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Microstructures, mechanical and oxidation behaviors of C/C composites modified by NiAl alloy

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Abstract: Carbon/carbon composites modified by NiAl alloy were prepared using vacuum reactive melt infiltration methods with NiAl and titanium mixed powders as raw materials. The microstructures were investigated by scanning electron microscopy. The fracture behavior, infiltration and oxidation mechanism were further discussed. The results indicated that NiAl alloy exhibited good wettability on the C/C preform because a TiC reaction layer formed at the interface. Multi-layer (PyC/TiC/NiAl+TiC) coating evenly and compactly distributed on the surface of the carbon fiber in tubular form. The penetration depth of molten NiAl alloys depended on the reaction between the PyC and titanium. The impact fracture was inclined to along the interface between the NiAl permeability layer and C/C matrix. Al_2TiO_5 and TiO_2 formed on the surface, while the interior multi-layer tubular structure partially remained after oxidation at 1773 K for 30 min.

Key words: carbon/carbon composites; interface; NiAl; multilayer structure; oxidation

1 Introduction

Carbon fiber reinforced carbon matrix composites (C/C) are expected to be applied as a potential high-temperature structural material for engineering and aerospace applications, due to their unique and excellent properties of high specific strength, high thermal conductivity and high specific stiffness at temperatures above 2273 K [1−3]. However, rapid oxidation above 673 K in an oxidizing environment restricts their high temperature applications [4]. Therefore, C/C composites need to be infiltrated with ultra-refractory materials such as metals and carbides/borides [5−8] to improve their anti-oxidation property at high temperature. Combined with the anti-ablation of ultra-high temperature ceramics and high ductility, thermal shock resistance of metallic materials to modify C/C composites may be an effective method for addressing this issue.

NiAl is recognized as a promising candidate for high temperature structural applications, due to its high melting point (1913 K), superplasticity (1073−1373 K, 480%) and excellent creep resistance at high temperature [9−11]. Using high temperature super-plasticity, high temperature oxidation resistance, and high temperature strength of NiAl intermetallic compound to improve the oxidation resistance of C/C composites has not been reported. However, it is very difficult to directly infiltrate NiAl to C/C composite because of the poor wetting [12]. Titanium possesses good wettability on the surface of C/C composites preform. RAN et al [13,14] prepared C/C−Cu composites through reactive melt infiltration (RMI) process using titanium as an active additive. It is revealed that the improvement of adding small amount of titanium for molten copper infiltration into the C/C composites preform results from the improvement of the chemical and physical adsorption characteristic of copper on the composites. The liquid copper alloy infiltrates into the preform under the capillary pressure. YU et al [12] also indicated that adding a small amount of Ti to NiAl may improve its wettability on carbon, and improve the interface bonding by forming a TiC layer. In the present research, using titanium as an active additive, a NiAl modified C/C composite was fabricated by vacuum reactive melt infiltration. The microstructures, fracture and oxidation behavior were obtained. The infiltration and oxidation mechanisms were investigated.

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2 Experimental

NiAl (150 μm, 99.95% purity) and titanium (50 μm, 99% purity) powders were used as raw materials. The infiltrating agent was mixed according to the composition of 15% Ti and 85% NiAl (mass fraction). The porous C/C preforms with a density of 1.35 g/cm^3 were embedded in the infiltrating agent in a crucible. They were placed in a vacuum furnace and heated at 2073 K for 1 h, then cooled to room temperature.

Phase analysis was conducted by X-ray powder diffraction using a D/Max2550 X-ray diffractometer (Rigaku, Japan) with Cu K_α radiation. Microstructural characterization was carried out using scanning election microscopy (SEM, NOVATM Nano SEM230 and JSM−6360LV) with energy-dispersive spectrometry (EDS). Electron probe microanalysis (EPMA, JEOLCO, JXA8530F) was performed to detect the distributions of the major elements in the composites.

The density of the composite was measured by the Archimedes water-immersion method using Sartorius cp224 s densitometer at room temperature. The open porosity was measured using air displacement method by filling the pores with kerosene under vacuum. Flexural strength was measured using a three-point bending test (Instron3369, UK) with a span of 40 mm. The crosshead speed was 2.00 mm/min. At least three specimens with dimensions of 55 mm × 5.0 mm × 3.0 mm were tested to obtain the average data. After three-point flexural tests, the fracture surfaces were observed by a scanning electron microscopy (SEM, JSM−6360LV).

The isothermal oxidation tests of the composites were carried out at 1773 K in air in an electrical resistance furnace. The samples after oxidation test for 30 min were analyzed by SEM and XRD with Cu K_α radiation.

3 Experimental

3.1 Microstructures

Surface SEM images and XRD patterns of the NiAl modified C/C composites are presented in Fig. 1. High densities of gray micron-sized particles are homogeneously distributed in the matrix surface. XRD pattern of the surface in Fig. 1(a) reveals that the phases of the composites mainly consist of NiAl, TiC and C, while the Ti phase is hardly detected. The composition of the white phases is determined by EDS to be 49.55 % Ni, 50.45 % Al. The composition of the gray large particles is 37.45 % C, 67.55 % Ti (mole fraction). Combining the EDS analysis and the results of XRD (Fig. 2(a)), it is obtained that the white phases and the gray large particles are determined as NiAl and TiC, respectively.

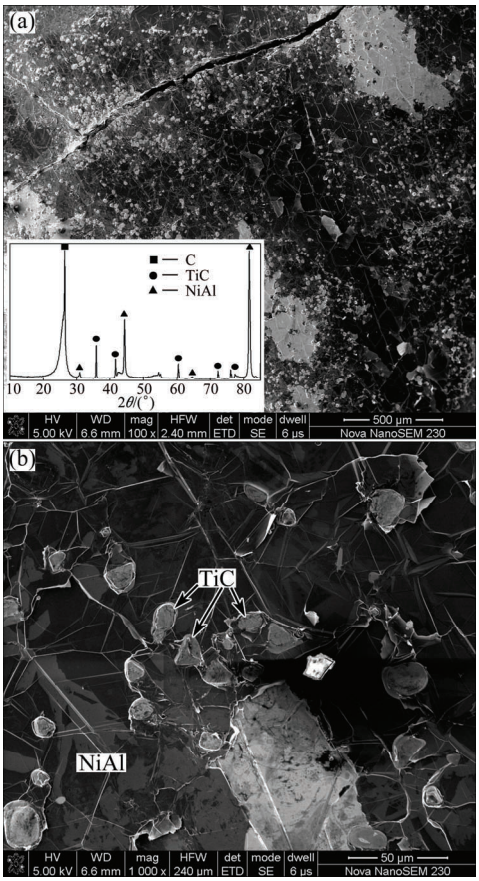

Fig. 1 Surface SEM images and XRD pattern of NiAl modified C/C composites

Cross-section SEM images of the composites are presented in Fig. 2. The metallic diffused layer of 0.8−0.9 mm in Fig. 2(a) shows that the inter-bundle and the inter-layer pores are not filled completely by the infiltrated NiAl metal. Combining the EDS analysis and the results of XRD (Fig. 2(a)), it is obtained that the white phases and the gray large particles are determined as NiAl and TiC, respectively. NiAl melts dispersed with micron-sized gray TiC particles are combined closely, while the interfaces between C/TiC and TiC/NiAl are perfectly bonded and free of pores and microcracks (Fig. 2(b)).

According to the EMPA results of the diffused layer in Fig. 3, Ni and Al elements are distributed around carbon fiber and Ti element, the distribution of Ni and Al is almost uniform. Titanium is mainly distributed around the carbon fiber and carbon matrix in the form of TiC layer or TiC particles. Combined with the XRD results shown in Fig. 1(a), it is inferred that the particles consist of TiC, surrounded by residual NiAl melt. TiC phases are resulted from the reaction between the PyC and titanium powder after reactive melt infiltration process. TiC distributes around PyC inside the pores in the C/C preform and NiAl locates in the middle of pores surrounded by TiC.