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Microstructure evolution of copper doped beryllium thin films

ZHOU Min-jie, LUO Bing-chi, LI Kai, ZHANG Ji-cheng, LI Jia, WU Wei-dong

Research Center of Laser Fusion, China Academy of Engineering Physics, Mianyang 621900, China

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Abstract: Copper (Cu) doped beryllium (Be) thin films were deposited on silicon substrates by using a simple ion beam sputtering method, which can also realize the varying of Cu doping concentration. Detailed morphological and structural characterizations of the samples clearly disclose a microstructure evolution of films upon doping Cu. Doping Cu can effectively suppress film grain growth, causing a small grain size as well as uniform size distribution. Furthermore, doping Cu affects the crystallographic texture of film, which leads to the formation of more compact film structure. In particular, the surface smoothness of the doped films is significantly improved, which makes them promising candidates for various applications.

Key words: beryllium; thin films; Cu doping; microstructure

1 Introduction

Beryllium (Be), one of the lightest elements (Atomic number Z=4), has long attracted great research interests because of its unique atomic structure and excellent physical properties [1,2]. Beryllium exhibits a number of technologically significant characteristics, including low mass density, low absorption for X-ray, low Poisson ratio, high elastic modulus and special alloying properties. Indeed, it has played an important role in nuclear power industry, aeronautics, mechanics, electronics and inertial confinement fusion experiments [3−7]. In particular, Be thin films are of special interests and many research efforts have been devoted to the synthesis, characterization, properties investigation of Be thin films [8−12]. It is known that the structure is a key factor that governs thin film properties, and the practical use of film requires fundamental knowledge on structure formation and structure evolution mechanism, which is crucial for tailoring thin film properties. Besides synthesis conditions, elemental doping is also an effective way to affect the microstructure of thin film, and thus leads to desired film properties [13,14]. Although doped Be film has received much attention [15,16], those researches focus on Be film with quite large thickness (~100 μm) and quite low doping content (<1% in mole fraction). In short, the study on modifying microstructure of Be thin film by elemental doping with a wider range is still rather limited, which is of both basic scientific and technological interests. In the present work, a series of beryllium films with different copper content are deposited by using a simple ion beam sputtering method. Chemical composition, microstructure, crystallinity and surface roughness of the films are examined, and it is found that doping copper strongly affects the microstructure of Be films. The evolution of the film surface roughness as a function of the copper doping content is correlated to the change of its grain size, size uniformity and crystallographic texture.

2 Experimental

A high vacuum system equipped with two 3 cm Kaufman-type ion guns (Ion Tech., model 3−1500−100) was employed to deposit copper doped beryllium thin film samples. The base pressure of the chamber was below 1.3×10^{-4} Pa. The Si (001) substrates were in-situ pre-sputter-cleaned using argon ion (Ar^+) beam (40 mA, 500 eV) before deposition. A high purity (99.5%) 4-inch (10.16 cm) beryllium target was used as a source material, and small Cu chips were fixed onto the Be target surface as mixed target for doped samples. The Cu doping content was manipulated by adjusting the quantity of Cu chips. During deposition, an Ar^+ beam

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Corresponding author: WU Wei-dong; Tel/Fax: +86-816-2480830; E-mail: lrcwuweidong@gmail.com
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(operating at 60 mA and 1000 eV) generated by the ion gun sputtered the target and contributed to the film deposition. The duration for the thin film deposition was kept 30 min for all samples. In total, a pure Be thin film and three doped samples with different Cu contents were deposited as sample series.

The chemical composition of samples was examined on a X-ray photoelectron spectroscope (XPS; PHI Quantum 2000). Detailed information of the films' microstructure was studied on a transmission electron microscope (TEM; JOEL 2010 FEG) with electron energy loss spectrometer (EELS, GIF) attached to the same microscope. Cross-section TEM sample was prepared by a focused ion beam (FIB) liftout technique [17], with a Pt protective layer deposited on the film surface. The general morphology and crystallinity of the films were investigated by scanning electron microscope (SEM; Quantum F400) and X-ray diffraction (XRD; Ragiku RU300), respectively. The film surface roughness was examined on a white light interferometer (Zygo NewView7000).

3 Results and discussion

The chemical compositions of all the thin films were examined by XPS and typical results are shown in Fig. 1. The films were sputtered off 20 nm to remove the surface contamination layer. Survey scan spectra reveal that all of the samples are high purity beryllium films with copper dopants. Fine scans were performed on both Be 1s and Cu 2p regions in order to find out the compositional binding states and copper doping content in the films. It is found that the peak centered at 111.8 eV can be observed in the Be 1s region, which corresponds metallic Be. For the feature observed in Cu 2p region, two peaks centered at 932.6 eV and 952.2 eV can be assigned to the metallic copper $2p_{3/2}$ and $2p_{1/2}$ peaks, respectively. Quantitative analysis reveals that copper contents of three samples are 0.8%, 2.1% and 4.5% (mole fraction), respectively, confirming the successful tuning of film composition.

The microstructure of film is disclosed by cross-sectional TEM study, and typical results are shown in Fig. 2. The full film can be seen in the image with Si substrate and Pt protective layer visible on the right and left (Fig. 2(a)), from which the film thickness is estimated at ~100 nm. High-resolution TEM image suggests the polycrystalline nature of the film (Fig. 2(b)). Furthermore, Be K-edge and Cu L-edge were used for EELS elemental mapping (Fig. 2(c) and Fig. 2(d)), by which the correlation between Be and Cu distribution in the film can be established. Both Be and Cu mapping images show quite homogeneous contrast in film region, indicating a fairly uniform Cu doping in the Be films over the whole area.

The surface of all films was examined by SEM, which gives a sense of the change in morphology films under the effect of Cu doping. Six-fold hexagonal pattering grains can be clearly observed in the pure Be film (Fig. 3(a)), and the grain size is found to be ~200 nm. Such hexagonal geometry becomes much less discernable due to Cu doping (Fig. 3(b)), which also leads to a grain size decreasing to ~150 nm. With the Cu doping content increases to 2.1%, the film surface exhibits a dense and faceted structure (Fig. 3(c)), and the grain size significantly reduces to ~50 nm. Further increase in the Cu doping content to 4.5% also shows faceting feature (Fig. 3(d)), while results in a more compact surface structure and smaller grain size of ~30 nm.

The crystallinity of all the films has been examined by XRD, and a structural evolution with Cu doping is observed. The XRD pattern of pure Be film can be indexed by assigning a hexagonal lattice (Fig. 4(a)), in which (100), (002), (101) and (012) reflections can be identified. Although no reflections corresponding to Cu phase can be detected due to the relatively low doping content (0.8%), Cu doping effectively suppresses the intensity of (002) reflection (Fig. 4(b)). When the Cu doping content increases to 2.1%, the Be (002) peak

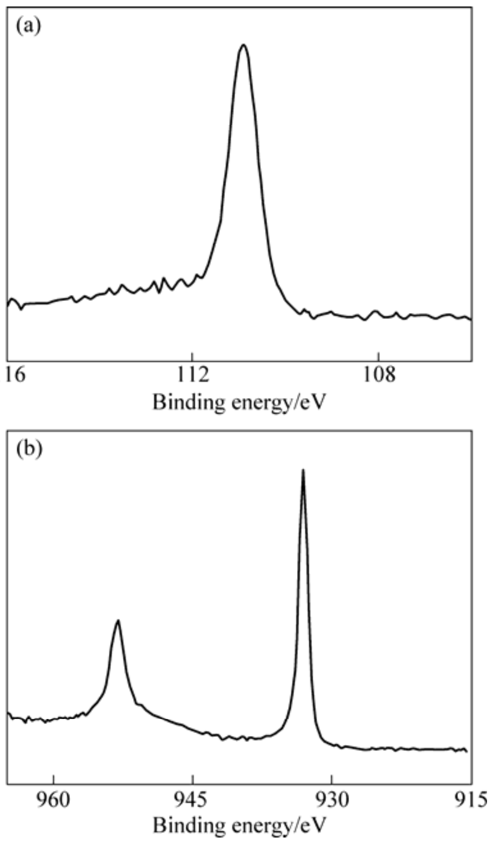

Fig. 1 XPS results of 4.5% Cu doped Be thin film: (a) Be 1s core level spectrum; (b) Cu 2p core level spectrum

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