COMPARISON OF TENSILE PROPERTIES OF TWO NiCoCrAl / YSZ MICROLAMINATES PRODUCED BY EB-PVD

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OVERVIEW

Electron-beam physical vapor deposition was employed to fabricate two kinds of metal / ceramic microlaminates: one was the multi-scalar microlaminate (MSML) with 5 thick layers of NiCoCrAl interspersed with 66 thin layer stacks of NiCoCrAl / ZrO_2-Y_2O_3 (YSZ), the other was the conventional microlaminate (CML) with only 50 layer stacks of NiCoCrAl / YSZ. Microstructures and fractographs were examined using SEM. Uniaxial tensile testing was performed to determine mechanical properties. It was found that both ceramic layers and metal layers consisted of columnar grains in the two composites. And columnar grains form clusters in which there was a relatively dense columnar grain structure. However there were gaps or holes between grain clusters. In room temperature tensile tests, both the composites exhibited brittle-like behavior without macroscopic plastic deformation, but the fracture strength of CML was greater than that of MSML. Fractography Examination of the tensile samples revealed that the ceramic layers of both composites failed by brittle intergranular fracture between columns, however, their metal layers differed in failure mode. In MSML, the metal layers failed mostly by ductile microvoid coalescence and brittle intergranular fracture, while only one predominat failure mode was observed in the metal layers of CML, namely, chisel point failure. It was also found that the dominant failure modes of metal-layers depended on their thickness and the critical thickness of metal layer was between 12 μm and 20 μm. Furthermore, Interfacial debonding in both microlaminates was apt to happen at the downside of metal layers rather than at their upside.

KEYWORDS: EB-PVD; microlaminate; microstructure; mechanical property

1. INTRODUCTION

Metal / ceramic composites have been a topic of interest for many researchers for various reasons [1-5]. The motivation for developing metal / ceramic composites is to fabricate structures that possess superior high temperature creep resistance compared to metals, simultaneously having better toughness and structural integrity compared to a monolithic ceramic [6, 7]. In various ductile / brittle composite systems, there are numerous reinforcement shapes employed, including particulates, fibres and laminate reinforcements. Early studies show that ductile layer reinforced composites generally promote greater levels of toughening than ductile particulate or fibre reinforced composites [8-10].

A microlaminate comprised of alternating layers of ceramic and ductile metal is a particularly attractive composite architecture. One major advantage of microlaminates is that the distribution and volume fraction of the phases can be readily controlled by altering the layer thickness [11]. The small size scales lead to increases in the ductile layer strength from enhanced constraint and, possibly, various strengthening mechanisms. In principle, smaller length scales may also increase the ceramic material cracking stresses by limiting the size of potential processing flaw [12]. But the dislocation motion that causes relaxation of residual stress and plastic deformation will be resisted if the individual layer thickness is too thin. To optimize the strength and toughness of microlaminates, multiscalar microlaminates have become interesting for some researchers [13, 14].

So far, only a few studies have been performed to investigate microlaminate composites [15-21], and most of them only concentrate on metallic / intermetallic systems. The investigation of metal / ceramic microlaminate is far insufficient. In the present study, we examine the tensile properties of two NiCoCrAl / YSZ microlaminates, and discuss the effect of layer thickness on their fracture behavior.

2. EXPERIMENTAL PROCEDURE

Electron beam physical vapor deposition was used to produce two NiCoCrAl / YSZ microlaminates. The deposition equipment consisted of six electron beam guns and a three-ingot continuous feeding system. A rectangular steel substrate was mounted on the holder and rotated at 25 rpm around the horizontal axis. YSZ and NiCoCrAl ingots of 70 mm in diameter and 200 mm in length were used as the evaporation sources. They were heated and evaporated by two different guns respectively. Another gun was used for preheating the rotating substrate. The maximum used EB power of each gun amounted to 60 kW. The process pressure during the deposition was in the range $6-10\times10^{-3}$ Pa.

During the deposition process, the substrate temperature was maintained at 950℃ approximately. To facilitate removal of the microlaminate from the substrate, the steel substrate was coated with 10－20 μm of ZrO_2 prior to depositing the microlaminates. YSZ and NiCoCrAl ingots were evaporated alternately to produce the microlaminates and layer thickness was determined by controlling their deposition time. The resulting MSML foil contained a total of 71 layers: thirty-six about 1 μm layers of YSZ, thirty about 1.6 μm thin layers of NiCoCrAl and five thick NiCoCrAl layers with different layer thicknesses, namely, layer Ⅰ, layer Ⅱ, layer Ⅲ, layer Ⅳ and layer Ⅴ. From layer Ⅰ to layer Ⅴ, layer-thicknesses were 35 μm, 20 μm, 12 μm, 10 μm and 6 μm respectively. And the resulting CML foil contained 50 ceramic layers (≈ 1.2 μm) and 50 metal layers (≈ 5 μm).

To assess the mechanical properties of the microlaminates, tensile specimens were milled from free standing microlaminate foils. Prior to tensile testing the edges of the specimens were mechanically polished. Tensile tests were conducted on the microlaminates using an INSTRON-5569 universal materials testing machine with a crosshead displacement speed of 0.01 mm/min at room temperature. Subsequently their fracture surfaces were observed with SEM.

3. RESULTS

Cross-sectional SEM micrograph of MSML shows that both ceramic layers and metal layers of EB-PVD microlaminates consist of columnar grains which are normal to layer planes (FIG 1). And columnar grains form clusters in which there are relatively dense columnar grain structures. However there are gaps or holes between grain clusters. And the pores are more or larger in thick metal layers than ones in thin layers, which results from the columnar grain size and porosity increasing with increasing coating thickness [22, 23]. The high porosity has an adverse effect on the strength of microlaminates.

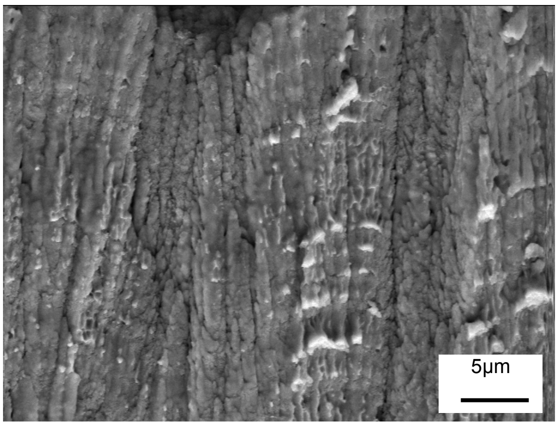

FIG 1. Cross-sectional microstructure of MSML

The stress-strain curve is one of the fundamental properties that determines mechanical properties in ductile phase reinforced composites. FIG 2 shows the stress-strain curves for the as-deposited NiCoCrAl / YSZ miocrolaminates tested at room temperature. Both MSML and CML exhibit typical characteristics of linear elastic fracture with no evidence of yielding. In the case, elastic loading is terminated by fast fracture at a peak load. CML shows a greater fracture strength (≈ 328MPa) and a greater failure strain (≈ 0.0022) than MSML. And the fracture strengths obtained for both CML and MSML are greater than those obtained by Vill. et al. on Mo / W multiscalar microlaminates [15]. The reason why the tensile strength of MSML is lower is that thicker metal layers of MSML has higher porosities.

The study on the fracture surfaces of tensile specimens shows the ceramic layers of both MSML and CML fail by intergranular brittle fracture between columns. And, secondary cracks vertical to layer planes in ceramic layers and bridging metal layers are occasionally observed, as shown in FIG 3b and FIG 4b.

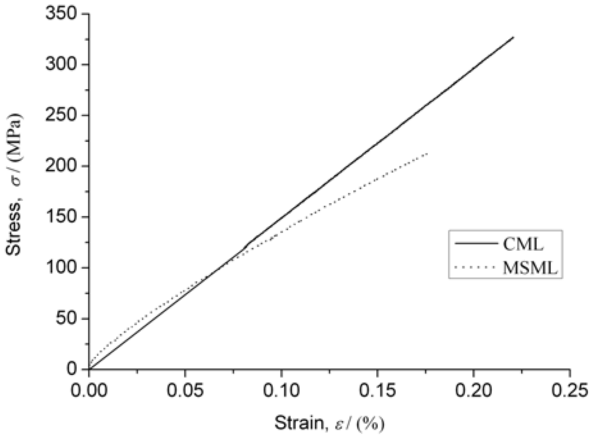

FIG 2. Stress-strain curves of NiCoCrAl / YSZ microlaminates

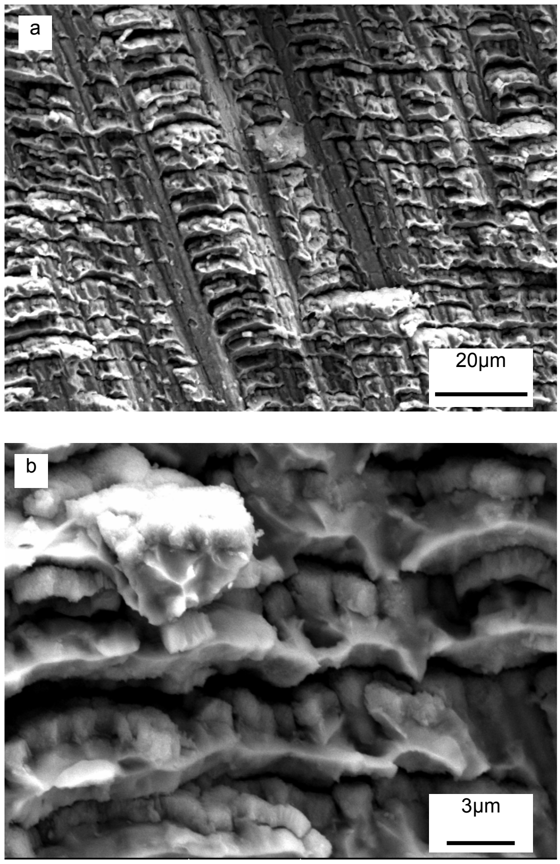

FIG 3. SEM micrographs of the fracture surfaces of CML tensile specimens tested at room temperature

The metal layers of MSML and those of CML display different failure modes. In CML, metal layers fail mostly by chisel point failure with fracture surfaces necked to 'knife edge' features (FIG 3). There are only very little regions of brittle intergranular fracture in the metal layers. Interfacial debonding is very evident and usually happens at the downside of metal layers rather than at their upside, which indicates the boundary adhesion strength formed by ceramic vapor depositing on metal layers is greater than the one formed by metal vapor depositing on ceramic layers.

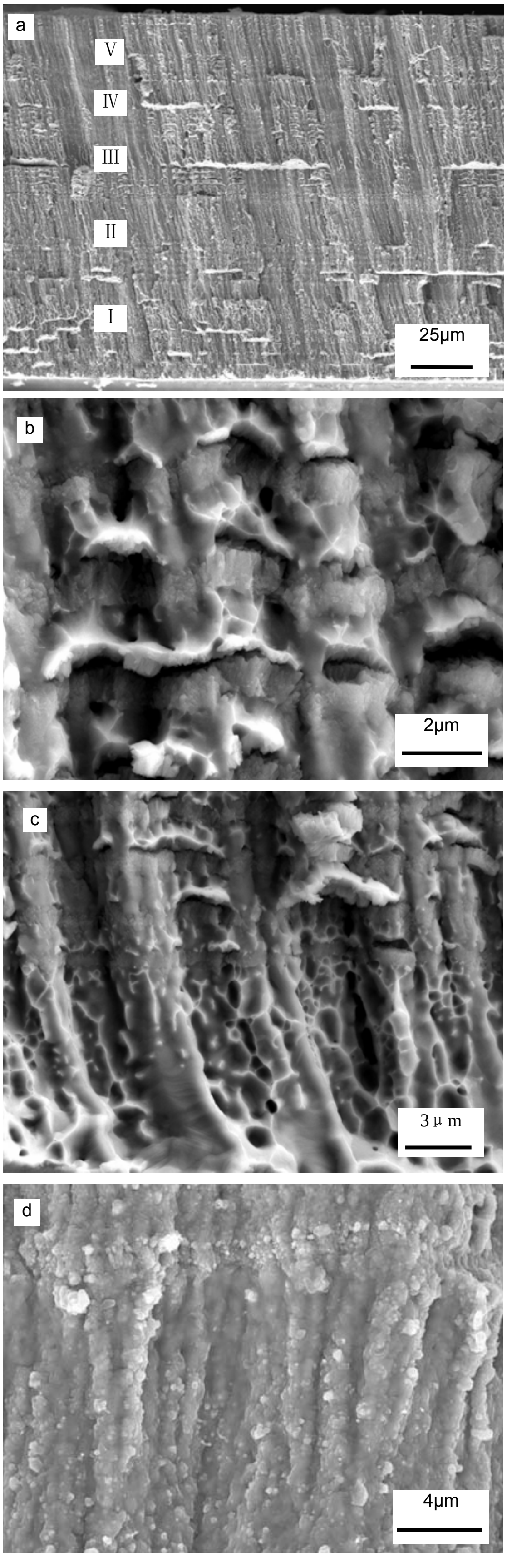

FIG 4. SEM micrographs of the fracture surfaces of MSML showing metal-layers with different thickness, (b) micrographs of the fracture surfaces of thin metal-layers, (c) micrograph of the fracture surface of layer Ⅲ, (d) micrograph of the fracture surface of layer Ⅱ

The metal layers in MSML display features of both ductile and brittle fracture as shown in FIG 4. Ductile failure occurs by the growth and coalescence of microvoids in the metal layers. Interfacial debonding is also observed at the downside of metal layers. Necking along layer thickness direction and shear lips are observed at the edges of thin metal layers near the debonding interfaces, but they don't occur at the edges near the firm combining interfaces due to the constraint of adjacent ceramic layers. There is little evidence of necking in ductile fracture region of thick metal-layer, but there is more evidence of interfacial debonding.

There is a distinct transition from ductile microvoids coalescence to brittle fracture associated with a corresponding transition between stable (slow) and unstable (fast) fracture in almost every metal layer of MSML, as shown in FIG 4a. The brittle failure also occurs by intergranular fracture in MSML due to the large gaps between columnar grain clusters (FIG 4d). Where the metal layers fail by brittle fracture, no interfacial debonding appears, which indicates the constraint imposed on the metal layer by the ceramic layer has an important effect on restricting plastic stretching and promoting brittle fracture. A similar response has been reported in ductile / brittle laminates [12, 13].

The size of brittle fracture region of metal-layer in MSML increases with the increase of layer-thickness. As a result, a large proportion of fracture surfaces are ductile fracture regions in layer Ⅲ, layer Ⅳ, layer Ⅴ and all thin metal layers with layer-thicknesses less than 12 μm. In other words, the thinner metal layers display mostly features of ductile fracture. While brittle fracture regions become dominant on fracture surfaces of Layer Ⅰ and layer Ⅱ whose thicknesses are larger than 20 μm. That is to say that the two thicker metal-layers fail mostly by brittle fracture. So it can be concluded that the dominant failure modes of metal-layers depend on their thicknesses and the critical thickness of metal layer is between 12 μm and 20 μm. When layer thicknesses are less than the critical thickness, the predominant failure mode of metal layers is ductile; when layer thicknesses exceed the critical value, metal layers have poor ductility and fail mostly by brittle fracture. The metal-layer thickness of CML is less than the critical value and its metal-layers fail mostly by ductile fracture, which is consistent with the above conclusion.

However, the ductile fracture mode of metal-layer in MSML and that in CML were different. The reason is related not only to metal-layer thickness but also to the distribution and volume fraction of metal-layer. Another study is underway to clarify the reason.

4. CONCLUSIONS

1) Two NiCoCrAl / YSZ microlaminates (MSML and CML) are fabricated by EB-PVD. Both of them exhibit a brittle-like behavior without macroscopic plastic deformation in room temperature tensile tests and CML shows a greater

fracture strength (≈ 328MPa) and a larger failure strain (≈ 0.0022) than MSML.

2) The ceramic layers in both microlaminates fail by intergranular brittle fracture between columns. Secondary cracks vertical to layer planes in ceramic layers and bridging metal layers are occasionally observed. However, the metal layer of MSML and that of CML display different failure modes. Metal layers fail mostly by chisel point failure in CML, while metal layers displayed two predominant fracture modes in MSML, namely, ductile microvoid coalescence and brittle intergranular fracture.

3) Interfacial debonding is seen in both microlaminates and usually happens at the downside of metal layers rather than at their upside.

4) The dominant failure modes of metal-layers depend on their thicknesses and the critical thickness of metal layer is between 12 μm and 20 μm. When layer thicknesses are less than the critical thickness, metal layers fail mostly by ductile fracture; when layer thicknesses exceed the critical value, metal layers fail mostly by brittle fracture.

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