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ELECTRICAL CONDUCTIVITY AND ELECTROMAGNETIC SHIELDING EFFECTIVENESS OF SILICONE RUBBER FILLED WITH FERRITE AND GRAPHITE POWDERS

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Abstract—There is increasing interest in electromagnetic interference (EMI) shielding due to the serious electromagnetic environment pollution caused by the continuously increased use of the electrical products and electronic devices. Electrical conductivity and EMI shielding effectiveness (SE) of composite materials made from silicone rubber with carbon powder and ferrite powder have been studied in microwaves and terahertz frequency ranges and the results are presented in this paper. In microwaves range, samples with higher electrical conductivity show a small variation of shielding performance with frequency, whereas the performance of samples with lower conductivity falls away with increasing frequency. It is shown that the variation of attenuation with frequency relates to the conductivity of the material.

1. INTRODUCTION

As the knowledge and technology progressed, the use of various types of electrical and electronic equipment in commercial, military, and scientific applications increased rapidly. These equipments are a source of electromagnetic radiation (EMR), which, depending on the needs, can be regarded either as a desirable or undesirable phenomenon. Thus, the problem of protection against electromagnetic radiation has a very important technical aspect concerning a reduction in the level of electromagnetic interference (EMI) that occurs between electronic instruments. Furthermore, an even more important aspect

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of protection against EMR is the health protection of persons present in the vicinity of equipment emitting EMR and exposed to its prolonged effects. This has increased the interest in developing effective materials for EMR shielding [1]. Electromagnetic shielding is used to confine electromagnetic energy within the bounds of a specific region and/or to prevent the propagation of such energy into a designated area. Depending on the interference due to any of forms of electromagnetic energy (radiated, static or time-varying electromagnetic force field), a variety of materials have been developed and used in the fabrication of shields. Shielding structures could be realized by using conductive and/or absorbing materials such as metallic conductor, polymeric composites with metallic and non metallic conducting inclusions, etc.. These materials must have specific requisites: first of all good shielding effectiveness (SE), lightness, good mechanical properties, good processability, and low cost [2].

It has been shown that promising shielding performance could be achieved with composite materials comprising an electrically non-conducting polymer matrix filled with conducting materials, such as stainless-steel, cooper, aluminum, nickel-coated carbon or neat carbon [3, 4]. Therefore, by increasing the conductivity of material a higher EMI shielding efficiency will be obtained [5, 6].

Silicone rubber is used today in a large variety of applications because it has low density, is easy to form, has good chemical resistance and is weather resistant. But, silicone rubber is electrically insulating and transparent to electromagnetic radiation. To achieve the desired absorbing properties, conductive fillers (metal powder, carbon black, graphite, ferrite, carbon nanotubes, ionic salts and others) are added into the silicone rubber matrix. Conductive silicone rubber can shield against electromagnetic interference and be applied more extensively in the field of electromagnetic compatibility [2, 7]. Conductivity may also be induced by adding conductive screen, plating, or paint to the finished product.

The conductivity of most metals is 1000 times greater than of carbon-based composites. The conductivity of a carbon-based composite material depends upon the current carrying capability and the amount of carbon. If additional conductivity is required for a specific application, conductive material may be added to the surface of the finished product or a layer of metallic material may be added as part of the laminate itself [8].

In this paper the electromagnetic interference shielding effectiveness (EMI SE) in the 1–18 GHz frequency range was studied on samples of ferrite and carbon-filled silicone rubber and conductive fabric. The paper also shows some results obtained by studying the same samples on terahertz frequencies, in the range 0.06–3 THz.

2. ELECTROMAGNETIC INTERFERENCE SHIELDING

EMI shielding involves three mechanisms: reflection, absorption and multiple-reflection [9, 10]. For a sheet of homogeneous conductive material (not a composite of a conductive filler and insulating matrix), reflection is the primary shielding mechanism. To shield by reflection, the material must have mobile charge carriers (electrons or holes) to interact with the incoming EM waves [11]. Absorption is the second important mechanism and it depends on the thickness of the shield. Shielding by absorption is enhanced when the shielding material has electrical or magnetic dipoles which interact with the EM waves. In conductive materials, absorption can also arise from resistive losses which consist in transforming the electromagnetic energy in heat by Joule effect. The third shielding mechanism is multiple-reflection. Typically, multiple-reflection decreases the overall shielding if the shield is thinner than the skin depth and can be ignored if the shield is thicker than the skin depth.

In polymer composites, shielding mechanisms are more complicated than those for homogeneous conductive materials because of the huge surface area available for reflection and multiple-reflection. The first reflection of an EM wave from a conductive material surface should be distinguished from the multiple-reflection mechanism which is the re-reflection of the waves already reflected [11].

The plane wave shielding theory developed by Schelkunoff [12] and Schultz et al. [13] defines the shielding effectiveness SE as

$$\mathrm{SE} = A + R + B, \tag{1}$$

where B is a term which take into account the loss caused by multiple-reflection inside the shield, R is the reflection loss, and A is the absorption loss. It is assumed that the shielding level from the composite is ultimately the same as the conductivity of an isotropic metal [14]. EMI shielding effectiveness (SE) is expressed in decibel (dB). A shielding effectiveness of 30 dB, corresponding to 99.9% attenuation of the EMI radiation, is considered an adequate level of shielding for many applications [15].

3. TERAHERTZ SIGNALS

"Terahertz (THz) fields" is a generic term for waves with a spectrum between 0.1 and 10 THz. THz fields have wavelengths extending from 3 mm up to 30 μm and wave numbers between 3 and 333 cm^{-1}; this wavelength interval ranges between the top edge of the millimeter wave spectrum to the bottom edge of the optical spectrum corresponding to the boundary of the far-infrared (FIR) spectral region.

The main two applications in which THz fields are involved are THz spectroscopy and THz imaging. These applications have contributed to a better knowledge of condensed matter, material properties and biology. The most common THz spectroscopy method is based on time domain techniques, which employ either photoconductive or electro-optical methods for emission and detection. The setup for THz time domain spectroscopy (TDS) in a transmission configuration is realized by placing a sample between the THz emitter and receiver, the THz radiation passing through it (Figure 1) [16].

In our work, we used THz spectroscopy to observe the behavior of our materials regarding the shielding effectiveness and electrical conductivity in THz domain.

4. EXPERIMENTAL

4.1. Samples Preparation

A two components silicon elastomer, which hardens at room temperature by a polycondensation reaction, was used as polymeric matrix. The uncured silicon elastomer has the aspect of a white viscous liquid and a density of 1.2 g/cm at 23°C and viscosity 35000 mPa·s at 23°C. After the addition of a catalyst, the silicon elastomer cures at room temperature and leads to a flexible and elastic material. To 100 parts of silicon elastomer, 5 parts of a catalyst with a demoulding time of 24 hours were added. Ferrite powder and graphite powder were

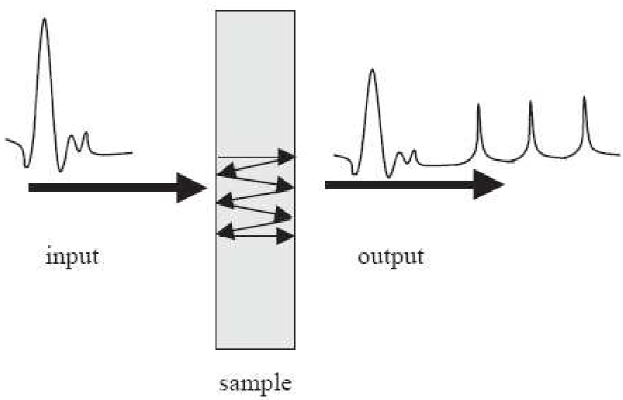

Figure 1. THz TDS spectroscopy.

used as fillers. The fillers were previously dried in an oven to eliminate hygroscopic humidity.

The preparation was performed at room temperature. The silicon rubber and filler were weighed and then mixed for 30 minutes for homogenization; next, the catalyst was added and mixed for another 15 minutes. The obtained composite material was stretched on a polyethylene foil using a technique similar with doctor blade technique. Doctor blade technique is a coating technique widely used for producing thin films on large area surfaces. Doctor blade is a flat knife blade that is held above the substrate onto which the material is to be deposited. A reservoir containing the material to be deposited is placed behind the blade so that the material can flow out through the gap between the blade bottom and the substrate. When a constant movement is established between the blade and the substrate, the material spreads on the substrate to form a sheet [17]. The sheet's thickness depends on the gap between the blade bottom and the substrate. On the top of the stretched material another polyetilene foil was placed and than pressed.

The composite materials were polymerized in open space at a temperature of approximative 22–25°C, for 24 hours.

Five samples, labeled PM, PMS, SR, SFU15, and SFUG20, were prepared for tests. The PM sample is a textile fabric made of polyester which is coated on one side with a thin metallic layer (Fe-Ni). The fabric is 300 μm thick, flexible and has open pores. PMS is a sandwich-type sample that contains a metallic fabric (same as PM) placed between two layers of silicone rubber (without filler), SFU15 was obtained by mixing 15 wt% ferrite powder with silicone rubber, and SFUG20 was obtained by mixing 20 wt% graphite powder + ferrite powder with silicone rubber. SR is a sample of silicon rubber which, in this work, was tested only in THz domain. All samples have the same size, i.e., $35 \times 35\,\mathrm{cm}^2$ and a thickness of 3 mm for PMS, SFU15, and SFUG whereas PM has a thickness of 300 μm.

4.2. Analysis Methods

The shielding effectiveness of the samples in the 1–18 GHz frequency range was determined using the method for large flat samples which implies the use of a shielded enclosure with an open window. This technique (described in IEEE-STD 299) involves irradiating a flat sample, mounted on the window of the shielded enclosure, with an electromagnetic wave over the frequency range of interest. Two measurements must be made for the shielding effectiveness assessment: the reference and load measurement. The reference measurement consists of measuring the test signal emitted by the transmitting

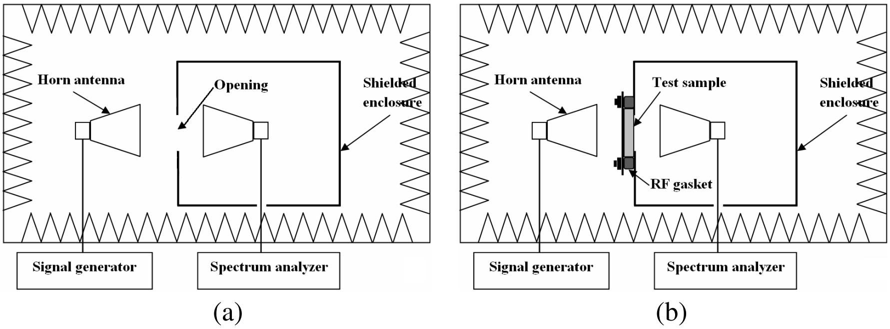

Figure 2. Experimental test setup used to determine shielding effectiveness. (a) Reference measurement (without sample) and (b) load measurement (with sample).

antenna through the shielded enclosure's opening without sample (Figure 2(a)). The load measurement is taken by transmitting the test signal through the opening with the sample placed over it (Figure 2(b)). During measurements, antennas will be kept in the same position.

The sample (whose dimensions are big enough to cover the window) is placed on the window using a metal plate with an aperture equal with the size of the window which is mounted over the sample using plastic screws to properly fix the sample in order to avoid the signal leakage between sample and wall. Shielding effectiveness is determined from equation

$$\mathrm{SE} = 20\log(E_1/E_2), \tag{2}$$

where E_1 is the field strength measured without sample and E_2 is the field strength measured with the sample covering the window. A similar equation can be derived for magnetic field shielding.

The test setup used to determine the shielding effectiveness is shown in Figure 2. The transmitting antenna and the shielded enclosure with the receiving antenna mounted inside it were placed in an anechoic chamber. Double-ridged horn antennas were used with a frequency range between 1 and 18 GHz. The transmitting and receiving antennas were connected to a signal generator respective to a spectrum analyzer (both placed outside the anechoic chamber) using coax cables type N. The frequency was scanned from 1 to 18 GHz and the field strength was measured at each frequency using the spectrum analyzer.

The samples were then studied in the 0.06–3 THz (2–100 cm^{-1}) frequency range using the TeraView TPS spectra 3000 terahertz transmission spectrometer. First, a reference spectrum was acquired with no sample mounted on the sample holder. After taking the reference spectrum, samples were mounted one by one on the sample holder and then measured. The sample compartment was purged during all measurements with dry nitrogen in order to remove the sharp lines in the spectra which are due to water vapor rotational lines. The samples measured in terahertz domain have the same thicknesses as the samples measured in the 1–18 GHz frequency range but smaller size ($3 \times 3\,\text{cm}^2$). Besides sample spectra, the spectrometer software can provide other quantities like transmittance, absorbance, real and imaginary dielectric constant etc.. The conductivity (in S/m) due to the alternating field is assessed using imaginary part of the dielectric constant obtained in terahertz frequency range using the following formula [18]:

$$\sigma_a = \omega\varepsilon'' \tag{3}$$

where σ_a is the conductivity due to the alternating field (S/m), ω is the angular frequency (rad/s), and ε'' is the imaginary part of the absolute permittivity.

5. RESULTS AND DISCUSSION

The electromagnetic shielding effectiveness for 1–18 GHz frequency range was determined using Equation (2) and the results are shown in Figure 3. PM sample presents an average electromagnetic shielding effectiveness of about 38 dB with the best efficiency for protection against electromagnetic radiation in the 10–16 GHz frequency range. Among the obtained samples characterized in the 1–18 GHz frequency range, the PM sample shows the best shielding effectiveness. The PMS sample follows the same graphic line, but with lower values, with an average shielding effectiveness of 33 dB. Polyester itself is not able to shield so we can say that the shielding effect shown by PM sample is due to the thin metallic layer deposited on one side of the fabric. Metals are the most common materials for shielding and function mainly by reflection due to the free electrons in them. The absorption loss is a function of the product $\sigma_r\mu_r$, whereas the reflection loss is a function of the ratio σ_r/μ_r, where σ_r is the electrical conductivity relative to copper and μ_r is the relative magnetic permeability. Elastomers can't shield either unless are coated with a conductor or filled with a conductive filler [9]. The lower shielding effectiveness showed by PMS compared with the one showed by PM suggests a decrease of sample's conductivity caused by silicon elastomer layers.

SFUG20 and SFU15 samples do not show a significant shielding effectiveness in this domain (Figure 3). In order to attain a high shielding effectiveness, highly conductive fillers are required (such as silver particles). Because fillers with high conductivity tend to be expensive, fillers with lower conductivity are used instead (like carbon

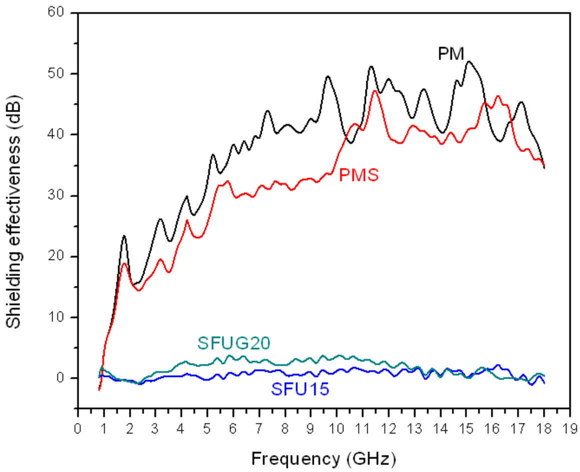

Figure 3. Shielding effectiveness in the 1–18 GHz range.

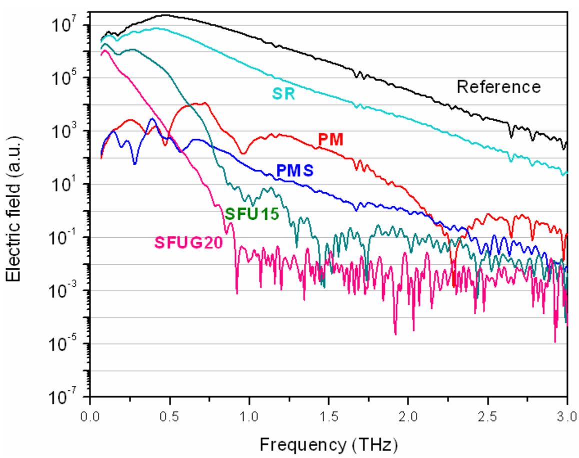

Figure 4. Reference spectrum and samples spectra in 0.06–3 THz ($2–100\,cm^{-1}$) range.

fibers). To achieve a high shielding effectiveness when using less conductive fillers, a large concentration of filler is needed [9]. Due to their high permeability, ferrites have the ability to absorb microwaves. By mixing ferrite powders with nonmagnetic polymers, absorption materials in microwave region can be achieved. According to the literature [19], permeability of such composite materials is mainly dependent on the ferrite volume concentration and less dependent of ferrite particle size. Therefore, we can consider that the poor shielding

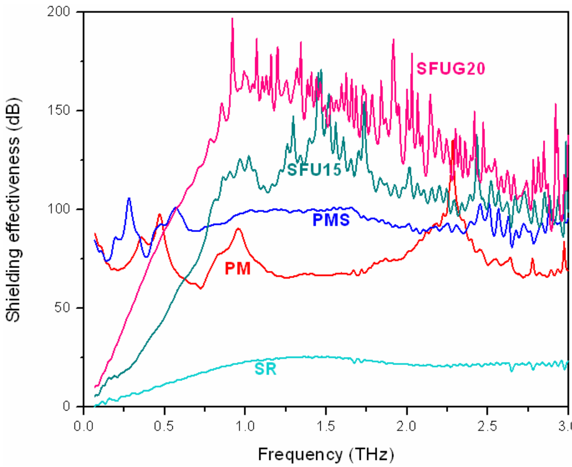

Figure 5. Shielding effectiveness in terahertz domain.

effectiveness showed by SFUG20 and SFU15 is due to the insufficient filler content.

To see the behavior of samples at higher frequencies, measurements were made in the range of 0.06–3 THz. The reference spectrum (measured without sample) and samples spectra were acquired (Figure 4) and the shielding effectiveness of the samples in this domain was calculated according to Equation (2) (Figure 5).

SFUG20 has the best shielding effectiveness and an optimal behavior in the 0.75–3 THz range with an average value on this domain of 140 dB. SFU15 follows the same graphic line but with lower values and an average shielding effectiveness on the same domain of 114 dB. PMS has a nearly constant shielding effectiveness on the 0.75–3 THz frequency range. PM has a lower shielding effectiveness in this frequency range with an average value of 75 dB. Still, this is a very good value. SR shows the lowest shielding effectiveness but with an average value of 20 dB between 0.7 THz and 3 THz.

Calculation of electrical conductivity in the 0.06–3 THz frequency range has been achieved using the Equation (3) and the results are shown in Figure 6. PM has a high conductivity with highest values between 0.06–0.5 THz and a flat zone between 1–2.25 THz. For the other samples (PMS, SR, SFUG20, and SFU15) electrical conductivity is much lower and almost constant between 0.75–3 THz with a higher average value for SFUG20.

Usually, the shielding effectiveness of shields with metal layers can be enhanced by increasing the physical thickness of the metal layer [20] thus increasing the conductivity. However, looking at Figures 5 and 6, it is observed that in the case of PMS sample which was obtained

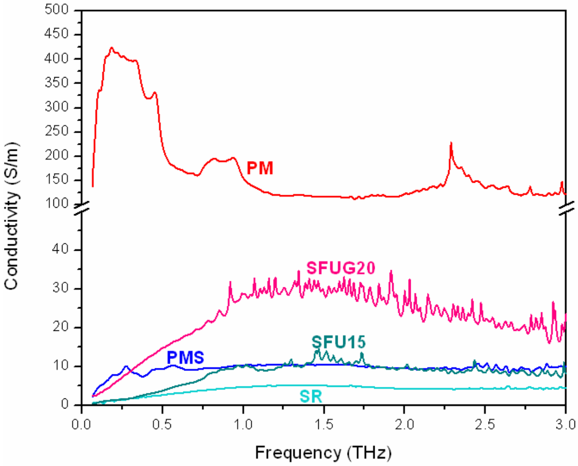

Figure 6. Samples conductivity in terahertz domain.

by adding silicon rubber (SR), which shows a very low conductivity, to the metalized fabric (PM), which shows a high conductivity, a higher shielding effectiveness was obtained although the conductivity decreased. This signalizes the shielding effect of silicon rubber in THz domain. The shielding effect showed by silicon rubber along with its low conductivity suggests that the main shielding mechanism for silicon rubber in THz domain is absorption.

The SFU15 and PMS samples show very close conductivity values in the 0.75–3 THz frequency range, but SFU15 shows a better shielding effectiveness than PMS. This indicates a positive influence of ferrite powder on both shielding and material's conductivity in this domain.

SFUG20 shows a higher conductivity and a better shielding effectiveness than SFU15 in THz domain indicating that the graphite powder enhances the shielding effectiveness by increasing the conductivity.

6. CONCLUSIONS

Five samples — PM, PMS, SR, SFU15, and SFUG20 — were used for the study of the electromagnetic shielding effectiveness and electrical conductivity in two different frequency ranges (1–18 GHz and 0.06–3.0 THz).

In the frequency range 1–18 GHz, the best shielding effectiveness was obtained for PM sample, with an average value of 38 dB. A close average value was obtained for PMS (33 dB).

In the terahertz domain, all samples, except SR, showed an adequate level of shielding for many applications. However, the silicon rubber (SR) also showed a good shielding effectiveness (20 dB) in this domain.

The obtained results regarding the electromagnetic shielding effectiveness of the samples show that for frequencies between 1–18 GHz, the optimal materials for use as electromagnetic shields are PM and PMS. The study of electromagnetic shielding effectiveness and electrical conductivity in the 0.06–3 THz frequency domain showed that the most efficient materials for shielding are SFUG20 and SFU15, and that the PM sample has the highest electrical conductivity. PMS contains metalized fabric (PM) and has a lower electrical conductivity, from where we can conclude that the silicon used to make the sample has a negative influence on electrical conductivity.

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