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Mechanical, dielectric and microwave absorption properties of FeSiAl/Al_2O_3 composites fabricated by hot-pressed sintering

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ABSTRACT

FeSiAl/Al_2O_3 composites were fabricated by hot-pressed sintering to evaluate the mechanical, dielectric and microwave absorption properties. The results show that the flexural strength of the composites increases with the increase of FeSiAl content, while the density and micro-hardness exhibit a downward tendency. The complex permittivity increases with increasing FeSiAl content in the X-band, which is resulted from the enhanced interfacial polarization, relaxation loss and conductance loss. Meanwhile, a rapid enhancement of complex permittivity for the composite with 10 wt.% FeSiAl is achieved due to the formation of conductive clusters and enhanced conductance loss. By calculating the reflection loss, the composite with 5 wt.% FeSiAl possesses the effective absorption bandwidth (<-10 dB) 1.9 GHz in 9.1 −11.0 GHz and the strong absorption peak of −34.2 dB is achieved at 9.4 GHz when the thickness is 1.9 mm due to its best impedance matching and appropriate electromagnetic wave attenuation.

1. Introduction

With the extensive applications of communication systems with gigahertz frequency range in the commercial, civil and military fields, such as mobile phone, intelligent transport, satellite broadcast systems and radar systems, the problems of radiation pollution and electromagnetic interference are becoming more serious [1–3]. Therefore, much attention has been paid to microwave absorbing materials for their electromagnetic attenuation capability. As a promising microwave absorber, it should not only have light weight, broad width, and strong microwave absorption, but also possess excellent oxidation resistance in the high temperature environments [4]. Generally, the loss mechanisms of the absorbents, such as carbonaceous materials, conductive metal particles and magnetic absorbents, are usually classified as dielectric loss, magnetic loss and conductance loss [5]. Recently, considerable effects have been made to investigate the hybrid absorbers with dielectric/magnetic loss (e.g., BiOI/Ni@C [6], $Ag_3PO_4/SrFe_{12}O_{19}$ [7], and Fe_3O_4@g-C_3N_4 [8]) and dielectric/conductance loss (e.g., ZnO/carbon nanotubes [9], and MoS_2/graphene [10]), which exhibit light weight and high-efficiently electromagnetic wave absorption. Owing to the weak oxidation resistance of carbonaceous materials and the invalid magnetism of magnetic absorbents in high temperature, conductive metal particles with dielectric (or conductance) loss and high-temperature resistance have the potential to be applied in high temperature environments.

In recent years, flaky FeSiAl powders have attracted considerable interests for applications in electromagnetic interference shielding and microwave absorption due to their low cost, large anisotropy, high permittivity and good temperature stability [11,12]. Up to now, the dispersion of FeSiAl particles into insulating matrix is an effective method widely used to fabricate microwave absorbing materials or/and improve the microwave absorption properties. Qing et al. [13] reported plasma-sprayed FeSiAl/Al_2O_3 coatings with the reflection loss less than -7 dB in the X-band and the minimum reflection loss −24.7 dB when the FeSiAl content was 20 wt.%. To further improve the reflection loss and density [14], flaky graphite was used as a second absorbent to fabricate FeSiAl/graphite/Al_2O_3 coatings by plasma spraying. It was found that the composite coating with 15 wt.%FeSiAl and 5 wt.% graphite presented excellent microwave absorption properties with the 80% absorption bandwidth (<-7 dB) in the whole K-band and the strong absorption peak −18.4 dB at 15.2 GHz when the thickness was 0.8 mm. Furthermore, many researchers investigated the microwave absorption properties of the composites filled with FeSiAl and

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carbonaceous absorbents (e.g. graphite, carbon black, and carbon nanotube) by various methods [15–18]. Even though the FeSiAl-based composite coatings exhibit outstanding microwave absorption properties, it still has the challenges for the applications in large-scale industries due to the generation of stress and the deterioration of bonding strength. In addition, the composites with carbonaceous material as a second absorbent in high temperature applications are restricted due to the weak oxidation resistance.

It is believed that cermets (e.g. Ni/YSZ [19], TiC/Fe [20], and Fe/WC [21]) are heterogeneous composites consisting of metal (or alloy) binder phase and ceramic phase, in which ceramic components make cermets high hardness and oxidation resistance [22]. Due to the low density, high hardness and excellent electrical insulation, Al_2O_3 has been widely used as a ceramic phase in cermets and an ideal candidate matrix phase for the composites with microwave absorption properties [23]. Among various technologies, hot-pressed sintering is an important approach to fabricate dense composites owing to its advantages of the inhibition of grain growth and the production of high precision and complex parts [24]. In this study, FeSiAl/Al_2O_3 composites were fabricated by hot-pressed sintering for microwave absorption applications. The phase composition and microstructure were characterized, and the influence of FeSiAl content on the mechanical, dielectric and microwave absorption properties of the composites was investigated. By analyzing the electrical conductivity, input impedance and attention coefficient, the dielectric and microwave absorption mechanisms of the composites in the X-band had been discussed in detail with the increase of filler content, frequency and thickness.

2. Experimental

2.1. Preparation of the composites

Commercial flaky FeSiAl powders (Si 9.6 wt.%-Al 5.4 wt.%-balance Fe, purity >99.0 wt.%) with a diameter of less than 30 μm were obtained from Chengdu YCE Technology Co. Ltd. α-Al_2O_3 powders with a purity of over 99.9% had an average diameter of 0.8 μm. The mixtures containing Al_2O_3 powders with different content of FeSiAl powders (0, 5, 10 and 15 wt.%) were milled for 8 h using a planetary ball mill (QM-3SP4, Nanjing Nada Instrument Co., Ltd.) with polytetrafluorethylene (PTFE) jar and zirconia balls in alcohol. The ball milling process was carried out in air atmosphere with the rotation speed of 500 rpm and the ball to powder weight ratio was approximately 5:1. After the oven-drying process at 80 °C for 3 h, the mixed powders were sieved with 100 meshes. Then, the mixtures were put into a graphite die and hot-press sintered at 1350 °C for 1.5 h under a load of 30 MPa in vacuum, with heating rates of 13 °C/min (0–900 °C), 5 °C/min (900–1200 °C) and 2.5 °C/min (above 1200 °C). Finally, the sintered composites were cooled with the furnace to room temperature, and the surface of the composites contaminated by the graphite die were mechanically removed with sandpaper.

2.2. Characterization

The microstructure of the composites was characterized with a scanning electron microscopy (SEM, VEGA III SBH, TESCAN, Brno, Czech Republic) in backscattering electron (BSE) mode on the polished surface. Prior to the microstructure characterization, the surfaces of the samples were sputtered with gold. The phase structure was determined by X-ray powder diffraction (XRD, Rigaku, D/Max2500, Tokyo, Japan) with Cu-Kα radiation in the 2θ range of 20 to 90°, operating at 40 kV and 40 mA. The porosity was tested using Archimedes method (Density determination instrument, Sartorius YDK03, Germany), and the porosity level of each samples was calculated according to the following equation [25]:

$$\text{Porosity}(\%) = 100 - \frac{\textit{coating density}}{\textit{theoretical density}} \times 100\% \tag{1}$$

The Micro-hardness testing was conducted in the polished cross sections using a Vickers tester with a load of 1000 g and a loading time of 10 s. Five individual measurements were made for each sample and the average was regarded as the micro-hardness value. The flexural strength P was measured by the three-point bending method with a size of 3 mm (thickness) × 4 mm (width) × 40 (length) mm at a cross-head speed of 0.5 mm/min, and P was expressed as follows:

$$\mathrm{P} = 3Fl/2bh^2 \tag{2}$$

where F is the load at fracture, l is the distance between the two supporters, b and h are the width and thickness of the sample, respectively.

Semiconductor parameter analyzer (Agilent 4155C and Keithley 651B) was used to test the conductivity of the composites. The complex permittivity was measured based on the rectangle waveguide method in the X-band using a E8362B PNA vector network analyzer with nonmagnetic model, and the testing samples were machined into rectangular blocks with a size of 22.86 mm (length) × 10.16 mm (width) × 2.00 mm (thickness).

3. Results and discussion

3.1. Crystalline phase and microstructure

XRD patterns of FeSiAl/Al_2O_3 composites are shown in Fig. 1. It can be observed that the composites consist of FeSiAl and α-Al_2O_3 crystalline phases without any additional phases. In addition, no Fe oxides or/and Si oxides are found, indicating that no other chemical reactions occurred and both the FeSiAl and α-Al_2O_3 with excellent chemical compatibility were retained during the sintering process [14]. For the FeSiAl phase, three diffraction peaks are associated with the (110), (200), and (211) planes of α-Fe (Si, Al) with bcc lattice structure [26], and the integrated intensity of the FeSiAl diffraction peaks increases with increasing FeSiAl content in the composites.

The cross-sectional microstructure of FeSiAl/Al_2O_3 composites with varying FeSiAl content is shown in Fig. 2. It is observed that the Al_2O_3 ceramic appears dark gray and metallic FeSiAl presents light

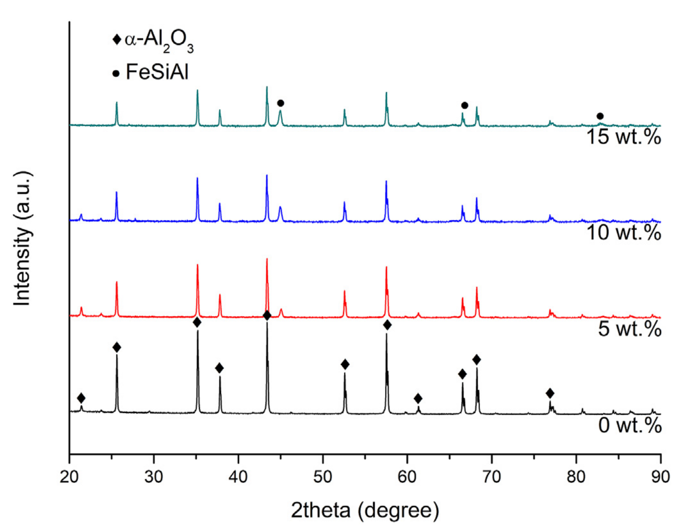

Fig. 1. XRD patterns of FeSiAl/Al_2O_3 composites.