



A self-setting particle-stabilized porous ceramic panel prepared from commercial cement and loaded with carbon for potential radar-absorbing applications

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Abstract

Porous ceramic materials are in a current research focus because of their outstanding thermal stability, chemical stability and lightweight. Recent research has widened the range of applications to radar absorption to utilize the advantages of porous ceramic materials. There has been long-standing interest in the development of lightweight radar-absorbing materials for military applications such as camouflaging ground-based facilities against airborne radar detection. Therefore, in this study, a novel lightweight radar-absorbing material for X-band frequencies was developed using a self-setting particle-stabilized porous ceramic panel composed with carbon. The panel was prepared using a commercial calcium aluminate cement (as a self-setting matrix), zeolite 13X particles with propyl gallate (as a particle-stabilized pore former) and carbon (as a radar-absorbing material). The panel contained macropores approximately 200 to 400 μm in size formed by zeolite 13X particles that are irreversibly adsorbed at liquid-gas interfaces. The self-setting particle-stabilized porous ceramic panels were characterized by scanning electron microscopy, mercury porosimetry, physisorption analysis, capillary flow porosimetry and network analysis. When 0.2 wt.% carbon was added to a self-setting particle-stabilized porous ceramic panel to fabricate a composite 7 mm thick, the maximum reflection loss was -11.16 dB at 12.4 GHz. The effects of the amount of added carbon and the thickness variation of a self-setting particle-stabilized porous ceramic panel on the radar-absorbing properties remain important issues for further research.

Keywords: self-setting particle-stabilized porous ceramic panel, radar-absorbing properties

I. Introduction

Porous ceramics have received recent attention [1] because of their unique properties, such as low density [2], low thermal conductivity [3,4] and low dielectric constant [5]. Among many applications of porous ceramics, a porous ceramic panel for radar-absorbing applications is a feasible candidate. This is because con-

ventional porous polymer panels that are used for radar-absorption applications (usually polyurethane foam) cannot be operated under high temperature and harsh environments owing to the limitations associated with polymer materials.

Radar-absorbing materials are specially designed and shaped to effectively absorb incident radio frequency (RF) radiation from as many incident directions as possible. Since World War II, radar-absorbing materials have attracted much attention because of their stealth characteristics in military applications such as aircrafts,

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land vehicles, naval vessels, and support facilities [6]. However, it is still very challenging work to develop a low-cost, lightweight radar absorbing material. Therefore, a few researchers have investigated low-cost materials such as electronic waste [7], rice husks [8] and forest fire ash [9]. Radar-absorbing materials can be prepared by introducing conductive particles such as carbon or metal particles to induce a dielectric loss by enhancing the conductivity of the material. In particular, carbon-based conductive particles have been widely used because of their good absorption performance in the high frequency bands [10]. These include carbon nanotube-based composites [11,12] and carbon-based composites [13,14]. Because porous ceramics are both inherently lightweight and inexpensive, it is worthwhile to study them as low-cost lightweight radar absorbing materials.

If a radar-absorbing material needs to be impregnated or adsorbed onto a porous ceramic matrix, the porous ceramic matrix should have an open-cell pore structure. In the impregnation method [15], a highly open-cell pore structure is soaked in a slurry consisting of both a radar-absorbing material and a binder, until the internal pores are filled in with the radar absorbing material. However, in general, the mechanical strength of an open-cell pore structure is significantly lower than that of a closed-cell pore structure because of the inherent limitations of the replica technique, which is mainly used when an open-cell pore structure is fabricated [2,16]. The minimum pore size is also limited to approximately 200 μm , because of the difficulty of impregnating an open-cell pore structure with excessively narrow cells [1].

However, if a radar-absorbing material is composited with a pore structure from the beginning of the preparation of a porous ceramic panel, the panel does not need to be an open-cell pore structure, which has an inferior mechanical strength, because no post-processing such as impregnation or coating is required. Hence, in this study, a particle-stabilized direct foaming method was introduced in order to have a porous ceramic panel composited with a radar-absorbing material from the start. With the rapid progress in colloidal chemistry, a closed-pore structure can now be fabricated by incorporating air into an aqueous suspension containing surfactant molecules, then drying and sintering the resulting sample. This processing method is known as a particle-stabilized direct foaming method [16–27] and it has been extensively studied as a promising route for fabricating porous materials because of its exceptional stability. Particles that are irreversibly adsorbed at a liquid-gas interface impede the coalescence or shrinkage of bubbles, stabilize the liquid foam, and do not assemble to yield aggregates, in the same way that surfactant molecules form micelles. This processing method offers an easy, inexpensive and rapid approach to fabricating highly porous ceramics with a closed-cell pore structure.

Carbon as a radar-absorbing material cannot withstand high temperatures because oxidation reactions occur, which means that a conventional sintering process is not appropriate for preparing a porous ceramic panel composited with carbon. Therefore, pore forming by a particle-stabilized method and self-setting by calcium aluminate cement should provide an easy, inexpensive and rapid approach to fabricating porous ceramic panels composited with carbon. In addition, because the particle shapes of calcium aluminate cement and zeolite are irregular, the self-setting particle-stabilized porous ceramic panel would have intermediate characteristics between an open-cell pore structure and a closed-cell pore structure, which means that further coating processes could be adopted if needed.

As far as the authors know, no research group has studied a self-setting particle-stabilized porous ceramic panel composited with a radar absorbing material, although several recent reports show the feasibility of self-setting particle-stabilized porous ceramics using calcium aluminate cement [26–28]. Therefore, the aim of this study was to clarify whether a porous ceramic panel composited with carbon can be prepared by a self-setting particle-stabilized method without a conventional sintering process.

II. Material and methods

2.1. Sample preparation

Zeolite 13X (Molecular sieve 13X, Sigma-Aldrich, USA) and propyl gallate (3,4,5-(HO)₃C₆H₂CO₂CH₂CH₂CH₃, 98% pure, Sigma-Aldrich, USA) were used as pore formers. We added 40 ml of distilled water, 0.6 g of propyl gallate, 20 g of commercial calcium aluminate cement (KS L 5201 standard, Tongyang Cement, Korea), and carbon (Nano carbon, Sigma-Aldrich, USA) to 20 g of the zeolite 13X. Optionally, 1 g of lithium carbonate (99% pure, Sigma-Aldrich, USA) was also added. We had determined by preliminary experiments that the maximum amount of carbon to avoid carbon agglomeration is 0.2 wt.% and the minimum amount of carbon to affect the reflection loss is 0.04 wt.%.

Foaming was carried out using a direct-driven motor at a speed of 1000 rpm. The slurry was poured into an acrylic mold and allowed to set for 48 h at room temperature for self-setting. For comparison, 40 g of commercial calcium aluminate cement and 40 ml of distilled water were used for the preparation of a self-setting ceramic panel.

2.2. Characterization

The pore characteristics of the self-setting particle-stabilized porous ceramic panel were investigated by scanning electron micrography (JSM-5800, JEOL, Japan), and mercury porosimetry (Autopore IV 9510, Micromeritics, USA). The compressive strengths were measured by a uni-axial mechanical tester (Instron

4206, Instron, USA) with $10\text{ mm} \times 10\text{ mm} \times 10\text{ mm}$ specimen dimensions, and a crosshead speed of 0.5 mm/s . To measure the complex permittivity of the self-setting particle-stabilized porous panel at X-band, an Agilent N5230A analyser (PNA-L Vector Network Analyzer) was used. The dimensionless permittivity was obtained from the scattering parameters for reflected and transmitted microwaves over the $8.2\text{--}12.4\text{ GHz}$ range using Agilent 85071E (Materials Measurement Software), which employs the Nicolson-Ross-Weir method. In detail: the complex permittivity, permeability, and reflection loss (electromagnetic wave absorbing performance) of the samples with dimensions of $150\text{ mm} \times 150\text{ mm} \times 10\text{ mm}$ were measured by a free space measurement system which consists of transmitting and a receiving spot-focusing horn antennas, a network analyser, and a sample holder. Compared to traditional measurements of electromagnetic properties such as coaxial, waveguide or cavity resonance methods, the contactless and nondestructive free space measurement system is highly effective for brittle samples like the open-pore ceramic specimens. The material parameters were determined from the measured transmitted and reflected scattering parameters.

Note that, hereinafter, CP denoted the self-setting ceramic panel, and PPCP denoted that self-setting particle-stabilized porous ceramic panel.

III. Results and discussions

The PPCP panel after 48 h of setting time and the acrylic mould that was used are shown side by side in Fig. 1. Although the authors used a $15\text{ cm} \times 15\text{ cm}$ square acrylic mould, the shape and size of the sample have no restrictions because of its self-setting ability.

Prior to analysing the PPCP samples, a CP was prepared for comparison. A typical scanning electron microscope (SEM) image of the CP sample after 48 h of setting time is shown in Fig. 2a. The CP had the typical microstructure of the hardened calcium aluminate cement. In preliminary experiments, a needle-like ettringite phase was formed as a result of the reaction of calcium aluminate with the calcium sulphate that is commonly present in the microstructure of the hardened calcium aluminate cement, as seen in Fig. 2b. It is

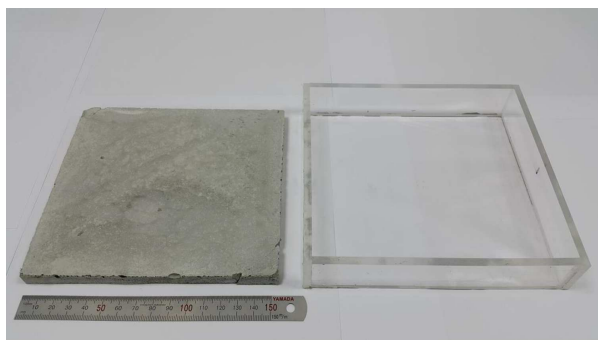


Figure 1. Image of the PPCP after 48 h of self-setting time and the acrylic mould that was used

well known that the ettringite phase does not contribute to strength [29]. Moreover, it should be noted that the voids within the strut walls have a detrimental effect on material strength. Hence, the presence of an ettringite phase in the PPCP would prevent it from having adequate strength for handling. Therefore, lithium carbonate was added to minimize the ettringite phase [28]. The authors expected that the crystallization of ettringite could be inhibited by accelerating the hydration process of the calcium aluminate cement.

Typical SEM images of the PPCP after 48 h of setting time are shown in Figs. 2c and 2d. The panel contained macropores, with a size range of approximately 5 to $50\text{ }\mu\text{m}$, formed by the zeolite 13X particles in the calcium aluminate cement matrix that were irreversibly adsorbed at the liquid-gas interfaces. If an ettringite phase had been generated, the majority of these macropores would have decomposed during the hydration process in the presence of lithium carbonate, similar to the case of barium carbonate [30]. The macropores were highly inter-connected with neighbouring macropores through voids within the strut walls. The microstructure of the PPCP sample shows voids within the strut walls with a few ettringite needles among the zeolite 13X particles. Therefore, the microstructure of the panel is typical of the particle-stabilized direct foaming method.

The particle-stabilized direct foaming method is intrinsically based on the idea that particles can be used to adsorb irreversibly on the surfaces of gas bubbles to stabilize wet foams. Upon adsorption, particles lower down the overall free energy of the system by replacing part of the highly energetic gas-liquid interfacial area with less energetic interfaces [31]. Therefore, if the particles are uniform and spherical, the surfaces of the gas bubbles are fully covered with a single layer of particles. If the particles are irregular, the surfaces of the gas bubbles are covered with several layers of particles. In this study, because zeolite 13X particles and calcium aluminate cement particles are non-uniform, there were many inter-particle voids induced by the irregular particles. Thus, the surfaces of the gas bubbles were covered with as many particles as possible, which is energetically favourable. Therefore, it was observed that the PPCP had intermediate characteristics between an open-cell pore structure and a closed-cell pore structure, as shown in Figs. 2c and 2d.

Figure 3 shows the pore size distributions of the PPCP and CP samples. The PPCP shows a peak at approximately 5 to $50\text{ }\mu\text{m}$ and another peak at approximately 100 to $1000\text{ }\mu\text{m}$. The former corresponds to inter-particle voids between the zeolite 13X particles, whereas the latter corresponds to the voids inside the strut walls, throats, or entrance openings of spherical macropores because mercury enters the macropores at a pressure determined by the entrance size rather than by the actual spherical pore size [32]. The CP shows few peaks because of the lack of macropores inside the microstructure, as shown in Fig. 2a.

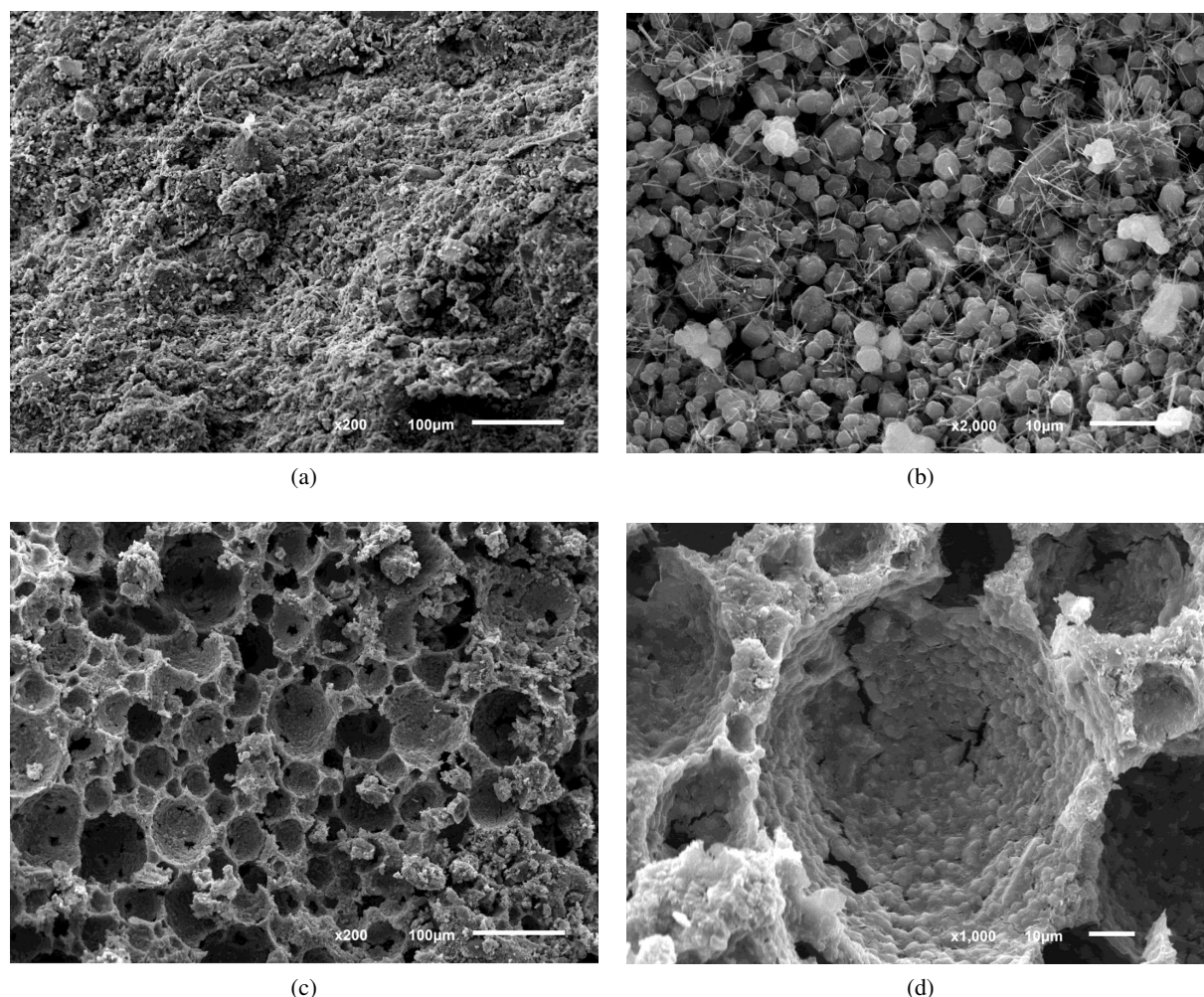


Figure 2. SEM images of: (a,b) the CP after 48 h of self-setting time, (c,d) the PPCP after 48 h of self-setting time

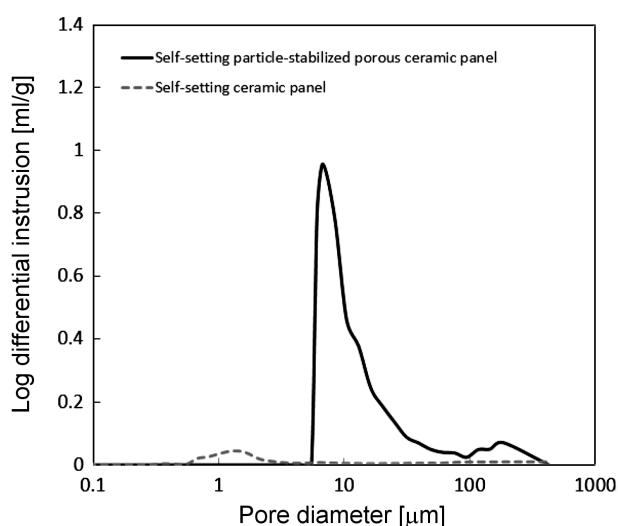


Figure 3. The pore size distributions of the PPCP and the CP samples

The authors could not expect good radar-absorbing properties of a self-setting ceramic panel regardless of the presence of macropores, unless carbon was added as a radar-absorbing material. Therefore, carbon was added to a self-setting particle-stabilized porous ce-

ramic panel. Typical SEM images of the PPCP composited with 0.2 wt.% of carbon after 48 h of self-setting time are shown in Figs. 4a and 4b. The PPCP composited with 0.2 wt.% of carbon contained macropores with a size range of approximately 5 to 50 μm formed by the zeolite 13X particles in the calcium aluminate cement matrix that were irreversibly adsorbed at liquid-gas interfaces as seen with the PPCP without carbon. However, it was difficult to observe the presence of carbon because the amount added was very low.

If the density is high (thus increasing the material weight) or if the surface area is not high enough to effectively impregnate material (thus reducing its radar-absorbing properties), this process would not be acceptable for radar-absorbing applications, even though the authors could obtain a PPCP as a rigid monolith. Therefore, the pore volumes of the PPCP, the CP and zeolite 13X (pore former) were measured using Barrett-Joyner-Halenda (BJH) analysis, and the results are shown in Fig. 5a. Figure 5b shows the Brunauer-Emmett-Teller (BET) surface area and density of each material. The density (1.80 g/cm^3) and the BET surface area ($359.67 \text{ m}^2/\text{g}$) of the PPCP are between those of a CP (3.16 g/cm^3 , $1.02 \text{ m}^2/\text{g}$) and zeolite 13X (1.43 g/cm^3 , $729.10 \text{ m}^2/\text{g}$). In the literature,

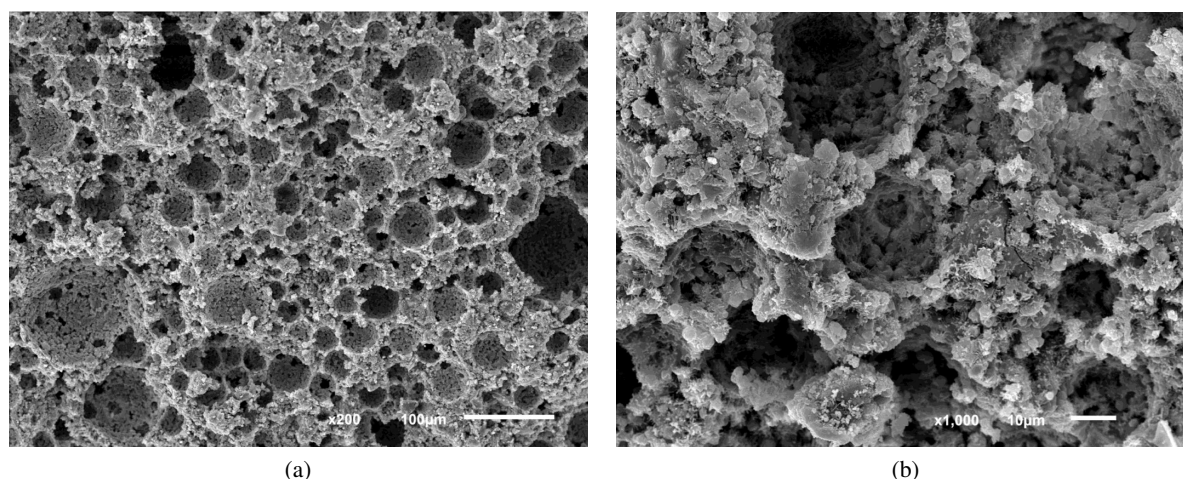


Figure 4. (a) SEM of the self-setting particle-stabilized porous ceramic panel composited with 0.2 wt.% of carbon after 48 h of self-setting time and (b) magnification of its macropore morphology

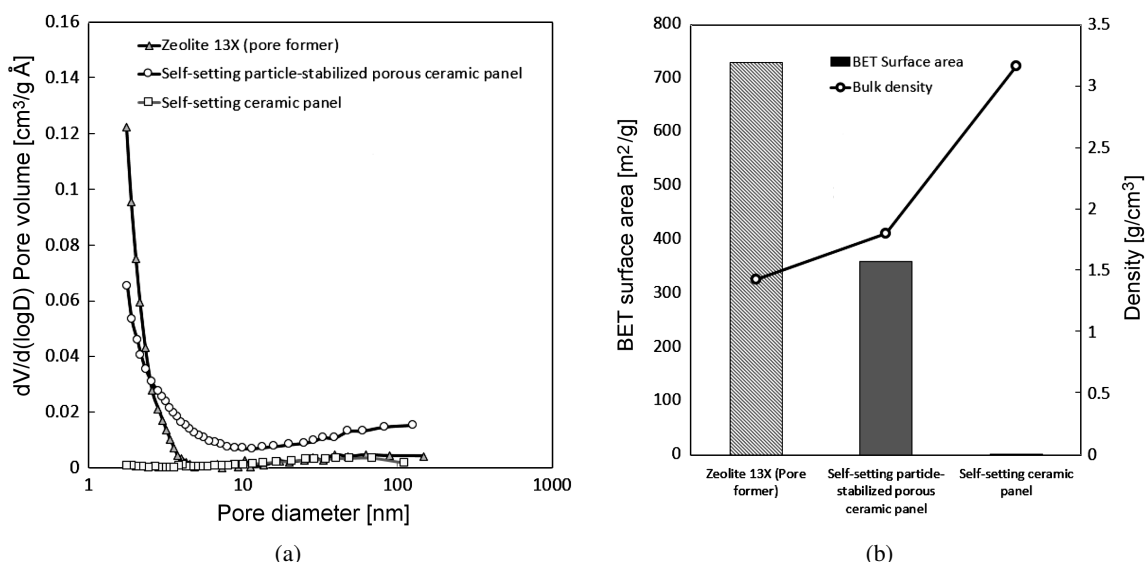


Figure 5. (a) The BJH adsorption pore volume and (b) the BET surface areas and bulk densities of the PPCP, the CP and the zeolite 13X powder (pore former)

self-standing zeolite monoliths such as faujasite zeolite (63.31 m²/g, and 19.6 MPa) [33], NaY (97.81 m²/g, and 57.60 MPa) [34], and Zeolite A (30 m²/g, and 0.7 MPa) [35] showed reduced BET surface areas, although the compressive strengths of these monoliths were significantly enhanced. Therefore, it is noteworthy that the PPCP, prepared in this study maintained its BET surface area (359.67 m²/g), following the rule of mixtures with calcium aluminate cement, and has an acceptable compressive strength under non load-bearing conditions (1.80 MPa).

Figure 6a shows the permittivities of the PPCP composite with 0.04 wt.% and 0.2 wt.% of carbon. The higher the carbon weight percent is, the greater the permittivity is. This might be explained by the conductive behaviour of the carbon particles inserted into the porous ceramic matrix. This trend corresponds well with the previously published data on carbon-based radar-absorbing materials [36,37]. Some of the incident

radar waves will be absorbed by the radar-absorbing material; the rest will start propagating through the medium with lower intensity than the incident wave [38]. The open-cell pore structure of a PPCP enables the free movement of waves inside the medium, and the radar-absorbing material (in this study, carbon) present inside the medium absorbs some of the radar waves.

The reflection losses of both the PPCP and the PPCP composited with 0.2 wt.% of carbon were measured in the range of 8.2 to 12.4 GHz (X-band), as shown in Fig. 6b. For a radar-absorbing material, the reflection loss is calculated from the complex permittivity and the permeability at a given frequency and absorber thickness according to transmission line theory. The measured reflection loss of the PPCP composited with 0.2 wt.% of carbon approaches -11.16 dB at 12.4 GHz for a 7.0 mm thickness, which corresponds to ≤ -10 dB reflection loss (i.e., more than 90% absorption of the incoming radar wave).

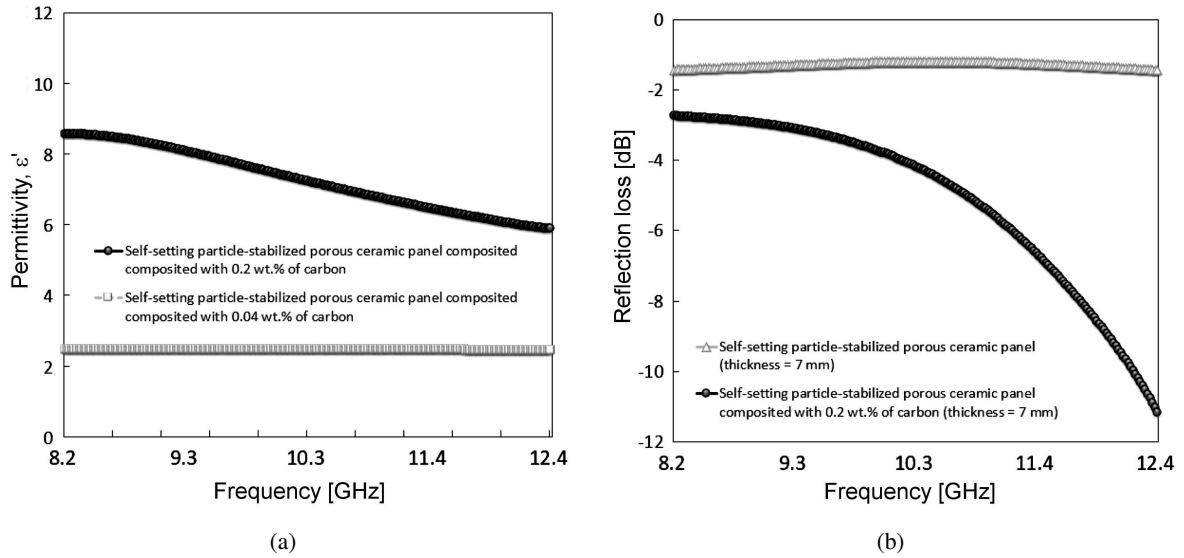


Figure 6. (a) Permittivities of PPCPs composed with 0.04 wt.% and 0.2 wt.% of carbon. (b) Reflection losses of PPCPs composed with 0.2 wt.% of carbon and without addition of carbon

These data are comparable with those of the carbon-based radar-absorbing materials reported in the literature, such as carbon nanotube-based composites (98.54% absorption at 11.1 GHz with a thickness of 2.0 mm [12], 98.4% absorption at 8 GHz with a thickness of 4.0 mm [11]), and carbon-based composites (99.5% absorption at 11.5 GHz with a thickness of 4.0 mm [13]). Compared to the above mentioned literature data, the reflection loss of the PPCP composed with 0.2 wt.% of carbon was acceptable, because a self-setting particle-stabilized porous ceramic panel is low-cost (the inexpensive raw materials), lightweight (the particle-stabilized pore structure), and easy to prepare (the self-setting fabrication method).

The reason behind the better absorption of the PPCP composed with 0.2 wt.% of carbon over a PPCP is the efficient combination of porous ceramic matrix and carbon particles. Usually, the radar-absorbing properties of dielectric materials are considered to be related to the efficient complementarity between the imaginary part and the real part of the permittivity in the materials [39]. Carbon is a conductive material with a large real part and imaginary part of its permittivity, hence, the poor impedance match results in weak absorption of the incoming electromagnetic wave. Therefore, controlling the amount of carbon added to a porous ceramic matrix is an effective way to tailor the permittivity and improve the impedance match. In this work the PPCP composed with 0.2 wt.% of carbon may possess better cooperative interaction than the other samples, resulting in better radar-absorbing properties.

In this study, the authors investigated the feasibility of a self-setting particle-stabilized porous ceramic panel as a low-cost, lightweight radar-absorbing material. Because radar-absorbing properties can generally be tuned by varying the thickness of the panel, the thickness is very important when dealing with electromag-

netic waves. Also, the self-setting method can provide a PPCP with a uniform surface and thickness. The effects of the amount of added carbon, and the thickness variation of a PPCP on the radar-absorbing properties remain important issues for further research.

IV. Conclusions

In summary, a self-setting particle-stabilized porous ceramic panel contained macropores formed by zeolite 13X particles that were irreversibly adsorbed at the liquid-gas interfaces. The density of the panel was reduced to that of typical lightweight porous ceramics because of the highly inter-connected macropores, while obtaining an acceptable BET surface area for any further coating. Furthermore, the self-setting particle-stabilized porous ceramic panel can be prepared without cracks or defects induced by a delicate and costly sintering process and free of shape and size limitations. Therefore, combination of the particle-stabilized direct foaming method (using zeolite 13X and propyl gallate) and the self-setting method (using calcium aluminate cement and lithium carbonate) can provide an easy, inexpensive, and rapid approach to fabricating a highly porous ceramic with intermediate characteristics between an open-cell pore structure and a closed-cell pore structure. Also, when 0.2 wt.% carbon was added to a self-setting particle-stabilized porous ceramic panel to fabricate a 7 mm thick composite, the maximum reflection loss was -11.16 dB at 12.4 GHz. These findings show the feasibility of using a self-setting particle-stabilized porous ceramic panel composed with carbon in potential radar-absorbing applications.

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