THE EFFECTS ON HEAT TREATMENT OF SiC COATED / GRAPHITE PLATE BY RF SPUTTERING SYSTEM

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Silicon carbide film was coated by RF sputtering system onto graphite plate substrate. Surface roughness of substrate was polished by wet polishing with sand paper, number of 3000. RF power was 200 W in 0.4 Pa of argon gas. Deposition holding time was 3hours in 600 °C temperature. Specimens were heat treated by 1200, 1400 and 1500 °C for 1 hour to research the effect of heat treatment on SiC coating layer. Atomic Force Microscope (AFM) was used to characterize the surface morphology and the Sq surface roughness. AFM was used to investigate the grain size and roughness of the surface of thin coating film. X-ray diffraction (XRD) was used for detecting the coated SiC thin film by thin film mode. FE-SEM was used to characterize the microstructure of SiC coating film.

Keywords: SiC coating, RF sputtering system, Atomic force microscope, Heat treatment effects

1. Introduction

Silicon carbide (SiC) is a low activation and the high temperature material. SiC_f/SiC composite is a candidate material of blanket in fusion reactors. Helium Cooled Solid Breeder (HCSB) Test Blanket Module (TBM) is designed to give the simulated performance for DEMO-relevant blanket¹. Graphite pebble has been considered as a reflector material for neutron irradiation. During operation of fusion reactor, the graphite pebble has to endure the harsh environment of neutron irradiation. According to mechanical properties of graphite, the strength and hardness are not enough to endure the irradiation environment in fusion reactor.

SiC coating is considered as an ideal protective method in several industrial sectors because of the physical, chemical and mechanical properties, high thermal conductivity and enough strength to endure the neutron irradiation on fusion reactor²⁻⁴. It is a important reason that its characteristics of high melting temperature, high oxidation resistance at high temperature and relatively low thermal coefficient of expansion. SiC has been researched for a reinforcement material in other materials⁵⁻⁶.

In this study, the characteristics of SiC coating layer on graphite plate was researched. SiC coating layers were deposited by RF Sputter system. The specimens were heat treated by 1200, 1400 and 1500 °C for 1 hour to research the effect of heat treatment on SiC coating layer. It is possible that SiC crystal growth on graphite substrate above 1100 °C⁶. We expected dendrite structures of SiC developed on the original SiC film by heat treatment above 1100 °C. AFM and X-ray diffraction (XRD) were used to characterize the surface morphology and the Sq surface roughness. Field Emission Scanning Electron Microscope (FESEM) was used to observe the microstructure of SiC coating surface.

2. Experiment

The SiC coatings were deposited by RF sputtering from a SiC target onto graphite plating substrate at 600 °C temperature. The target size is 2 inch diameter while the substrate is 20 mm by 20 mm. The graphite plate was polished with sand paper, number of 3000, and cleaned by acetone and alcohol in an ultrasonic bath for 5 min.

The vacuum chamber was evacuated below 4×10^{-2} Pa. Pure argon (99.99%) was introduced into the vacuum chamber after the vacuuming. The argon flow meter was by 50 sccm to keep the vacuum in 0.4 Pa. The cylindrical chamber size is 450 mm diameter and 350 mm height. The distance between target and substrate was about 30 mm. The RF power was 200 W. The temperature of substrate was 600 °C and depositing time was 3 hours. The specimens were heat treated in vacuum chamber after the coating process. The heat treatment temperature were none-HT, 1200, 1400 and 1500 °C for 1 hour.

The surface morphology and the Sq surface roughness of SiC coated specimens were carried out with an atomic force microscope system in contact mode. Only one probe was used for analyzing all specimens in order to avoid the modification in the process of measurements. The probe consists of a V-shaped silicon nitride level and an integral

sharp tip. Both the level and tip are coated with gold. All the presented images are 1×1 μm square. The brightness contrast in the AFM images testifies the height of SiC particles. The Sq surface roughness was evaluated by using specific software. And the microstructure was observed with the FESEM for research the crystallization by the heat treatment on microstructure.

3. Results and Discussion

The roughness (Ra and Sq) of heat treated SiC coating layer is shown in figure 1. The SiC coatings were deposited on graphite plate substrate and heat treated at different temperature for 1 hour. The AFM image of heat treated SiC coating layer is presented in figure 2. The temperatures of heat treatment were none-HT, 1200, 1400 and 1500 $^{\circ}\text{C}$. Ra and Sq were decreased with increasing of heat treatment temperature. The surfaces were very smooth. The Ra and Sq roughness of no heat treatment surface were about 16.5 and 22.5 nm. Ra and Sq roughness were decreased to about 5.5 and 7 nm after heat treatment at 1500 $^{\circ}\text{C}$.

In figure 2, it shows that the surface became smoother at high temperature heat treatment. The particles of surface at no heat treatment was composed no shape with holes. SiC coating layer was composed of ellipse shaped particles after high temperature heat treatment. The average diameter of these particles were about 100 ~ 300 nm.

After 1200 $^{\circ}\text{C}$ heat treatment, the particles were composed of ellipse shaped and the average diameter was about 300 nm. These particles grow not absolutely perpendicular to the substrate. In case of 1400 $^{\circ}\text{C}$ heat treatment, the particles became smaller than 1200 $^{\circ}\text{C}$. The average diameter was about 200 nm. Ra and Sq roughness of SiC film were similar with the particles of 1200 $^{\circ}\text{C}$ heat treatment. But the particles of 1500 $^{\circ}\text{C}$ heat treatment were decrease to the average diameter about 50 nm and Ra and Sq roughness of surface were decreased to about 5.5 and 7 nm. Furthermore, the shape of particle was changed to globular shape.

SiC layer was coated onto graphite plate by RF sputtering system in XRD results in figure 3. According to figure 3, SiC peak list was not found on specimen before heat treatment. The Specimens after heat treatment show SiC peak list. SiC coating layer before heat treatment was amorphous. The SiC layer was re-crystallized by heat treatment.

Figure 4 shows the microstructure image by FESEM after heat treatment of SiC coating layer by RF sputtering method system with 50,000 times magnification. In the image of (a), no crystalline was shown, because the layer was amorphous before heat treatment. SiC coating layer was composed of thread shape in image of (b). The thin film was re-crystallized by high temperature heat treatment. The shape of SiC layer was changed to globular shape at 1400 and 1500 $^{\circ}\text{C}$ heat treatment. And the shape of 1500 $^{\circ}\text{C}$ was bigger than 1400 $^{\circ}\text{C}$.

Fig. 1 The roughness of Ra and Sq of heat treated SiC coating layer

Fig. 2 The AFM image of heat treated SiC coating layer

Fig. 3 XRD results of SiC coating layer

Fig. 4 The microstructure image of SiC coating layer

4. Conclusion

The characteristics of SiC coating layer by RF sputtering system was researched. Specimens were heat treated by no heat treatment, 1200, 1400 and 1500 °C for 1hr. AFM system and XRD was used to characterize the surface morphology and surface roughness. The microstructure was observed by FESEM.

In the results, sputtered SiC coating layer was re-crystallized by high temperature heat treatment. The roughness was decreased with increased temperature of heat treatment. The Ra and Sq roughness of no heat treated SiC coating layer were measured about 16.5 and 22.5 nm and decreased to about 5.5 and 7 nm after heat treatment at 1500 °C. The particles of 1200 °C heat treatment specimen were composed of ellipse shaped and the average diameter was about 300 nm in AFM results. According as the increasing of temperature in heat treatment, these particles grow to globular shape. The amorphous SiC

is coated by RF sputtering. The layer is re-crystallized by heat treatment in high temperature about 1400 and 1500 °C. In the SEM image, the thin film was re-crystallized by high temperature heat treatment. The shape was thread shape but changed to globular shape at 1400 and 1500 °C heat treatment.

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