Investigating the structural changes induced by SHI on W-SiC samples

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Highlights

- In this study, Tungsten (W) thin film was deposited on 6H-SiC wafer.
- The W-SiC samples were then irradiated with swift heavy ions at energies of 167 MeV.
- The XRD results indicated that W reacted with SiC to form WC and WSi_2 after irradiation.
- The W-SiC surface morphology before irradiation was uniform and flat.
- After irradiation with SHI the surface was more textured and had identifiable grains on the surface.
- The SiC before and after annealing had biaxial compressive stress.
- The as-deposited stresses, σ_{11} and σ_{22} for SiC were 536 ± 128 MPa and 531 ± 121 MPa, respectively.
- After Irradiation at the highest fluence of 10^{14} cm^{-2}, the stress increased by ~30%.

Abstract

The structural modification of tungsten-SiC samples irradiated with Xe^{26+} swift heavy ions (SHIs) was investigated. Tungsten (W) thin films were deposited on 6H-SiC using e-beam. After deposition, the W-SiC samples were irradiated by 167 MeV Xe^{26+} ions to fluences of 10^{12} cm^{-2}, 10^{13} cm^{-2} and 10^{14} cm^{-2} at room temperature. The sample composition, phase identification, residual stress component and surface morphology were investigated with Rutherford backscattering spectrometry (RBS), X-ray diffraction (XRD) and scanning electron microscopy (SEM). The results indicated that the as-deposited samples were composed of W and SiC, with no reaction between them. The samples irradiated to a fluence of 10^{12} cm^{-2} showed that a

reaction between W and SiC took place resulting in the formation of WSi_2 and WC phases. The samples irradiated to fluences of 10^{13} and 10^{14} cm^{-2} showed further reactions between W and SiC with WSi_2 and WC being the only phases formed. The SiC substrate had bi-axial compressive stress which did not excess 700 MPa after irradiating to the highest fluence. The W layer deposited on SiC was flat and homogeneous after deposition. A textured surface with identifiable grains was observed after the SHI irradiations.

Keywords: tungsten, thin film, SiC, SHIs, irradiation, reaction

1. Introduction

Metal-silicon carbide (m-SiC) composites have a vast load of technological applications such as in nuclear reactors, electronics and aerospace [1]–[5]. The metals commonly used in these applications are tungsten (W), palladium, zirconium, copper, cobalt and iron. The use of these metals is due to their exceptional physical, chemical, electrical and mechanical properties. In nuclear reactors and aerospace applications, these composites are continuously exposed to different irradiation at elevated temperatures. These irradiations include ions with energies in the swift heavy ion region i.e <1 MeV/amu. The investigation of m-SiC properties is still part of the ongoing research for many researchers [6]–[8]. These investigations are meant to enhance the already existing body of knowledge on m-SiC composites and further understand the composites behaviour in these severe environments.

Extensive work has focused on studying the interaction between metals and SiC caused by different deposition methods and annealing (heat treatment) [8]–[10], while others have focused on identifying the best method to deposit metal films on SiC [11]–[13]. More recently, a number of studies have focused on the effects of swift heavy ions (SHIs) irradiation on m-SiC composites [14]–[16].

As part of the thermal annealing studies, Goesmann and Schmid-Fetzer [17] investigated the stability of W electrical contacts on 6H-SiC by looking at interfacial reaction and microstructure. They deposited W thin films on 6H-SiC and the samples were annealed in vacuum between 4–29 days and analysed using scanning electron microscopy (SEM), energy dispersive X-ray analysis (EDX) and X-ray diffraction (XRD). Their XRD results showed that the as-deposited

samples contained mainly W_5Si_3 with some traces of W_2C, WSi_2 and WC indicating that W-SiC reacted during deposition. No changes in the phases present were observed after annealing the as-deposited samples at 1000 °C. Annealing at 1300 °C led to an increase in the volume of the WSi_2 and WC phases at the interface, which resulted in the decrease of the W_5Si_3 phase composition at the interface.

Njoroge et al. [16] reported on the solid state interactions between Zr and SiC induced by SHI irradiation. In their work they deposited Zr on SiC and irradiated with Xe^{26+} SHIs to different fluences (10^{12} cm^{-2} to 10^{14} cm^{-2}) at energies of 167 MeV. Their results indicated that the reaction between Zr and SiC took place after the initial irradiation to fluences of 10^{12} cm^{-2}. Further reactions were observed when the irradiation fluence was increased to 10^{14} cm^{-2}. Njoroge et al. [15] also reported on the interactions between Pd and SiC induced by SHI irradiation. Pd was deposited on SiC and the Pd-SiC sample was irradiated with Xe^{26+} SHI at different fluences from 10^{12} cm^{-2} to 10^{14} with energy of 167 MeV. They reported that irradiating with SHI at the fluences of 10^{12} cm^{-2} to 10^{13} cm^{-2} did not trigger any reaction. The samples irradiated to a fluence of 10^{14} cm^{-2} resulted in the reaction between Pd and SiC. Similar results were observed by Thabethe et al. [14] when they studied the effect of SHI on Pd-SiC samples. The researchers deposited Pd on SiC and irradiated the sample with Xe^{26+} SHI energies of 167 MeV. The fluences used were 1×10^{13} cm^{-2} and 3×10^{14} cm^{-2}. They observed a reaction between Pd and SiC after irradiating with a fluence of 10^{14} cm^{-2}.

To the best of our knowledge, no SHIs irradiation has been reported for W-SiC which is vital in the behaviour of this composite in harsh environments where it is used. However, the effect of SHIs (with energies of 120 MeV) in W-Si contacts has been investigated at different fluences [18]. The reaction between W and Si were only observed after irradiation at a fluence of 10^{13} cm^{-2}. The sample irradiated at a lower fluence of 10^{12} cm^{-2} was identical to the as-deposited. In this paper, the effect of SHIs irradiation on W-SiC was investigated and the following properties where studied: the structural changes, interaction between W and SiC and the stress on SiC caused by SHIs irradiation.

2. Experimental Procedure

The substrate used in this study were cut from a semi-insulating 6H-SiC wafer of 2 inch diameter, 330 μm thickness, with a micro pipe density of <10 cm^{-2} and a root mean square roughness surface roughness of < 0.5 nm. The substrates were then chemically cleaned before deposition. They were degreased by boiling them in tri-chloroethylene, methanol and deionized

water sequentially. They were etched by dipping them in hydrofluoric acid solution and dried with nitrogen gas.

The W thin films of about 10 nm were deposited on 6H-SiC substrate using electron beam deposition. The base pressure of the chamber was 5×10^{-7} mbar and pressure of the chamber during the evaporation process was 8×10^{-6} mbar. After the deposition, W-SiC samples were irradiated with Xe^{26+} swift heavy ions of 167 MeV at room temperature. The ion fluences were set at 1×10^{12}, 1×10^{13} and 1×10^{14} ions/cm^2. The irradiation was performed at the Joint Institute for Nuclear Research (JINR), Dubna using the IC-100 FLNR cyclotron. The as-deposited and irradiated samples were analysed using Rutherford backscattering spectrometry (RBS), X-ray diffraction (XRD) and scanning electron microscopy (SEM).

The RBS spectra obtained were simulated using the RUMP code [19] to obtain the thickness and elemental composition of the deposited layer. The energy of the He^+ ions used during the RBS analysis was 1.6 MeV. The silicon surface detector was set at a backscattering angle of 165° and beam current of 15 nA was used. The surface morphology of the W-SiC samples before and after irradiation were analysed using the Zeiss Ultra 55 field emission scanning electron microscope. The beam used was set at 1 kV so as to disclose surface features before and after irradiation.

A Bruker D8 Discover diffractometer was used for phase identification and residual stress investigation. The as-deposited sample was used to determine the angle of incidence with respect to the surface of the X-ray beam. Since the peaks of the single crystalline SiC substrate would be much larger than the reaction product peaks and would dwarf them. The angle was reduced from 5° to the point where the SiC peaks just disappeared. The final angle used for analysis was 0.3°. The diffractometer is equipped with a theta-2theta goniometer and ¼ Eulerian cradle. A copper tube (energy = 8 keV) operated in point focus mode was used for the analysis with the diffracted beam collected using an area detector, Vantec500. The primary optics included a 0.8 mm collimator mounted to a graphite monochromator, with no optics on the secondary side. Grazing incident X-ray diffraction (GI-XRD) was used for phase identification analysis. The stress-strain measurements were done in a side-inclination mode. To access the full stress tensor SiC at an angle of 49.7°, the plane (102) was measured at three azimuth angles ϕ, i.e. 0°, 45° and 90°. A set of 8 tilt angles, in steps of 10°, was measured for each azimuth angle. To access negative tilt angles, each ϕ was rotated by 180°. Measurements were done on two lateral points across the sample surface and a stress-strain determination was carried out using the manufacturer's proprietary Leptos v6.02 software.

3. Results and Discussion

3.1 Rutherford Backscattering Spectrometry

Figure 1 shows the as-deposited RBS spectrum (black) and the RUMP simulated spectrum (red) of the W-SiC sample. The as-deposited sample was composed of C, Si and tungsten elements with the surface channel positions indicated by the arrows in figure 1. The thickness of the W thin film obtained from RUMP simulation was about 10 nm. The Si and C peaks are below their normal surface position due to W layer deposition. There was no intermixing observed between W and SiC in the as-deposited samples.

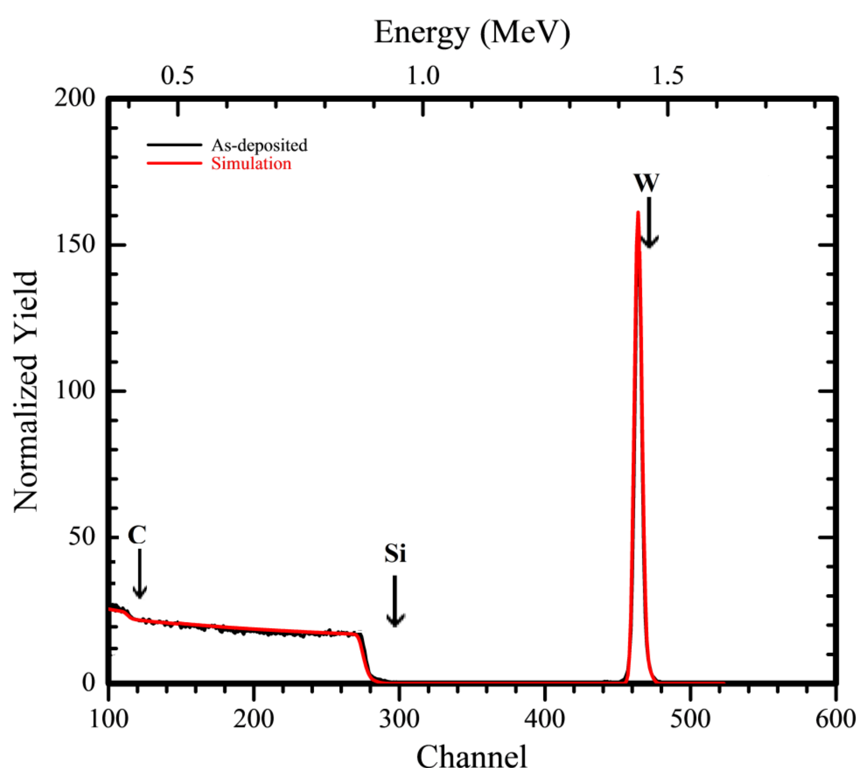

Figure 1: *RBS spectrum of as-deposited W thin film on SiC substrate, the black and red represent the measured and RUMP simulated spectra respectively.*

Irradiating the sample to a fluence of 1×10^{12} cm^{-2} indicated no significant change in the W peak (see figure 2). Irradiating to a fluence of 1×10^{13} cm^{-2} resulted in the reduction of the W peak height and slight broadening. Further reduction in the W peak height and broadening was observed after irradiating to a fluence of 1×10^{14} cm^{-2}. These reductions on W peak intensities and broadening after irradiating to fluences of 10^{13} cm^{-2} and 10^{14} cm^{-2}, were not accompanied by any visible change in the Si edge. In figure 2, the W peak after irradiating to fluences of 10^{13} cm^{-}

^{2} and 10^{14} cm^{-2} shifted slightly towards the bulk. The broadening of W peak towards the lower channels was an indication of interdiffusion at the W/SiC interface.

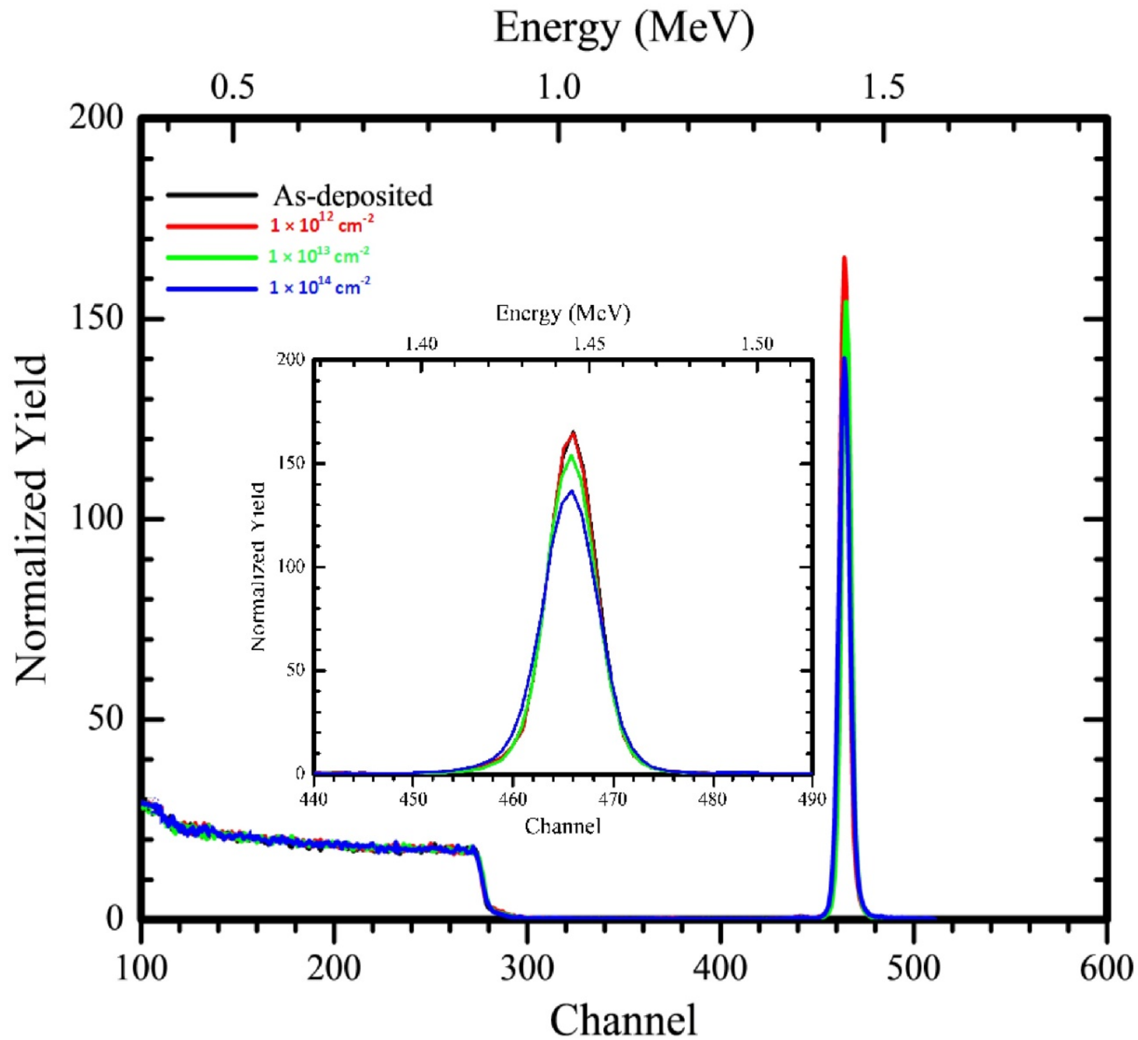

Figure 2: *The RBS spectra of the W-SiC samples before and after irradiation with* Xe^{26+} *swift heavy ions to different fluences. Inset of the W peak has been included.*

RBS depth resolution is usually between 5 – 10 nm. Because of the limitation, RBS results could not give us much information regarding the interaction between W and SiC. It was unclear as to whether the reduction in W was due to a reaction between W and SiC or it was caused by sputtering during SHI irradiation. The sputtering yield was calculated using SRIM to check whether the reduction on the W peak was due to sputtering. The sputtering yield of 3.32×10^{-4} nm and 3.32×10^{-3} nm was obtained for the fluences of 1×10^{13} cm^{-2} and 1×10^{14} cm^{-2} respectively. This sputtering value is too low to result in a visible change on the W peak. The integration of the tungsten peak before and after SHI irradiation was performed and was found to be almost the

same for all the samples. Since there was negligible sputtering, the changes observed in the W peak width might be an indication that some interdiffusion took place.

3.2 X-ray Diffraction

XRD at grazing incident geometry was performed to identify the phases present in the W-SiC samples before and after irradiation. Figure 3 shows the XRD patterns of W-SiC samples before and after irradiation. The XRD pattern of the as-deposited sample showed that the sample was composed of W and no impurities were detected. The W peaks were located at 2 theta positions of 40.3° and 73.2° indexed to (110) and (211) planes respectively. The peaks were small and broad, indicating that the W deposited on SiC was polycrystalline. The XRD pattern for the low fluence irradiated samples, i.e. 10^{12} cm^{-2}, indicated a reaction between W and the SiC had occurred. The reaction resulted in the formation of WC with diffraction peaks located at the 2 theta positions with the corresponding miller indices of 38.3° (002), 48.5° (101) and WSi_2 at position of 41.3° (111). The initial phases observed from our analysis after the first irradiation are in line with the phases predicted by Seng et al. [11]. They found that at lower temperatures (300 <T< 970 K) WC and WSi_2 are the dominant phases.

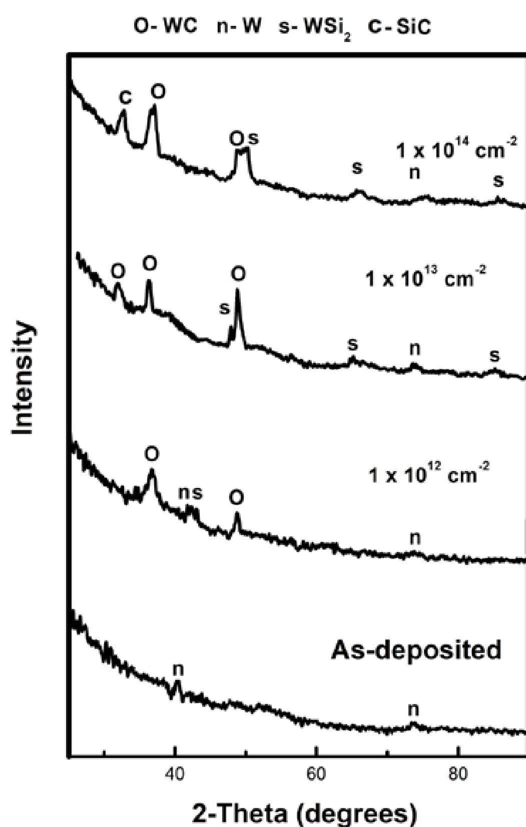

Figure 3: *The XRD patterns obtained from W-SiC samples before and after irradiating with Xe^{26+} swift heavy ions at different fluences.*

Irradiating the W-SiC sample with Xe^{26+} to a fluence of 10^{13} cm^{-2} resulted in the formation of four peaks at positions 31.7°, 47.9°, 68.2° and 85.3° attributed to WC (001), WSi_2 (112), WSi_2 (212) and WSi_2 (116), respectively. The formation of new peaks indicated that further reactions took place between W and SiC. An increase in the WC peak intensities at positions 38.3° and 48.5° was observed. The increase in peak intensity can be attributed to the crystal structure improvement of the polycrystalline WC phase caused by the SHI irradiation. The improvement in the crystal structure of the WC phase can be attributed to the increase in temperature caused by the irradiation. To confirm whether this is viable, the ion ranges and energy loss for W were calculated using the Monte Carlo computer programme SRIM-2013 [20]. The electronic energy loss for a 167 MeV xenon ions in W was 42.0 keV/nm, while the nuclear energy losses are 0.20 keV/nm. The electronic stopping was the dominant energy loss mechanism for the incident xenon ions and not nuclear stopping.

When SHIs traverse through a material they lose energy through localized electronic excitations (electronic energy loss) along the ion trajectories which induces temperature rise which leads to a transient melt known as a thermal spike [20]. This thermal spike raises the temperature of the W-SiC sample but it also rapidly cools.

The tungsten silicide peak at 47.9° overlaps with the WC peak at 48.5° creating a kink on the WC peak at position 48.5°. The WSi_2 phase probably consists grains with different orientations, resulting in the broadening and reduction in peak intensity of the WSi_2. The WSi_2 peak at 41.3° reduces in intensity and becomes broad.

The samples irradiated at a fluence of 10^{14} cm^{-2} had two additional peaks. SiC (015) at position 33.1°, which was an indication that a large amount of the W had reacted exposing the SiC substrate. The WSi_2 peak at position 52.3° with reflection (620) indicates further reaction between W and SiC took place. The change in the XRD patters after irradiating to fluences of 10^{13} cm^{-2} and 10^{14} cm^{-2} is due the grains orientation being different and allows for diffraction to occur from other planes.

The stress investigation was performed to study the residual stress caused by the SHIs irradiation on the SiC substrate. Table 1 depicts the normal stress components σ_{11} and σ_{22}. The results show that the observed stress was compressive and bi-axial for all the samples, but did not exceed -700 MPa. The measurements indicated that the stress on SiC was not homogeneous. The stress along the x- direction (σ_{22}) was more compared to the stress along the σ_{11} y-direction. The radiation-

induced stress on SiC was compressive. An increase in compressive stress of the irradiated samples was observed when compared to the as-deposited sample.

Table 1: *Residual stress and radiation-induced stress on SiC compared with as-deposited sample.*

Irradiation Fluence	Stress components (MPa)		Radiation- induced stress (MPa)	
	σ_{11}	σ_{22}	σ_{11}	σ_{22}
As-deposited	-536 ± 2.6	-531 ± 2.6	-	-
1×10^{12} cm^{-2}	-563 ± 19.9	-520 ± 20	-27	-11
1×10^{13} cm^{-2}	-609 ± 11.7	-650 ± 27	-73	-119
1×10^{14} cm^{-2}	-686 ± 10.6	-691 ± 3.0	-150	-160

We can explain the stresses in our samples (caused by the SHI irradiation) to be due to the mismatch thermal expansion coefficient values between W and SiC which results in the compressive stress seen in SiC. The coefficient of thermal expansion (CTE) of SiC is 2.8×10^{-6} K^{-1} and 4.5×10^{-6} K^{-1} for tungsten at room temperature (25 °C) [21]. This means that during SHI irradiation with the subsequent temperature increase, the W film expands faster compared to SiC because of its higher CTE value. During the rapid cooling processes, the thermal contraction of the W layer will cause compressive stress in the adjacent layer (SiC) and tensile stresses within itself. Also, the energy deposited by the SHI on W-SiC sample is used to trigger a reaction between W and SiC. This reaction results in residual stresses in both the W layer and SiC layer. Within the W layer, tensile stresses develop due to the formation of new phases causing expansion in the W layer, while compressive stresses will be generated in the SiC layer.

3.3 Scanning Electron Microscopy.

SEM analysis was done on the samples before and after SHI irradiation, and the images are depicted in figure 4. The images were taken in order to study the surface morphology of the samples. The as-deposited surface structure of W deposited on SiC was uniform and flat. This indicated that the W layer was evenly distributed on the SiC substrate. The samples irradiated with Xe^{26+} SHIs to fluences of 10^{12} and 10^{13} cm^{-2}, showed a slight change in the surface morphology. That is, the surface was more textured and had identifiable grains on the surface compared to the as-deposited sample surface. Irradiating the W-SiC sample at 10^{14} cm^{-2} resulted in the formation of patches on the surface which were lighter than the other parts of the surface. This was as a result of the reduction in the W quantity because of the reaction between W and

SiC. These results are in agreement with the XRD data of the sample irradiated at 10^{14} cm^{-2} where exposed SiC substrate was observed.

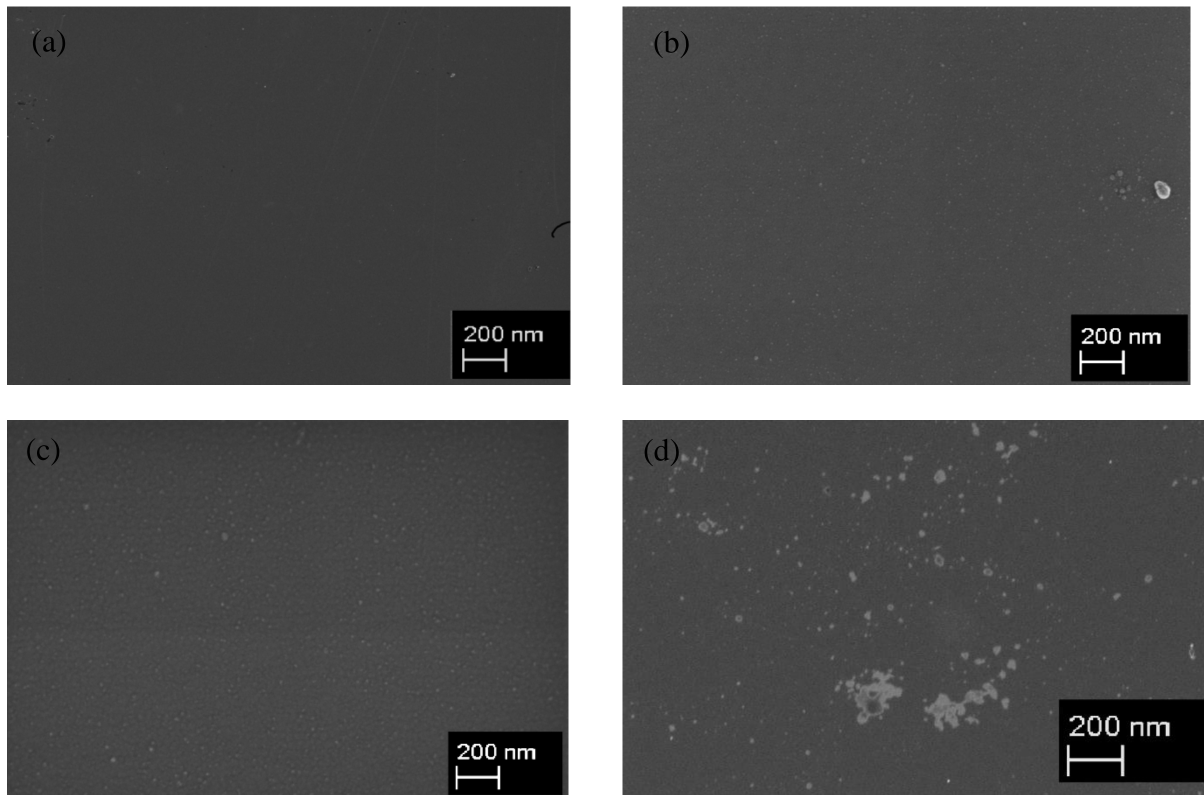

Figure 4: SEM micrographs of W-SiC samples (a) before and after SHI irradiation up to fluences of (b) 1×10^{12} cm^{-2}, (c) 1×10^{13} cm^{-2} and 1×10^{14} cm^{-2}.

4. Conclusions

The effect of irradiating W thin film deposited onto SiC substrate with Xe^{26+} SHIs was investigated. W thin films were deposited on SiC substrate at room temperature and irradiated with Xe^{26+} SHIs to different fluences. The RBS results indicated that the as-deposited sample was composed W, C and Si. The sample irradiated to a fluence of 10^{12} cm^{-2} did not show any significant change, while samples irradiated to fluences of 10^{13} and 10^{14} cm^{-2} indicated a broader W peak with almost the same number of counts which was attributed to the reaction between W and SiC.

The GI-XRD results showed only the presence of W on the as-deposited samples. Tungsten carbide and tungsten silicide phases were detected in the SHI irradiated samples. This indicated

that a reaction took place between W and SiC after irradiation. WC and WSi_2 where the only phases observed in all the SHI irradiated samples. This indicated that they are stable phases throughout the chosen irradiation fluences. SEM images before and after irradiation showed that the surface structure was composed of tiny W granules. A textured surface structured was observed for the irradiated samples when compared to the as-deposited sample.

The SiC before and after irradiation had biaxial compressive stress. The σ_{11} and σ_{22} stress components for SiC in the as-deposited sample were 536 ± 128 MPa and 531 ± 121 MPa respectively.After SHI irradiation to the fluence of 10^{14} cm^{-2}, the stress was seen to have increased by ~30%. The actual stress introduced by irradiation was compressive stress and increased with irradiation fluence.

5. Acknowledgement

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