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Ni/Ni_3Al microlaminate composite produced by EB-PVD and the mechanical properties

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Abstract

Microlaminates with alternating layers of Ni and Ni_3Al were fabricated by using EB-PVD method. The influences of deposition rates and substrate temperature on morphology of the microlaminates were investigated. The results showed that in order to produce Ni/Ni_3Al microlaminate with lower porosity and prevent the formation of columnar grains, higher deposition rates and substrate temperature were required. The mechanical properties of the microlaminates were examined using tensile tests. The fracture behavior was temperature and interlamellar spacing dependent, with a lower tensile strength at higher temperature or with larger interlamellar spacing.

Keywords: Electron beam physical vapor deposition (EB-PVD); Composite materials; Multiplayer structure; Tensile test

1. Introduction

High temperature structural intermetallic compound, Ni_3Al has been intensively studied due to its potential high strength and high temperature applications in the aerospace industry, but it has been vexed with persistent physical limitations, such as low temperature brittleness. One approach to compensate for its inherent limitations is to reinforce the brittle intermetallic matrix with modest volume fractions of ductile refractory metals [1–3]. A microlaminate comprised of alternating layers of intermetallic and ductile metal is one particularly attractive composite architecture, which exhibits the high temperature properties superior to intermetallics or superalloys [4–6]. Moreover, microlaminates have the advantage that the distribution and volume fraction of the phases can be readily controlled by altering the layer thickness.

Previous investigations mainly focused on fabricating microlaminates using sputtering methods [7,8]. The main disadvantages of sputtering process are the slow deposition rates and the difficulty to apply oxide coating efficiently. In recent years, electron beam physical vapour deposition (EB-PVD) method, which is usually used for fabricating coatings and films, opened a new era of fabricating microlaminate composites. The principle of this operation is to heat a target to evaporation point by electron beam in a vacuum chamber. The evaporated atoms emanating from the target then condense onto the substrates. This process allows the deposition of metallic coating, cermets or oxide compounds and offers many desirable characteristics such as flexible deposition rates (10 nm/min up to 150 μm/min, depending on the materials), dense films or coatings, precise composition control, capability of producing multilayered metallic and ceramic laminates on large components, low contamination and high thermal efficiency [9].

In this paper, we detail the processing of Ni/Ni_3Al microlaminates composites by EB-PVD method and investigate their mechanical properties.

2. Experimental procedure

Fig. 1 shows the EB-PVD equipment used in the present study. Six electron beam guns (I–VI) are installed in the two gun chambers isolated from the working chamber and having an independent pumping-down system. A stainless

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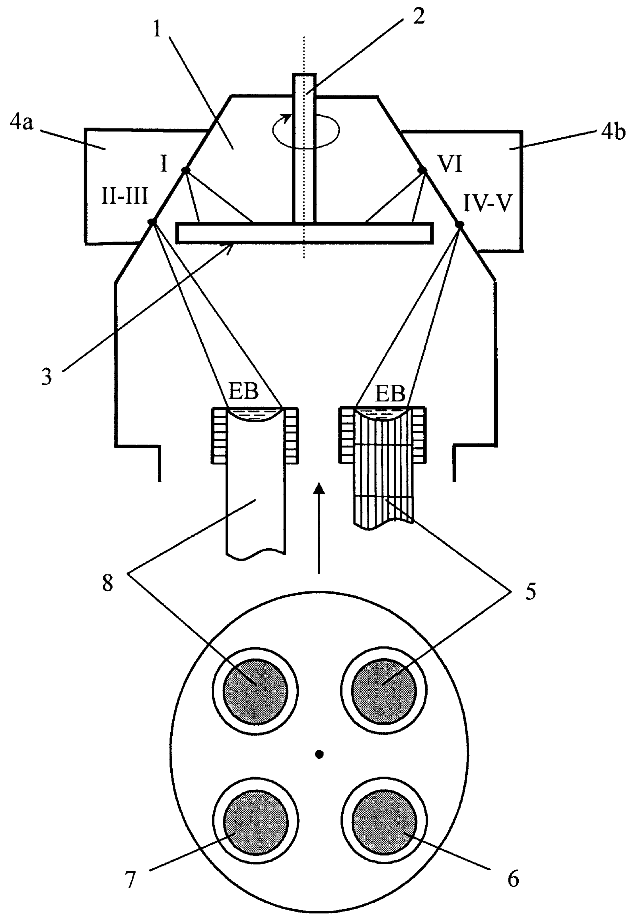

Fig. 1. Schematic drawing of EB-PVD equipment. 1—Working chamber; 2—vertical shaft; 3—rotating substrate; 4a,4b—gun chambers; 5,6—reserve ingots; 7—Ni ingot; 8—Ni_3Al ingot; I–VI—electric beam guns.

steel disk substrate with a diameter of 350 mm is mounted on the holder and rotates around the vertical axis. Ni and Ni_3Al ingots of 50 mm in diameter and 200 mm in length were used as the evaporation sources. They were heated and evaporated by guns II and III, respectively. Guns IV and V are used for evaporation of reserve ingots 5 and 6. Guns I and VI are 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 $6-10 \times 10^{-3}$ Pa.

The process of deposition of the Ni/Ni_3Al microlaminate composite is conducted as follows: First, guns I and VI are used to preheat the substrate and maintain the temperature at a certain value. Then to facilitate removal of the microlaminates from the substrate, a small amount of CaF_2 is evaporated from the surface of reserve ingot 5, and a thin (5–10 μm) separating layer of CaF_2 is deposited on the substrate surface. After that, guns II and III are switched on. Ingots 7 and 8 (Ni and Ni_3Al) are evaporated alternatingly and microlaminates with alternating layers of Ni and Ni_3Al were deposited of equal thickness. The total thickness for the microlaminate was 1 mm. In the experiment, the evaporation rates of Ni and Ni_3Al were about 5–10 and 2–5 μm/min, respectively.

After completion of the deposition process and substrate cooling, the Ni/Ni_3Al microlaminate is separated from the substrate. The resulting laminates had interlamellar spacings ranging between 0.2 and 2 μm.

To assess the mechanical properties of the microlaminates, tensile specimens were milled from the as-deposited samples directly and from the samples after heat-treatment at 1050 °C for 3 h. 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, 500 and 600 °C. In this work, four to five specimens were tested for each temperature to get an average value. The microstructure of the cross-sections of as-deposited specimens and the fracture

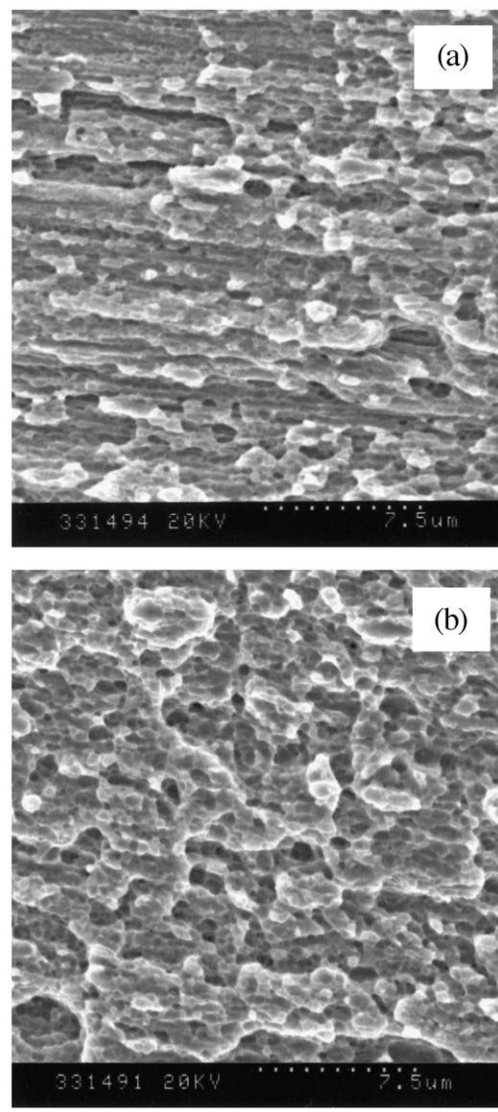

Fig. 2. SEM micrograph of Ni/Ni_3Al microlaminates deposited at different deposition rates of Ni (a) 2.0 μm/min (b) 5.0 μm/min.

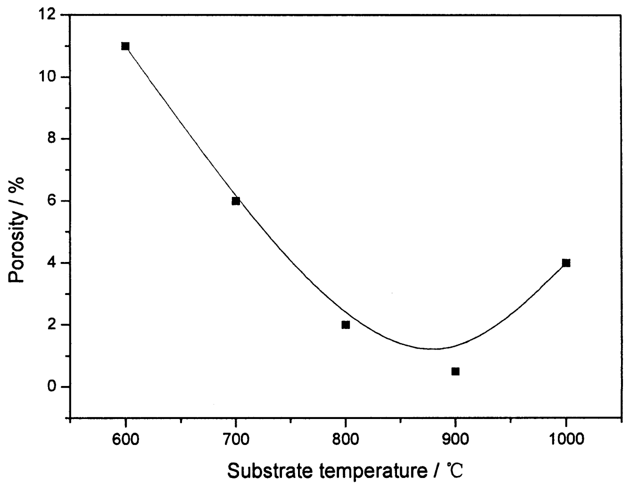

Fig. 3. The dependence of porosity on the substrate temperature.

surfaces of the tensile specimens were observed with SEM. The porosity volume fractions were measured by the precision density method (Archimedes' principle).

3. Results and discussion

3.1. Deposition rate

A stable EB-PVD technology requires the generation of a complete molten surface of the evaporant without solid zones at the edges, and a splash-free evaporation from the pool on the top of the ingot which is moved for feeding the pool. The deposition rate is varied by changes in the electron beam gun power. However, increasing beam power excessively to raise the deposition rates may result in an unstable melt pool and melt spitting. So a long-time splashless evaporation process is generated by 40–60 kW EB power. The feeding rate of the rod amounted to 2.3–5.5 mm/min. Consequently, a changed deposition rate from 2 to 10 μm/min was obtained.

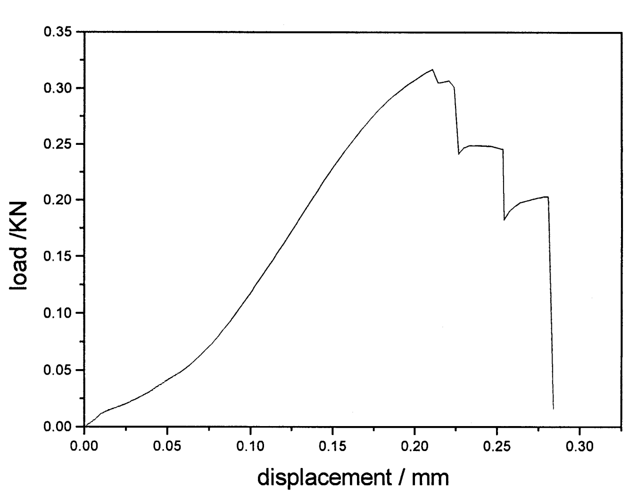

Fig. 4. Load–displacement curve of Ni/Ni_3Al microlaminate.

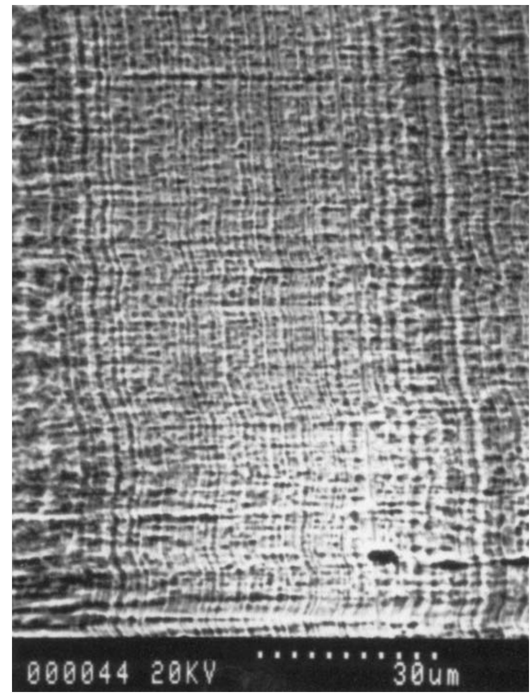

Fig. 5. Cross-sectional SEM micrograph of Ni/Ni_3Al microlaminate.

The morphology of the microlaminates depend on process parameters, primarily deposition rates and substrate temperature. It is known that metal, alloy, intermetallic compound and ceramic condensates, produced at high deposition temperature, as a rule, possess columnar structures. The microstructure of the microlaminates deposited at different rates was shown in Fig. 2. Fig. 2a exhibited a columnar microstructure. This yields an easy fracture path through the materials due to intergranular fracture between columns and transgranular fracture through columns. While

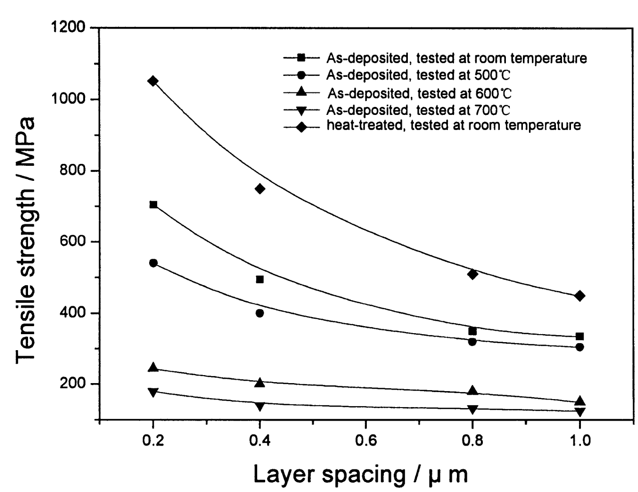

Fig. 6. Tensile strength, as a function of interlamellar spacing, of as-deposited and heat-treated microlaminates tested at room temperature and of as-deposited microlaminates tested at 500, 600 and 700 °C.

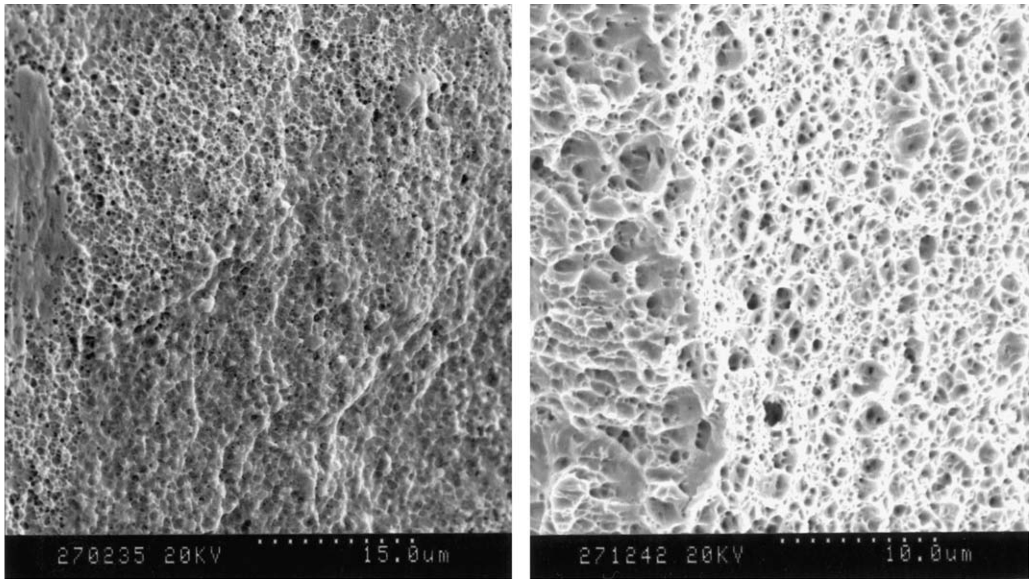

Fig. 7. Fractographs of as-deposited and heat-treated Ni/Ni_3Al microlaminates.

in Fig. 2b, a denser, non-columnar microstructure was observed. Thus, higher deposition rates resulted in more dense, less columnar microstructures, probably as a result of changed adatom mobility [10].

3.2. Substrate temperature

During the deposition process, the substrates were preheated to 600, 700, 900, and 1000 °C, respectively. Fig. 3 shows the porosity for the samples deposited as a function of substrate temperature. It appears that the porosity decreased as the substrate temperature was increased. This is primarily the result of coarsening. As a result of the low substrate temperature, very little coarsening occurred which was supported by the high porosity. At higher temperatures, the volume of voids decreased which suggests better surface mobility, and resulted in a more tightly packed microstructure indicated increased density as expected with increasing deposition temperature. However, further increasing the substrate temperature up to 1000 °C resulted in a highly porosity. The change in microstructure is attributed to the coalescence of "shadowed" sections [11].

3.3. Mechanical properties

The load–displacement curve is one of the fundamental properties that determines mechanical properties in ductile phase reinforced composites. Fig. 4 is the curve of load–displacement for Ni/Ni_3Al microlaminate tested at room temperature. It showed typical characteristics of multilayered materials [12].

An initial load drop coincides with the cracking of the tensile layer. The lower load is the load sustained by the crack arrested at the interface. Further increase in displacement causes the load to increase nonlinearly to a load at which another crack nucleates in the microlaminate. Then the crack continues to arrest and renucleate. The final load drop represents overall specimen failure. A maximum tensile stress of 705 MPa was achieved from the peak load. The curve indicates that the microlaminates exhibit a non-brittle failure manner at room temperature. Fig. 5 shows clear interfaces between Ni_3Al and Ni in microlaminated materials, which is considered to be helpful in improving the toughness of the materials.

Tensile tests at different temperatures were performed on the as-deposited and laboratory heat-treated (aged) EB-PVD microlaminates. Fig. 6 shows the relationship between tensile strength and interlamellar spacing. The tensile strength of the microlaminates at elevated temperatures were much lower and showed less dependence on the interlamellar spacing in comparison with that of at room temperature. In addition, the strength level was enhanced by heat-treating at 1050 °C for 3 h.

The fracture morphologies of as-deposited and heat-treated microlaminates are shown in Fig. 7. Fracture in sample after heat-treated appeared to involve ductile fracture with dimples. While in as-deposited samples, less and smaller dimples were produced indicated lower stress level.

4. Conclusions

Ni/Ni_3Al microlaminates were fabricated with distinct layers by EB-PVD method. The results show that deposition rates and substrates temperature have great influence on the morphology of the microlaminates. The combination of strength and ductility exhibited by the Ni/Ni_3Al microlaminates suggests that EB-PVD may be a valuable method for the production of ultra-high strength materials for specific application.

Acknowledgements

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