



Microstructure and He desorption behaviors of He charged FeCrNi-based films fabricated by direct current magnetron sputtering



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ABSTRACT

He-charged FeCrNi-based films were prepared at different temperatures in a mixed atmosphere of He and Ar by direct-current magnetron sputtering method. X-ray diffraction and energy dispersive spectrometry analysis confirmed the typical austenitic structure of the deposited FeCrNi films and the compositions were in good accordance with 304 stainless steel target. Cross-sectional scanning electron microscopy images revealed the dense columnar nanocrystalline structure of the fabricated FeCrNi films. Nanoindentation measurements showed that the film fabricated at 300 °C exhibited the highest hardness value of 11.5 GPa. He desorption from FeCrNi-based films was traced by thermal desorption spectroscopy; the relatively low He desorption temperature range (150 °C–450 °C) implied that the charged He atoms were mainly located in interstitial sites of FeCrNi-based films.

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1. Introduction

FeCrNi-based austenitic alloys, especially the AISI 316 and 304 stainless steels [1], have been widely used as the main structural material in tritium storage containers and nuclear reactors because of their outstanding combination of mechanical formability with good high temperature strength as well as oxidation and corrosion resistance. However, performance degradation under neutron irradiation for austenitic stainless steels had been reported, which inevitably compromises their serviceable lifetime [2,3]. One of the main reasons associated with irradiation induced performance degradation was attributed to helium (He) embrittlement. Owing to hydrogen isotope decay, (n, α) reaction or bombardment of α plasma particles, He atoms can be easily generated and accumulated in FeCrNi-based structural material [4,5]. These accumulated He atoms are prone to be captured by various defects because of their insolubility and high mobility in metals, leading to the formation of He–vacancy (He–V) aggregations [6,7]. With the increase of accumulation amount, He atoms captured by defects have a strong tendency of precipitating into bubbles, which will consequently cause performance deterioration of structural material, such as swelling and embrittlement [8,9]. Therefore, a detailed investigation linking He accumulation in structure to properties of austenitic steel is very important.

To be able to introduce He atoms into investigated materials, traditional techniques including radioactive decay of tritium in metal tritides [9], neutron irradiation [10] as well as He-ion implantation [11] are frequently used. However, because of the radioactivity and requirement of special equipment, rigorous preparations are necessary for such helium introduction methods. For example, although radioactive decay of tritium in metal tritides can uniformly introduce He atoms into materials, tritium has a long half-life radioactive period of 12.3 years, making it hard to be widely applied. As for neutron irradiation, serious irradiation induced damage will be caused, resulting in trapping of transmuted He atoms by concurrent irradiated defects and thus appending the complexity of He behavior investigation [12], especially for interstitial diffusion of He atoms. Ion implantation method has been used to investigate the irradiation effects on various structural materials, but it is difficult to obtain uniform distribution of He atoms with satisfying depth owing to its limited irradiation range.

Magnetron sputtering method under a gas mixture of He and Ar, initially reported by Mattox and Kominak [13], had been recently developed as an easy and convenient technique to introduce He into metal films, such as He charged Ti film and Al film by Liu et al. [12,14], Jia et al. [15] and Shi et al. [16]. Through magnetron sputtering in a He/Ar mixed atmosphere, the He concentration introduced into titanium films can be even up to 45 at.% with uniform distribution [12]. It was reported that during the sputtering process, the sputtering yield of He atom to metal was much smaller than that of Ar atom, but the backscattering probability of He atom by the heavy nuclei was greatly

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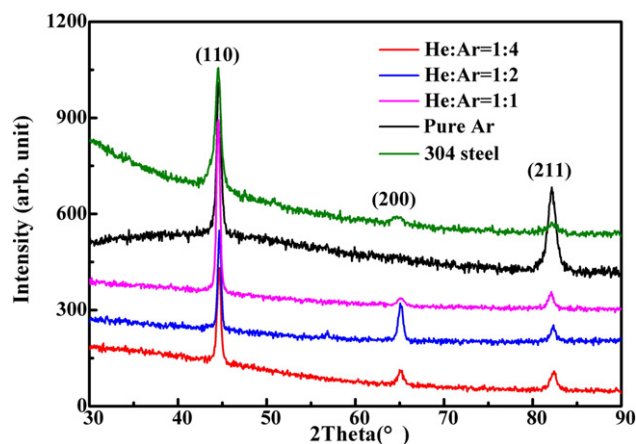


Fig. 1. XRD patterns of FeCrNi-based films fabricated under different He/Ar ratios at room temperature on Si substrate. XRD pattern of the bulk 304-stainless steel used as target is also presented for comparison.

larger than that of Ar atom [12,17,18]. In this condition, the role of ionized Ar in magnetron sputtering was mainly to sputter out the target atoms for depositing films, while the ionized He that bombarded the target would be backscattered and implanted into the growing film. Moreover, the He concentration can be conveniently controlled by varying He/Ar ratio.

In this investigation, He charged FeCrNi-based films were prepared by direct current (DC) magnetron sputtering method in a mixed atmosphere of He and Ar, and commercial 304-stainless steel was chosen as the sputtering target. Microstructure and He behavior of FeCrNi-based film were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), thermal desorption spectroscopy (TDS) and nanoindentation measurement, respectively.

2. Experimental

Cylindrical disc of about 3 mm in thickness and 60 mm in diameter cut from a commercial 304-stainless steel bulk was used as the sputtering target. The target was polished on abrasive papers of 5 types of roughness: P800, P1500, P2000, W10 and W5, sequentially. The silicon (Si) single crystal sheets with a (111) preferred orientation were chosen as substrates for the characterization of surface and cross-sectional morphology, nanoindentation and microstructures of the deposited films. After being cut into small pieces, Si substrates were boiled in cleaning fluid ($\text{HCl}:\text{H}_2\text{O}_2:\text{H}_2\text{O} = 1:1:6$) at 70 °C for 10 min and then rinsed by deionized (DI) water for several minutes.

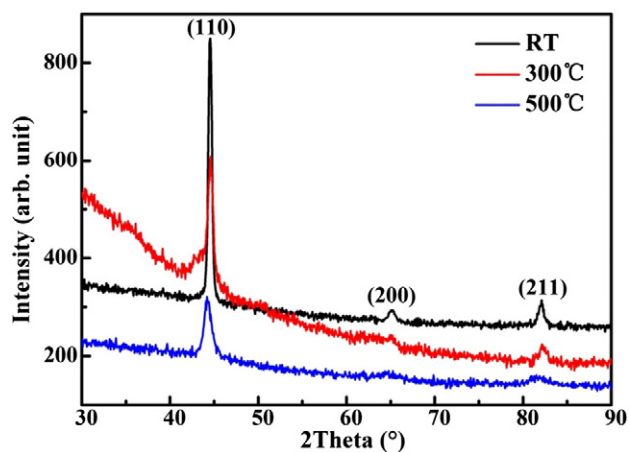


Fig. 2. XRD patterns of FeCrNi-based films fabricated on Si substrates at three different temperatures and He/Ar = 1:1.

Table 1

The main chemical composition of 304-stainless steel used as target evaluated by energy dispersive spectrometer.

Element		Cr	Ni	Si	Mn	Fe
Content	wt. (%)	18	9	0.7	1	Bal.
	at. (%)	19	8	1.4	1	Bal.

The pretreated silicon substrates were further ultrasonically rinsed in ethanol for 10 min, blow-dried and then stored separately for different usage.

He-charged FeCrNi-based films with a thickness of 5–7 μm were fabricated on Si substrates through DC magnetron sputtering method in a mixed atmosphere of He and Ar. The amounts of He and Ar in the sputtering chamber were monitored by two separate mass flow controllers in order to control the He concentration implanted into FeCrNi-based films. Meanwhile, the substrates were heated by a resistance furnace to adjust the depositing temperature of the films. During the sputtering process, the ionized He will be scattered and deposited into the films, which is similar to the preparation of He containing Ti film and Al film reported by Liu et al. [12] and Jia et al. [14]. Prior to depositing He-charged FeCrNi-based films, the working chamber was evacuated to a pressure of below 8.0×10^{-4} Pa using a turbo molecular pump. 304-stainless steel target was initially pre-sputtered in Ar atmosphere in order to remove metallic oxides on the polished target surface. During the process of film preparation, the total He/Ar mixed pressure in the chamber was adjusted to about 1.0 Pa and the sputtering power applied to the target was controlled at 60–70 W. To obtain FeCrNi-based films with different He concentrations, He/Ar ratio was adjusted at 0, 1:4, 1:2 and 1:1, modulating He partial pressure at 0, 20%, 33% and 50%, respectively. The temperature of the substrates was controlled at room temperature (RT) as well as at 300 °C and 500 °C heated by an electric resistance furnace, respectively.

Structural property of the films deposited on Si substrates was characterized by X-ray diffractometer (XRD, A Philips X'Pert PRO) at a small incident angle of 1° with a $\text{Cu K}\alpha$ radiation (wavelength: 0.15418 nm). XRD patterns were collected in a step-scanning mode of 0.06°/step over an angular range of 30°–90° at 1 s per step. Surface morphology and chemical compositions of the prepared films were analyzed by field emission scanning electron microscopy (FESEM, Sirion 200FEG) equipped with an energy dispersive spectrometer (EDS, Oxford INCA). The accelerating voltages for SEM and EDS analysis are 10 keV and 15 keV, respectively, and the detector for SEM image is a secondary electron detector. Cross-sectional microstructure of FeCrNi-based films deposited on Si substrates was observed on fresh and natural fractures by breaking down the substrates with scratches marked on the backside.

Table 2

Weight and atomic percentage of Cr, Ni, Si, Mn and Fe elements in FeCrNi-based films fabricated under different He/Ar ratios at room temperature (RT), 300 °C and 500 °C, respectively.

T (°C)	He:Ar	Cr wt./at. (%)	Ni wt./at. (%)	Si wt./at. (%)	Mn wt./at. (%)	Fe wt./at. (%)
RT	0	18/19	9/8	0.8/1.6	1/1	71/70
	1:4	18/19	8/7	0.7/1.4	1/1	72/71
	1:2	18/19	8/7	0.7/1.4	1/1	72/71
	1:1	18/19	8/7	0.6/1.2	1/1	72/71
300	0	18/19	8/7	0.6/1.2	1/1	72/71
	1:4	18/19	9/8	0.7/1.4	1/1	71/70
	1:2	17/18	8/7	0.7/1.4	1/1	73/72
	1:1	18/19	8/7	0.7/1.4	1/1	72/71
500	0	17/18	9/8	0.6/1.2	1/1	72/71
	1:4	18/19	9/8	0.8/1.6	1/1	71/70
	1:2	18/19	9/8	0.7/1.4	1/1	71/70
	1:1	18/19	9/8	0.6/1.2	1/1	71/70

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