Influence of the Permittivity on Carbon Fiber Particulates Applied in Radiation Absorbing Materials

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Abstract

Carbonaceous materials are widely applied as materials that absorb electromagnetic radiation, whether in the form of carbon fibers, nanotubes and graphene. In this work the carbon fiber from raw material textile polyacrylonitrile was used in two distinct forms, felt and particulates. The carbon fiber felt samples showed real and imaginary permissiveness about four times higher than those in particulate form and also a reflection of up to 93

Index terms—

1 Introduction

he measure which electronic technology information by means unguided and use of electronic devices develop also growing problems with electromagnetic interference, making it a serious problem for communication between devices that communicate at the same frequency. The occurrences of interference leads to a malfunction of electronic devices [1,2,3]. In order to resolve these interference problems, many materials have been developed, so that a coating on the equipment to be used. These materials are known as radiation absorbed materials (RAM) which have ability to convert electromagnetic energy into heat. Basically RAMs are made of dielectric and or magnetic materials, that when processed conveniently promote high power loss in certain frequency bands. These materials have been used to solution the interference problem in most varied materials, such as covered, copper covered, polymer composites, carbon fiber, activated carbon fiber and deposit thin films [4,5,6].

Other areas which received great attention from the industries and academic research centers, due to their RAM applications in the most diverse areas are military, aeronautics, aerospace and telecommunications [7,8,9]. In the aeronautical and military areas the RAMs have been extensively studied in the frequency bands of 8 -12 GHz, known as X-Band [10,11]. Materials such as carbon, ceramic oxides, ferromagnetic and conductive polymers are traditionally applied to RAM and are thus used as centers for the absorption of unwanted radiation [12]. In particular, carbon is traditionally applied as RAM in the frequency range in GHz because it is an excellent reflector of electromagnetic radiation [13]. Therefore, many researches on this frequency were conducted with carbon in its different allotropic forms for the production of RAMs, whether in the form of activated carbon fibers [14], felt fabrics of carbon fiber in rectangular shape [15], particles dispersed in a matrix [16], cobalt oxide deposited [17], composite of activated carbon fiber with polymer [18], nickel particulates covered by carbon layer [19] and carbonaceous material in pyramidal form [20]. However, the use of these absorbers centers present as major disadvantages the weight and the volume occupied by the absorber final material. Hence, RAMs based carbon fibers, have been explored to improve these characteristics, as is known by its low density which facilitates applications in aerospace industry.

The RAMs are characterized by their ability to convert electromagnetic waves into thermal energy, so that the permittivity (?) and the magnetic permeability (µ) are parameters related to the electrical and magnetic properties of the material, which in turn are directly associated with the interaction of the wave with matter. When an electromagnetic wave propagates in a medium, the electric field of this wave polarizes the material. However, when a material is lossy, there is a delay between the electric field and the polarization of the medium, causing losses. The level of the losses depends on the difference between the phase electric field and polarization. These materials are classified into two types depending on the interaction with the wave. T Abstract-Carbonaceous materials are widely applied as materials that absorb electromagnetic radiation, whether in the form of carbon fibers, nanotubes and graphene. In this work the carbon fiber from raw material textile polyacrylonitrile was used in two distinct forms, felt and particulates. The carbon fiber felt samples showed real and imaginary permissiveness about four times higher than those in particulate form and also a reflection of up to 93% of the incident radiation. The study of the particulate fibers was carried out with particles of sizes smaller than 25um and 25-53um and embedded in an epoxy resin matrix in two concentrations of mass, 25% and 50% of carbon fiber. The best attenuation occurred for samples with particulate size 25-53um, where the concentration of 50% attenuated until 60% and the samples with 25% carbon fiber concentration until 75%. these characteristics are presented intrinsic properties of the materials, there are still features about the material geometry that also influence the attenuation of the wave such as: irregular features on the sample surface (example: pores), distribution of particulates and size of these particulates [21]. The way in which the matrix material is distributed in the samples improves the effect of wave absorption ??22. In the CF particles embedded in a matrix and CFF in case have different materials distributions which is directly related with geometrical characteristics of the material, producing a material that allows or not allows the wave penetration, causing different in side interaction with radiation incident. Others authors have been study the role of the carbon fiber concentration by a matrix of high and low concentrations, but not with reference made studies of the particulate size [23,24]. Thus, this study aims to understand the behavior of the electromagnetic wave in frequency range of 8.2-12.4GHz (x-band) for carbon fibers (CF) in different distributions in epoxy resin (ER). For this is first produced a carbon fibers felts (CFF) so that impede the wave of entry into the test body, and then the CFF will be pulverized and molded in epoxy resin with different concentrations of CFF and particulate size to modify the way in which the wave interacts with the particles.

2 II.

3 Materials and Methods

4 a) Production of Samples

In order to produce CF it was used textile PAN, due to its low cost compared with other raw materials for the production of CFs. The commercial 200 ktex tow of 5.0 dtex textile PAN fibers, was thermal oxidized in a laboratory scale oven set by, aiming the production of flame resistant fibers. The oxidation process was performed in two steps, the first at 200°C and the second at 300°C. The total time process were 50 minutes for each step. After that, the oxidized PAN produced used as a raw material to produce a CFF having 200 g/m². During the carbonization process, the oxidized PAN loses about 50% in mass and shrinks linearly about 10%. The shrinkage is an important parameter and must be controlled because an inadequate shrinkage result in poor mechanical characteristics and the fiber can't be handled. For this purpose, the CFF sample was cut into pieces of about 0.7 x 0.25m and placed in a special sample holder that can control the sample shrinkage in two dimensions.

The set was introduced in an electrical furnace. Both ends of the furnace tube were closed by flanges, which allow the insertion and the purge of processing gas to provide an atmosphere condition necessary for the carbonization and activation. The carbonization was performed in argon atmosphere at a final temperature of 1000°C by using a heating rate of 30°C/min. The process time at maximum temperature was set in 20 min to complete the carbonization process. After finishing the carbonization process, the furnace was turned off and maintained in Ar atmosphere. This condition of inert atmosphere was maintained until the room temperature inside the furnace reactor was reached.

5 b) Experimental Procedure

Different from granular or powdered carbon, CFs are composed of carbon filaments that may have different properties from other types of carbon materials due to the possibility of being transformed to fabric, felt or textile medium. The second stage is to powdering the CF before becoming felt, powdering it, and separates it into different particles sizes and embed them in epoxy resin (ER). The samples were separated in two particulates dimensions < 25um and in the range of 25-53um. Besides, two different mass fraction was studied, 25 and 50% of CF. The samples were produced with thickness of 1.5mm and dimensions of 10.22 x 22.70mm. The experiments were summarized in Table 1.

6 c) Electromagnetic Properties

The electromagnetic properties of the samples were studied through a waveguide technique in the frequency range 8.2-12.4 GHz. A rectangular waveguide (calibration kit WR-90 X11644A -Agilent) with a flexible cable 50? (85132F -Agilent) coupled to an analyzer Microwave PNA-G networks, model N5232A 20GHz was used to perform the reflection measurements and the scattering parameter (S parameter). The measured reflection gave information about the attenuation of the wave in the samples. The S parameter gives information about the material properties such as permittivity ? and permeability µ through the reflection coefficient (S 11) and transmission (S 21). The ? and µ we obtained from a specific software (85071E -Agilent), based on Nicolson Ross Weir (NWR) algorithms [25]. These values are essential to learn how the material reacts to electric and magnetic fields of the electromagnetic wave [26].

Also was possible to compare the experimental results of the reflection with transmission line model. Through of transmission line model the normalized input impedance ?? ???? of a metal-backed microwave-absorbing layer is given by [27]:Year 2017 F ?? ???? = ? µ ?? ?? ?? ?? ????????2?? ? µ ?? ?? ?? ð ??"ð ??"ð ??"ð ??"?**(1)**

Whereµ ?? and ?? ?? are the relative permeability and permittivity, respectively, of the composite medium, f is Year 2017 F the frequency of microwaves, and d is the thickness of the absorber. The reflection is related to ?? ???? as [27]. It is well known that electrical permittivity and magnetic permeability are parameters related to reflection and attenuation characteristics of electromagnetic wave absorbers. The real part ?' and µ' represents the energy storage capacity and the imaginary part of the complex ?" and µ" account for the energy loss dissipative mechanisms in the materials. In other word, the ?' is related with the material capacitance which is proportional to charge stored into the system under an applied electric field. The measurement of the ? and loss tangent dielectric? ??? ?? ? for a pure ER in frequency range of 8.2-12.4GHz is around 3.5 and 0.020. These results are in very good agreement with the results reported in the literature [28]. The low value of loss tangent dielectric indicates that the ER does not present a good dissipative property, therefore isa material that has not electromagnetic property enable to attenuate the electromagnetic wave at X-band. It is important to emphasize that the carbon fiber and epoxy resin are materials with exclusively electrical properties and then the real and imaginary permeability were not shown in this work.

The studies of the electromagnetic characterrization begin with the intrinsic properties and reflectivity of CFF impregnate in ER. According to Figure 2 the CFF present different results of particulate CF, because despite the material are the same, the form which the material are introduced in ER influence in the measurements. The CFF present ?' relative in range of 90-100, while the imaginary part have been a crescent behavior in frequency function of 50-90. It was also observed that through that the carbon in felt form are a good reflector, resulting in a reflectivity range of 85-90%. The fact that this sample exhibits a plate behavior is due to the carbon being known as a reflective material, and because it is in the form of felt it was also observed that corroborates with the reflection of the electromagnetic radiation [24]. The measures of the ?' and ?" of the composite based in ER and CF with different particles sizes and concentrations are shown in Figure 3.

7 III.

8 Results and Discussions

In Figure 1 (a) it shows the SEM image of the CF in the felt form with magnification of 1000x. CF filaments are distributed randomly throughout felt, thus not presenting a distribution in one direction. It is also worth noting that the textile PAN does not show cross section in cylindrical shape which can be seen in Figure 2 (b). According to the Figure 3, keeping the concentration of CF in 25% and varying only the size of the particles we can observe some situations: (I) an increase in ? 'from about 12 (<25um) to 23 (25-53um) and from 0.5 (<25um) to 1.8 (25-53um) in ? ". This increase is observed as a function of the increase of the particulate size from <25um to 25-53um. (II) For the 50% concentration was observed that the variation in ? 'was from 14 (<25um) to approximately 23 **??**25-53um). For ? "this variation from 0.5 (<25um) to on average 1.5 **??**25-53um).In summary, we can conclude that for both concentration of 25% and of 50% there was a significant increase in ??' due to the increase in the size of the particles. However, another way of analyzing these results is to fix the particle size at <25 um and observe the variation in ? '. In this way it is found that increasing the concentration from 25% to 50% favors an increase from about 12 (25%) to 14 (50%) in the ? ', but this linear increase in relation with concentration is not observed for particles size between 25-53um. The ? ' decrease from 23 (particulate size 25-53um) to 18 (particulate size <25um), and the same decreasing behavior is observed for ?". Same observations can be performed in relation this results. Firstly, the diminution the particulate size in both concentrations increase the transversal area of the CF with resin epoxy. Other observation is that the particulate dispersion in composite, because for low concentrations like that 25% and particulate size <25um the dispersion cannot produce a connections between the particulate CF. Then, the concentration of 50% have more probability to interact resulting in a particulate network. Figure 4 shows the schematic representation of the samples with different particulate concentrations and in the felt form. Is important highlight that in the Figure 3 (a) and (b) were observed some peaks in certain frequencies. Perhaps this peaks are associated with some process of absorbance by resonance due to the particulates presence. Besides, it was notice that the peak intensity and position are related with the concentration and particulates size. In order to investigate theses results was realized an fitting in the ?' and ?" through the classic Lorentz model. The real and imaginary permittivity in frequency function is showed in the equations below.?? ? (ð ??"ð ??") = ?? ?? + ? ?? ?? ð ??"ð ??"² ?? (ð ??"ð ??"² ???? ? ð ??"ð ??"²) (ð ??"ð ??"² ? ð ??"ð ??"² ????)² + ?² ?? ð ??"ð ??"² ?? (4) ?? ?? (ð ??"ð ??") = ? ?? ?? ð ??"ð ??"² ?? ? ?? ð ??"ð ??" (ð ??"ð ??"² ? ð ??"ð ??"² ????)² + ?² ?? ð ??"ð ??"² ?? (5)

Whereð ??"ð ??" ?? is denominated plasma frequency which is associate with the charge q, ð ??"ð ??" ?? is called resonance frequency, n is the number of difference resonance frequency contribute due to the different charges q in the system and is the damping constant ? [29]. Through these equations it was possible to perform the adjustment with the experimental data. Figure 5 shows an adjustment made for the 50% with particle size <25um sample using the sum of three equations. According to ours first conclusions, ours results are in good correspondence with the results obtained Dang at al [30]. The authors investigated the dielectric properties of

CF/polyethylene matrix composite. According to the authors there is a direct relationship between the dielectric constant with the increase of volume fraction of the CF for particle size distribution of approximately 100um. In order to explain the quantity of charge stored to justify the increase in the dielectric constant the authors concluded that the charge accumulated was related with the increase of CF/ER interfaces. In other words, by increasing the CF volume fraction the amount of interfaces increases causing an increase in the dielectric constant. According to our results there is an increase in the real part of permittivity when the CF volume fraction increases from composite with CF concentration of 25% and 50% for particle size (<25um). However, fixing the particle sizes in the range Year 2017 F of (25-53um) the permittivity decreases with the increase of CF volume fraction, which is not in agreement with the results proposed Dang at all. More recently, Hong et all investigated the dielectric properties the carbon fiber randomly distributed [31]. The authors reported that the higher the volume fraction of carbon fiber the higher is the permittivity. Besides, by increasing the fiber length the real and imaginary part of dielectric constant increases. By increasing the particles size the polarization effect is enhanced and smaller is the depolarization effect. The decrease in the ?' as reported in our work may be related with a limit for CF/ER concentration. This limit also may be associated with the lack of space to allow multi-reflection between the fibers to enhance the polarization.

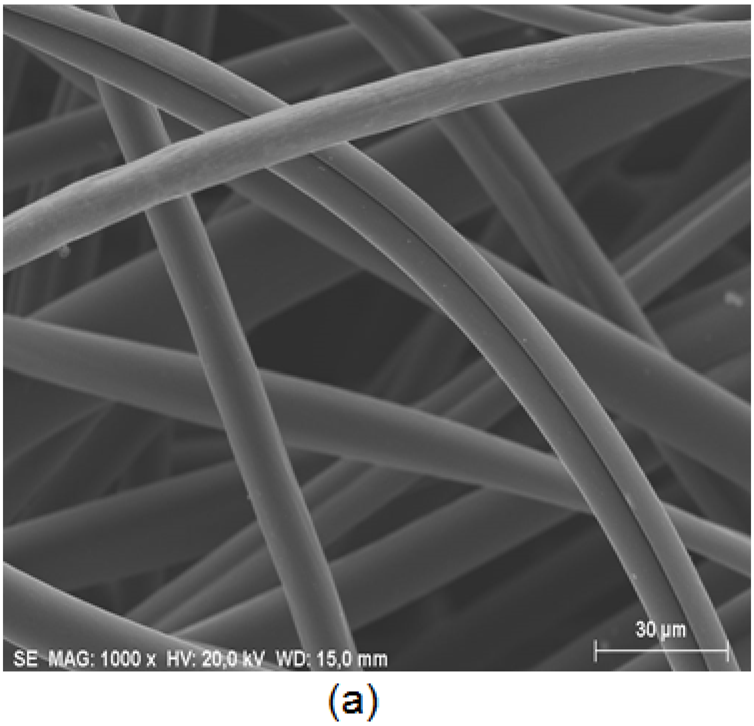
The results were summarized in the Table 2. In Figure 6 are showed the results of reflectivity measurement of the samples with metal-backed. According to Figure 6 for concentration from 25% to 50% CF for particulates size <25um not have significant changes in the reflectivity, both exhibited a decreasing behavior as a function of frequency resulting in a minimum reflection of 60% in approximately12.4GHz. However, for particulates size between 25-53um the concentration increase reveal significant change. For theses particulates size, the results showed that for larger particulate sizes with 25% CF concentration the attenuation was higher in the frequency of 10.18 and 10.88GHz than for 50% CF concentration. In frequency of 10.18GHz the reflectivity was approximately 10% and in 10.88GHz was 6.4%. For 50% CF concentration were observed three peaks; 8.83, 10.18 and 11.65GHz with their respective reflectivity32.5, 25 and 35.6%. Despite there are more peaks of attenuation in 50% CF concentration than 25%, there is a greater attenuation for samples with 25% CF concentration than 50%.According to the results it was possible to observer that the material is more sensitive to the variation of granulometry than concentration of CF, this for high CF concentrations. Using the permittivity and permeability of the samples in transmission line model for short load it was possible to estimate the reflectivity by the Equation (1) and (**??**). The results are showed in Figure 7 and they are good concordances with the measured results (Figure 6). However, it was observed a peak shift in the relation of the measured results. Huang et al [32 also use the model described in Equation (1) to compare the calculated and the measured reflectivity for nickel encapsulated carbon particles for frequency range of 2-18GHz. Huang highlighted some reasons that can be influenced in this discrepancy, as different characteristics of the elements used in the manufacture of the composite and even device instrumental limitation. In contrast, the model showed really effective when the experimental conditions are accurate, for example the uniformity and thickness of the samples. Thus, the electromagnetic wave in the frequency range of 8 -12.4GHz cannot fully penetrate the CFF. This is due to the large number of intertwined fibers in random directions, then occurring reflection to the source. However, minority, some of the signal can still penetrate the fiber occurring multiple reflections inside, these multiple reflections are gradually attenuating the signal by dielectric loss of CFF. Therefore, with the samples in particulate fixed by epoxy resin electromagnetic wave can penetrate the material and suffer attenuation by multiple reflections and other process like resonance.

IV.

9 Conclusion

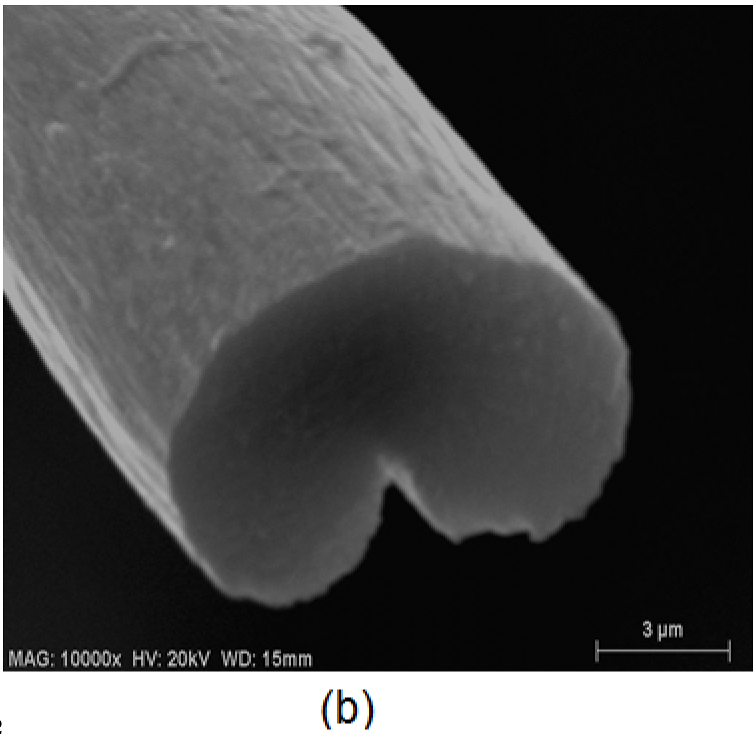
The study of how the carbon fiber is disposed in the resin, whether in permittivity the form of felted or particulate, showed a strong influence in the behavior of the permittivity and consequently in the attenuation of the electromagnetic wave. The carbon fiber in felt form presents properties of reflector material and it was not observe the attenuation peaks in the frequency range of 8.2-12.4GHz. According to the results it is possible to observe that the variation of particle size has different contributions in behavior of the permittivity and reflectivity. Too was observed in imaginary permittivity peaks give rise of some absorbance process. This peaks are more salient for samples with particulate 25-53um than <25um. The particulates size 25-53um and 25% carbon fiber concentration present peaks position approximately in 8.75, 10.85 and 11.65GHz and the samples with 25-53um with 50% present peaks in 9, 10.10 and 11.25GHz. Theses resonates peaks are contribute the reflective results. The best attenuation occurred for samples with particulate size 25-53um, where the concentration of 50% attenuated until 75% and the samples with 25% carbon fiber concentration until 93.6%. The transmission line model showed to be a good method to estimate the reflectivity since it has accuracy in the measurement of the thickness. Therefore, is a good technique to estimate thickness of the sample before of the production.

[1] [2]



1

Figure 1: Fig. 1 :



2

Figure 2: Fig. 2 :

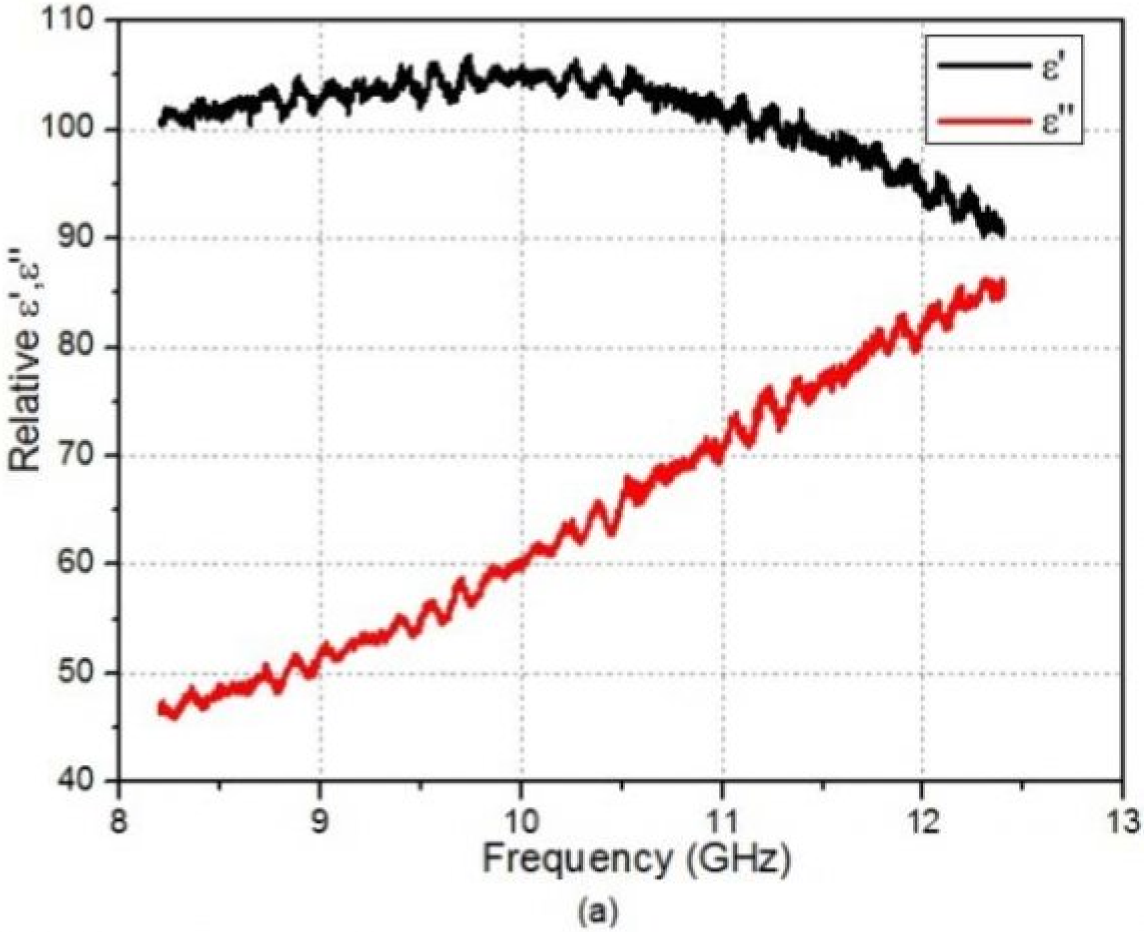


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Figure 3: Fig. 3 :

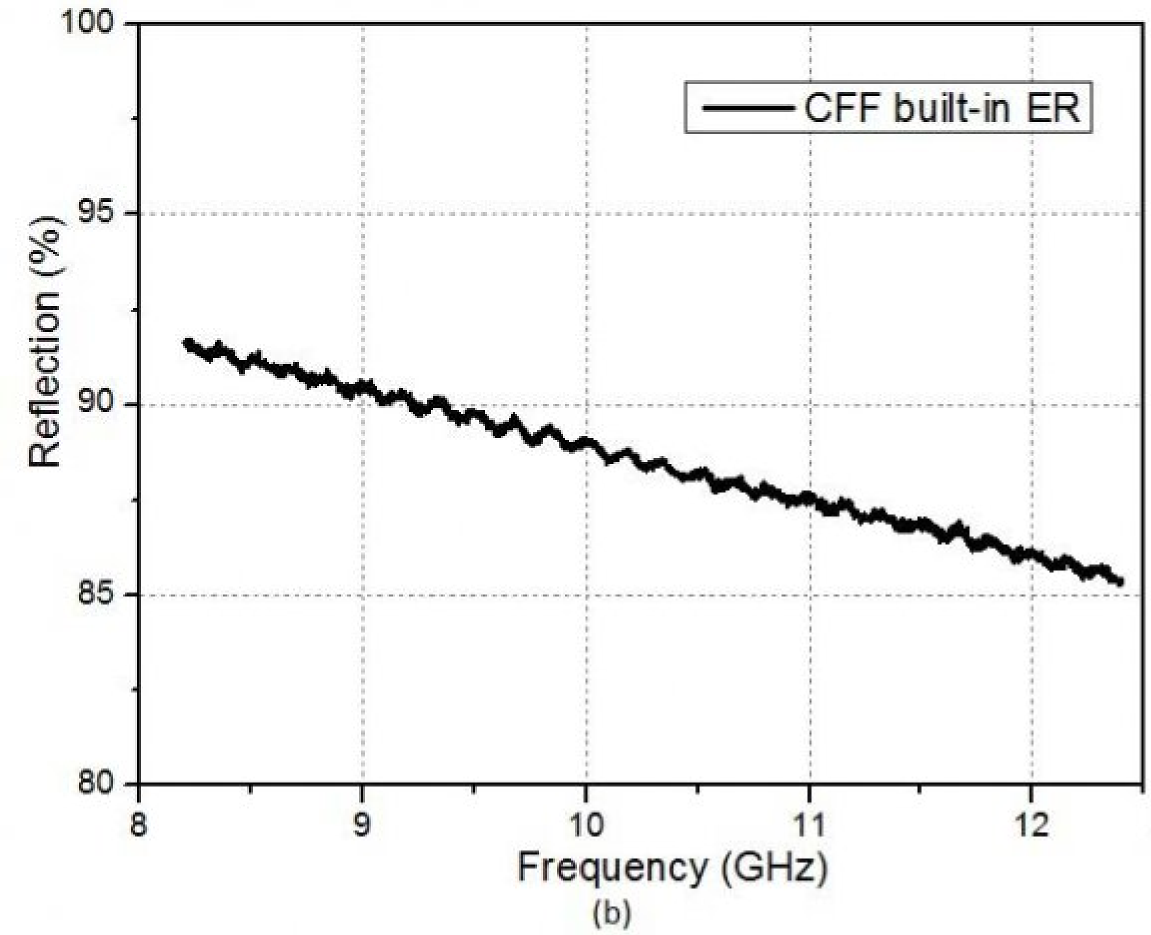

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Figure 4: Fig. 4 :

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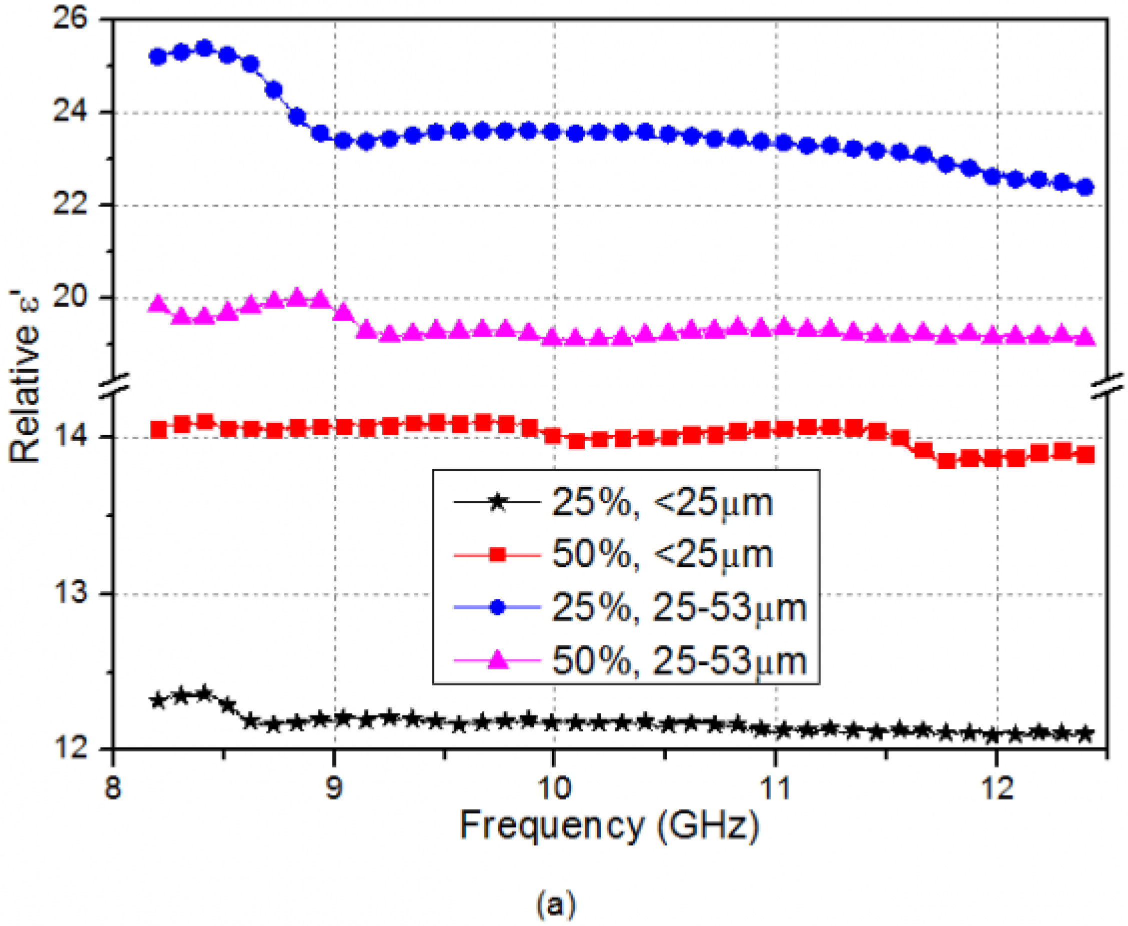

Figure 5: Fig. 5 :

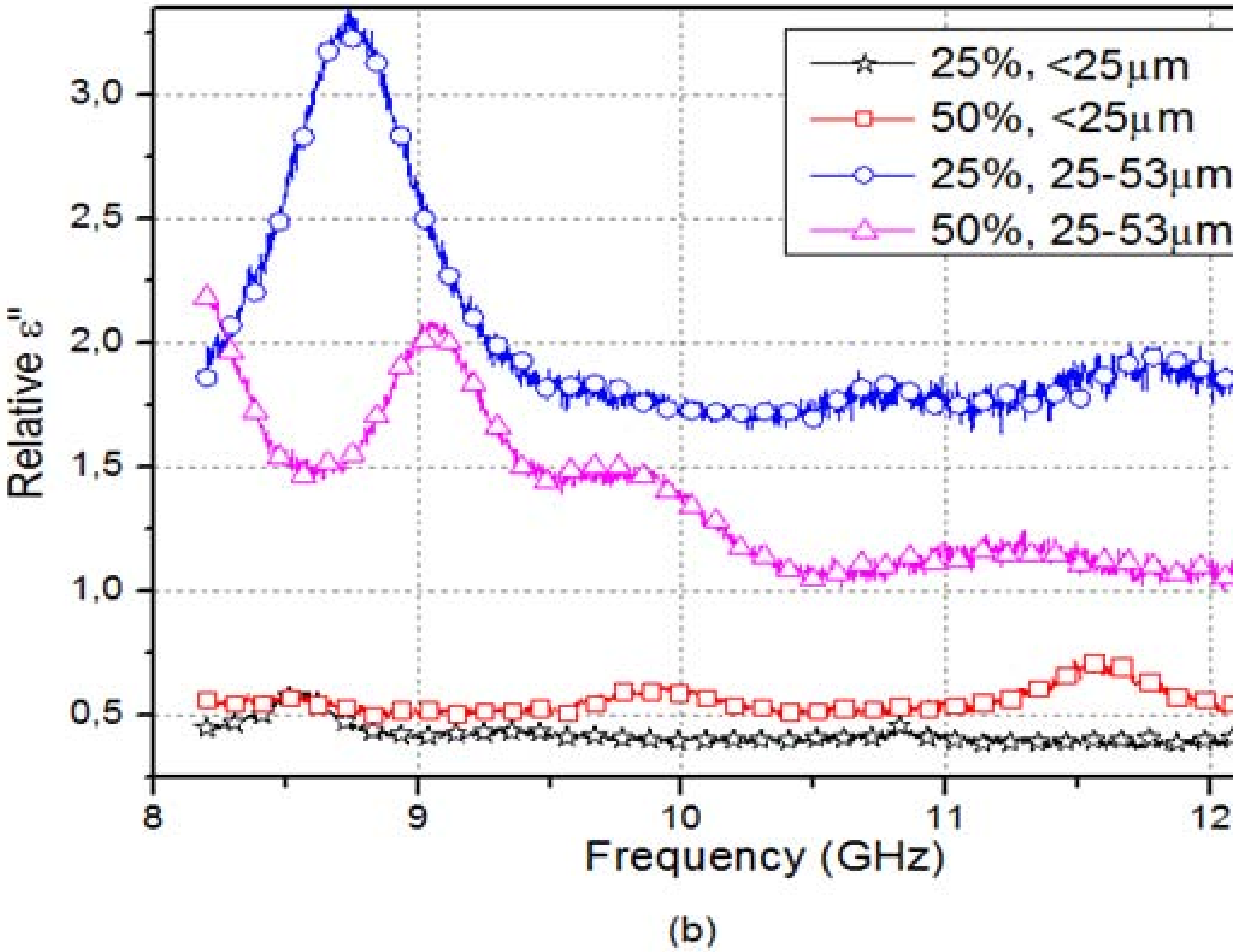

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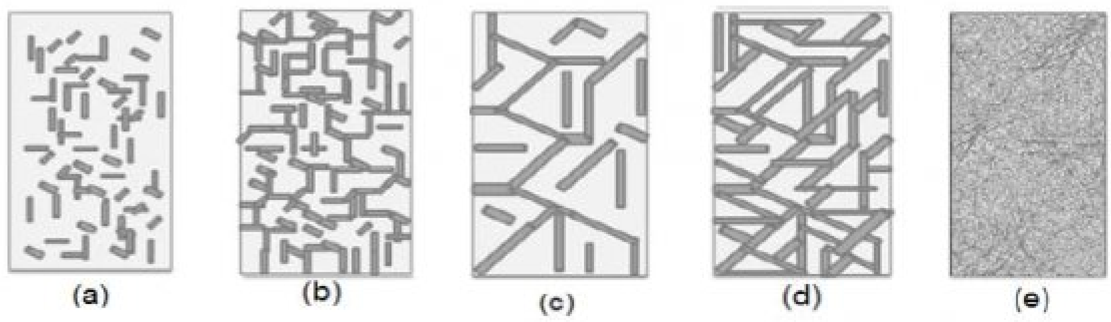
Figure 6: Fig. 6 :



7

Figure 7: Fig. 7 :

1

Samples	CF Concentration	Particulate Size
1	25%	<25um
2	25%	25-53um
3	50%	<25um
4	50%	25-53um

Figure 8: Table 1 :

2

CF Concentration	Particulate Size	?'	?"
25%	<25um	12.50	0.47
50%	<25um	14.00	0.50
25%	25-53um	24.00	2.00
50%	25-53um	20.00	1.50

Figure 9: Table 2 :

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