Effects of Multi-Components on the Microwave Absorption and Dielectric Properties of Plasma-Sprayed Carbon Nanotube/Y₂O₃/ZrB₂ Ceramics

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Abstract: Carbon nanotube (CNT)-reinforced Y₂O₃/ZrB₂ ceramics were fabricated via planetary ball milling and atmospheric-pressure plasma spraying for the first time. The phase composition, micromorphology, and electromagnetic (EM) wave absorption performance of the Y₂O₃/ZrB₂/CNT hybrid was investigated from 8.2 to 12.4 GHz. Both the real and imaginary parts of the complex permittivity were enhanced as the ZrB₂ and CNT content increased. The Y₂O₃/ZrB₂/CNT hybrids corresponded to a ZrB₂ content of 15 wt.%, and the CNT content was 2 wt.%, and showed an exceptional EM wave absorption capability, with a minimum reflection loss of –25.7 dB at 1.9 mm thickness, and the effective absorption band was in a full X-band. These results indicate that an appropriate CNT or ZrB₂ content can tune the complex permittivity and absorption performance of the Y₂O₃/ZrB₂/CNT ceramics.

Keywords: Y₂O₃/ZrB₂/CNT ceramics; plasma spraying; dielectric property; microwave absorption

1. Introduction

Electromagnetic (EM) pollution has become a serious problem to human health and the environment due to the growth of the electronic industry and radio communication technologies [1–3]. To solve this problem, designing and fabricating suitable microwave absorption materials is an urgent task for researchers in the field of materials science [4–8]. Currently, polymer-based absorbing materials used as fillers have limited utility under special environments due to their relatively poor mechanical performance. In contrast, ceramic-based absorbers have received extensive attention due to their excellent mechanical properties [9–11].

Y₂O₃ is a sintering aid that can be added into ceramics such as SiC, SiAlON, and Al₂O₃; this doping promotes the solid-phase sintering of materials and can effectively improve the mechanical properties of ceramics [12,13]. In addition, zirconium diboride (ZrB₂) has a high strength and electrical conductivity (~6 × 10⁴ Ω⁻¹·cm⁻¹), high melting point (3200 °C), good thermal shock resistance, and good oxidation resistance at a high temperature. Thus, it has attracted attention as a microwave-absorption material [14].

Such excellent features can be attributed to the unique crystal structure of ZrB₂. Typically, the graphite-like layered structure of B atoms and the electronic structure of the Zr outer layer leads to the formation of highly conductive ZrB₂ [15]. The high conductivity implies a large value of ϵ″ so that the material has a good dielectric loss capability, thus causing the EM energy to be dissipated in the form of Joule heat by inducing a high current [16]. However, the strong covalent bonds of ZrB₂ imply that hot pressing or spark plasma sintering at temperatures over 2000 °C are usually required to meet the densification of ZrB₂-based ceramics [17]. To solve the densification problem, coatings.

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deposited by the atmospheric-pressure plasma spraying (APS) process can effectively tune the multilayer structure and porosity and thus obtain a high bonding strength; this has become a key method for preparing ZrB$_2$-based ceramic coatings.

APS outperforms traditional methods of ceramic coating, such as thermal spraying [18], laser cladding [19], micro-arc oxidation [20], and combustion synthesis [21]. It is an exceptional technology that can meet commercial requirements due to its reduced cycle times, high deposition efficiencies, low cost, and excellent microwave absorption performance. More importantly, the integration of highly conductive carbon nanotubes (CNTs) can be further optimized to control the complex permittivity and the microwave-absorption capabilities of the Y$_2$O$_3$/ZrB$_2$:ceramic [22–24].

Here, Y$_2$O$_3$/ZrB$_2$:CNT hybrids with a low CNT and ZrB$_2$: content were fabricated by APS technology for the first time. The phase composition, micromorphology, and EM properties of the hybrid were investigated via X-ray diffraction (XRD), scanning electron microscopy (SEM), and a vector network analyzer (VNA), respectively. Comparative analysis of the EM properties of ceramics doped with different contents of ZrB$_2$: and CNT showed that the plasma-sprayed Y$_2$O$_3$/ZrB$_2$:CNT hybrids, corresponding to a ZrB$_2$: content of 15 wt. % and the CNT content of 2 wt. %, have the best EM wave-absorption capability, with a minimum reflection loss (RL$_{\text{min}}$) of $-25.7$ dB at 1.9 mm thickness, as well as an effective absorption band (EAB) in the full X-band. Therefore, Y$_2$O$_3$/ZrB$_2$:CNT ceramics prepared via plasma-spraying could provide a theoretical basis and technical support for mass production in the field of ceramic-based high-temperature materials.

2. Experimental

Y$_2$O$_3$ (99.99%), ZrB$_2$ (99.5%) and CNT were used as raw materials. The Y$_2$O$_3$/ZrB$_2$:CNT ceramics were fabricated with different Y$_2$O$_3$: contents (76, 78, 81, 83, and 85 wt. %); the corresponding samples were denoted as Y76, Y78, Y81, Y83, and Y85 for simplicity. The mass percentages (wt. %) of the different Y$_2$O$_3$/ZrB$_2$:CNT ceramics are listed in Table 1. The desired amount of Y$_2$O$_3$, ZrB$_2$: particles, and CNT were evenly mixed using planetary ball milling. The fluidity of the powder was increased with spray drying technology, leading to spray-dried powders with a size of 30–80 μm. The Y$_2$O$_3$/ZrB$_2$:CNT ceramic coating was prepared via the APS process, in which Ar and N$_2$: were used as plasma gases, with flow rates of 50 and 10 standard liters per minute (slpm), respectively. Experimental parameters for the spray conditions were optimized for good deposition efficiency. The net arc input power and powder flow rate were 25 kW and 4 g/min, respectively; the spraying distance and coating thickness were 100 mm and 2 mm, respectively. Finally, the ceramic samples were polished to a thickness of 1.2 mm for measuring EM parameters.

Table 1. The mass percentage (wt. %) for different Y$_2$O$_3$/ZrB$_2$:CNT ceramics.

| Sample | Y$_2$O$_3$ (wt. %) | ZrB$_2$: (wt. %) | CNT (wt. %) |
|--------|-------------------|-----------------|-------------|
| 1      | 85                | 15              | -           |
| 2      | 83                | 15              | 2           |
| 3      | 81                | 15              | 4           |
| 4      | 78                | 20              | 2           |
| 5      | 76                | 20              | 4           |

The phase compositions of the plasma-sprayed samples were characterized through XRD with Cu-Kα radiation. The microstructures of the samples were observed by SEM (JEOL JSM-6360LV). The EM parameters of the Y$_2$O$_3$/ZrB$_2$:CNT specimens with a dimension of 22.86 mm (length) × 10.16 mm (width) × 1.2 mm (thickness) were investigated in a full X-band (8.2–12.4 GHz) by VNA (Agilent technologies E8362B).
3. Results and Discussion

Figure 1 shows the XRD pattern of the plasma-sprayed Y₂O₃/ZrB₂/CNT samples. The different Y₂O₃/ZrB₂/CNT ceramics were only composed of Y₂O₃, ZrB₂, and CNT crystalline phases. The peak intensity of Y₂O₃ obviously but gradually decreased with a decreasing Y₂O₃ content. The peak intensity changes in the ZrB₂ and CNT were the same as Y₂O₃ when studied in the Y₂O₃/ZrB₂/CNT specimens. There was no oxide peak for ZrB₂ in the XRD spectrum, suggesting that the plasma-spray parameters were suitable, and the deposition efficiency was satisfactory.

Figure 1. XRD spectra of Y₂O₃/ZrB₂/CNT specimens with different Y₂O₃ contents.

SEM images of Y₂O₃/ZrB₂/CNT ceramics are shown in Figure 2. ZrB₂ and CNT were evenly mixed with the Y₂O₃ matrix. The presence of a microporous structure during the plasma-spraying process may be related to unmelted agglomerates or resolidified agglomerates. Here, the theoretical density of Y₂O₃/ZrB₂/CNT ceramics could be deduced according to the density of ZrB₂ (6.08 g·cm⁻³) and Y₂O₃ powder (5.01 g·cm⁻³). Correspondingly, the relative densities of the Y₂O₃/ZrB₂/CNT hybrid decreased with the Y₂O₃ powder content. Furthermore, the decrease in Y₂O₃ content increased the porosity of the material, but the increase in CNT content better filled the pores of the Y₂O₃/ZrB₂/CNT ceramics (Figure 2a,b versus Figure 2c,d).
Figure 2. SEM images of Y₂O₃/ZrB₂/CNT ceramics with (a,b) 83% Y₂O₃ particles (Y83 sample) and (c,d) 81% Y₂O₃ particles (Y81 sample).

In general, EM waves can induce two kinds of currents in the absorber: conduction current and displacement current. Within that, the conduction current comes from moving charge carriers and increased conductivity, which in turn, increases the value of $\varepsilon''$. The $\varepsilon''$ value is then related to the dielectric loss ($\tan \delta = \varepsilon''/\varepsilon'$) capability of the absorber [25,26]. The displacement current is impacted by the dielectric polarization of local charge carriers, and the enhanced $\varepsilon'$ value can be attributed to the dielectric polarization and space charge polarization effects [27,28].

Figure 3 presents plots of $\varepsilon'$ and $\varepsilon''$ over 8.2 to 12.4 GHz, as the content of Y₂O₃ changed. In the full X-band, both $\varepsilon'$ and $\varepsilon''$ of the five samples slightly decreased as the frequency increased. In the Y85 sample, the $\varepsilon'$ was about 12.5, and the $\varepsilon''$ was only 0.8, indicating that the high content of Y₂O₃ and the relatively low content of ZrB₂ lead to lower complex dielectric properties when there was no CNT. This also shows that the Y85 sample may possess a lower dielectric loss ability and poor absorbing ability.

When the ZrB₂ content was 15 wt.%, the $\varepsilon'$ and $\varepsilon''$ values of the Y83 sample were slightly enhanced; the Y81 sample had significantly improved $\varepsilon'$ and $\varepsilon''$ values. We made a control Y85 ceramic without CNT, and we found that the $\varepsilon'$ and $\varepsilon''$ of Y85 could increase from 12.8 to 34.6 and 0.8 to 18.0, respectively. When the content of ZrB₂ was 20 wt.%, the $\varepsilon'$ and $\varepsilon''$ of Y78 were higher than the Y83 sample. The relationship of Y76 and Y81 was the same as the result of CNT at 2 wt.%. These data indicated that Y76 had the highest values of $\varepsilon'$ and $\varepsilon'': 41.1$ and $23.4$ versus the other four samples. The Y85, Y83, and Y81 samples had $\varepsilon'$ and $\varepsilon''$ values that gradually increased as the content of Y₂O₃ decreased. These changes were attributed to the enhancement of the CNT content, which in turn, increased the conductivity of the material and improved the dielectric properties of Y₂O₃/ZrB₂ ceramics.

The $\varepsilon'$ and $\varepsilon''$ values increased significantly in Y83 and Y78 samples as the content of Y₂O₃ decreased at a constant CNT content. The increased ZrB₂ content increased the
complex permittivity of the material. Y81 and Y76 samples showed a similar trend. In the Y78 and Y76 samples, the $\varepsilon'$ and $\varepsilon''$ values of the materials exhibited a significant increase when the content of ZrB$_2$ (20 wt.%) remained the same, due to the increase in the content of highly conductive CNTs. These results show that increasing the content of ZrB$_2$ and CNT improves the $\varepsilon'$ and $\varepsilon''$ of the Y$_2$O$_3$/ZrB$_2$/CNT ceramic. The improved conductivity of the mobile electrons in the absorber was affected by defects such as dangling bonds and vacancies, especially those caused by the interface between the ZrB$_2$ and Y$_2$O$_3$ phases [29–31]. Under the effect of the EM field, free carriers in the absorber could accumulate at these interfaces, thus leading to space charge polarization and an increasing $\varepsilon'$. Therefore, the enhanced $\varepsilon'$ value was related to an increased ZrB$_2$ content and the number of ZrB$_2$/Y$_2$O$_3$ interfaces. Furthermore, a higher ZrB$_2$ and CNT content would also lead to more permeable electrical pathways, thus improving the $\varepsilon''$ value. Moreover, when the content of CNT was equal, the enhanced ZrB$_2$ content increased the value of $\varepsilon'$ and $\varepsilon''$. The increase in CNT content enhanced the $\varepsilon'$ and $\varepsilon''$ values of the Y$_2$O$_3$/ZrB$_2$/CNT ceramic when the content of ZrB$_2$ was equal. Therefore, we concluded that the presence of ZrB$_2$ or CNT enacted a significant effect on the dielectric properties of Y$_2$O$_3$/ZrB$_2$/CNT ceramics.

The EM wave absorption performance of the plasma-sprayed Y$_2$O$_3$/ZrB$_2$/CNT ceramic coatings from 8.2 to 12.4 GHz are shown in Figure 4a–e. In Figure 4a, the minimum reflection loss ($R_{\text{L min}}$) of the Y85 sample was only $-3.8$ dB at 9.4 GHz, with a 2.2 mm thickness. The Y83 sample (Figure 4b) had a $R_{\text{L min}}$ value of $-25.7$ dB at 12.3 GHz, with a 1.9 mm thickness. The improved microwave absorption ability may have been caused by the enlarged $\varepsilon'$ and $\varepsilon''$ values from CNTs, forming a dense conductive percolation network. This network increased the conductivity loss. Furthermore, the effective absorption band (the $R_{\text{L min}}$ was lower than $-10$ dB) of Y83 ceramics covered the full X-band when the absorber’s thickness was 2.0 mm. However, the Y81 ceramic showed an $R_{\text{L min}}$ value of $-13.0$ dB at 1.3 mm thickness from 8.2 to 12.4 GHz (Figure 4c). The decreasing $R_{\text{L min}}$ value may be attributed to the highest conductivity based on excessive CNT content. Excessive CNTs cause more EM waves to be reflected from the emitting surface; thus, the waves cannot be absorbed and consumed, i.e., impedance mismatching.

When the ZrB$_2$ content increased to 20 wt.%, the $R_{\text{L min}}$ values of Y78 and Y76 were $-22.0$ dB at 12.2 GHz with a 1.9 mm thickness and $-9.8$ dB at 12.4 GHz with a 1.2 mm thickness, respectively (Figure 4d,e). Such results further prove that an appropriate CNT content is critical to an optimized absorbing performance in Y$_2$O$_3$/ZrB$_2$/CNT ceramics. Therefore, the excellent RL value of the Y$_2$O$_3$/ZrB$_2$/CNT material corresponded to the ZrB$_2$ content (15 wt.%) and the CNT content of 2 wt.%. The loss tangent (tan $\delta$) of the prepared composite samples, which is representative of the power loss in the material with respect to the stored reactive power, was estimated as the ratio between the imagi-

![Figure 3](image-url). The (a) $\varepsilon'$ and (b) $\varepsilon''$ plots of the plasma-sprayed Y$_2$O$_3$/ZrB$_2$/CNT hybrids in the full X-band.
nary and real part of the complex effective permittivity [32,33]. The computed values of \(\tan \delta\) are reported in Figure 4f. We notice that \(\tan \delta\), which increased with the contents of ZrB\(_2\) and CNT in the composite, was always greater than Y85 in the whole frequency range for samples Y76, Y78, Y81, and Y83. As we know, the highest \(\tan \delta\) values for the sample represent the impressive storage and loss capabilities for electrical energy. The dielectric loss behavior is mainly related to conductivity loss and polarization loss. Among that, ionic polarization and electronic polarization always work in the frequency of \(10^3\)–\(10^6\) GHz, and dipole orientation polarization from the frustrated reorientation of dipoles prefers frequency dispersion [34,35]. Thus, the dielectric loss should be determined by the interfacial polarization between Y\(_2\)O\(_3\), ZrB\(_2\), and CNT. Furthermore, the highest dielectric loss capability prompts it to consume electromagnetic waves and obtain an excellent reflection loss.

Figure 4. (a–e) Reflection loss with different absorber thicknesses and (f) dielectric loss curves of the plasma-sprayed Y\(_2\)O\(_3\)/ZrB\(_2\)/CNT hybrids.
To better analyze this phenomenon of different microwave absorbers, two essential factors, the EM attenuation constant (\(\alpha\)) and impedance matching (\(|Z_{in}/Z_0|\)), need to be considered [36–38]. The maximum value of \(\alpha\) suggests that more EM waves can enter the material and transform EM energy into thermal energy, as shown in Equation (1) [39]:

\[\alpha = \frac{\sqrt{2}}{c} \pi f \times \sqrt{\left(\mu'' \varepsilon'' - \mu' \varepsilon'\right)} + \sqrt{\left(\mu'' \varepsilon'' - \mu' \varepsilon'ight)^2 + \left(\varepsilon'' + \mu' \varepsilon'\right)^2}\]

(1)

Figure 5a presents the \(\alpha\) value of the five samples in the X-band. The Y85 sample showed an average \(\alpha\) value of about 19.0, but the \(\alpha\) value of doped-CNT samples had a major effect relative to the lowest \(\alpha\) value seen in the undoped-CNT Y85 ceramic. These data further prove that the addition of CNT can improve EM attenuation, leading to good microwave absorption. The average \(\alpha\) values of Y76 and Y81 were 319.8 and 267.1, respectively. The average \(\alpha\) values of Y78 and Y83 were 188.8 and 176.4, respectively. The \(\alpha\) values of Y76 and Y81 showed a larger \(\alpha\) value versus Y78 and Y83, suggesting an exceptional EM wave attenuation ability. However, Y78 and Y83 ceramics had higher reflection loss values than Y76 and Y81: the attenuation constant only indicates that the material has a strong EM attenuation ability. No other factor could be considered, i.e., impedance matching. Figure 5b–f shows the impedance matching of five samples simulated at different absorbing thicknesses. Values of \(|Z_{in}/Z_0|\) of Y85, Y81, and Y76 ceramics were far from 1, but Y83 and Y78 approached 1 as the absorbing thickness changed. These observations suggest that Y83 and Y78 samples absorb more EM waves. Therefore, good microwave absorption is related to both EM attenuation loss and impedance matching.
Figure 5. Frequency dependence of (a) attenuation constant and (b–f) impedance matching of Y$_2$O$_3$/ZrB$_2$/CNT ceramics with the different absorber thicknesses.

4. Conclusions

Y$_2$O$_3$, ZrB$_2$, and CNT powders were uniformly mixed via a planetary ball mill, followed by spray drying. The Y$_2$O$_3$/ZrB$_2$/CNT ceramics were fabricated by APS technology. The $\varepsilon'$ and $\varepsilon''$ values of Y$_2$O$_3$/ZrB$_2$/CNT ceramics increased when the ZrB$_2$ or CNT contents increased, because Y$_2$O$_3$/ZrB$_2$/CNT ceramics have dense conductive percolation pathways. These pathways can respond to the presence of conductance losses and thus improve the microwave absorption ability. The $RL_{\text{min}}$ value of the Y83 sample was $-25.7$ dB at 12.3 GHz with a 1.9 mm thickness. One could control the EM properties of Y$_2$O$_3$/ZrB$_2$/CNT ceramics by tuning the mass percentage of Y$_2$O$_3$, ZrB$_2$, and CNT. This could lead to a high attenuation capability and good impedance matching. This unique Y$_2$O$_3$/ZrB$_2$/CNT hybrid had excellent EM wave absorption properties and is a good candidate for designing ceramic-based high-temperature absorbers.
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