PREPARATION AND MICROWAVE ATTENUATION PROPERTY IN THE X BAND OF SiC–C COMPOSITES

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For developing an effective, low-cost method on preparation of microwave attenuation material, SiC–C composites with different additions of graphite grain were fabricated with solid phase sintering and liquid phase sintering, respectively. The microwave attenuation and the relative electrical properties of composites in the X band were measured. The results show that both methods are cost-effective and easily controllable processes and Solid phase sintering was more suitable to obtain SiC-C attenuation composites than the liquid phase sintering. The composite with 3 wt.% C prepared by solid phase sintering exhibited the best microwave attenuation, the biggest attenuation was -40.5 dB and most attenuations were above -30 dB in the whole X band. Furthermore, the microwave attenuation of composites depended strongly on the C additions; Match of interface of wave impedance and high wave consumption are both necessary for microwave attenuation materials.

INTRODUCTION

SiC fiber, because of its excellent attenuation property, has become an important microwave attenuation additive for a long time. However, the composites including SiC fiber are obtained conventionally by complex process at expensive cost [1-2]. With increasing applications of attenuation materials such as applied in microwave communication and aviation, it becomes necessary to develop composites with SiC matrix (not fiber) prepared by cost-effective and easily controllable manufacturing process.

It is well known that the sintering mechanisms of SiC can fall into two broad categories: solid phase sintering and liquid phase sintering, which are strongly correlative to the sintering assistants. Currently, lots of references on the sintering mechanisms of SiC composites have been reported [3-10]. However, effects of sintering methods on microwave attenuation of composites are seldom investigated.

Figure 1 shows propagation of microwaves in the interface of two media. To obtain maximal microwave attenuation in the medium 2, the wave impedance of two media should match each other to ensure that the reflectivity of the microwaves in the interface is as minimal as possible [11-12]. Furthermore, the medium 2 should have an excellent microwave attenuation property to effectively consume the transmission and refracted waves. Therefore, it is best realized by controlling proper electric properties to obtain excellent microwave attenuation of the medium 2.

In this paper, for obtaining excellent SiC microwave attenuation composites in the X band (8-12 GHz, the most extensively applied band in all applications), which also have cost-effective and easily controllable manufacturing process, SiC–graphite (SiC–C) composites were fabricated by solid phase sintering and liquid phase sintering under pressure-less condition, respectively. And the effects of different sintering methods on their microwave attenuation were studied.

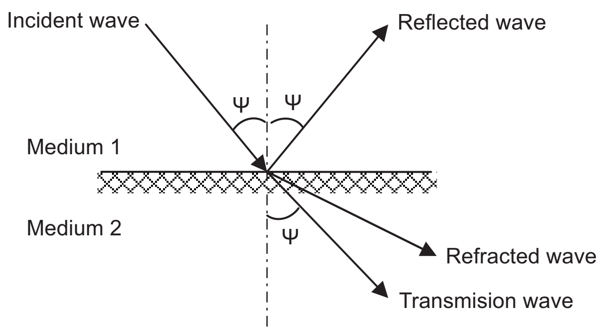


Figure 1. Propagation of microwaves in the interface of two media.

EXPERIMENTAL PROCEDURES

Materials preparation

β-SiC powder (0.6 μm, H.C.Stack, Germany) was used as the matrixes. B_4C with 3.5 μm particle size and graphite C with 6 μm particle size for solid phase sintering, but Al_2O_3 with 0.7 μm particle size and Y_2O_3 with 5.6 μm particle size for liquid phase sintering, were added into the matrixes as sintering assistants, respectively. Moreover, different additions of graphite C were used as microwave attenuation materials (For solid phase sintering, the sintering assistant C would have been consumed during the sintering period [6], so it could not have been considered as the attenuation material), the mass fractions were 0, 3, and 6 wt.% of the total sum. Thus three different SiC-C composites with B_4C and C assistants were marked SBC0, SBC3, and SBC6, but the composites with Al_2O_3 and Y_2O_3 assistants were marked SAYC0, SAYC3, and SAYC6, respectively (They were also marked SBC and SAYC series of composites, respectively).

The composite powders were dry-press molded at room temperature. Then the SBC and SAYC compacts were sintered without pressure at 2200°C and 1950°C, respectively.

Apparatuses and methods of analysis

The density D of the composites after sintering was measured according to Archimedes law, as well as the theoretical density D_T of them was determined from mixture-law of composites (The calculated D_T of SBC0, SBC3, and SBC6 were 3.152 g cm^{-3}, 3.125 g cm^{-3} and 3.098 g cm^{-3}, respectively; and the values of SAYC0, SAYC3, and SAYC6 were 3.304 g cm^{-3}, 3.273 g cm^{-3} and 3.241 g cm^{-3}, respectively). Correspondingly the relative density RD was calculated by Equation (1) and the remaining porosity included both open and closed holes (Also refer to Figure 6):

$$RD = D/D_T \times 100\% \tag{1}$$

The electric resistance R of the samples was measured based on two-electrode method, and the dimensions of the samples were 20 mm thickness and 2 mm × 4 mm section for decreasing measuring error caused by transition resistance of Ag electrode-sample. Then the electric resistivity ρ was calculated by Equation (2):

$$\rho = \frac{R \cdot S}{d} \tag{2}$$

where S is the sectional area and d is the thickness of the samples.

The real part ε' and imaginary part ε'' of dielectric constant ε in the X band were tested by wave-guide methods (HP8722ES vector-net-analyzer). Before test the samples were first cut into 22.86 mm × 10.16 mm × × 2 mm blocks. The dielectric loss angle tangent tg δ was calculated by Equation (3):

$$\mathrm{tg}\,\delta = \varepsilon''/\varepsilon' \tag{3}$$

The microwave attenuation property of the samples in the X band was tested by wave-guide method (Wiltron 54169A scalar-net-analyzer). The size of samples was 22.86 mm × 10.16 mm × 12 mm.

JXA-8100 electron probe analyzer (EPA) was used to observe the morphologies of samples.

RESULTS

Relative Density (RD)

RD of SBC0, SBC3, and SBC6 composites were 94 %, 89 % and 86 %, respectively; and RD of SAYC0, SAYC3, and SAYC6 composites were 94 %, 87 % and 84 %, respectively. It is clear that the density of the composites decreased with the C additions after sintering, whatever for SBC composites or SAYC ones. And the reason is not the key point in this paper and has been discussed in lots of references [3-10].

In particular, the tested results show that two sorts of sintering procedures mentioned previously are both reasonable for fabricating expecting SiC composites, and RD of SBC0 and SAYC0 are clear evidences.

Electric properties

The effects of C content on Electric Resistivity ρ of SBC and SAYC composites is shown in Figure 2. The real part e' of dielectric constant as well as the dielectric loss angle tangent tgδ of two series of composites in the X band are shown in Figures 3 and 4, respectively.

The results are drawn as follows: 1) The eletric properties depended strongly on the C addtions. With the C addition increasing, ρ decreased obviously but ε' and tg δ increased, whatever for SBC or SAYC composites; 2) In Figures 3 and 4, all curves of the composites except SAYC6 series were relatively steady and the waving

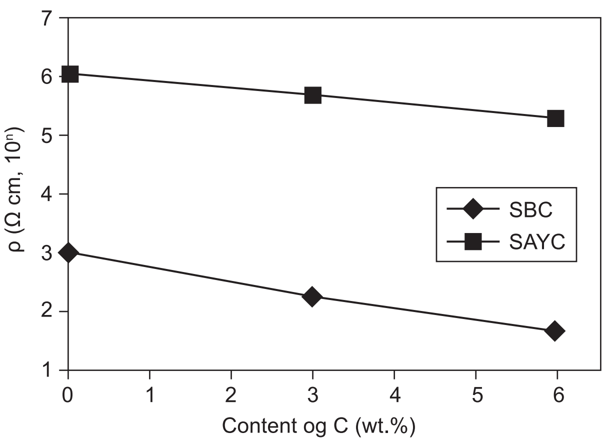


Figure 2. Relationship between C content and electrical resistivity (ρ) composites.

range was less than 1 magnitude throughout the whole X band. So we conclude that the above composites are ideal wide-band attenuation materials; 3) ρ of SBC and SAYC were in the range of 10^1-10^4 Ω×cm and 10^5-10^7 Ω cm, respectively; 4) For SBC3 composites, ρ was about 10^2 Ω cm, ε' and tg δ were in the range of 22.59-72.25 and 8.9-16.6, respectively, which are expected results. [11-12] (The reason will be explained later)

Microwave attenuation properties in the X band

Figure 5 shows the microwave attenuation properties of SBC and SAYC composites in the X band. The results show: 1) Under identical conditions, SBC composites were better in the microwave attenuation properties than SAYC composites; 2) The microwave attenuation properties of SBC3 are the best, the highest value is -40.5dB and almost all values are more than -30 dB through the whole X band.

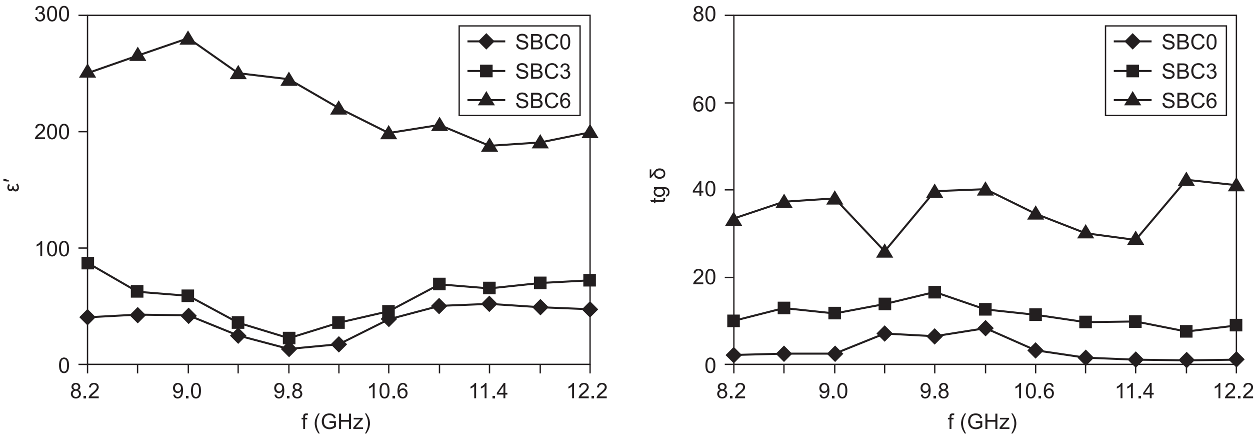
Micrograph

By analyzing the above electric properties and microwave attenuation properties, we conclude that SBC composites were better in the microwave attenuation properties than SAYC composites. Therefore, we observed the micrographs of the surface of SBC composites (Shown in Figure 6). C aggregation and connection in SBC6 composites are obvious; whereas most C is isolating each other in SBC3 composites. Besides, open holes were observed in the surface, so we conclude both open and closed holes existed after sintering.

DISCUSSION

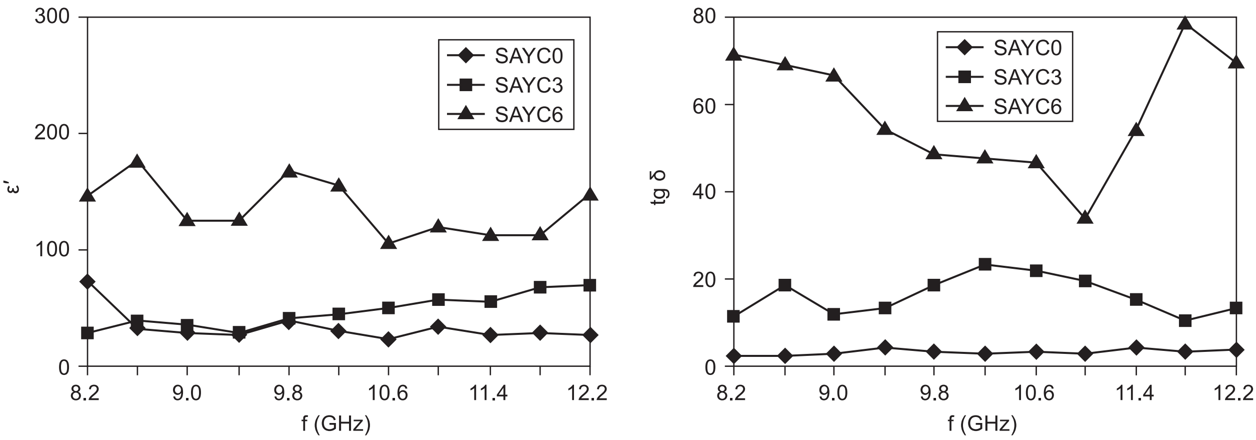
Match of interface of wave impedance [11-12]

We have emphasized the match of wave impedance in two media mentioned in the introduction. For most applications, microwaves always propagate from air to



a) ε′ b) tg δ

Figure 3. Real part of dielectric constant ε' and dielectric dissipation angle tangent tg δ of SBC composites in the X band.



a) ε′ b) tg δ

Figure 4. Real part of dielectric constant ε' and dielectric dissipation angle tangent tg δ of SAYC composites in the X band.

an attenuation material. Thus, to avoid the reflection of waves on the interface of two media, the wave impedance of attenuation material should match with that in the air. The wave impedance Z is defined by Equation (4):

$$Z_i = (\mu_i \mu_0 / e_i e_0)^{1/2} \tag{4}$$

where e_i and μ_i are dielectric constant and magnetic permeability of medium i, ε_0 and μ_0 are electric and magnetic constant, respectively.

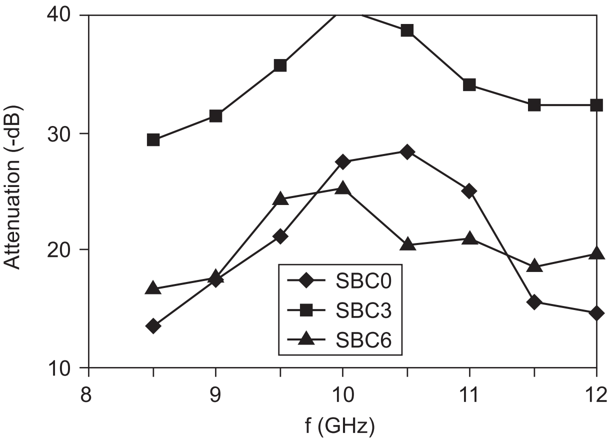
Z in the air can be calculated by Equation (3) and is equal to 377 W because of e = μ = 1 here. So, for a good match of the wave impedances and low reflection in the interface, the best way is to improve the electric properties of attenuation materials.

Relationship between ρ and microwave attenuation [11-14]

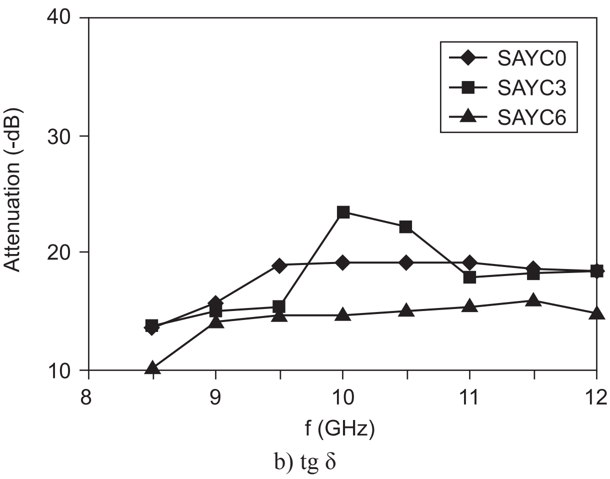
For microwave attenuation materials, the match of interface of wave impedances should been first considered to avoid the further reflection. Generally Z is hard to be exactly calculated but could be estimated by Equation (5) [6]:

$$Z \approx (1 + j)\sqrt{\frac{\varpi\mu\mu_0\rho}{2}} \tag{5}$$

where ω is angular frequency.

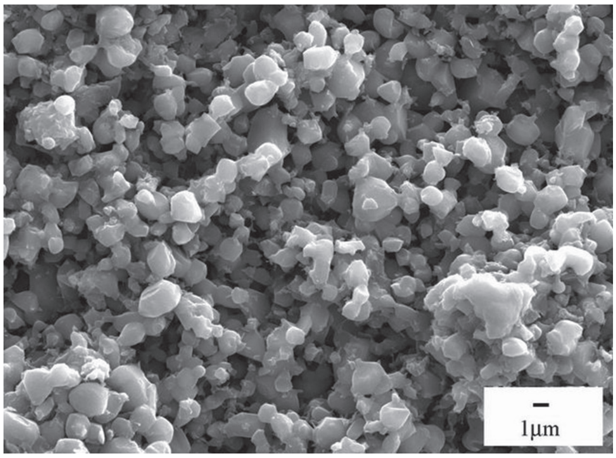


a) ε′

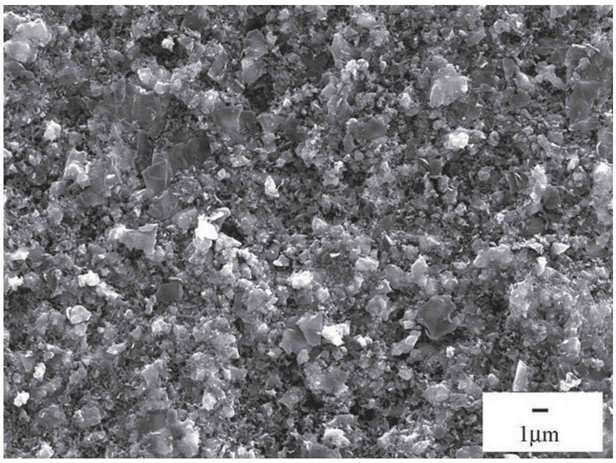


b) tg δ

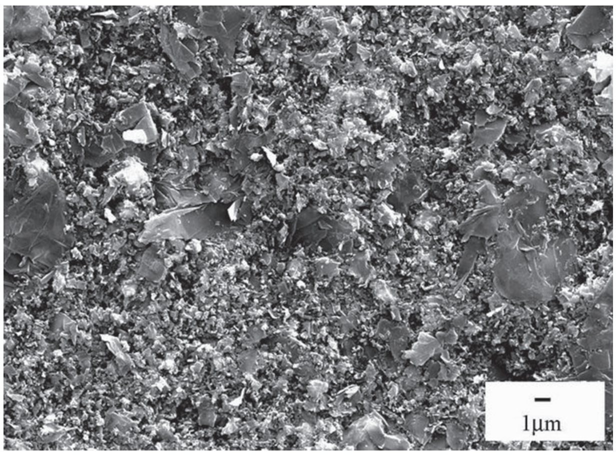
Figure 5. Microwave attenuation (-dB) of SBC and SAYC composites in the X band.



a) SBC0



b) SBC3



c) SBC6

Figure 6. Micrographs of surface of SBC composites.

Therefore, if ρ is rather low, it consequentially results in a mismatch of wave impedance between two media, accordingly brings lots of reflection of microwaves. Of course, the microwave attenuation property of composites is poor.

The microwave is mainly consumed by leakage conductance after they enter the attenuation composites. Theoretically, high C content will result in the further consumption of microwave.

Microwave can be effectively consumed by controlling proper electric properties or magnetic properties for different attenuation mechanisms. For SiC–C composites, the microwave is attenuated depended strongly on leakage conductance but not on magnetic activity, so electric parameters such as ρ were taken into account but magnetic parameters such as μ were not considered.

Match of interface of wave impedance and good leakage conductance are both necessary for good microwave attenuation. Therefore, proper ρ of composites is also necessary. For SiC matrix materials, it is reported that an ideal ρ is 10^1-10^3 Ω cm.

Relationship between dielectric properties and microwave attenuation [11-14]

For dielectric constant ε, ε' means a capability of depositing charges and ε'' a capability of consuming charges. Theoretically, the higher e' and e'' of composites are, the more the consumption of microwave is.

Similar to the effect of ρ, high ε' and ε'' also result in a mismatch of wave impedance between two media, which could be expressed by the definition of Z in Equation (4). The best microwave attenuation and the most suitable match of interface of wave impedance are always contradictive of each other. To obtain low reflection and high consumption of microwave in the attenuation composites, proper e is also necessary.

According to its definition (See Equation (3)), high dielectric loss angle tangent tg δ brings a good attenuation of microwaves. However, high tg δ may derive from two cases, one is a high ε'' but the other is a low ε'. The former results in a mismatch of wave impedance and the latter bring a low deposition of charges.

According to references [11], an ideal attenuation material should have a middle modulus of e and at best ε'' > ε'. For SiC matrix materials, an ideal ε' should be less than 100 and tgδ should be 1-20.

Effect of C distribution on microwave attenuation property [15]

Undoubtedly, the best contribution of C for microwave attenuation materials is to improve their electric properties and accordingly to obtain an excellent attenuation. Moreover, the distribution of C in the composites also affects their attenuation.

When C is isolated in the composites, it shapes an isolated “island”, these “islands” result in increased reflection, refraction and attenuation of the microwave in the body, effectively consuming the energy of the waves.

Besides, as a second-phase, C increases the polarization of the interface in the body, also effectively consuming microwaves.

Combined with the test results, when C was 3 wt.%, the isolated C has resulted in the excellent attenuation, but when C was over 6 wt.%, the aggregate of C and the changes of electric properties have decreased the microwave attenuation.

Effect of sintering methods on microwave attenuation property of composites

Combined with the above results and discussions, it is clear that solid phase sintering was more suitable to fabricate SiC-C composites with excellent microwave attenuation than liquid phase sintering in our test. Better microwave attenuation of SBC series was obtained due to a more proper dielectric property in the material.

CONCLUSIONS

1. Both methods are cost-effective and easily controllable processes and solid phase sintering was more suitable to fabricate excellent SiC–C attenuation composites than liquid phase sintering; i.e. SBC composites were more suitable for microwave attenuation material than SAYC composites.
2. Electric properties of SiC–C composites depended strongly on C additions and sintering methods. Therefore, the proper electric properties of composites could be obtained by the optimization of compositions and sintering process.
3. Match of interface of wave impedance and high wave consumption are both necessary for microwave attenuation materials. By controlling electric parameters, SiC–C composites with excellent microwave attenuation could be obtained.
4. SBC3 prepared by solid phase sintering was the best microwave attenuation composite in the test, the biggest microwave attenuation was -40.5dB and most attenuation values were above -30dB in the whole X band.

Acknowledgements

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