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NiCoCrAl/YSZ laminate composites fabricated by EB-PVD

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ABSTRACT

Two NiCoCrAl/YSZ laminate composites (A and B) with different metal-layer thickness (≈35 μm and 14 μm, respectively) were fabricated by electron beam physical vapor deposition (EB-PVD). Their microstructure was examined and their mechanical properties were compared with the 289 μm thick NiCoCrAl monolithic foil produced by EB-PVD. Both the YSZ and NiCoCrAl layers of the laminate composites had columnar grain structure. But the periodic layer interfaces limited the columnar grain size. Some pores between the columns were also observed. It was found that the strength of the laminate A was equal approximately to that of the NiCoCrAl monolithic foil, and that laminate B had the greater strength. Moreover, the density of the foils decreased with the increasing thickness ratio of YSZ/NiCoCrAl layers and the increasing the layer number. Thus, comparing with the NiCoCrAl monolithic foil, the NiCoCrAl/YSZ laminate composites not only had the equal or greater strength, but also had the much greater specific strength.

1. Introduction

Previous reports [1] of the extraordinary toughness and strength of mollusk shells have inspired much research because the shells, consisting of 99% calcium carbonate ($CaCO_3$), are hundreds of times tougher than simple polycrystalline limestone. These remarkable properties are related to the fine-scale structure of the shell, a laminate of thin calcium carbonate crystallite layers and tough biopolymers, arranged in an energy-absorbing hierarchical microstructure [2,3]. Based on the bio-inspired structure, a lot of researchers have produced the some ductile/brittle laminate composites by various techniques for applications in the aerospace industry [4–7].

Up to now, the study on the ductile/brittle laminate composites has focused mostly on metallic/intermetallic or metallic/ceramic systems [6–9]. At high temperature, the metallic/intermetallic multilayer structure is relatively instable due to the relatively easy diffusion of the elements between the metallic and intermetallic phases, which limits its application. While the layer structure in metallic/ceramic system is relatively stable due to the relatively difficult element diffusion between the two phases. However, the large residual stress resulting from the large difference in physical properties between metallic/ceramic phases has a great adverse effect on the mechanical property of the laminate composite. It has been reported that the ceramic coating or film produced by electron beam physical vapor deposition (EB-PVD) generally has the columnar grain structure, offering high strain tolerance [10]. As a result, the residual stress in the EB-PVD metallic/ceramic multilayer system may be relieved effectively.

In the present study, the NiCoCrAl/ZrO_2–Y_2O_3 (YSZ) laminate composites were fabricated by EB-PVD. And the microstructure and mechanical properties of the laminate composites were analyzed and discussed in detail.

2. Experimental

Two NiCoCrAl/YSZ laminate composites (A and B) were produced by EB-PVD. The EB-PVD equipment used in the present study contained two electron beam guns. A stainless steel disk substrate with a diameter of 1000 mm was mounted on the holder and rotated around the vertical axis. Ni–20Co–12Cr–4Al (wt%) and ZrO_2–8 mol%Y_2O_3 ingots of 68 mm in diameter and 250 mm in length were used as the evaporation sources. They were heated and evaporated by the two electron beam guns, respectively. The rotating substrate was preheated by resistance-wire. The maximum used EB power of each gun amounted to 60 kW. The process pressure during the deposition was in the range $6–10 \times 10^{-3}$ Pa. For comparison, a 289 μm thick metal monolithic foil was deposited from the Ni–20Co–12Cr–4Al (wt%) ingot by EB-PVD.

The process of deposition of the NiCoCrAl/YSZ laminate composites was conducted as follows. First, the substrate was heated and the substrate temperature was maintained at 650 °C approximately. Then to facilitate removal of the laminate composites from the substrate, a small amount of CaF_2 was evaporated and a thin

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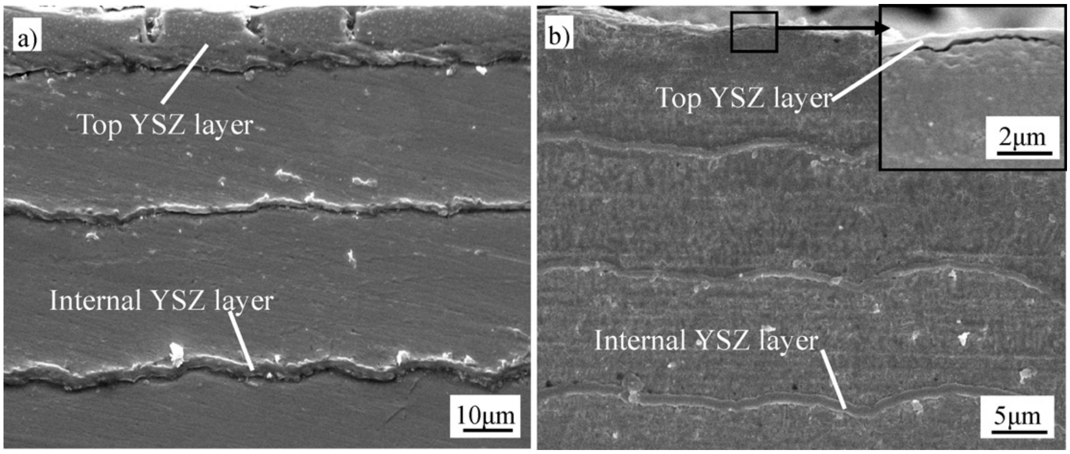

Fig. 1. SEM micrographs of the cross-sections of (a) laminate A and (b) laminate B.

(5–10 μm) separating layer of CaF_2 was deposited on the substrate surface. After that, the ingots of NiCoCrAl and YSZ were evaporated alternately to produce the laminate composites and the layer-thicknesses were controlled by their deposition time. The evaporation rates of NiCoCrAl and YSZ were about 1.4–1.8 and 0.4–0.6 μm/min, respectively. After completion of the deposition process and substrate cooling, the foils were stripped mechanically from the substrate.

The resulting layering and microstructures are shown in Fig. 1. It could be found that the layers were very distinct. Laminate A had 10 NiCoCrAl layers with thickness of 35 μm and 10 YSZ layers. Among the ceramic layers, the top YSZ layer was 6 μm thick, and the other 9 internal YSZ layers were 1 μm thick. Laminate B consisted of 13 NiCoCrAl layers with thickness of 14 μm, 12 internal YSZ layers having thickness of 1 μm and a top YSZ layer. But the top YSZ layer of laminate B was only 0.3 μm thick due to an accidental trouble of the EB-PVD equipment.

To assess the mechanical properties of the laminate composites, tensile specimens were milled from the as-deposited laminate composites. Tensile tests were conducted on the foils using an INSTRON-5569 universal materials testing machine with a crosshead displacement speed of 0.05 mm/min at room temperature. In this work, five specimens were tested to get an average value. Fracture surfaces of the specimens were examined by SEM. The cross-sectional microstructure of the specimens was observed with TEM and the phase identification was performed by X-ray diffraction (XRD). Moreover, the density of the laminate composites was measured by Archimedes method.

3. Results and discussion

3.1. Microstructure

XRD patterns were performed on the top surfaces of the laminate composites, as shown in Fig. 2. The diffraction patterns confirmed that the ceramic layers were predominantly of the non-transformable tetragonal phase (t' phase) and that the metal layers were mostly composed of γ phase. Because the top YSZ layer was too thin, t' peak of laminate B was not obvious.

Fig. 3a shows a typical cross-sectional microstructure of the YSZ layer of the laminate. In general, the EB-PVD YSZ coatings show an equiaxed structure during the early stages of nucleation, and then the columnar structure becomes more evident with increasing coating thickness [10–12]. The nucleation zone was very thin; as a result, the vast majority of the YSZ layer was the columnar grain zone. It has been reported that the wavy or banana-type microstructure was formed in EB-PVD YSZ coatings when the rotation speed of the substrate was relatively low [13]. The formation of the irregular column was attributed to the oscillation of the substrate temperature between maximum and minimum during one single revolution due to radiation heating from the evaporation ingot and crystallization heat. However, the YSZ layer did not form wavy columns in this study. Instead, straight columns were formed with a banded structure, as seen in Fig. 3a. This was due to the relatively high rotation speed of the substrate. Increasing the rotation speed reduces the amount of vapor particles arriving at the top of the columns and therefore the interval between each curve becomes shorter resulting in a banded structure [14]. Fig. 3b shows a typical fractured cross-section of the NiCoCrAl layer of the laminate. The straight columns with a banded structure were also observed on the fractured cross-section of the EB-PVD NiCoCrAl layer; and the ductile fracture zone indicated a microstructure differing from the columnar grain structure at the bottom of the NiCoCrAl layer.

From the TEM micrographs of laminate A shown in Fig. 4, the different microstructure between the upper and the lower zones of the NiCoCrAl layers was observed. The lower zone consisted of small size equiaxed grain structure. And the upper zone consisted of large size columnar grain structure. The columnar grains of the individual layers were periodically interrupted due to the multilayer structure, limiting the columnar grain size. In addition, nano

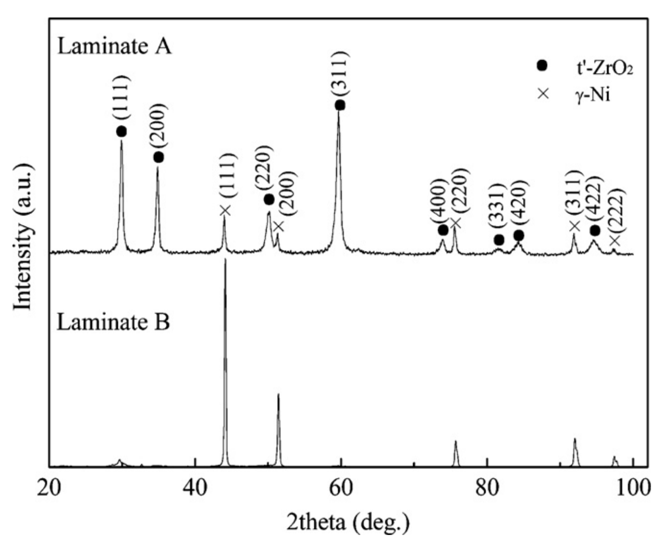

Fig. 2. X-ray diffraction patterns of the laminate composites deposited by EB-PVD.