Interfacial Reactions of Ion Beam Mixed SiC film deposited onto Hastelloy X substrate

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1. Introduction

As we reported previously [1], the materials used for the SO_3 decomposer in Iodine-Sulfur cycle for Nuclear Hydrogen Production System require excellent mechanical properties as well as a high corrosion resistance in SO_2/SO_3 environment at an elevated temperature up to 950℃. So far, no metallic materials have been proved to be useful in such an environment. We have studied a surface modification of Hastelloy X by a SiC coating and an ion beam mixing. It is expected that only deposited SiC film is easily peeled off during heating at the elevated temperature. However, the SiC coating on Hastelloy X followed by ion beam mixing (IBM) is sustained at the elevated temperature above 900℃. The mechanisms of the good adhesion at the interface was studies using X-TEM, AES, XPS, XRD, X-SEM and EDS are presented.

2. Experiments

2.1 Coating and Ion Beam Mixing apparatus

An electron beam gun with peak power ratings 10 kV and 500 mA evaporate SiC source to deposite on the Hastelloy X substrate and an ion implantation setup with 120 kV and 15 mA is attached for the same vacuum chamber. The coating and the ion beam bombardment were done repeatedly to produce the less abrupt interface and to increase the density of the film.

2.2 Sample preparations

Hastelloy X sheets with dimension 20x20x0.5 mm were first polished by diamond paste up to 1 micron on both sides. In the vacuum work chamber, the sputter cleaning of the sample was performed for 10 minutes with N ion energy of ~10keV. Finally, the simultaneous evaporation of SiC and IBM treatment at 70keV was performed for about 500nm thickness. The coated coupon was later subjected to high temperature annealing investigations by annealing at 900 - 950℃ for 2 hrs in air or in vacuum and thermo-cycling in air at 400 - 900℃ for 12 hrs.

2.3 Secondary Electron Microscopy and Cross sectional transmission electron microscopy attached with an energy dispersive X-ray spectroscopy

For cross-section analyses of the IBM processed SiC coated Hastelloy X coupons, the filed emission transmission electron microscope (FE-TEM) modeled FB-2100F (HR) manufactured by JEOL Ltd., was used. Prior to XTEM analyses, the IBM processed specimen was prepared by state of the art dual beam focused ion (Ga+) beam (FIB) equipment modeled NOVA200, manufactured by FEI. For cross sectional SEM analysis, JEOL model JSM6300 was used.

2.4 Auger electron spectroscopy(AES)/X-ray photoelectron spectroscopy (XPS)

Scanning Auger microprobe Phi model 670 with an ion sputter gun was used for the line scanning across the film/substrate interface. XPS was conducted with Kratos Model AXIS-NOVA.

2.5 X-ray Diffraction(XRD)

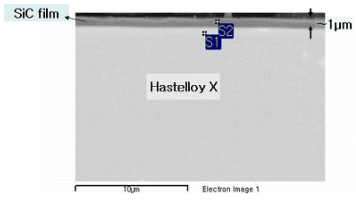
XRD was conducted with a Rigaku Geiger count diffractometer in this work. The characteristic X-ray was CuKα and a monochromatic beam was obtained by a curved single crystalline graphite monochromator. The step size for data acquisition was 0.01° in 2θ and X-ray tube voltage and current were 40KV and 30mA, respectively.

3. Results

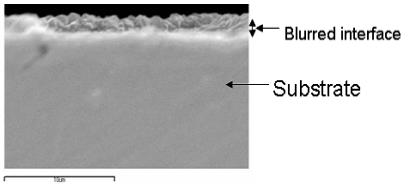
3.1 X-SEM and X-TEM with EDS

The cross section of the 1 μm SiC film deposited on Hastelloy X sample shows a clear interface between the film and the substrate (Fig. 1a). However, the IBM treated and annealed sample shows a blurred interface (Fig. 1b).

XTEM image and EDS elemental mapping for Cr, Ni, Fe, Mo, and Si near the film/substrate interface regime are shown in the Fig.2. It is evident that a significant Cr out-diffusion from the Hastelloy X substrate to the SiC film is observed. In addition, Fe, Ni, Mo , and O elements are also found in the deposited.



(a)



(b)

Fig. 2. Cross-sectional SEM images of as-deposited SiC/ Hastelloy X sample (a) and IBM SiC /Hastelloy X after heating (b).

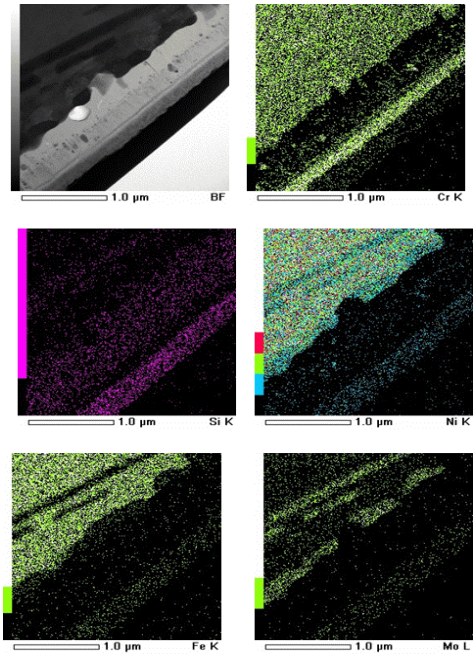


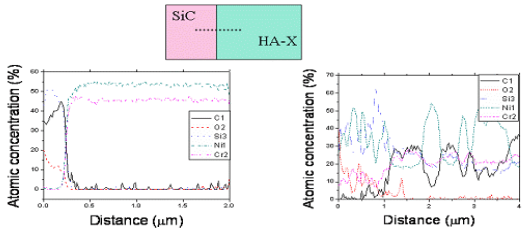
Fig. 1. X-TEM image and EDS elemental mapping at the film/substrate interface

Table.I. EDS analysis result from the sample cross section

	C	O	Si	Cr	Fe	Ni	Total
S1	30.49	3.36	5.58	15.00	12.52	30.24	100.00
S2	55.52	20.16	17.64	1.98	1.45	3.24	100.00

3.2 AES/XPS

Fig. 3 shows an Auger line scanning data obtained across the interface on the cross sectional SiC/Hastelloy X sample, in which as-deposited SiC film on Hastelloy X is abrupt (Fig. 3a) while the interface is not clearly seen in the IBM treated SiC/Hastelloy X after heating (Fig. 3b).



(a) (b)

Fig. 3 Auger line scanning across the interface.

Fig. 4 shows Cr existence on the surface of SiC film, which suggests Cr was out-diffused from the Hastelloy X substrate to the surface of SiC film.

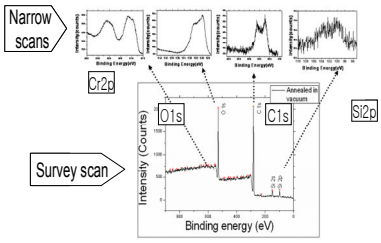


Fig. 4 X-ray photoelectron spectroscopy

3.3 X-ray Diffraction

The Hastelly X FCC peaks, SiC FCC peaks, amd SiO_2 formed on the surface of the deposited film were identified. However, there are many unidentified peaks (arrow marked) in Fig.5. which stand for the compounds formed among Ni, Cr, Si, C and/or O.

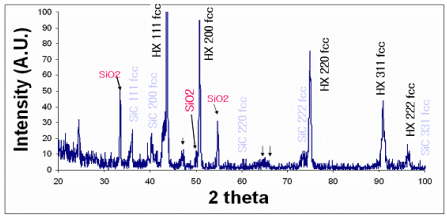


Fig. 5 X-ray diffraction pattern

4. Summary and conclusions

IBM plays a role of fastening the SiC film on the Hastelloy X substrate until the interfacial reaction takes place.. Once the reaction takes place, new phases are developed at the interface under the consumption of the film and the substrate materials.

ACKNOWLEDGEMENTS

This work was sponsored by the Nuclear Hydrogen R & D Program and Proton Engineering Frontier Project through the Korean Ministry of Education, Science and Technology. Some of the ion beam irradiation work was done using 400keV ion accelerator in TIARA (Takasaki Ion Accelerators for Advanced Radiation Application), Japan Atomic Energy Agency, Japan.

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