surface of the GC.

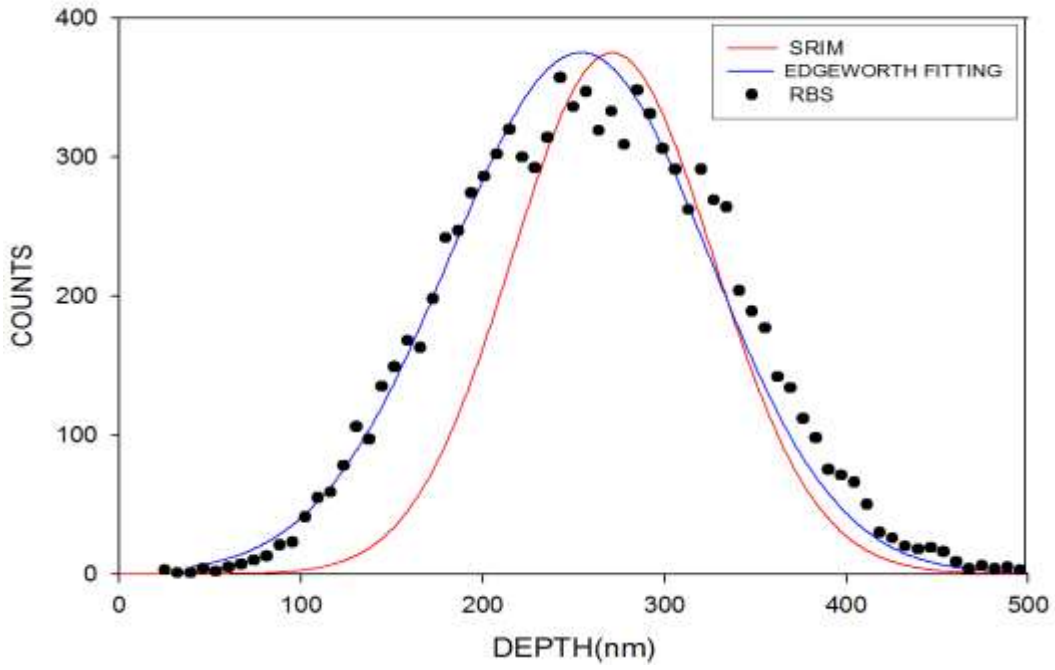
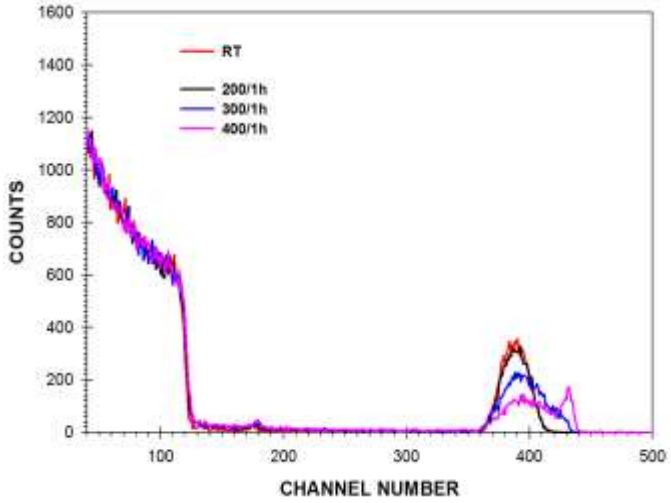



*Fig. 3: RBS spectrum of the as-implanted strontium peak compared to a SRIM simulation.*

The RBS spectra of vacuum annealed GC implanted with strontium are shown in Figure 4. After annealing the Sr-implanted glassy carbon at 200 $^{o}$C for 1h, no change in the RBS spectrum for the strontium wasdetected and the strontium peak remained approximately Gaussian. This indicates that no visible diffusion of the Sr occurred. After further isochronal annealing for 1 h of the sample to 300 $^{o}$C and 400 $^{o}$C respectively, the Sr profile became broader and skewed towards the surface of the GC. A distinct strontium peak was formed on the surface of the GC after annealing at 400 $^{o}$C. No broadening of the Sr profile towards the bulk of the GC was observed for both of these annealing temperatures. This indicates that diffusion of the implanted Sr occurred only towards the surface of the GC and not into the bulk of the glassy carbon. Since one can expect that most of the radiation damage induced by the bombarded Sr ions will roughly correspond to the implanted Sr profile, this diffusion towards the surface of the glassy carbon is an indication that the diffusion of strontium in glassy carbon was induced by the radiation damage. The Raman results confirmed that some

damage remained in the glassy carbon after annealing allowing this radiation-induced diffusion to occur.




## 4. SUMMARY

360 keV Sr ions were implated into glassy carbon to a fluence of $2\times10^{16}$ ions/cm$^2$ at room temperature. Raman spectroscopy was used to investigate the effects of implantation and heat treatment on the structure of the glassy carbon. It was observed that the sample bcame damaged after implantation with Sr-ions. After implantation, the Raman peaks (D and G ) of the glassy carbon merged, signifying damage in the material. After annealing at 200 $^{o}$C, the peaks reappeared although they were not as prominent as in the un-implanted glassy carbon. The reappearance of these peaks indicates that some of the damage was annealed out after heat treatment of the sample.

The diffusion of strontium implanted in glassy carbon was also investigated using RBS. Diffusion towards the surface of the glassy carbon occured at 300 $^{o}$C and 400 $^{o}$C. At 400 $^{o}$C, a distinct strontium peak formed on the surface. This diffusion/segregation towards the surface is probably due to radiation-induced diffusion. SEM investigations of the samples showed that the topography of the surface was small enough not to affect the RBS spectra.

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