

The Preparation of NiFe₂O₄ Thin Film Prepared by E-Beam Evaporation Method for Simulated Specimen in PWR System

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1. Introduction

Decontamination technology is necessary to reduce radioactive component and contamination of complex equipment during decommissioning of nuclear power plants. There are a number of methods for decontamination process including chemical, electrochemical, foam, and mechanical contamination technologies. Among them, decontamination foam is considered to have potential application for the cleaning of radioactive contaminants from metallic walls, overhead surfaces, and complex components [1]. For the effective decontamination, the understanding of the fundamental mechanisms responsible for corrosion and stress corrosion of austenitic and nickel base alloys is necessary. Austenitic 304L and 316L stainless steel owe their high corrosion resistance in water to the formation of a continuous oxide layer, the passive film, only a few nanometer thick at ambient temperature, strongly enriched in Cr(3⁺) and separating and protecting the alloy from the corrosive medium [2,3]. Many researches have been investigated the influences of various parameters such as water chemistry, materials composition and stress, on the initiation and growth of stress corrosion cracking. Most studies reported the observation of a duplex oxide layer with a Fe-rich outer layer and a Cr-rich inner layer. The outer oxide layer is composed of magnetite (Fe₃O₄) or iron-nickel spinel oxide. For the Cr-rich inner layer, composition and phase are subject to controversy.

In this study, we prepared the NiFe₂O₄ film on the stainless foil (SUS 304) using E-beam evaporation technique using NiFe₂O₄ target. Compared to the other technique, the materials can be simply deposited by E-beam evaporation and is capable of providing advantages such as large area deposition, convenient replacement of source targets, and controllability of precise deposition thickness as low as 1 nm.

2. Experimental

NiFe₂O₄ thin film was deposited on stainless foil (SUS 304, thickness 25 μm) by using an electron beam evaporation system at room temperature. NiFe₂O₄ pellet (purity 99.9%) was used as a target. Prior to deposition process, the chamber was evacuated down to 3 x 10⁻⁶ Torr. The deposition rate and thickness of NiFe₂O₄ thin film were 0.5 Å/s and about 490 nm monitored by a thickness sensor during the evaporation process. The substrates were sputter-etched with Ar ions for 5 min before deposition in order to remove any oxide layer on the SUS foil surface. For the high crystallinity of NiFe₂O₄ thin film, as-deposited sample was annealed at 700 °C for 1h in Ar atmosphere.

The morphology of the surface and cross-section of NiFe₂O₄ thin film was investigated by a field emission scanning electron microscopy (S-4800, Hitachi) working at 30 kV. A high resolution transmission electron microscope with energy dispersive spectroscopy (JEOL-2100F HRTEM) with a 200 kV operating voltage was also used to capture the morphology and elemental analysis of the NiFe₂O₄ sample. The thin film X-ray diffraction (X-pert PRO MRD, Philips) pattern was conducted with Cu Kα radiation (λ = 1.5406 Å) operating at 40 kV and 30 mA between 10° and 90° at a scan rate of 0.01°, 2θ/min. Raman measurement (NTEGRA SPECTRA, NT-MDT) was conducted with a Ar laser excitation source emitting at wavelength of 514 nm. The X-ray photoelectron spectroscopy (K-alpha, Thermo VG Scientific) analysis was performed to evaluate the chemical status of the thin films and the binding energy was referenced to the C 1s peak from carbon at 284 eV.

3. Result and discussion

The crystallographic structure and phase purity of

as-prepared sample are examined by X-ray diffraction (XRD). Fig. 1 shows the XRD patterns of NiFe_2O_4 thin film after heat treatment at 700°C for 1h. The XRD pattern of as-deposited film was also shown for comparison. Three broad peaks with low intensity at 37° , 43° , and 62° in the as-deposited sample were observed, which is corresponding to the amorphous phase of the Ni and Fe oxide. The amorphous phase is could be due to the low energy for crystal formation during deposition process. However, when the sample was heated at 700°C , the film began to crystallize at high temperature as indicated by the most intense peak (311). This is assigned to those of the NiFe_2O_4 cubic spinel, which could be indexed to the inverse spinel (NiFe_2O_4 , JCPDS Card # 10-0325). The spinel oxide (440) peak indicated the spinel oxide dominated by FeCr_2O_4 . No peaks related to any other phase or impurities could be observed. After the heat treatment, the reflections become narrower with an increase of crystallite size. The impurity peak at 45° was also observed, indicating the stainless foil (denoted by star).

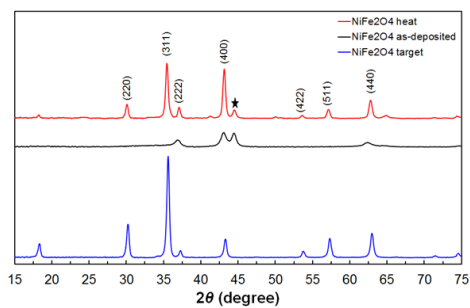


Fig. 1. XRD patterns of as deposited and heat treatment of NiFe_2O_4 and NiFe_2O_4 target.

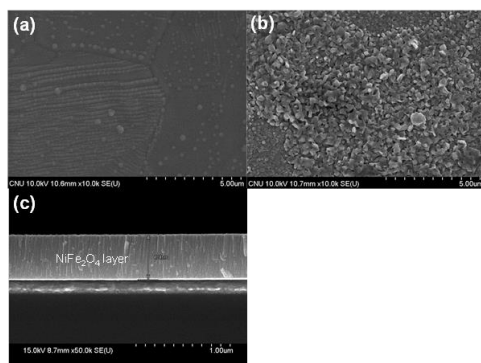


Fig. 2. SEM images of the NiFe_2O_4 thin film; (a) before and (b) after heat treatment, (c) cross-sectional view of NiFe_2O_4 thin film, respectively.

SEM images in Fig. 2a-c clearly show the surface of the as-deposited NiFe_2O_4 thin film and cross-section image, respectively. Fig. 2a and b shows the

surface morphology of NiFe_2O_4 thin film before and after heat treatment at 700°C . As shown in Fig. 2a, NiFe_2O_4 thin film present smooth surface along with base metal and a few droplets is observed due to the incomplete elimination of target splashing during deposition. After heat treatment, the NiFe_2O_4 thin film shows different morphology suggesting that the film may be undergoing change of orientation. The crystallized grains with increase in size are also observed. For the thickness measurement, the NiFe_2O_4 thin film was also deposited on indium tin oxide (ITO) glass. Fig. 2c shows the NiFe_2O_4 thin film which is vertically grown on the ITO glass without any cracking and thickness of film was about 484 nm.

4. Conclusion

Equations should be placed on separate lines and numbered:

REFERENCES

- [1] L. Nunez, L. Kaminski, Foam and gel methods for the decontamination of metallic surface, US patent, 166, 758 (2007).
- [2] V. Maurice, W.P. Yang, P. Marcus, J. Electrochem. Soc. 145, 909 (1998).