Journal of Nuclear Materials 481 (2016) 73–80

Contents lists available at ScienceDirect

Journal of Nuclear Materials

journal homepage: www.elsevier.com/locate/jnucmat

Be/W and W/Be bilayers deposited on Si substrates with hydrogenated Fe-Cr and Fe-Cr-Al interlayers for plasma facing components

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HIGHLIGHTS

- Be/W and W/Be bilayers were deposited on Si with hydrogenated Fe(Cr-Al) interlayers.
- Metallic Fe, Fe and W oxides and other binary phases were found in the interlayers.
- Specific atomic intermixing processes in Be/W and W/B structures were reported.

ARTICLE INFO

Article history:
Received 28 April 2016
Received in revised form
7 September 2016
Accepted 9 September 2016
Available online 10 September 2016

Keywords:
Plasma facing components
Be/W and W/Be bilayers
Atomic interdiffusion
XPS
CEMS
GIXRD
XRR

ABSTRACT

Be/W and W/Be bilayers, of interest in regard to the specific behavior of plasma facing components (PFCs) were deposited on Si substrates by thermionic vacuum arc, with Fe, Fe-Cr and Fe-Cr-Al interlayers. The interlayers, with compositions approaching the one of the reduced activation steels used in supporting PFCs, were subsequently annealed in hydrogen atmosphere. The multilayers were characterized with respect to morphologic, structural, diffusional and atomic intermixing aspects via XRD, XRR, X-ray photoemission spectroscopy and Mössbauer spectroscopy. All as-prepared samples present partially amorphous structures. A main α-Fe phase is observed, as well as (superparamagnetic) secondary Fe oxides, metallic Fe with Si, Cr, W and Be neighbors, Be-rich Fe-Be and Fe-Si phases. High amounts of tungsten and tungsten oxides were also evidenced in the Fe layer. The strong atomic intermixing of W and Be layers was indirectly supported by the unusual densities of W and Be layers and ^{57}Fe Mössbauer spectroscopy results.

1. Introduction

Plasma facing components (PFCs) [1–3] are part of the test blanket modules in tokamak-type fusion reactors, such as ITER [4], which is expected to start plasma experiments in 2020, and future DEMO applications [5,6]. These complex materials are envisioned for operation in extreme temperature and irradiation conditions, with minimal damages on their lifetime and device performances. However, the droplet spraying from melted material from the PFCs caused by plasma-wall interactions may induce contamination of the plasma core and negatively influence the plasma discharge [7,8].

Tungsten-based materials, presenting low sputtering yields under ion bombardment, high melting point, good thermal conductivity, low tritium retention, are good candidates for plasma facing components materials [9,10]. However, the impact on the plasma core is significant. On the other hand, light elements such as Be [11], present low influence on the plasma core but have high sputtering yields. Therefore, a viable approach for the top structure of the PFCs would be a heterostructure composed of these two materials with very distinct behavior. In order to improve the mechanical properties of the top layers, which are greatly affected by high temperatures, special reduced-activation ferritic-martensitic (RAFM) steels (e.g., Eurofer) [12–14], are used as underlayers [15].

During fusion experiments, an important effect induced by temperature and irradiation is the redeposition of intrinsic

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http://dx.doi.org/10.1016/j.jnucmat.2016.09.010
0022-3115/

elements on the PFCs (some elements contained in the PFCs, such as Be or W or in the bottom structures, like Fe and other elements in the RAFM steels). The new structures may have different properties related to the designed PFCs. It is expected that such processes (induced by annealing and irradiation) will have a negative influence on the mechanical properties of the entire structures and a good knowledge of their effects is of great importance. Also, the generation of displacements per atom, inducing dislocation loops and transmutation reactions (which can generate He and H) may affect the brittleness of the steels.

In order to give more insight into redeposition processes possible to occur in PFCs, investigations of atomic intermixing in specifically designed thin films and multilayers represent a suitable approach. In some of our previous works [16,17], effects of diffusion and atomic intermixing in as deposited and thermally annealed Be/W and Be/C bilayers as grown on Si(0 0 1) substrates with Fe thin interlayers, have been studied. The influence of special hydrogenation treatments of alloy films approaching the composition of Eurofer (Fe-Cr based layers) has also been previously investigated in Refs. [18,19], emphasizing a higher degree of crystallization and an increased diffusion of different atoms in the alloy structure.

The purpose of this paper is to study the effects induced by atomic intermixing and diffusion in some thin multilayers which might be considered as case studies for components in nuclear fusion technology, such as complex structures of Be/W and W/Be bilayers deposited on Si substrates with Fe-Cr and Fe-Cr-Al interlayers. The characterization of the local phenomena and interfacial intermixing in the Be/W and W/Be layers deposited on Si substrates was made by means of grazing incidence X-ray diffraction (GIXRD), X-ray reflectometry (XRR), X-ray photoemission spectroscopy (XPS) and conversion electron Mössbauer spectroscopy (CEMS).

2. Experimental details

Fe, Fe-Cr and Fe-Cr-Al thin films (4 nm thick), partially enriched in the ^{57}Fe Mössbauer isotope, were deposited for 3 minutes on Si(0 0 1) substrates by radio-frequency (RF) sputtering. The substrate was held at room temperature, the discharge power was 100 W and the working Ar pressure was 5×10^{-2} mbar. The native Si oxide layer was not chemically removed from the substrate, but an etching process of 30 min was applied before deposition. The films were subsequently reduced in hydrogen atmosphere (the label of samples obtained after hydrogenation carries the suffix "_h"), prior to the deposition of the desired bilayer structure, in order to improve the crystallinity of the films and to reduce oxidation. A commercially available volumetric Sievert apparatus provided by Advanced Material Corporation, Pittsburgh, USA has been used in this respect. The hydrogenation was performed at 300 °C for 90 min under 20 bars H_2 pressure after 20 subsequent cycles of vacuum cleaning and purging in hydrogen at a lower temperature. According to our previous experience, the hydrogenation degree is very sensitive to the film thickness and, while errors of at least 10% in this parameter are expected among subsequently obtained Fe films, their phase composition was analyzed via Mössbauer spectroscopy prior to any subsequent deposition. Two different bilayers, Be/W and W/Be structures, were deposited on the Fe, Fe-Cr and Fe-Cr-Al interlayers by means of the thermionic vacuum arc (TVA) method which consists of plasma ignition in the vapors of the metallic anodes (Be, W) under vacuum conditions. The vaporization of the anode material is continuously maintained by electron bombardment [20–22]. The sample codes (related to films deposited on the Si substrates) and treatments performed prior to the Be/W and W/Be deposition are shown in Table 1.

Table 1
Sample codes (related to films deposited on the Si substrates) and treatments performed prior to the Be/W and W/Be deposition.

Sample codes	Treatments
Fe/Be/W	–
Fe/W/Be	–
$Fe_{0.89}Cr_{0.11}$/Be/W	–
$Fe_{0.89}Cr_{0.11}$/W/Be	–
$Fe_{0.84}Cr_{0.11}Al_{0.05}$/Be/W	–
$Fe_{0.89}Cr_{0.11}$/Be/W_h	hydrogenated
$Fe_{0.89}Cr_{0.11}$/W/Be_h	hydrogenated
$Fe_{0.84}Cr_{0.11}Al_{0.05}$/Be/W_h	hydrogenated

Both grazing incidence X-ray diffractometry (GIXRD) and X-ray reflectometry (XRR) measurements were performed with a Bruker type (AD 8 ADVANCED) diffractometer working with Cu(K_α) radiation of 0.154 nm wavelength, in order to derive the phase composition and the film thicknesses. The Bruker LAPTOS software package was used for fitting the XRR spectra whereas the qualitative analysis of the diffraction pattern was performed via the EVA program.

The X-ray photoemission spectroscopy (XPS) measurements were performed in a surface science cluster (Specs) also comprising a photoelectron spectroscopy chamber. The base pressure in all ultrahigh vacuum (UHV) chambers was in the 10^{-10} mbar vacuum range. The depth profiling using XPS was performed in an analysis chamber equipped with a 150 mm Phoibos hemispherical electron energy analyzer, a dual anode (Mg/Al K_α) X-ray gun, a monochromatized (Al K_α/Ag L_α) X-ray source and a high power UVS 300 UV lamp. A flood gun operating at 1 eV electron energy and 100 μA electron current was employed to ensure sample neutralization for all measurements. Monochromatized Al K_α radiation was used for this experiment (1486.7 eV). The analyzer operated in fixed analyzer transmission (FAT) mode with pass energy of 30 eV. Etching for depth profiling was performed with a high intensity Ar^+ gun (IQE 12/38) generating a ion current of 10 μA at the sample surface, under an

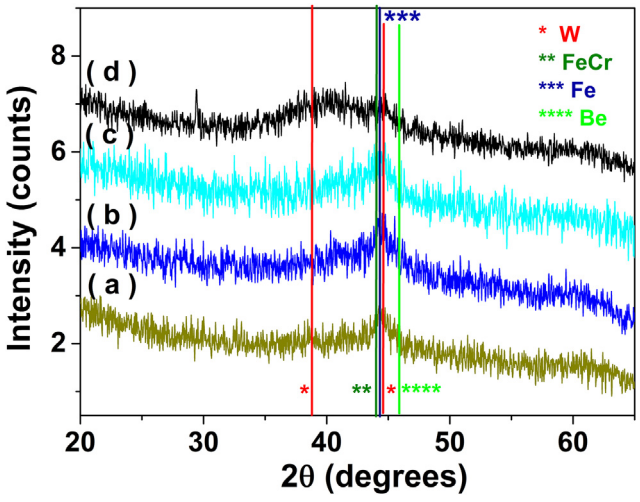

Fig. 1. GIXRD patterns of samples: (a) $Fe_{0.89}Cr_{0.11}$/Be/W, (b) $Fe_{0.89}Cr_{0.11}$/W/Be, (c) $Fe_{0.84}Cr_{0.11}Al_{0.05}$/Be/W, and (d) Fe/W/Be.