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Effects of substrate rotation on the microstructure of metal sheet fabricated by electron beam physical vapor deposition

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ABSTRACT

The effects of substrate rotation speed and rotation mode on the microstructure of large-sized metal sheet fabricated by electron beam physical vapor deposition technique were investigated. Helical and columnar microstructures were found in the deposited sheet. Both types of microstructures exhibit no preferential crystallographic orientation. The column inclination under asymmetric vapor incidence pattern was discussed. Integrated vapor incidence angle was found to be effective in evaluating the column inclination.

1. Introduction

Electron beam physical vapor deposition (EBPVD) is widespread in fabricating coatings and metallic sheets such as yttria stabilized zirconia thermal barrier coating, SixCy high emissivity coating, high temperature alloy sheets, and so on. In most of the application, the substrate was rotational. However, the effect of substrate rotation on microstructure has not been clarified [1–4].

The microstructure of inclined columns is generally found in EBPVD prepared thermal barrier coatings and obliquely deposited or sputtered thin films. It causes anisotropy and strongly influences crystallography and properties of the deposits. And the orientation of intercolumnar gaps is closely related with column inclination, which is of great concern for fabricating porous materials or thermal barrier coatings [5,6].

As for deposits with stationary substrate, the tangent rule is especially effective in predicting the relationship between column inclination angle and vapor incidence angle (VIA) in the VIA range of less than 60° [7–12]. However, as for the column inclination in deposits with rotational substrates, especially with asymmetric vapor incidence pattern, little work has been done.

In this paper, EBPVD technique is employed to fabricate large-sized metal sheet. This paper mainly discusses the effect of substrate rotation on the morphology and the column inclination in the sheet under asymmetrically changing vapor incident pattern.

2. Experimental details

A carbon steel sheet was fabricated by a GEKONT L5 electron beam facility in a vacuum chamber evacuated to 1×10^{-3} Pa. The arrangement of the facility was illustrated in Fig. 1. The diameter of the stainless steel substrate was 1000 mm and the vertical distance from the vapor source to the substrate was 550 mm. A carbon steel vapor source was placed 250 mm far from the central axis of the substrate.

Three substrate rotation speeds of 1.6, 10.7, and 25.4 rpm were applied in the deposition and each speed lasted about 15 min. The substrate temperature was 635 °C with a 10 °C error range. The electron beam current was controlled to be 1.85 A with a 0.05 A error range.

Samples were fractured respectively along the tangent direction (TD) and radius direction (RD) at different positions of the sheet in liquid nitrogen. In this paper, the plane determined by RD and the substrate normal is denoted as RP. The plane determined by TD and the substrate normal is denoted as TP. The cross-sections were observed on a HITACHI S-570 Scanning Electron Microscope.

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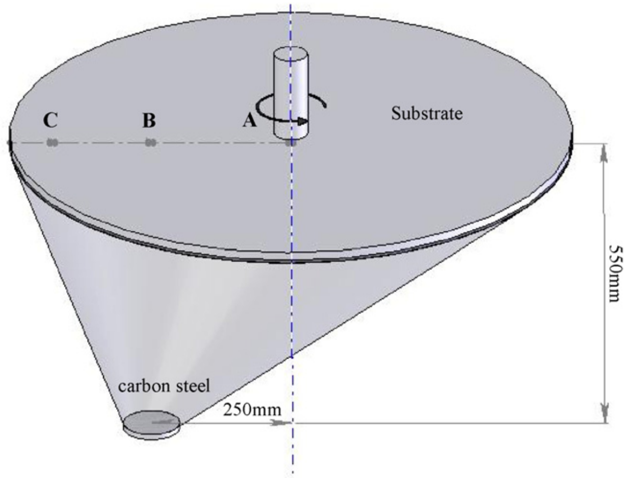

Fig. 1. Arrangement of the substrate and the vapor source.

X-ray diffraction was conducted for both sides of the sample at 250 mm from the center of the sheet on a D/max-rB X-ray diffractometer with Cu Kα radiation.

3. Results

3.1. Helical microstructures and columnar microstructures

It is observed from Fig. 2(a) that the cross-section is mainly composed of a helical microstructure zone and a columnar microstructure zone. The thickness of the helical microstructure zone is about 61.8 μm, one third of the total thickness of the sheet. It is thus taken that the helical microstructures were formed under conditions when the substrate rotation speed was 1.6 rpm, as shown in Fig. 2(b). And columnar microstructures were formed under conditions when the substrate rotation speed was 10.7 rpm and 25.4 rpm, as shown in Fig. 2(c).

The deposition time was 15 min for the substrate rotation speed of 1.579 rpm, which means the substrate finished about 24 cycles of rotation during low rotation speed deposition. The thickness of one helix is about 2.4 um, and there are about 25 helixes in the low rotation speed zone. Which indicates that one helix may correspond with one cycle of substrate rotation.

3.2. Inclination of columns

Fig. 3(a) shows cross-sectional fractures along the radius of the sheet at positions 100, 200, and 300 mm far from the center of the sheet. Samples were firstly sliced at the appointed positions, and then fractured respectively along TD and RD in the liquid nitrogen. Three samples were examined for each distance. It is indicated that the position right above the vapor source exhibit inclined columns while the position that is the rotation center with inclined incident vapor exhibit vertical columns. Fig. 3(b) shows cross-sectional fractures vertical to the radius of the sheet at same positions with Fig. 3(a). It is indicated that the columns are parallel to the surface normal in the plane defined by the surface normal and the tangent at any position of the sheet.

The inclination of the columns does not change with the substrate rotation speed obviously.

3.3. Crystallographic orientations

Fig. 4 shows XRD patterns examined both from the near-substrate side and from the deposit surface of the sample at 250 mm from the center of the sheet. The patterns indicate no obvious preferred orientation for the two sides.

4. Discussion

4.1. Effect of substrate rotation on column inclination and morphology (growth mode)

Fig. 5 illustrates the deposit growth at three typical positions on the substrate, which are 0, 250, and 425 mm from the center of the substrate, denoted as A, B, and C, respectively. We are going to interpret the column inclination by tracking the displacement of the column tips, as inspired by idea of K. Wada in terms of the formation of crescent-shaped grains [13]. Assuming that the columns grow in the direction of incident vapor flux and thus shadowing from neighboring grains has a negligible effect on the growth of column tips, which means that the growing direction follows the variation of VIA. In the actual situation the vapor source is stationary and the substrate is rotated around a vertical axis. To better understand the variation of their relative positions it is supposed that the substrate is stationary while the vapor source moves around the vertical axis. So the incident vapor fluxes for position A move along the generatrix of a symmetric cone. The time of each substrate revolution is divided into many minute durations, each arrow-headed line section indicates the column growth for certain duration. So as the substrate rotates through 2π radian, the column tips finish a complete helix. As for position B and C, the situation is similar except that the incident vapor fluxes move along an asymmetric cone thus the tips finish an inclined helix.

In this assumption, the variation of deposition rate at different VIA and with different distance from the vapor source was taken into account. The column inclination was caused by the different displacement of column tips along different direction and can be estimated by the inclination of helix axis.

As for any position on the substrate whose distance from the center is d, the instantaneous VIA during one substrate revolution can be expressed by the following equation:

$$\mathrm{VIA} = \arctan\frac{\sqrt{250^2 + d^2 - 500d\cos\theta}}{550} \tag{1}$$

where d is the distance from the point on the substrate to the center of the substrate and is in millimeters; θ represents the instantaneous rotation angle of the substrate and is in degrees. VIA is the instantaneous vapor incidence angle and is in degrees too.

Because the column inclination is observed from RP and TP, VIA is also calculated in terms of its projections on the two planes. The instantaneous VIA projected on RP can be expressed as:

$$\mathrm{VIA}_r = \arctan\frac{d - 250\cos\theta}{550} \tag{2}$$

where VIA_r is the instantaneous VIA projected on RP and is in degrees. When the value is positive, it means the incident vapor flux inclines towards the center of the substrate. While the value is negative, it means the incident vapor flux inclines towards the edge of the substrate.

The instantaneous VIA projected on TP can be expressed as:

$$\mathrm{VIA}_t = \arctan\frac{250\sin\theta}{550} \tag{3}$$

where VIA_t is the instantaneous VIA projected on TP and is in degrees.

The instantaneous VIA projected on RP and TP during one substrate revolution are separately illustrated in Fig. 6(a) and (b). As for position A, the value of VIA keeps constant to be 24.4°, while