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Rigid composite materials for anechoic chamber application

C. Méjean, M. Badard, R. Benzerga*, C. Le Paven-Thivet, A. Sharaiha

Institut d'Electronique et de Télécommunications de Rennes, Université de Rennes 1, IUT de Saint Brieuc, 18 rue Henri Wallon, 22004 Saint-Brieuc, France

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ABSTRACT

An electromagnetic absorbing material is developed to replace the polyurethane foam currently used in anechoic chambers. In order to solve issues related to the polyurethane foam (flexibleness, imprecise cut and inhomogeneous load dispersion), we propose the synthesis of new absorbent composites made of epoxy foam loaded with carbon particles. Our elaboration method leads to homogenous and rigid materials which allow a precise cut into pyramids and other complex shapes. Two kinds of carbon are used in association with epoxy foam. The dielectric characterization of these composites highlights low dielectric losses, contrary to expectation. In order to see if a chemical reaction between epoxy components and carbon black occurs, epoxy and polyester resins loaded with carbon are compared. Conductivity measurements on loaded epoxy resins revealed a percolating network, but this one doesn't show any effect on dielectric properties; the occurrence of chemical reactions between carbon and epoxy matrix is proposed.

1. Introduction

Electromagnetic wave absorbing materials show their interest in multiple fields of applications such as radars [1], electromagnetic compatibility [2] and anechoic chambers. In the literature, several works have focused on the optimization of absorbing materials. Two solutions are currently studied: the first one consists in optimizing the material's geometry [3,4], and the second one consists in optimizing the material itself, in particular its chemical composition [5]. Carbon based (graphite, graphene, nanotubes, particles . . .) polymer absorbing materials have been extensively studied because they show good electrical conductivities combined to a great abundance and to a relatively low cost and low weight [6–8]. Magnetic based materials have also raised interest thanks to the combination of magnetic and dielectric losses; they generally show good absorption performances at low frequencies ($<10^9$ Hz) [9] or at microwave frequencies [10,11]. However, two major drawbacks are related to magnetic materials: they are heavy and tend to oxidize. In consequence, hybrid materials combining magnetic particles and dielectric particles have also been studied [12,13].

Today, the most used absorbers in anechoic chambers are made of carbon loaded polyurethane (PU) foam cut into pyramidal shapes. These absorbers show a lot of advantages, like a low density thanks to the presence of porosities and good absorption performances for frequencies between 80 MHz and 40 GHz [14] thanks to the addition of carbon which enhances the electric conductivity of the material and leads to high dielectric losses (tan δ) [1]. Nevertheless these absorbers present several drawbacks such as the difficulty to machine the PU foam (because of its flexibleness) which induces a non-reproducibility of the forms and thus, an unreliability in the absorption properties. The flexibility of the PU foam also limits its use for the achievement of complex shapes required to enhance the absorption performance [3]. Furthermore, the loaded PU foams are made by impregnation in an aqueous solution containing carbon particles. This method gives rise to an inhomogeneous dispersion which limits the reproducibility of the performances of the pyramids. Moreover, by this elaboration technique, the load is located inside the open porosities and thus can easily escape from the foam. This leads to a bad ageing of the absorber and risks to human health. However, a solution consisting of a plastic paint coating added to the absorber is proposed to limit this effect [15].

We propose here to use another material to replace the PU foam: the epoxy foam. On one hand, it shows better mechanical properties, especially rigidity which enables the achievement of complex shapes since almost all the cells of the foam are closed [16]. On the other hand, the elaboration method for this epoxy

* Corresponding author.
E-mail addresses: ratiba.benzerga@univ-rennes1.fr (R. Benzerga), claire.lepaven@univ-rennes1.fr (C. Le Paven-Thivet).

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foam lowers the inhomogeneity since the load is added before the foaming step. The load being confined inside the walls of the cells provides a very good ageing of the absorber.

This paper concerns the synthesis, electric and dielectric characterization of epoxy foam loaded with carbon. The foamed composites are compared to non-foamed composites (epoxy and polyester resins loaded with carbon) in order to point out possible chemical reactions between the used reactants and the carbon black explaining the observed electric/dielectric properties.

2. Experimental methods

2.1. Composites elaboration

For this study, two types of carbon CB1 (graphite) and CB2 (carbon black) having different granulation sizes were used with different weight percentages in the foam: 0.1 wt%, 0.5 wt%, 0.92 wt %, 2.23 wt%, 3.58 wt%, 5 wt%, 7 wt%. The carbon particles are mixed to 10 g of epoxy resin (PB170 from Sicomin); then 3.6 g of hardener (DM02 from Sicomin, with *Diaminodicyclohexylmethane Polypropylene-Triamine* formulation) is added. The mix is allowed to rest for 6 h at ambient temperature to enable the foaming and the polymerization process. After, the foams are cured in an oven at 60° C for at least 6 h. This step achieves the polymerization of epoxy and fixes the mechanical properties which improves the rigidity of the foam. Foams are finally cut to show a flat surface to be characterized. It has to be noted that this elaboration method is completely different from the loaded PU foams one: in our case, the load is added in-situ, before the foaming step.

Two other types of composites, consisting in carbon loaded resins (non-foamed), have been made. Two types of resins were used: epoxy resin IP purchased from PRESI (20 g of resin mixed to 2 g of catalyst) and polyester infusion resin (20 g of resin mixed to 0.4 g of catalyst). Different weight percentages (0 wt%, 0.92 wt%, 2.23 wt%, 5 wt% and 7 wt%) of carbon were used to achieve the composites. The elaboration process of these materials is similar to the one of the loaded foams, but without the foaming step.

The morphology of the two carbon powders and the different foams was observed with a JEOL 5600 scanning electron microscope (SEM) using a 10 kV voltage. The specific surface area of the carbon powders was measured from the multipoint BET (Brunauer, Emett and Teller) method using a Tristar Micrometrics apparatus.

2.2. Dielectrical and electrical characterization of the samples

The complex permittivity ($\varepsilon' - j\,\varepsilon''$) of the samples was measured in the range 500 MHz – 18 GHz with an Agilent 85070E coaxial probe linked to an Agilent 8510C vector network analyzer. For this characterization, specific attention has to be paid to the sample's surface. The surface in contact with the probe has to be as flat as possible in order to avoid any air gap which would induce an underestimation of the complex permittivity. Polyester and epoxy resin samples were thus polished using different SiC disks (from P320 to P4000) to achieve a very smooth and flat surface. Also, to minimize the measurement uncertainty, 8 measurements were run for each sample; the mean value was then calculated and plotted in the presented figures.

Two points electrical conductivity measurements were made via a measurement cell made of two copper plates ($15 \times 26\ mm^2$) separated partially with 3 mm of PVC (Fig. 1). A DC 2000-scan Keithley multimeter has been used to measure the resistance R of the materials from which the conductivity σ was calculated using

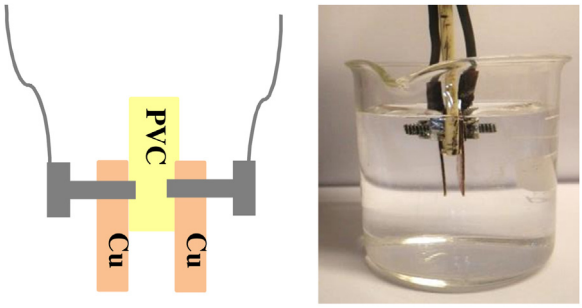

Fig. 1. (a) Layout and (b) photo of the conductivity cell used for the electric characterization.

Eqs. (1) and (2):

$$\rho = R * \frac{S}{e} \tag{1}$$

$$\sigma = \frac{1}{\rho} \tag{2}$$

with ρ: the resistivity of the mixture, S: the surface of the copper plates in contact with the mixture and e the space between the two plates.

3. Results and discussion

3.1. Morphological characterization

SEM pictures of the CB1 and CB2 powders are presented on Fig. 2. Fig. 2(a) shows that the CB1 carbon is constituted of particles having dimensions comprised between 5 and 10 μm while the CB2 carbon (Fig. 2(b)) presents agglomerates having dimensions which can reach several tens of micrometers. They seem to be constituted of numerous particles and pores with dimensions below the SEM resolution limit, i.e. below 1 μm. A specific surface area measurement, with the BET method, was done in parallel. The measurement shows that CB1 has a specific surface area of $10.1 \pm 0.2\ m^2/g$ whereas for CB2 it is equal to $226 \pm 6\ m^2/g$. This supports the SEM observation: the CB2 carbon has a higher specific surface area which means a finer structure (nanometric particles) than CB1 (micrometric particles).

Foams made with these two kinds of carbon are presented on Fig. 3. The ones obtained with CB2 (Fig. 3(b)) are darker than the ones obtained with CB1 (Fig. 3(a)). This is due to the difference between the specific surface areas of CB1 and CB2, the light being

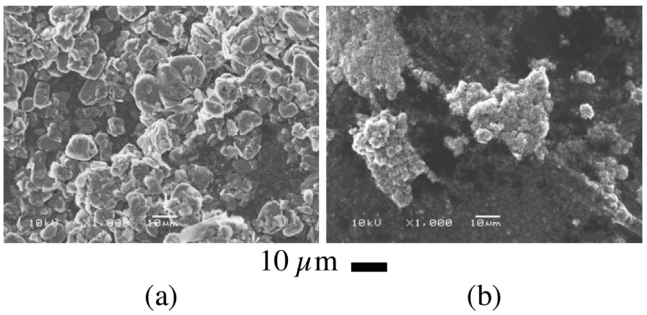

Fig. 2. SEM observations of (a) carbon graphite CB1 and (b) carbon black CB2.