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## Morphology and texture evolution of FeCrAlTi-Y<sub>2</sub>O<sub>3</sub> foil fabricated by EBPVD

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#### ABSTRACT

An electron beam physical vapor deposition method was used to fabricate freestanding  $\rm Y_2O_3$  dispersive strengthened FeCrAlTi foils for high-temperature applications. The vapor incidence mode was found to have great impact on the morphology and crystallographic orientation of the foils. Under symmetric vapor incidence mode, an out-of-plane <100> fiber texture was formed. While under asymmetric vapor incidence mode, both out-of-plane preference of <111> direction and several in-plane preferences were developed. As the deposition proceeded, the extent of in-plane orientation increased, and the preferred out-of-plane orientation increasingly deviated from the surface normal. The vapor incidence mode played a role on the growth rate of <100> direction and <110> direction, by which the morphology and crystallographic orientation of grains were modified.

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#### 1. Introduction

With nanoscaled oxide particles dispersed in the base alloys, Oxide Dispersion Strengthened (ODS) high-temperature alloys are attracting intense attention because of their exceptionally high creep and oxidation resistance at temperatures above 1000 °C[1–3]. Large-sized ODS alloy foils are in great demand for the metallic thermal protection system as panel material. Currently, the ODS alloys are mostly fabricated by mechanical alloying (MA) method. However, it will be exceptionally complicated and expensive to fabricate large-sized alloy sheets with small thickness, because repetitive hot rolling processing is required and the strong anisotropy introduced by rolling needs to be considered.

As a candidate method for the fabrication of ODS high-temperature alloy foils, the electron beam physical vapor deposition (EBPVD) technique has superiority in its simple technical process especially for the production of large-sized foil with chosen composition and tailored microstructure [4–8]. Therefore, the fabrication of ODS high-temperature alloy foils by EBPVD has attracted much attention [9–11]. The work was developed in our group and in GE India Company [9–11]. In our previous work, we have succeeded in preparing  $\rm Y_2O_3$  dispersion strengthened Ni-based alloy by EBPVD and found that, after treated by HIP, the ultimate strength of the foil arrived at 1230 MPa [9], which is superior compared with alloys fabricated by other methods. However, the microstructure evolution and the crystallographic orientation which govern the mechanical behavior are seldom investigated.

The texture types of ODS alloys fabricated by MA method have been extensively explored and are found to significantly influence the

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creep behavior of the material and bring into plastic anisotropy [12–16]. It is reported that at 900 °C and strain-rates of above  $10^{-5}$  s<sup>-1</sup> the tensile strength of alloy with <111> texture is about 30% higher than that of an alloy with <100> texture [16]. In the past, a lot of work has been conducted on the texture of thermal barrier coatings fabricated by EBPVD [17–21]. Texture distribution and evolution in materials fabricated by EBPVD method will be totally different from that of materials fabricated by MA method because the texture evolution mechanisms are completely different. For MA method, the textures are induced by the deformation processing and recrystallization; while for EBPVD method, the textures are contributed by preferential growth of the deposits [21,22]. But, the mechanisms governing the texture evolution in ODS alloy foils fabricated by EBPVD have never been discussed.

The texture evolution for ODS alloy foil with thickness in the magnitude of several hundreds of micrometers has both similarity and great differences with thin films or thick coatings. For thin films, the growth of low energy crystallographic planes dominates the film growth and the texture evolution. And there will be a critical thickness, surpassing which the texture types will not vary [23]. However, the texture types in this study change with thickness until the total thickness of about 150 µm. As for texture evolution in thermal barrier coatings grown by EBPVD, lots of works have been conducted. Substrate temperature [17], vapor incidence angle [18], substrate rotation speed [19] and rotation mode [20] are found to strongly influence the microstructure and crystallographic texture evolution of coatings grown by EBPVD. Both the "evolutionary selection" regulation and the "equal flux" requirement are always used to interpret the texture evolution of thermal barrier coatings fabricated by EBPVD [21]. However, the energy factors governing the nucleation and initial stage growth were seldom taken into consideration. Whether the energy factor plays a dominant role

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during growth of thick coatings, and whether the equilibrium shapes (Wullf shape) of grains composing of low energy crystallographic planes keep during the following stage of growth remain unclear.

In order to investigate the texture evolution and accordingly to improve the mechanical performances of large-sized ODS alloy foils, three small-sized foils are fabricated under three different vapor incidence modes in this paper. The microstructures and the crystallographic orientation evolutions under different vapor incident modes are investigated. This work firstly characterized the texture evolution of large-sized ODS alloy foils fabricated by EBPVD method. The roles of different mechanisms governing the texture evolution during the growth of ODS foil including low energy requirements and competitive growth were discussed and evaluated in details. The relationships between the morphology and the crystallographic texture were discussed in terms of the growth mechanisms.

### 2. Experimental details

#### 2.1. Material fabrication

The foils were prepared by a GEKONT L5 electron beam facility in a vacuum chamber evacuated to  $1\times10^{-3}$  Pa. The arrangement of the substrates and the vapor sources is shown in Fig. 1. A substrate barrier with a diameter of 1000 mm was placed 550 mm above the vapor sources. Three separate carbon steel substrates in the size of 25 mm  $\times$  25 mm were placed at 0 mm, 250 mm, and 425 mm from the center of the substrate barrier, as indicated in Fig. 1 by A, B, and C. The substrate barrier was rotated around the vertical axis at 12 rpm. The crucibles were 250 mm far from the rotation axis.

A 98.5 mm diameter FeCrAlTi (Fe-19.7Cr-5.3Al-5.5Ti) bar and a 68.5 mm diameter  $Y_2O_3$  bar were placed into two water-cooled copper crucibles and evaporated by two electron beams simultaneously and separately.

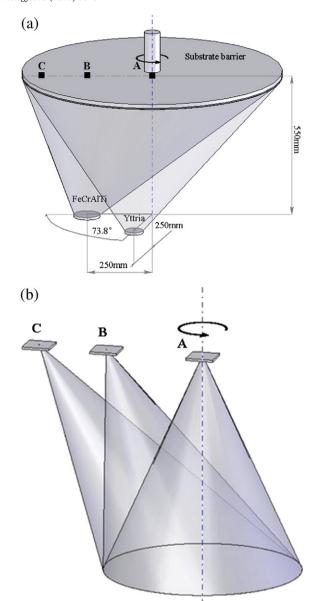
The temperature of the substrate was controlled at 600 °C with a 10 °C error range by thermo-heaters during deposition. The size of the thermo-heater was 1100 mm in diameter. Three thermocouples were placed at positions A, B and C to monitor the substrate temperature. The deposition time was 15 min. To separate the deposited foil from the substrate, a  $\text{CaF}_2$  interlayer was deposited prior to the deposition of metal foil.

#### 2.2. Vapor incidence modes

The three vapor incidence modes adopted in this paper represent the typical modes in the fabrication of large-sized ODS alloy foils by EBPVD. As for sample A, during the substrate rotation, the incident vapor fluxes move along the generatrix of a symmetric cone. While for sample B and sample C, the incident vapor fluxes move along an asymmetric cone. Thus during the fabrication of large-sized foil by EBPVD, only at the center the foil grows under symmetric vapor incidence mode and at other positions the foil grows under asymmetric vapor incidence modes. The variations of vapor incidence angle during substrate rotation for the three samples are shown in Fig. 1, the calculation is detailed in [8]. The influence of the yttria vapor source on the texture evolution was neglected in this work since the composition of yttria in the deposit was as low as 0.8 wt.%.

#### 2.3. Material characterization

The deposited samples were separated from the substrates. Both of the cross-sections and the top views were observed on an S-4700 Cold Field Emission Scanning Electron Microscope (Cold FE-SEM). Two groups of cross-sectional specimens were obtained by being fractured in liquid nitrogen, with one group fractured along the tangent direction (TD) of the substrate barrier, and another group along the radius direction (RD) of the substrate barrier. The composition of



**Fig. 1.** (a) The arrangement of the substrate and the vapor sources. (b) The schematic drawing of variations of vapor incidence angle during substrate rotation for samples A, B, and C.

sample B was examined by X-ray Fluorescence Spectroscopy, which was Fe 17.451 Cr 5.076 Al 0.023 Ti 0.642 Y wt.%.

The microstructures of the three samples were firstly studied by Xray diffraction, with Cu Kα radiation on a D/max-rB X-ray diffractometer for both sides, and then characterized by X-ray pole figure analyses on a Philips PW3040 diffractometer with iron filtered Co  $K\alpha$ radiation for three different depths in terms of each sample. The specimens were abraded from the surface side to examine the texture evolution along the thickness direction. Because none of the three Xray diffraction patterns for the near-substrate surfaces of the three samples indicated preferred orientation, only the substrate surface of sample A was characterized by X-ray pole figure. With different distances from the vapor source and with different vapor incidence angle, samples A, B and C had different thicknesses, which were respectively 110 μm, 130 μm, and 75 μm. As for sample A, the texture characterization was conducted at near-substrate surface, 70 µm and 110 µm (deposit surface) from the near-substrate surface. For sample B, it was conducted at 85 μm and 130 μm (deposit surface) from the near-substrate surface. And for sample C, it was conducted at 40 µm

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