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Crack propagation in NiCoCrAl/YSZ multilayer film produced by EB-PVD in crack arrester orientation

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Abstract. A multilayer film containing 10 NiCoCrAl layers with thickness of about 35 μm and 9 $\text{ZrO}_2\text{-Y}_2\text{O}_3$ (YSZ) layers with thickness of about 1 μm was fabricated by electron beam physical vapor deposition (EB-PVD). Considering the difficulty to machine a surface notch in the thin film, a 6 μm thick YSZ layer with the columnar structure was deposited as the outer layer of the multilayer film, which was used to introduce the initial crack tip normal to the film surface. The crack propagation in the crack arrester orientation was analyzed by in situ examining the effect of microstructure. The R-curve and the interface toughness for delamination were determined. It was found that crack propagated along the columnar boundaries in the YSZ layers with the negligible crack growth resistance and that a small part of NiCoCrAl columnar boundaries provided relatively low resistance to crack growth due to the intercolumnar pores. However, most of NiCoCrAl columnar boundaries provided relatively high resistance to crack growth. Moreover, the resistance to crack-growth increased with increasing crack-length. The crack deflection at the layer interfaces and the plastic deformation of NiCoCrAl layers were the important factors resulted in the increasing resistance.

1. Introduction

Current interest in multilayer films is motivated by a number of factors such as the desire to advance microelectronics by moving to smaller device sizes, the need for protective or functional coatings, and the high degree of promise for high temperature structural materials in the aerospace industry [1-4]. Physical vapor deposition (PVD) is one of the most commonly used preparation technologies for the multilayers [5-8]. However, the PVD multilayers usually show a columnar structure which was not desired for multilayer films, as cracks may develop along vertical grain boundaries and cause the films failure between columns [5-8]. Therefore, it is necessary to study the crack propagation in the PVD multilayer films.

Crack propagation in multilayer materials can be studied in two extreme orientations. In the crack arrester orientation (crack propagating perpendicularly to the interfacial planes), an initial notch/crack tip ends within an individual layer of a test multilayer sample such that the crack front “sees” each layer interface sequentially during loading. In the crack divider orientation (crack propagating parallel to the interfacial planes), the initial notch/crack tip intersects all the layers of the test sample such that the crack front “sees” all the layer interfaces simultaneously. Some articles have been published on crack propagation in crack divider orientation for multilayer films [9, 10]. Due to the small dimension of the films in thickness, micron-level initial notches/cracks normal to their surfaces are needed in



order to study their crack propagation in crack arrester orientation, which are very difficult to be prefabricate. Furthermore, there are many standard tests that enable precise measurement of the fracture properties of materials at a macroscopic length-scale, yet few available tests that enable direct measurement of crack propagation in multilayer films or coatings at a microscopic length-scale. Recently, X-ray imaging method has been used to detect in situ the micro-cracks in multilayer ceramic capacitors and composite laminates [11, 12]. Moreover, In-situ SEM is also an efficient and commonly used method to study the micro-crack propagation [13, 14].

In this paper, we reported a new method to prefabricate the initial crack tip normal to the surface of a NiCoCrAl/YSZ multilayer film fabricated by electron beam physical vapor deposition (EB-PVD). Then the crack propagation of the multilayer film in the crack arrester orientation was studied by in situ bending test carried out in a Scanning Electron Microscopy (SEM) chamber. Moreover, the influence of the columnar structure on the crack propagation was discussed in detail.

2. Experimental procedure

A NiCoCrAl/YSZ multilayer film was produced by EB-PVD. The deposition parameters and process have been described in our previous studies [15]. The resulting multilayer film was 0.37 mm thick, consisting of 10 NiCoCrAl layers (about 35 μm thick) and 10 YSZ layers. Due to the small dimension in thickness, it was too difficult to prefabricate the initial notch/crack tip normal to the film surface. A number of researches have demonstrated that the EB-PVD YSZ films or coatings generally had a columnar structure and that the intercolumnar interfaces were weak and easily cracking [16]. Based on the feature, the outer YSZ layer (6 μm thick) of the multilayer film was designed to be much thicker than the other 9 YSZ layers (1 μm thick). Thus, the initial crack tip normal to the film surface could be easily formed in the outer YSZ layer under relatively low bending load, and the crack propagation behavior in the crack arrester orientation could be observed.

Transmission Electron Microscope (TEM) was used to observe the microstructure of the multilayer film. Several samples were cut from the prepared multilayer film for three-point bending tests. The thickness, W , of a bend sample was the one of the multilayer film (0.37 mm). Its width, B , and span, S , were 6 mm and 30 mm, respectively. Three-point bend test was performed in the vacuum chamber of the SEM that was equipped with a loading stage with a displacement range of 10 mm. First, the three-point bending test was used to prefabricate an initial crack of a bend sample with a bending load of 15.0 N. After the sample was unloaded, a single-edge cracked sample was obtained. The initial crack was located in the outer YSZ layer of the sample such that the crack fronts “saw” each layer interface sequentially during loading, as illustrated in Figure 1. Resistance curve toughness (K_{Ic}) vs. crack extension (Δa) measurements were made further by three-point bending test on the single-edge cracked samples. The precracked samples were incrementally loaded to the maximum displacement range with a crosshead displacement speed of 0.5 mm/min. Crack propagation was monitored as a function of load increase, and the interaction of the propagating crack with surface microstructure was recorded by taking photographs. After the in situ bend test, the fracture surface was examined by SEM. For comparison, fracture property of a 0.3 mm thick NiCoCrAl monolithic foil produced by EB-PVD was also studied by the in situ bend test, using a load direction normal to the film surface.

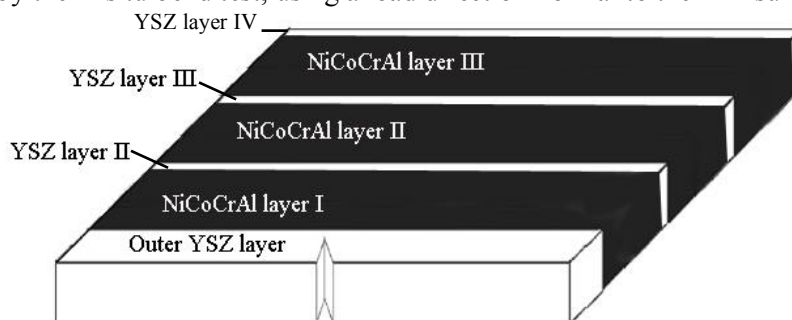


Figure 1. Schematic representation of sample geometry.

3. Results and discussion

Our previous studies have demonstrated that both the EB-PVD YSZ and NiCoCrAl deposits had columnar structures and the heights of the columns were approximately equal to their layer-thicknesses [15]. And a number of intercolumnar pores or gaps in the YSZ deposits have also been reported [17]. Figure 2 shows the TEM micrograph of the NiCoCrAl deposit produced by EB-PVD. Most of NiCoCrAl columnar grains bonded tightly. However, some micro-pores were observed on a few column boundaries. The micro-pores were distributed discontinuously along the NiCoCrAl column boundaries. It had been reported that the micro-pores along the EB-PVD column boundaries were formed due to so-called shadowing effect dependent on vapor incidence pattern or vapor incident angles of vapor flux [18, 19].

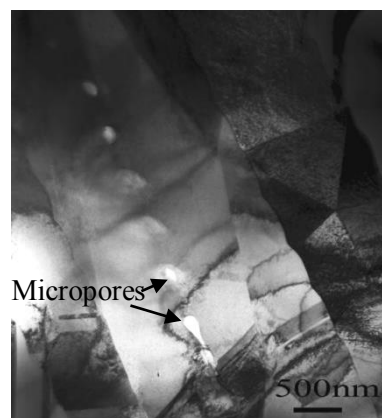


Figure 2. TEM micrograph of the NiCoCrAl deposit produced by EB-PVD.

Figure 3 shows a typical single-edge cracked bending load-displacement curve for the NiCoCrAl/YSZ multilayer film. Figure 4a-f shows a typical process of crack evolution in the NiCoCrAl/YSZ multilayer film during the three-point bending test. The initial crack was prefabricated with a three-point bending load of 15.0 N as shown in Figure 4a. It propagated in the outer YSZ layer but was arrested at the interface between the outer YSZ layer and NiCoCrAl layer I. Thus the precrack length was equal to the outer YSZ layer thickness. Before bending load reached 25.0 N in the subsequent three-point bending test, the initial crack didn't grow evidently, which proved that the outer YSZ layer was more easily cracking than the other layers. At the load of 25.0 N, the crack grew into the NiCoCrAl layer I about 3 μm and then was blunted (Figure 4b). With the load increasing to 25.3 N, two (or more) cracks nucleated in the YSZ layer II, as indicated by the white arrows, and propagated rapidly forward and backward across the layer interfaces (Figure 4c). But the cracks were blunted very soon due to the plastic deformation of the NiCoCrAl layers, and the unbroken NiCoCrAl layer I generated a zone of bridging ligaments. This proved that the YSZ layers were easily cracking and assisted in crack propagation in the multilayer film. Afterward, the small increment (1 N) in load resulted in that the crack renucleated in the YSZ layer III and propagated forward and backward (Figure 4d). At 27 N, the debonding along the interface between NiCoCrAl layer III and YSZ layer IV was observed (Figure 4e). Before the load increased to 29.5 N, the main crack didn't grow evidently, but some microcracks nucleated and propagated along the direction vertical to the interfacial planes in the NiCoCrAl layers. At the same time, the interface debonding between NiCoCrAl layer III and YSZ layer IV became more and more obvious. When the load reached 29.5 N, the sample failed by the abrupt fracture of the bridging NiCoCrAl layers (I-III), as shown in Figure 4f and g. A number of secondary microcracks near the fracture surface in the fractured NiCoCrAl layers indicated that main crack could advance rapidly by the linking up of microcracks ahead of the main crack in the NiCoCrAl layers. Considering the intercolumnar micropores could cause local stress concentration, they probably acted as sites for microcrack initiation and/or assist in microcrack propagation forward

and backward in the NiCoCrAl layers. Moreover, some bridging ligaments between the microcracks were observed, which offered the potential to shield an advancing crack and to absorb energy through plastic deformation.

The NiCoCrAl monolithic film specimen did not break in the three-point bending test because of the limited displacement range (10 mm) provided by the loading stage. Figure 5 shows the micrograph of the NiCoCrAl monolithic film specimen at the maximal displacement in the three-point bending test. During the displacement increased from 0 to the maximum (the corresponding maximal load being 28.2 N), no crack was observed on the cross-section of the NiCoCrAl monolithic film, which indicated that the crack nucleation and propagation along the NiCoCrAl column boundaries was relatively difficult for the film without a relatively large precrack. Moreover, the NiCoCrAl/YSZ multilayer film was deposited at 650 °C [15]. After cooling of the multilayer film, there were a tensile residual stress in the NiCoCrAl layers and a compressive residual stress in the YSZ layers, resulting from the large difference in thermal expansion coefficients between metallic/ceramic layers, which made the NiCoCrAl layers easier to fracture than the NiCoCrAl monolithic film.

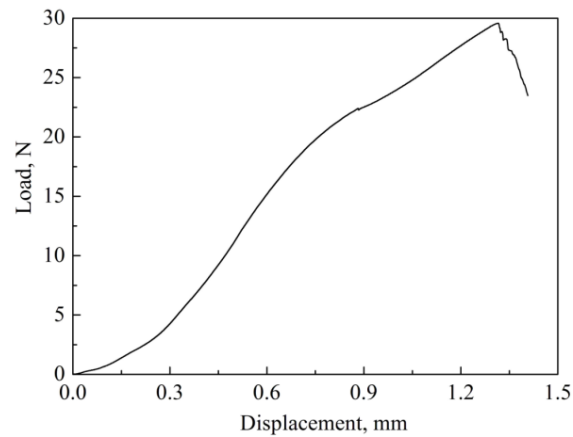


Figure 3. Typical single-edge cracked bending load-displacement curve for the NiCoCrAl/YSZ multilayer film.

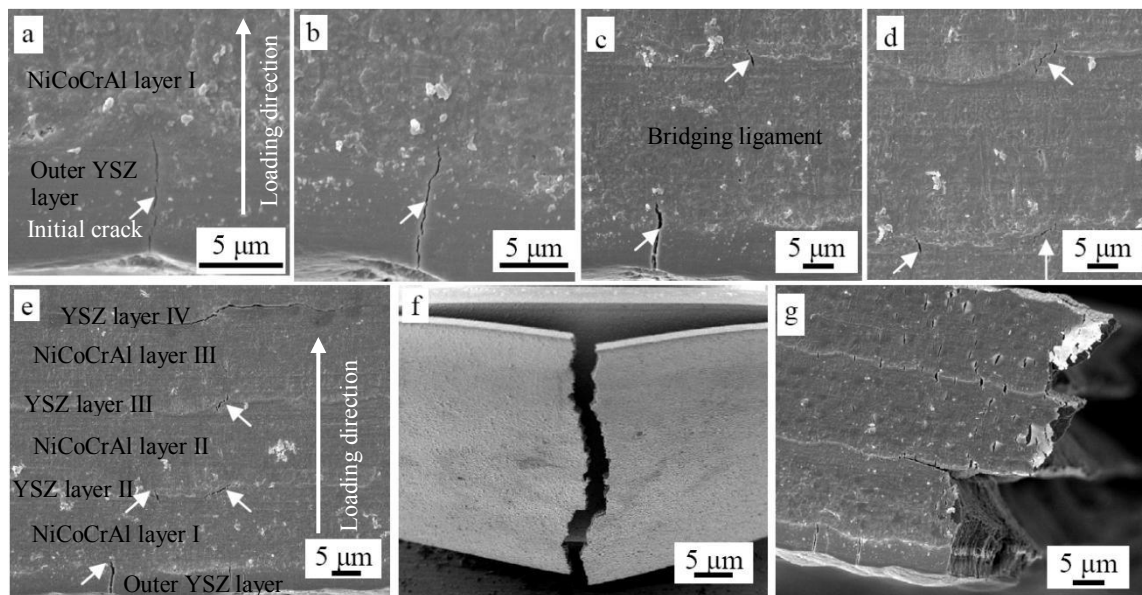


Figure 4. Crack growth in the NiCoCrAl/YSZ multilayer film under different bending loads: a) 15 N, b) 25 N, c) 25.3 N, d) 26.1 N, e) 27 N, f) and g) 29.5 N.

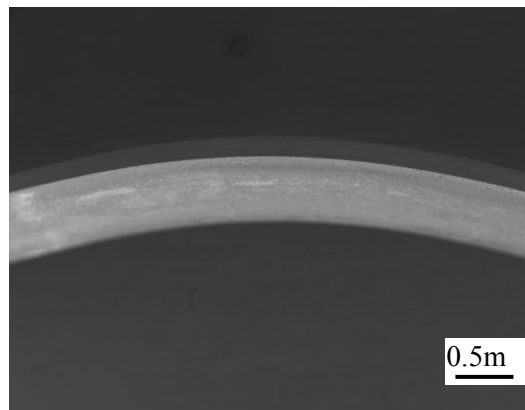


Figure 5. Microstructure of the NiCoCrAl monolithic film specimen at the maximal displacement in the three-point bending test.

The SEM micrograph of the fracture surface of the multilayer film sample is shown in Figure 6. It was found that the fracture surface of every NiCoCrAl layer was divided into two different zones. The upper parts of NiCoCrAl layers which cracked first showed the features of brittle intercolumnar fracture. With the further expansion of the crack, the brittle fracture changed into the ductile shear fracture in the middle and lower parts of the NiCoCrAl layers. It indicated that the crack propagation took place on weak intercolumnar interfaces in the NiCoCrAl layers at the early stage of crack propagation, however, ductile failure occurred by the growth and coalescence of microvoids at the later stage of propagation. It was reported that the columnar structure was more evident in the upper parts of EB-PVD coatings than that in their lower parts [17]. This may be why the upper parts of NiCoCrAl layers failed mostly brittle intercolumnar fracture.

The effect of columnar structures on the crack propagation in the NiCoCrAl/YSZ multilayer film was significant. YSZ layer II and III fractured prior to the NiCoCrAl layers during the bending test of the multilayer film. This demonstrated that the YSZ intercolumnar interfaces were weak and easily cracking, consistent with the previous report [16]. The brittle intergranular fracture in the upper parts of NiCoCrAl layers indicated that more evident columnar structure provided lower resistance to crack growth. Because the columnar structure became more evident with increasing EB-PVD coating thickness [13], decreasing the NiCoCrAl layer thickness may be beneficial to improve the crack growth resistance of the NiCoCrAl/YSZ multilayer film. Although the upper parts of NiCoCrAl layers were relatively easily cracking, the cracks could still be blunt and deflected at the layer interfaces.

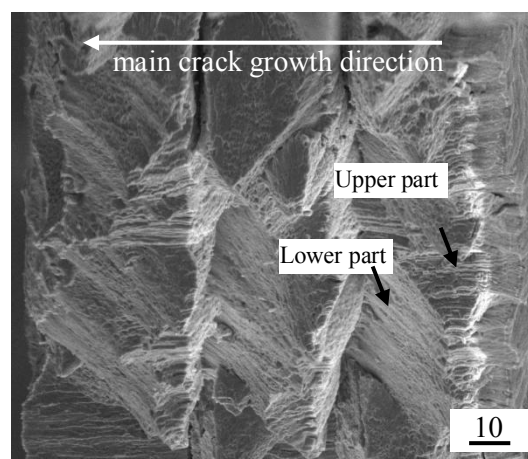


Figure 6. SEM micrograph of the fractured cross-section of the multilayer film.

Stress intensity factor was calculated as follows, in accordance with the ASTM standard E399-90:

$$K_I = PS / (BW^{3/2}) \times f(a/W) \quad (1)$$

where K_I is the stress intensity factor, P is the load, a is the crack length, and $f(a/W)$ is the function defined as:

$$f(a/W) = 3(a/W)^{1/2} [1.99 - (a/W)(1-a/W) \times (2.15 - 3.93a/W + 2.7a^2/W^2)] / [2(1+2a/W)(1-a/W)^{3/2}] \quad (2)$$

The typical resistance curve for the NiCoCrAl/YSZ multilayer film is plotted in Figure 7. The initial stress intensity factor value was $6.5 \text{ MPa m}^{1/2}$. The crack-growth resistance increased with the increased crack-length, and reached $29.2 \text{ MPa m}^{1/2}$ before NiCoCrAl layer I~III fractured. The crack deflection at the layer interfaces (in Figure 4e, f and g) and the plastic deformation of NiCoCrAl layers (in Figure 6) were believed to be the important factors resulted in the increasing crack-growth resistance.

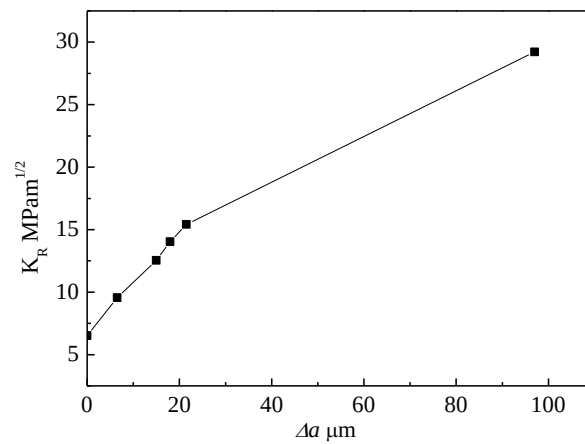


Figure 7. R-curve for the NiCoCrAl/YSZ multilayer film.

Interface toughness of the multilayer film can be determined based on the critical load for delamination. The analytical expression for the energy release rate of a bend sample is given by [20]

$$G = \frac{M^2(1-\nu^2)}{2E} \left(\frac{1}{I_2} - \frac{1}{I_1} \right) \quad (3)$$

where the inertia of the laminate I_1 , and that of the uncracked ligament I_2 , are given by

$$I_1 = \frac{h_1^3}{12} + \frac{h_2^3}{12} + \frac{h_1 h_2 (h_1 + h_2)}{4} \quad (4)$$

$$I_2 = \frac{h_2^3}{12} \quad (5)$$

where E is the elastic modulus (200 GPa), ν is the Poisson's ratio (0.3), M is the applied bending moment which linearly varies from zero at the supports to

$$M = \frac{PS}{4B} \quad (6)$$

at the midspan in which the load P is applied. The geometric parameters h_1 and h_2 were the thicknesses of the cracked part and uncracked part of the sample, respectively.

The stress intensity factor characterising the interface favourable to delaminate is obtained using the following equivalence [21]

$$K_c = \left(\frac{G_c E}{1-\nu^2} \right)^{1/2} \quad (7)$$

At the load of 27 N, the interfacial debonding occurred, as shown in Figure 3d. Thus, the critical stress intensity factor for delamination was determined to be about $14.6 \text{ MPa m}^{1/2}$. It is notable that the interface toughness value was slightly higher than the corresponding K_{Ic} ($\approx 14.0 \text{ MPa m}^{1/2}$). This was attributed that a lot of interior microcracks caused by intercolumnar pores were distributed in the NiCoCrAl layers, which could not be observed on the surface and side of the sample.

4. Conclusions

The initial crack tip normal to the layer interface of the NiCoCrAl/YSZ multilayer film was prefabricated in the three-point bending test, through the deposition of a $6 \mu\text{m}$ thick YSZ layer with the columnar structure on the surface of the multilayer film. It was found that the initial crack could form in the outer YSZ layer under the relatively low bending stress, at the same time, no evidence of any damage was observed for the internal YSZ and NiCoCrAl layers of the multilayer film. This method of prefabricating the initial crack in the crack arrester orientation is also applicable for other films. Crack propagation behavior was studied in the crack arrester orientation. It was found that crack propagated in the YSZ layers with very small resistance, but in the NiCoCrAl layers with much greater resistance. The intercolumnar pores were observed and were believed to accelerate the crack growth in the NiCoCrAl layers. The R-curve behavior and interface toughness of the multilayer film was investigated. The initial stress intensity factor value was $6.5 \text{ MPa m}^{1/2}$. The crack growth resistance increased with increasing crack-length. The critical stress intensity factor for delamination of the interface was $14.6 \text{ MPa m}^{1/2}$.

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