Transactions of the Korean Nuclear Society Spring Meeting
Jeju, Korea, May 29-30, 2014

Effect of Ion Beam Bombardment on Vacuum Sublimation of Low Density Films

Jae-Un Kim, Jae-Won Park, Min-Hwan Kim, Sung-Deok Hong and Yong-Wan Kim*

Korea Atomic Energy Research Institute, Daejon-City, South.Korea
**Corresponding author: pjw@kaeri.re.kr*

1. Introduction

It is known that a commercial bulk SiC can be grown by sublimation at a temperature range of 1600 ℃ - 2500 ℃ under $1x10^{-3}$ torr to $2x10^{-2}$ torr pressure. In such a temperature regime, the film growth rate is as high as a few tens of µm/h. In this work, we found that the sublimation occurred even below 1000 ℃ under low pressure ($\leq 2x10^{-5}$ torr) conditions for films deposited by e-beam evaporation, although the sublimation rate was much lower.

The original object of this work was to develop SiC coating to protect metallic components to be used above 900℃ in a vacuum [1], but unexpectedly the coating experienced sublimation even at lower temperatures. In this work, therefore, we studied systematically the low temperature sublimation behavior of an e-beam deposited SiC film by annealing the film below 1000 ℃ under high vacuum, and in this endeavor, developed a method to reduce the sublimation through ion beam treatment.

2. Experiments and Results

2.1 Experiments

SiC films were deposited by electron beam evaporation on an Alloy HX sheet. The system used in this work is equipped with an e-beam evaporator and a 150 keV ion accelerator in the same chamber such that the coating and ion beam process could be conducted in-situ without breaking the vacuum. Prior to SiC deposition, the sample surface was sputter-cleaned. The base chamber pressure was $\sim 1x10^{-5}$ Torr and the operational pressure during e-beam deposition was $\sim 5x10^{-5}$ Torr. The SiC film was then deposited to a thickness of 50 nm by e-beam evaporation, followed by ion beam mixing with N^{+}-ions at 70keV to a fluence of $\sim 5x10^{16}$ ions/cm^2. Additional SiC was then deposited to make the total film thickness ~ 1 µm at a deposition rate of ~3 Å/s with ~0.15 A electron beam current. Subsequently, the deposited film was bombarded by 70 keV N^{+}-ions to a fluence of ~ $1x10^{17}$ ions/cm^2 to $4x10^{17}$ions/cm^2. The samples were then placed in an alumina boat and heated in a quartz tube vacuum furnace at temperature ranges of 550 ℃ to 950 ℃ for 5 to 10 hrs at ~ 1.5 $x10^{-5}$ torr. The metal specimens were weighed before and after SiC deposition, and before and after annealing to determine the density of as-deposited SiC and the sublimation rate of annealed SiC, using a micro balance with a readability of 0.01 mg (a repeatability of 0.02mg).

2.2 Results and Discussion

From the measurements of the weight changes, SiC film thickness, and the surface area of the sample, the density of the SiC film was found to be about 1.92 g/cm^3. This value is about 40% lower than that of the bulk SiC (3.217 g/cm^3).

The as-deposited SiC film showed a smooth surface (Fig. 1a), presumably amorphous, but transformed into well-defined faceted crystalline cones after annealing at 950 ℃ at $\sim 5x10^{-5}$ torr (Fig.1b). In addition, although the original alumina boat had coarse grains (Fig. 1c), much finer particles were decorated on the alumina boat in which the sample was placed (Fig. 1d). XPS analysis (Fig. 2) reveals that the chemical state of the particles shown in Fig. 1d is identical to that of the deposited film, indicating the apparent sublimation of the SiC from the sample and redeposition on the boat.

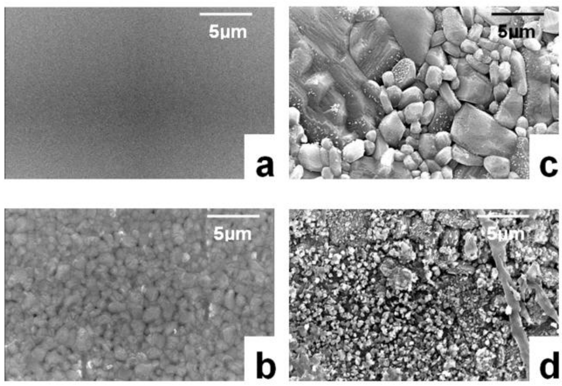

Fig. 1. Surface morphologies of as-deposited SiC film (a), SiC film annealed at 950℃under ~ 1.5 $x10^{-5}$ Torr pressure (b), as-received alumina (c), and deposition of sublimated SiC on the alumina surface (d).

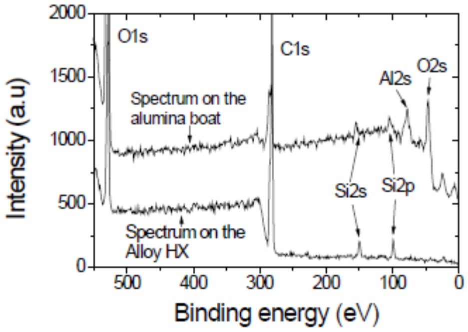

Fig. 2. XPS spectra :: Si and C exist on both surfaces of the Hastelloy X and the alumina boat.

In Fig. 3a, as-deposited specimens show no apparent weight loss below ~ 700 ^{o}C during a 5 hour annealing period, but the film weight loss increases drastically with increasing temperature, ~ 5% at 750 ^{o}C, ~ 7% at 850 ^{o}C, and ~ 30% at 950 ^{o}C. When the deposited film was bombarded by 70 keV N^+ ions to the a fluence of $1x10^{17}$ ions/cm^2 onto the deposited film, and then annealed under ~$1.5x10^{-5}$ torr pressure for 5 hr at 950 ℃, there were no green color deposits on the alumina boat after annealing signifying the reduced sublimation. Nonetheless, there was still ~ 10% weight loss at 950 ^{o}C despite the ion beam treatment. In the same way as above, the resultant sublimation rate can be estimated to be ~23nm/h. A significant finding is, however, that the weight loss was limited to ~10% up to a fluence of $4x10^{17}$ ions/cm^2 (Fig. 3b), and that there was virtually no further weight loss with extended heating beyond 5 hours (Fig. 3c). The 5 hour annealed specimen in Fig. 3c shows a somewhat higher ~14% weight loss, but its abnormality is believed to be due to an experimental scatter.

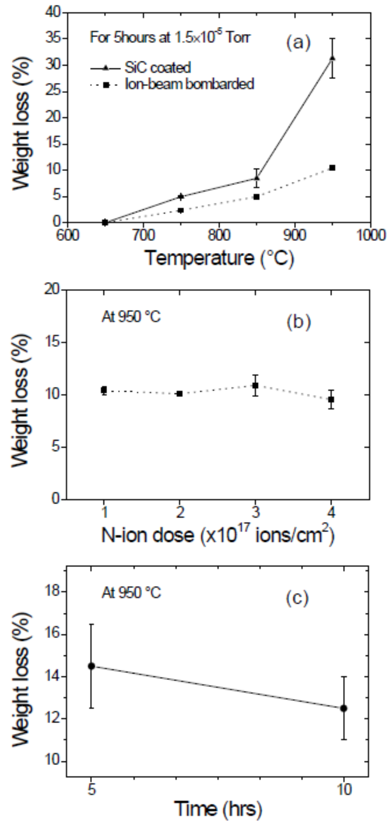

Fig. 3. An annealing of as-deposited SiC film at a pressure of ~1.5 x10^{-5} torr for 5 h shows no apparent weight loss below ~700 ℃, but the film weight loss increases drastically with increasing temperature, ~5% at 750 ℃, ~7% at 850 ℃, and ~30% at 950 ℃ (a). Ion beam treatment (70 keV, N ion) reduces the sublimation rate to ~10% at 950 ℃ at a fluence of ~1 x 10^{17} ions/cm^2, neither further decreasing as increasing the ion fluence up to 4 x 10^{17} ions/cm^2 (b) nor further reducing the weight loss rate with extended heating beyond 5 h (c).

3. Summary and conclusions

The sublimation behavior of the e-beam deposited SiC in a high vacuum was investigated to explore the potential use of SiC as a coating material for metals in a vacuum. However, the e-beam deposited SiC has a low density and sublimes below 1000 ℃, although its sublimation rate is low. This shortcoming is mitigated by ion beam packing.

Acknowledgement

This study was supported by Nuclear Hydrogen Development Project sponsored by Ministry of Education, Science and Technology, Republic of Korea

REFERENCES

[1] Jae-Won Park, Youngjin Chun, and Jonghwa Chang, J. Nucl. Mater. 362, Issues 2-3 (2007) 268