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Influence of substrate temperature on the growth and electrical properties of thermally evaporated Be films

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HIGHLIGHTS

- The Be films were prepared on the water-cooled substrates and heated substrates by thermal evaporation, respectively.
- The Be grains grown on Si substrates were refined than those grown on glass substrates in the experiment of water-cooled substrate due to the different thermal conductivity of substrate.
- The Be films grown on water-cooled glass substrates were under pressure stress and it gradually decreased with increasing the heating temperature.
- The electrical resistivity of Be films decreased with increasing the heating temperature in the experiment of water-cooled substrate due to the gradually improving crystallinity.
- However, the electrical resistivity of Be films increased with increasing the substrate temperature in the experiment of heated substrate due to gradual oxidization at high temperature.

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ABSTRACT

The Be films were prepared on the water-cooled substrates and heated substrates by thermal evaporation, respectively. The experiment of water-cooled substrate showed the grains in Be films grown on Si substrates were refined than those grown on glass substrates and the surface roughness of Be films grown on Si substrates was also improved. The electrical performance test suggested that the electrical resistivity of Be films grown on water-cooled substrates decreased with increasing the heating temperature and was bigger than that grown on non-water-cooled substrates. For the experiment of heated substrate, the Be films appeared the loose flocculent structure, and the surface roughness firstly increased from 8 nm to 45–65 nm in the range of 150–300 °C and then decreased to 10 nm at 400 °C. Meanwhile, its electrical resistivity sharply increased from 0.6 Ω/□ to 206.6 Ω/□ due to the gradual oxidization at high temperature, especially 400 °C.

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1. Introduction

Owing to its many unique merits, beryllium, one of the lightest nuclear metals has been applied remarkably in aeronautics and astronautics, atomic energy industry, national defense industry, and science and engineering research. As is well known that beryllium also was an armour material for the in-vessel components of the International Thermonuclear Experimental Reactor (ITER). As far as the Be films and deposits were concerned, they were used as foil windows in X-ray instruments, especially used as capsule in ICF for its high density, low opacity, high thermal conductivity and high mechanical property [1]. Concerning the preparation method, they were frequently prepared by the ordinary PVD method, i.e., thermal evaporation and magnetron sputtering [2,3]. In the thermal evaporation, the biggest factor was the heating temperature of evaporation source so that a small change of it would cause the evaporation rate to increase greatly and the properties of prepared Be films to change largely as shown in our previous work [4]. Moreover, the substrate temperature was also an important processing parameter influencing the growth and properties of films, e.g., electrical properties, magnetic properties, optical properties, etc. [5–9]. In these literatures about the influence of substrate temperature on films, they generally studied the effects of heated substrate at high temperature on the growth and properties of films. However, the

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influence of cooled substrate at low temperature on the growth and properties of films was hardly investigated, especially for the Be films.

The electrical resistivity of materials is often related to its microstructure and compositions, the change of the latter at different condition will directly give rise to the change of resistance. For the metallic thin film, the conductive mechanism of it totally depends on the electron mobility that is often influenced by the crystallinity, the film thickness, the crystal defects or voids, the surface roughness, the compositions, etc. [10–14]. So, the researchers can get further results about structure and composition of materials via studying on the electrical resistivity and establish the correlation between them.

In this paper, the authors prepared the Be films on the water-cooled substrates and heated substrates, respectively, and analyzed the influence of substrate temperature on the growth and electrical resistivity of Be films. The results would give some meanings to the study of Be film grown on the substrate at low temperature and high temperature.

2. Experimental

The Be films were prepared by the thermal evaporation method. The experimental instrument was the same with that in the literature [4] except that the substrate at this time was heated by a heater or cooled by the circulated cooling water as shown in Fig. 1. The experiments were divided into two groups, one was the experiment of water-cooled substrate that the substrates were cooled by the 20 °C circulated cooling water as the heating temperature of crucible was varied between 980 and 1120 °C, the other was the experiment of heated substrate that the substrates were heated to 200–400 °C by a heater as the heating temperature of crucible was kept at 1050 °C. In each experiment, glass slides and silicon wafers were placed into chamber together as substrates. The distance between the evaporation source and the substrate was 90 mm. The deposition time was 3 h for all samples and the background pressure of the chamber was 5×10^{-5} Pa. The other aspects of experiment, e.g., the structure of equipment, the heating method, the temperature measurement method, the method of cleaning substrate were the same with those in literature [4], so not mentioned here.

The morphology of the prepared Be films was characterized using a scanning electron microscope (FEI Sirion200), the surface roughness of Be films was characterized by a stylus profiler (Dektak 6M), the crystalline phase was examined using X-ray diffraction (Philips X'Pert PRO, Cu Kα radiation, λ = 0.154 nm), the composition of heated Be films was characterized by a X-ray energy dispersive spectrometer (INCA Energy 200), the electrical resistivity of Be films was characterized by the four-point probe method using Agilent 34401A, respectively.

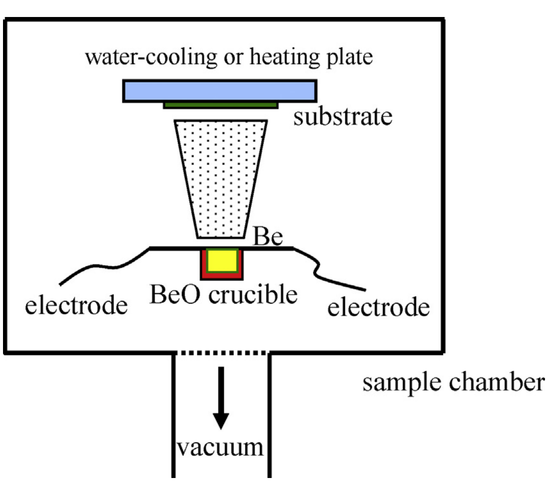

Fig. 1. Schematic diagram of the Be film grown on a water-cooled substrate or heated substrate by thermal evaporation.

3. Results and discussion

3.1. The experiment of water-cooled substrate

3.1.1. Morphology

The morphologies of Be films grown on water-cooled substrates are shown in Fig. 2. The grains in the Be films grown on Si and glass substrates all increased with increasing the heating temperature. But the grains in the Be films grown on Si substrates were refined in comparison with those grown on glass substrates, especially at high heating temperature. However, in our previous work [4], the morphologies of Be films grown on the two kinds of non-water-cooled substrates were basically the same. The reason for grain refinement was that the low substrate temperature in this experiment inhibited grain growth. Especially, the thermal conductivity of glass and Si varies widely (i.e., 0.7 W/(m K), 150 W/(m K), respectively), the high thermal conductivity caused the much higher undercooling of Si substrate and resultant smaller critical nucleation radius of the grain in Be film. This process ultimately leaded to the significant grain refinement for the Be films grown on water-cooled Si substrates.

In particular, the Be films grown on the water-cooled Si wafer even sometimes fell off the substrate as shown in Fig. 3. Generally, the excess thermal stress in the deposition often causes the film to wrinkle or to curl up. The thermal stress in film during the course of deposition is generally described as below [15,16]:

$$\sigma_f = \frac{E_f}{1 - \nu_f} \times \Delta a \times \Delta T \quad (1)$$

where σ_f is the thermal stress, E_f is the elastic modulus of film, ν_f is the Poisson ratio of film, Δa is the difference of thermal expansion coefficient between the film and substrate, ΔT is the difference between the deposition temperature and measurement temperature. The thermal expansion coefficients of Be, Si and glass are 12.3×10^{-6}/K, 2.5×10^{-6}/K and 9×10^{-6}/K, respectively. So the Δa of the Be film grown on Si substrate was bigger than that grown on glass substrate due to the different thermal expansion coefficient as listed above. Furthermore, the ΔT of the Be film grown on Si substrate was also bigger than that grown on glass substrate due to the high thermal conductivity and better cooling of Si substrate. Taken together, these two factors jointly leaded to higher thermal stress in the Be film grown on the water-cooled Si wafer and caused it to curl up and to detach from the substrate.

3.1.2. Surface roughness

Due to the different morphologies of Be films grown on the two kinds of water-cooled substrates, the surface roughness of theirs also differs very big as shown in Fig. 4. The roughness of Be films grown on glass substrates increased from several nanometers to about 50 nm, however, the roughness of Be films grown on Si substrates just increased a little. The examination on surface roughness was in good agreement with the SEM characterization. The results suggested that the temperature decrease in substrate that has the characteristic of high thermal conductivity would help to prepare the smooth film. Furthermore, the film thickness in this experiment was basically the same with the result of non-water-cooled substrate in literature [4], suggesting that the decrease of substrate temperature had little influence on the growth rate of Be films that still mainly depended on the heating temperature.

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