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ARTICLE

Influence of Substrate Temperature on the Microstructure of Sc and ScD_2 Films

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Abstract: Scandium films with thickness of ~ 2~3 μm were deposited on Mo substrates with a surface roughness $R_q \approx 5$~8.63 nm. Subsequently, the films were converted to scandium dideuteride (ScD_2) in the Sievert's apparatus. A novel substrate preparation process has been developed for a simple observation of the cross-sectional images. The effects of substrate temperatures (623, 823, 1023 K) on the initial Sc-metal film microstructure and the following ScD_2 microstructure were investigated. Results indicate that Sc films show a columnar microstructure with a preferred orientation of the crystallites along the [002] direction at all the three tempertures and the (002) texture increases with increasing of the temperature. The grain sizes of Sc films deposited at higher temperature are larger than those deposited at lower temperature, consistent with the published structure zone models (SZMs). As expected, the stronger (002) texture of Sc-metal film deposited at higher temperature results in the stronger (111) texture of deuteride film, indicating that the deuteride nuclei might inherit their texture from the initial metal grains' structure. Unexpectedly, an inverse correlation appears that larger Sc-metal grains lead to smaller deuteride grains due to the improved deuterium diffusion kinetics in the more defective and fine-grained metal structure

Key words: scandium film; deuteride; substrate temperature; microstructure; electron beam evaporation

Scandium films loaded with the hydrogen isotopes (deuterium, D or tritium, T) are under consideration to be a potential candidate material used as neutron targets. It is important for a variety of applications including defence programs, fuel cells, down-hole oil-logging devices, and neutron sources for medical imaging and diagnostics[1]. Although the ScD_2 film has a relatively low neutron emission rate, it can reach steady-state yield more quickly than other deuterides, and it also has good thermal stability and does not produce crater on the target surface when bombarded by a deuterium beam[2]. In addition, ScH_2 contains 7.31×10^{22} H atoms/cm^3 and it may be useful as a high temperaure neutron moderator[3].

Neutron targets are usually composed of a metal-hydride (either -deuteride or -tritide) film on a copper[2] or molybdenum[4] substrate. The consistency of generated neutrons is influenced by the preparation of the target materials. Different preparation processes will lead to various morphologies and microstructures in the final samples, which in turn affect the final properties of the deposited film. The property of the metal hydride films fabricated by a two-step process involving evaporation of metal and subsequent hydrogen loading, will be affected by the above process. Among them, the influence of substrate temperature on the initial metal film microstructure is particularly important and has been studied for a long period. The earlier study on the effect of substrate temperature for evaporated films (20~2000 μm) was done by Movchan and Demchishin and the structure zone models (SZMs) were first proposed for the broad description of polycrystalline film structure, which consisted of three zones, separated by two boundary temperatures at T_s~0.3 T_m and T_s~0.5 T_m (T_s and T_m are the substrate and the material melting temperatures in Kelvin, respectively)[5]. Subsequently, many researchers turned to study the effect of substrate temperature on various systems[6-10]. However, up to now, few efforts have been made in studying the effect of substrate temperature on the grain size and microstructure of metal films loaded with the hydrogen isotopes. The

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relationship between the deposition temperature and the microstructure of deuteride film is not clear yet so far. In this paper, we studied the influence of substrate temperature not only on the microstructures of scandium films in order to assess the extent to which they are consistent with the SZMs, but also on the grain size and structure of ScD_2 films.

1 Experiment

Scandium films of 2~3 μm thickness were deposited on Mo substrates with a surface roughness $R_q \approx 5$~8.63 nm by electron beam evaporation. Scandium with the purity of ~99.95 % was from Hunan Rare Earth Technical Development Co., Ltd. The substrate temperatures were ~ 623, 823 K, and 1023 K, corresponding to the three regions of the structure zone models in order to examine the extent to which the morphologies were consistent with the SZMs, and the deposition rate was ~5 nm/s.

The molybdenum substrates were heated in a H_2 furance at 1123 K for 1 h to remove the oxide discoloration. Just before being used, all substrates and scandium target were ultrasonically cleaned in acetone and then in ethanol for 10 min. In addition, before deposition, the substrates and substrate holder were degassed at 1023 K for 2 h, as well as the scandium source was degassed by the electron beam with the shutter closed. Then, the temperature was cooled down to the desired deposition temperature, and during the process the background pressure in the chamber was less than $\sim 2.3\times10^{-4}$ Pa. After finishing all above, the deposition began with the incidence direction of Sc flux pependicular to the substrate surface and with the distance of 23 cm between the target crucible and substrate. During deposition, the pressure was decreased to 8.1×10^{-5} Pa due to the gettering property of Sc. The deposition rate was monitored by a IC5 thin film deposition controller (Inficon, USA) and controlled by feedback to the evaporation source. Film thickness was monitored by a quartz crystal unit. To obtain uniform films, the substrate holder was rotated during deposition under the revolution voltage of 30 V. After deposition, the samples were held at the corresponding substrate temperature for 30 min and then cooled down to room temperature before being removed from the chamber.

After they were deposited, Sc films were immediately transferred to the Sievert's apparatus to alleviate oxidization in air, because the scandium films have a strong affinity to oxygen. Before the reaction of scandium and deuterium, the scandium films were degassed by increasing temperature from room temperature to 923 K, and during this process, the base pressure was controlled to be less than 5.4×10^{-4} Pa. An initial deuterium pressure of 3500 Pa was introduced into the chamber at 923 K with the soaking time of 10 min. Then, the temperature was decreased to room temperature at the rate of 30 K/min.

The above process was repeated several times and the reproducibility of the results was confirmed. The deposition parameters for preparing film samples as well as the the grain sizes of Sc and ScD_2 films are listed in Table 1.

The microstructures of these films were obtained using a Panalytical XRD X'pert PRO MPD Diffractometer (Cu Kα radiation) with a step size of 0.04 ° and count time of 1 s per step. Surface morphologies and cross-sections were characterized by means of SEM (Apollo 300 electron microscope of CamScan Company). Grain width and film thickness were measured from electron micrographs using ImageJ software. The accuracy of this method used for measuring grain sizes was low but the variation trend was confirmed.

Due to the very likely reaction of the scandium films with water or solvents containing even a small amount of humidity, standard metallographic cross-sectional techniques cannot be used for the Sc system. Here, modified Mo substrates were used (shown in Fig.1), which offered a relatively easy method to obtain the cross-sectional images and avoided the contamination to the greatest extent. The reverse side of the Mo substrate was machined with a three-cornered groove which didn't penetrate the substrate. When the scandium films were deposited onto the above substrate, the samples were fractured into two parts along the dashed line shown in Fig.1, and the cross-sectional morphologies can be easily observed using half of the samples. Although the samples were tore into two parts by the external force, the morphologies and grain sizes changing with the substrate temperatures were affirmed to explain the actual growth mode of the films.

2 Results and Discussion

2.1 Characterization of scandium film

Fig.2 shows the representative surface and cross-sectional morphologies of the three Sc-metal samples. The Sc-metal films show columnar structures which are generally consistent with the structure-zone models (SZMs)[5]. At 623 K (Zone 1), the scandium film consists of narrow inverted conelike fine-grains with the average grain size of ~103 nm, which are developed from the bundles of many individual grains at the lower temperature and extend through the thickness of the film. These narrow columns are usually separated by grain boundaries containing a high

Table 1 Deposition parameters for preparing Sc films and the grain sizes of Sc and ScD_2 films

Substrate	Substrate temperature/K	Deposition rate/nm·s^{-1}	Grain sizes/nm	
			Sc film	ScD_2 film
Mo	623(T_s/T_m=0.35, Zone 1)	5	~103	~287
Mo	823(T_s/T_m=0.46, Zone 2)	5	~198	~134
Mo	1023(T_s/T_m=0.57, Zone 3)	5	~246	~115

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