



Sintering and microstructure evolution of columnar nickel-based superalloy sheets prepared by EB-PVD

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ABSTRACT

A ~0.15 mm-thick columnar nickel-based superalloy sheet was obtained by electron beam physical vapor deposition (EB-PVD). The as-deposited alloy sheet was sintered at different conditions. The microstructure of the specimens before and after sintering was characterized by using scanning electron microscopy. An X'Pert texture facility was used to determine the crystallographic orientation of the as-deposited alloy sheet. The phase transformation was investigated by X-ray diffraction. Tensile tests were conducted at room temperature on as-deposited and sintered specimens. The results show that the as-deposited sheet is composed of typical columnar structures. After sintering, however, the columnar structure degrades. The degradation depends on sintering temperature and time. Both the ultimate tensile strength and the elongation percentage are effectively improved after sintering.

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1. Introduction

Electron beam physical vapor deposition (EB-PVD) is a high-efficiency and non-equilibrium deposition technique [1]. It is commonly used to deposit thermal barrier coatings (TBCs) on rotating blades and some high-pressure turbine section vanes, which require surfaces with a smooth finish and the ability to withstand high thermo-mechanical strain [2–5]. The unique columnar microstructure is typical of TBCs obtained by EB-PVD. The loosely bonded columnar grains provide outstanding resistance against thermal shock and mechanical strains, which are associated with high-temperature, high-pressure turbine blades [6]. Recently, a significant amount of research work has been conducted on the application of EB-PVD technology in the production of microlaminate material [7] and thin alloy sheets. He et al. reported on oxide dispersion strengthened (ODS) Ni-based superalloy foils produced by EB-PVD [8]. Chen et al. studied the microstructure and mechanical properties of Ni-based superalloy foil with nanocrystalline surface layer produced by EB-PVD [9]. Li et al. investigated the isothermal oxidation behaviors of Ni-based alloy sheets produced by EB-PVD [10]. In these studies, columnar structures were obtained. These structures significantly affected the mechanical

properties of alloy sheets. The ultimate tensile strength and elongation percentage of as-deposited alloy sheets were small because of cracks propagating along the interface of columnar grains. Therefore, to understand the full potential and performance benefits offered by EB-PVD, it is necessary to reduce the effects of columnar structures on the mechanical properties. Earlier studies have examined the effects of sintering on the properties of the topcoat. Some researches have been done to reveal the mechanisms underlying topcoat sintering and the associated changes in coating microstructure [11,12]. Guo et al. reported that the columnar structure of a 4.0 mol% Y_2O_3 partially stabilized ZrO_2 coating material degraded after thermal exposure, and both hardness and Young's modulus significantly increased [13]. Similar results in an 8 wt% Y_2O_3 partially stabilized ZrO_2 coating material were also reported by Wellman et al. [14]. These studies are essential to correlate sintering-related changes in the topcoat to coating failure. However, this kind of research has seldom been done for EB-PVD thin alloy sheets.

In the present study, a Ni-based superalloy sheet with a typical columnar structure was prepared by EB-PVD. The microstructural evolution and tensile properties of the alloy sheet before and after sintering in vacuum were investigated.

2. Experimental

A large-scale EB-PVD equipment (GEKONT L5) was employed to prepare a Ni-based superalloy sheet. A Y_2O_3 ingot and a Ni–20Cr–1.4Al (wt%) ingot were selected as vapor sources. Deposition was performed under high vacuum (1×10^{-3} Pa) using a turbo-molecular pump. During preparation, the deposition rate was adjusted by changing the currents of the electron beams. The deposition speed was ~3 $\mu\text{m}/\text{min}$

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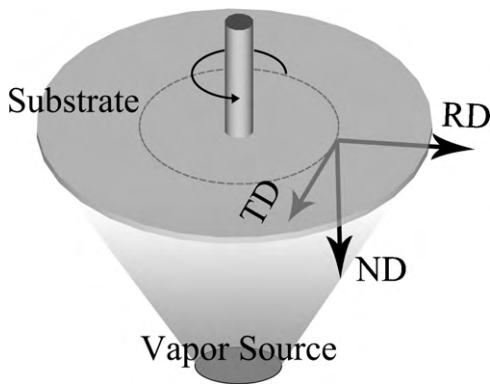


Fig. 1. Illustration of the local rectangular coordinate system on specimens.

and the total deposition time was 50 min. The rotation speed was 12 rpm and the substrate temperature was 720 °C, with a 20 °C error range. After deposition, the Ni-based superalloy sheet was mechanically stripped away from the stainless steel substrate. The as-deposited samples were then sintered at 760 °C for 3 h, 760 °C for 20 h and 1050 °C for 3 h. Prior to sintering, samples were encapsulated in quartz tubes and evacuated to less than 1×10^{-3} Pa.

Microstructural investigations of as-deposited and sintered specimens were performed by scanning electron microscopy (SEM) and X-ray diffraction (XRD). The chemical composition of the alloy sheet was determined by X-ray fluorescence spectrometry (XRF). Pole figures were obtained using a PANalytical X'Pert Pro diffractometer using Cu radiation with a nickel filter in the diffracted beam. Textural data were collected to a psi angle of 70° and corrected for contributions from the background and for defocusing. Data were then analyzed using a PANalytical X'Pert texture software in order to produce complete pole figures for the crystallographic orientation type. The reference frame is illustrated in Fig. 1. The radius direction, tangent direction, and normal direction were denoted as RD, TD and ND, respectively.

Tensile tests were conducted at room temperature on as-deposited and sintered specimens. The sample size and shape in the tensile tests are shown in Fig. 2. Specimens were strained at a rate of approximately 0.02 s^{-1} with an extensometer clipped to the gauge length until failure.

3. Results and discussion

3.1. Microstructures

A Ni–18Cr–0.6Al superalloy sheet strengthened with 0.4Y₂O₃ (wt%) was deposited. The thickness of the sheet is ~150 μm.

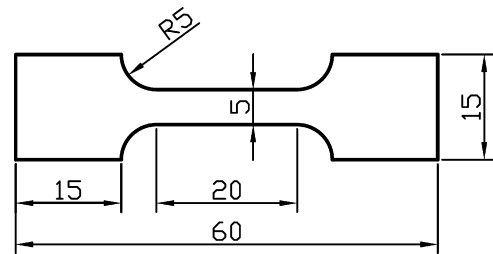


Fig. 2. Sample size and shape in tensile tests.

Fig. 3(a) shows the cross-sectional morphology (fracture surface) of the as-deposited sheet. Molecules of the vapor flow with a certain kinetic energy, when they collide with the condensation surface, go into an adsorbed state and exchange the energy with surface atoms, and move jump-like over the surface. The surface temperature determines the level of thermal activation of the adsorbed atom, number of jumps, probability of collision, interaction with other adsorbed atoms, and formation of their respective atomic configurations. According to an experimentally established schematic of structural zones [15], substrate temperature (T_s) is one of the main parameters that determine the structure of thick condensates. When $T_s/T_m < 0.3$ (T_m is the melting temperature of condensates in K), an amorphous or nanosize structure is formed. In the second high-temperature zone ($0.3 < T_s/T_m < 0.5$), condensates are characterized by a columnar structure with a predominantly crystallographic orientation. In the third high-temperature zone ($T_s/T_m > 0.5$), the columnar structure gradually changes to an equiaxial grain structure. T_s/T_m in this study belongs to the third temperature zone. However, as can be seen in Fig. 3(a), a typical columnar structure is formed. This is caused by the addition of Y₂O₃ in the condensate since Y₂O₃ can effectively inhibit grain growth in the matrix by pinning grain boundaries [16]. The surface morphology of the as-deposited alloy sheet is shown in Fig. 3(b) and (c). The terminal faces on top of the columns exhibit a pyramidal shape. At first glance, they look like octahedral planes on crystals with a cubic symmetry. It can be seen from Fig. 3(c) that each pyramidal tip corresponds to a columnar grain. Cracks can easily propagate along the interface of the columnar grain.

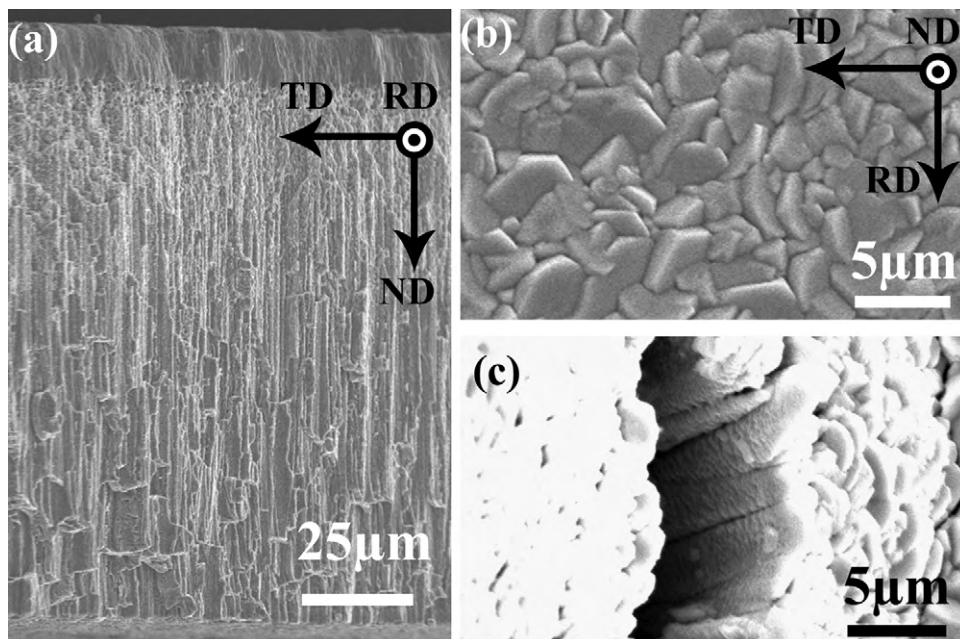


Fig. 3. SEM micrographs of the as-deposited Ni-based superalloy sheet: (a) cross-sectional morphology, (b) top surface and (c) crack on the top surface.

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