



High-temperature microwave bilayer absorber based on lithium aluminum silicate/lithium aluminum silicate-SiC composite

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Abstract

The microwave absorbing properties of lithium aluminum silicate (LAS) and LAS–SiC double layer composite absorbers were investigated within the frequency range of 8.2–12.4 GHz at 300–500 °C. The composite absorbers for use as high-temperature radar wave-absorbing materials were developed by hot-pressing LAS glass–ceramic and LAS–SiC composite, which were used as the impedance transformer layer and the low-impedance resonator layer, respectively. Nanometer-size β -SiC powders were fabricated at the relatively low sintering temperature of 1450 °C in argon atmosphere. The structure and morphology of the SiC powders were characterized using thermogravimetry-differential scanning calorimetry (TG-DSC), X-ray diffractometry (XRD) and scanning electron microscopy (SEM). The electromagnetic absorbing properties of the double layer at different temperatures and SiC contents were measured at normal temperature (27 °C) and high temperature by a vector network analyzer. The results indicate that the contents of nanometer-size β -SiC can increase the relative permittivity and dielectric loss of the double layer microwave absorbers. At high temperatures, the microwave absorber consisting of a 2-mm-thick layer of LAS ceramic and a 2-mm-thick layer of LAS–SiC composite with 10 wt% SiC content exhibited excellent performance with a minimum absorption at –42.8 dB at 10.5 GHz and the absorption bandwidth (reflection loss less than –10 dB) of 3.5 GHz in the X-band. The minimum reflection loss measured at 300–500 °C was less than –24 dB.

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1. Introduction

Recently, radar absorbing materials (RAMs) used as military stealth materials which have attracted significant attention for radar cross section (RCS) reduction applications [1–3]. Stealth materials in weapons can be categorized into normal-temperature and high-temperature applications according to their optimal uses. Most magnetic materials are constrained by their respective Curie temperatures and easily lose their low reflectivity characteristics at high temperatures because of the associated loss of their magnetic properties. Thus, magnetic absorbing materials are generally used only in the normal-temperature stealth region of weapons applications but are not suitable for hyper-velocity missiles, rockets or aircrafts in

which the engines output significant heat. In comparison to magnetic materials, however, ceramic dielectric composites perform with suitable lossy capabilities and relatively stable complex permittivities in the range of room temperature to high temperature [4]. Silicon carbide (SiC) was studied as a dielectric structural ceramic due to its high strength and hardness, low density, good resistance to oxidation, high thermal stability and high thermal conductivity at elevated temperatures [5–7]. Additionally, SiC is considered to be one of the most important microwave absorbing materials due to its sufficient dielectric loss in microwave radiation [8–10].

The β -SiC phase is widely used in industrial applications and is the subject of a variety of research efforts due to its superior properties. Further, it is inorganic and acts as a high-temperature wave absorbing material; consequently, it can be used as an absorber matrix. Typically, silicon carbide is produced by the carbothermal reduction of silica using the

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Acheson process, which involves heating silica sand and carbon to high temperatures (2500 °C) in an Acheson graphite resistance furnace [11]. The commercial product has a large grain size and is invariably contaminated with oxygen. Several other fabrication processes can be used to obtain SiC, including polymer pyrolysis [12], chemical vapor deposition (CVD) [13] and hot pressing. Most of the above-mentioned approaches require temperatures higher than 1500 °C and consume significant quantities of energy. In contrast, sol–gel processing is one of the most advanced methods for the fabrication of β -SiC powders and can be performed at room temperature by homogenizing a sol system derived from the initial liquid components [14–24]. In comparison to ferrites, SiC is used in high temperature conditions, and its loss factor increases with temperature [10,25]. Unfortunately, few systematic studies on the microwave absorption properties of SiC-based absorbers have been performed in high-temperature environments, and most documented characters refer to normal temperatures.

In this work, nanometer-size β -SiC powders were fabricated at a relatively low temperature by the sol–gel process and were expected to exhibit better microwave absorption properties than the commercially produced powders. The powder phase components and morphology were studied with X-ray diffraction (XRD) and scanning electron microscope (SEM), respectively. To achieve a broadened absorption bandwidth of the dielectric absorber, a double layer absorber was developed in which the β -SiC powders were mixed with lithium aluminum silicate (LAS) glass–ceramics to form the SiC–LAS composite layer as a low impedance resonator, and the other layer from the LAS glass–ceramics acts as the impedance transformer layer [4,20]. In this study, an enhanced microwave absorption characteristic was observed for the LAS/LAS–SiC composite absorbers. The microwave absorbing properties of this material are further discussed below.

2. Experimental

2.1. Preparation of the β -SiC powders

A fixed quantity of phenolic resin (a condensation polymer of methanol and phenol with a softening temperature of 80–90 °C) was mixed with ethanol and stirred until it dissolved. Then, the reagent TEOS (tetraethyl orthosilicate, $\text{Si}(\text{CH}_3\text{CH}_2\text{O})_4$) was mixed with several drops of HCl in a solution of deionized water and ethanol. The HCl solution was slowly poured into the phenolic resin/ethanol solution, followed by 48 h of stirring at room temperature to hydrolyze the TEOS and form the sol. Finally, a small amount of hexamethylenetetramine ($\text{C}_6\text{H}_{12}\text{N}_4$) in ethanol solution (35 wt%) was added to the sol dropwise to accelerate the polymerization and form the gel. The gel was then baked in an oven at 100 °C for 24 h, aged and dried to form the xerogel precursor. The xerogel was then placed in a quartz boat, which was inserted into the central position of a high-temperature tube furnace. The tube was covered at both ends, and the gas flow into the tube was controlled by a valve in the cover. The tube was first

purged with argon; then, the argon flow was fixed at 40 cm^3/min . The xerogel was heat-treated at 1450 °C for 2 h with a heat ramping speed of 2 °C/min in argon. Under these conditions, a carbothermal reduction occurred in the xerogel, forming the SiC powders. The phenolic component in the xerogel served as a carbon source in the carbothermal reduction. Another component, NH_4NO_3 , released gases upon heating, which prevented condensation and formed the internal pores. The relatively low temperature employed in this method (other methods require 1550 °C or higher) suppresses crystal grain growth. The formation of pores by gas release suggests that porous nanometer-size SiC powders can be achieved with this method. The products are also expected to possess relatively high dielectric dissipation. The synthesized raw SiC powders were calcined in air to remove carbon residues and washed with HF to remove SiO_2 . Additionally, the LAS glass was prepared from analytical grade reagents of SiO_2 , Al_2O_3 and Li_2O_3 in a molar ratio of 4:1:1, fabricated by the hot-pressing method.

2.2. Preparation of the LAS/LAS–SiC double-layer composite absorber

The high-temperature microwave composite absorbers composed of a LAS layer and a LAS–SiC layer were fabricated by the hot-pressing method. Hot-press sintering of the LAS glass–ceramic formed the outer layer, which is an impedance transformer layer. The inner layer was formed by hot-press sintering of SiC powders evenly distributed within the LAS glass–ceramic. The inner layer worked as a low impedance resonator. The width and length of both layers were 15 cm with thicknesses of 1 mm, 2 mm or 3 mm, depending on the specific experimental design. The layers were laid on the substrate as follows: the inner layer (i.e., the LAS–SiC layer or low impedance resonator) was applied to the substrate first; then, the outer layer (i.e., the LAS layer or impedance transformer layer) was deposited on top. The resulting composite is a so-called “double-layer microwave absorbing material.” The reflection loss or reflectivity was then measured at normal and high temperatures. The structure of the double-layer microwave absorber is shown in Fig. 1.

2.3. Characterization

X-ray diffractometry (XRD) was used to analyze the crystallographic composition and poly types of the carbothermal reduction products. Thermogravimetry-differential scanning calorimetry (TG-DSC) measures weight changes in materials for determining the composition, thermal stability and related phenomena. The morphologies of the powders were observed by scanning electron microscopy (JEOL,

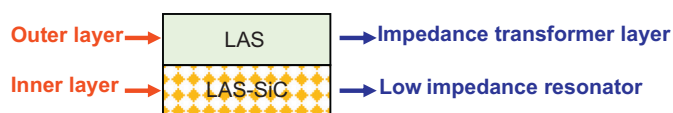


Fig. 1. The structure of the double layer microwave absorber.

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