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Fabrication and interfacial structure of Al₂O₃/Ni laminar ceramics

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Abstract

 Al_2O_3/Ni laminar ceramic composite was fabricated by aqueous tape casting with close control of the thickness of Al_2O_3 and nickel layers. Owing to a high flexibility and strength, green tapes of Al_2O_3 and Ni could be laminated easily at room temperature. The compact, defect free interfacial structures of Al_2O_3/Ni laminar composites sintered by hot-pressing (HP) at $1400\,^{\circ}C$ were investigated by X-ray diffraction (XRD) and energy dispersive X-ray analysis (EDX). It is shown that the diffusion of Al and Ni caused a progressive boundary between Al_2O_3 and Ni layers. The Al_2O_3/Ni laminar composite was mostly composed of Al_2O_3 and Ni phases, together with a small amount of $MgAl_2O_4$, Al_4Ni_3 and $NiAl_{10}O_{16}$ phases.

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1. Introduction

Although ceramics with high strength are good structural materials, the lack of damage tolerance ability constrains their application. From a biomimetic point view, laminated composites have an advantage of insensitivity to defects. Clegg and co-workers [1–4] had produced laminated SiC with graphite interface layers, which composite showed stepped stress–strain behavior with higher apparent toughness and work of fracture than monolithic SiC. The ceramic/metal composite as a part of laminated composites combines some advantages of the ceramics and metals. When crack stretches to the metal interlayer, due the ductility of the metal, they can be deflected, bridged, etc. which behaviors generally improve the toughness of the composition.

Thick layers of laminar ceramic composites are usually prepared by tape casting [5,6], slip casting [7], centrifugal casting [8] and self-propagating high temperature synthesis [1]. Thin layers can be realized by screen printing [9],

electrophoresis deposition (EPD) [10–12], tape casting [6], plasma spraying [13], electroless plating, etc.

We tried using aqueous tape casting to produce nickel thin layers and Al_2O_3 thick layers. Al_2O_3/Ni laminate materials were obtained by hot-pressing (HP) sintering technology. The diffusion of interfacial elements and the interfacial structure were investigated by X-ray diffraction (XRD) and EDX.

2. Experimental procedure

Aqueous tape casting of Al_2O_3 and Ni was used to produce green tapes due to environmental and health considerations. The composition of the high solid (56.4 wt%) alumina tape casting slurry is given in Table 1. Ni electrolytic powders ($d_{50} = 26.5~\mu m$, Shanghai Jiuling Smelting Co. Ltd., China) with amido, fatty acid and alcohol group show good dispersiton and plasticity in the PVA water solution [14,15]. Thus, Ni tape casting slurry was composed by Ni electrolytic powder, water and PVA.

Tape casting was performed on Procast Precision Tape Casting Equipment (Division of the International Inc.,

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Table 1 Composition of the alumina tape casting slurry

Ingredient	Function	Manufacture
Al ₂ O ₃ ($d_{50} = 0.6 \mu m$)	Ceramic powder	Shanghai Wusong Fertilizer Factory, China
Deionized water	Solvent	Home-made
Kalium polyacrylate (Lopon 895)	Dispersant	Bk guiulini chemic representative office
MgO	Sintering assistant	Shanghai Chemical Reagent Corp., China
PVA	Binder	Shanghai Chemical Reagent Corp., China
$1,2-C_3H_8O_2$	Plasticizer	Shanghai Chemical Reagent Corp., China



Fig. 1. Schematic illustration of the Al₂O₃/Ni laminate.

Ringoes, New Jersey) with a blade height of 200–500 μ m. Al₂O₃/Ni laminar composites were fabricated by periodically stacking Al₂O₃ and Ni layers (Fig. 1). Every layer contained some green films of Al₂O₃ or Ni. Subsequently,

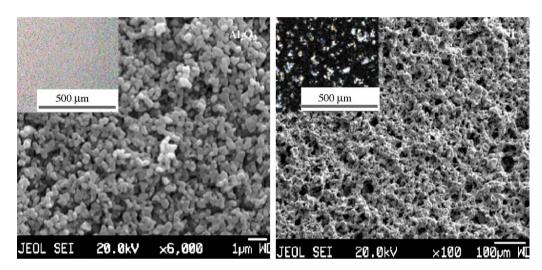
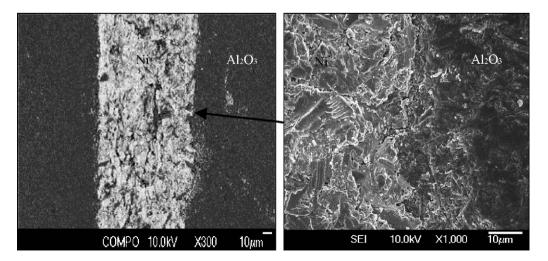


Fig. 2. SEM micrographs of Al₂O₃ and Ni green films after binder removal (left upper: optical graphs of Al₂O₃ and Ni green films).



 $Fig. \ 3. \ SEM \ micrographs \ of \ the \ Al_2O_3/Ni \ interface \ (right \ is \ the \ magnified \ view \ of \ a \ small \ area \ from \ the \ left \ image).$

organic additives were removed at 800 $^{\circ}$ C for 2 h in a vacuum drying oven. Then Al₂O₃/Ni laminates were sintered by hot pressing at 25 MPa under an argon atmosphere at 1400 $^{\circ}$ C for 1 h.

Surface properties and microstructure characterizations of Al_2O_3 and Ni green films were measured by optical microscopy (Olympus BX51M, Japan) and by SEM JSM-6700F, Japan). Energy dispersive X-ray analysis (EDX, EPMA-8705QH₂) was performed across the interface in order to determine the diffusion of elements from adjacent layers. The crystal structure was obtained from X-ray diffraction (Rigaku D/max 2550 V X, Cu K α).

3. Results and discussion

The qualities of green film, such as homogeneity, surface quality, absence of bubbles and cracks greatly affect the properties of Al₂O₃/Ni laminar compositions. Optical graphs of Al₂O₃ and Ni green films and SEM micrographs after binder removal are given in Fig. 2. Films do not present bubbles and cracks (left upper graphs of Fig. 2). The dark areas in SEM micrographs are holes formed during the organic matter removal, especially PVA. The homogeneous distribution of holes indicates the homogeneity of the green film. These films have very good strength and flexibility, and can be easily cut and stacked together.

SEM micrographs of interfaces between Al_2O_3 and Ni layers are shown in Fig. 3. The grey area is Al_2O_3 and the dark area is Ni. Al_2O_3 and Ni layers are all compact without delamination and voids. However, it is difficult to detect exactly the boundary between the Al_2O_3 and Ni layer from the left graph of Fig. 3, which is the magnified view of a small area from the right image. This might be due to the diffusion of elements.

To observe clearly the diffusion phenomena, samples with the thicker of Ni interlayer were characterized by EDX, as shown in Fig. 4. Al and Ni elements diffusion lines exhibit

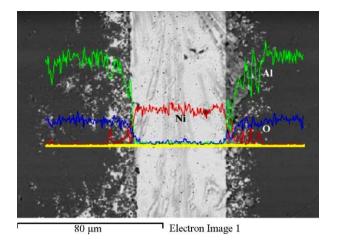


Fig. 4. Element analysis from a scan through an interface of the Al₂O₃/Ni laminar composite.

a similar gradually varying trend, suggesting some diffusion of Al and Ni species. Based on estimation, the diffusion distance of Ni in Al_2O_3 layer is about $10\text{--}25~\mu\text{m}$. The diffusion distance of Al is longer than the length of Ni layer (Fig. 3) and the distribution of Al in Ni layer is not homogenous. The reason is the lower solid content of Ni green film. After binder removal, holes in Ni film form the route for Al diffusion.

XRD analysis (Fig. 5) was used to detect compounds in the Al_2O_3/Ni laminar composites. In order to detect the different compounds with changing the thickness ratio of Ni/ Al_2O_3 layer, samples from $Al_2O_3/Ni-1\#$ to $Al_2O_3/Ni-4\#$ with decreasing thickness ratio were used. The Al_2O_3/Ni laminar composite is mostly composed of Al_2O_3 and Ni phases, together with little $MgAl_2O_4$, Al_4Ni_3 and $NiAl_{10}O_{16}$ phases. This result confirms the diffusion of Al and Ni elements. And contents of Ni compounds decreasing from $Al_2O_3/Ni-4\#$ indicate that the element diffusion benefit from a thicker Ni interlayer.

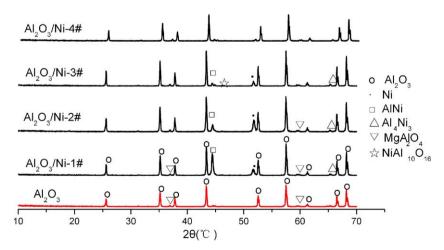


Fig. 5. XRD analysis of Al₂O₃/Ni laminar structures.

4. Conclusions

Tape casting is a useful technique in fabricating laminar ceramic composites with close control of the thickness of green film. Green films exhibit good qualities, such as homogeneity of composition, smooth surface, being free from bubbles and cracks, and so on. The interface bonding of the Al₂O₃/Ni laminar composite seems almost macro defect free. Al and Ni elements diffuse near the boundary region, forming an interphase area composed of Al₂O₃, Ni, MgAl₂O₄, Al₄Ni₃, NiAl₁₀O₁₆ phases.

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