Ciência e Engenharia de Materiais

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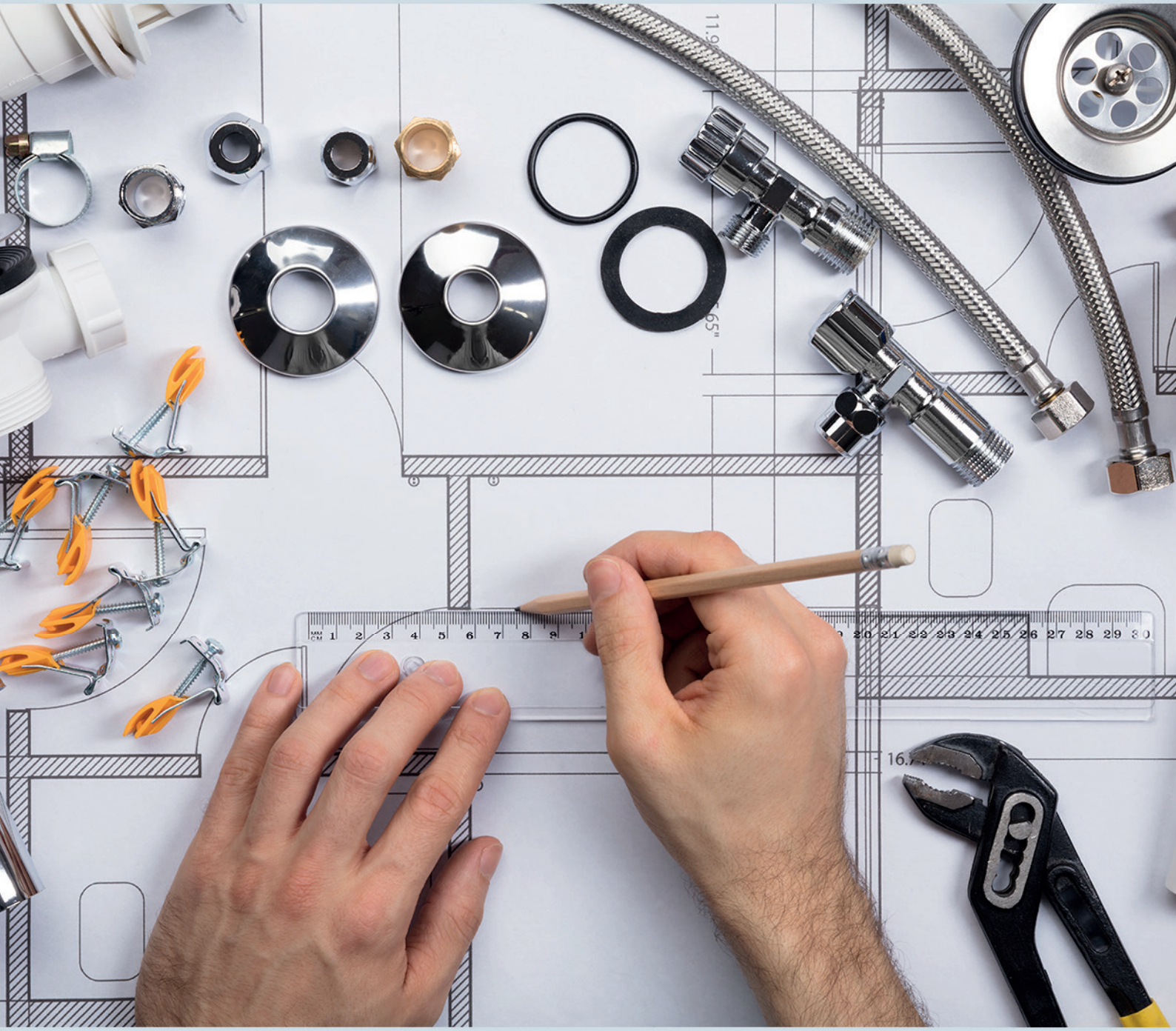
Marcia Regina Werner Schneider Abdala
(Organizadora)



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MARCIA REGINA WERNER SCHNEIDER ABDALA
(Organizadora)

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APRESENTAÇÃO

Você já percebeu a importância dos materiais na sua vida diária? Os materiais estão provavelmente mais imersos na nossa cultura do que a maioria de nós imagina. Diferentes segmentos como habitação, saúde, transportes, segurança, informação/comunicação, vestuário, entre outros, são influenciados em maior ou menor grau pelos materiais.

De fato a utilização dos materiais sempre foi tão importante que os períodos antigos eram denominados de acordo com os materiais utilizados pela sociedade primitiva, como a Idade da Pedra, Idade do Bronze, Idade do Ferro, etc.

A humanidade está em constante evolução, e os materiais não são exceções. Com o avanço da ciência e da tecnologia a cada dia surgem novos materiais com características específicas que permitem aplicações pormenorizadas e inovação nas mais diferentes áreas.

Todos os dias centenas de pesquisadores estão atentos ao desenvolvimento de novos materiais e ao aprimoramento dos existentes de forma a integrá-los em tecnologias de manufatura economicamente eficientes e ecologicamente seguras.

Estamos entrando em uma nova era caracterizada por novos materiais que podem tornar o futuro mais fácil, seguro e sustentável. O campo da Ciência e Engenharia de Materiais aplicada está seguindo por novos caminhos. A iminente escassez de recursos está exigindo inovações e ideias criativas.

Nesse sentido, este livro evidencia a importância da Ciência e Engenharia de Materiais, apresentando uma coletânea de trabalhos, composta por quatro volumes, que permitem conhecer mais profundamente os diferentes materiais, mediante um exame das relações entre a sua estrutura, as suas propriedades e o seu processamento.

Considerando que a utilização de materiais e os projetos de engenharia mudam continuamente e que o ritmo desta mudança se acelera, não há como prever os avanços de longo prazo nesta área. A busca por novos materiais prossegue continuamente...

Boa leitura!

Marcia Regina Werner Schneider Abdala

SUMÁRIO

CAPÍTULO 18

A NEW PROCEDURE TO DETERMINE THE PERMITTIVITY OF RADAR ABSORBING MATERIALS

Tamara Indrusiak Silva
Brazilian Army Technological Center, Av. das Américas, 28705 - Barra de Guaratiba, Rio de Janeiro - RJ, 23020-470,
tammy.indrusiak@gmail.com.

Iaci Miranda Pereira
Brazilian Army Technological Center, Av. das Américas, 28705 - Barra de Guaratiba, Rio de Janeiro - RJ, 23020-470.

Jorge A. Mitrione Souza
Pontifical Catholic University of Rio de Janeiro

Marbey Manhães Mosso
Pontifical Catholic University of Rio de Janeiro

Ângelo M. Leite Denadai
Federal University of Juiz de Fora, Campus Governador Valdares

Jéferson Gomes da Silva
Federal University of Juiz de Fora, Campus Governador Valdares

Alan Reis de Oliveira
Federal University of Juiz de Fora, Campus Governador Valdares

Bluma Guenther Soares
Federal University of Rio de Janeiro

ABSTRACT: Permittivity is an important electromagnetic (EM) property of materials used as radar absorbing material (RAM). Nowadays, there are many methods for measuring this property although they require very complicated test sets. Most of the techniques usually execute EM characterization of RAM with waveguides and vector network analyzer (VNA) that involves calibration procedures time consuming, complexity and are always frequency band limited according waveguide high pass and dominant mode operation. However, this combination holds certain complexities such errors or noise. Besides, waveguides, even at X band, are configured as large hollow metal pipes with very difficult assembling during measurements procedures. To reduce those complexities and problems, this paper proposes a new approach, using time domain reflectometry (TDR), for measuring the EM properties. The TDR evaluates the reflections in a transmission line and permits to analyze the properties that cause the reflection. Thus, in the present work, the scalar reflection loss techniques are used to characterize epoxy and epoxy/nanoferrite systems to be used as RAM. The epoxy/nanoferrite systems were produced by modifying the epoxy resin with nanoparticules of Fe_3O_4, $NiFe_2O_4$, $Ni_{0.5}Zn_{0.5}Fe_2O_4$ at concentrations of 0%, 10% and 20%. Results certify that TDR techniques can be used for EM characterization, being a less time consuming measurement and broadband measurement method.

KEYWORDS: radar absorber material, permeability, permittivity, vector network

analyzer, time domain reflectometer, iron oxides.

1 I INTRODUCTION

As mentioned before, permeability and permittivity are important electromagnetic properties of materials used as Radar Absorber Material – RAM (CULLITY; GRAHAM, 2009; DIAS; MARTIN; REZENDE, 2012). Nowadays, there are many methods for measuring those properties, which several times requires complicated test sets or limits the dynamic range (MORADI, 2007). Nevertheless, the majority of electromagnetic characterization of absorbing materials is executed using a diversity of waveguides or transmission lines and the parameters are measured by vector network analyzer (VNA) (COLLIER; SKINNER, 2007). However, this method holds certain complexities which may create errors or noise that are ignored by the researcher. For example, as many as 15 complex error terms can be found during the calibration of a two-port VNA, i.e.: wrong calibration between analyzer and waveguide through connecting cables, adapter and connectors (BARMUTA et al., 2014). Another problem is the accuracy required for sample holders and all waveguide sections. Therefore, the sample must be exactly the same size of those sections, reducing the characterization to a small number of samples (BROWN, 1946). As the manufacturing tolerance becomes a significant portion of the waveguide size, its dimensions restricts the frequency band (AGILENT TECHNOLOGIES, 2004; COLLIN, 1992) Thus, measurements at different frequency bands require different waveguides and calibrations, and, therefore, the quantification may become impossible. For instance, for lower frequencies, the waveguide dimensions become impractically large, and for higher frequencies the dimensions become impossibly small (AGILENT TECHNOLOGIES, 2004). In these situations, the RAMs are mainly characterized using calibration kit in the X-band (8.2-12.4 GHz).

Besides calibration, the quality and stability of the cables, adapters and connectors can affect the measurement (DUNSMORE, 2012) and may result in wrong calibration and inaccuracy. As highlighted at Handbook of Microwave Component Measurements (DUNSMORE, 2012), the first-order effect of cables add loss and mismatch in a measurement. For short cables, the loss is not significant but the mismatch can add directly to the source match and directivity of the VNA to degrade performance. With error correction, the effect of mismatch can be substantially reduced (to the level of the calibration standards quality) with cable stability, but since cable instability limits the repeatability of the cable mismatch and often is the dominant error in a return-loss measurement. Furthermore, when applying the VNA technique, the permittivity is obtained by Nicolson-Ross-Weir (NRW) algorithm based on ASTM D5568, that may become unstable signal when S11 results are close to zero (PAULA; REZENDE, 2011; PEREIRA, 2007).

To overcome the disadvantages associated to the VNA measurements in this work a new approach using microstrip line is being presented. The new method uses basic concepts of transmission line allied to a much more simple assembly and calibration.

2 I MATERIAL AND METHODS

2.1 Sample Preparation

Fe_3O_4, $NiFe_2O_4$, and $Ni_{0.5}Zn_{0.5}Fe_2O_4$ were prepared through the hydrothermal reaction of metal sulfates or mixture of them in a highly alkaline and oxidative environment ([NaOH] = 1.25 M and C_{H2O2} = 0.625 % v/v) at 120 °C during 2 h. The reactions were conducted in an automatized isothermal reactor containing a stainless metallic vessel of 1.2 L coupled with a collecting real-time data of T (°C), pH and pressure (kgf/cm^2). Following, the materials were washed and removed from aqueous environment by filtration. Then, they were incorporated in a commercial epoxy resin at proportion of 10% and 20% wt. Each composite resin was applied onto an aluminum plate with dimensions of 5 cm x 5 cm in order to obtain specimen with 1 mm of thickness. The curing time of resin was 24 h and a specimen containing pure epoxy resin was also prepared and used as reference.

2.2 TDR Method Theory

In Time Domain Reflectometry (TDR) approach, an incident pulse (quantified as voltage) is propagated into a transmission line in order to detect the reflections (PACKARD, 1969). Using this approach, a directional coupler and a sampling gate linked to an oscilloscope, can describe, in the time domain, the incident and reflected signals. The amplitudes of both signals yield the desired information. As the excitation signal is a real pulse, the signals are called real pulse TDR (MORADI, 2007). The variation (peaks) of the observed reflected signal indicates the physical location of the impedance or reflection coefficient discontinuity (PACKARD, 1969). Since the method detects the discontinuity, it is possible to analyze the EM properties of the material that causes the reflection (LIHUA et al., 2003), allowing to study how the input voltage step is affected by the presence of the sample in which it travels (MERIEM et al., 2010). The measuring platform consists of a terminated transmission line with a 50 Ω load.

The first task is to measure a terminated microstrip transmission line supported only by the connectors. This procedure corresponds to use the free space as a substrate ($\mathcal{E} = \mathcal{E}_0$). The TDR screen, displaying the curve Reflection Coefficient versus distance indicates the 50Ω characteristic impedance of the line. In the second task, the sample is introduced between the microstrip line and ground plane and the plot Reflection coefficient versus distance indicates the characteristic impedance of the line with the sample as a substrate.

Figure 1 shows the measuring platform of the TDR operation system.

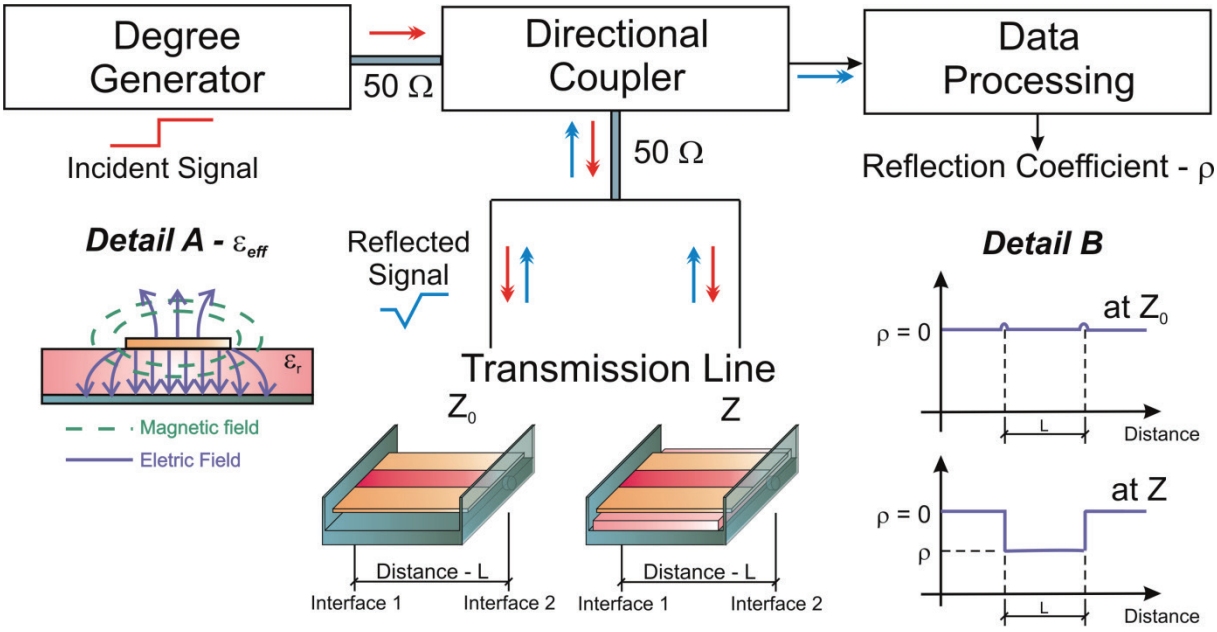

Figure 1- Scheme of test setup typical of a time domain reflectometer and geometry of a microstrip line (COLLIER; SKINNER, 2007).

The two tasks indicated above can be better explaining assuming that when a step voltage signal is introduced in a transmission line configuration, the signal propagates until an impedance discontinuity produces a reflection signal. This reflection signal is detected and sampled by a directional coupler.

The pulse propagates toward the microstrip line, whose characteristic impedance (Z_0) is designed in such a way as to yield a similar impedance of the genarator (MATTEI et al., 2008). The TDR screen shows the discontinuity according the plot at Figure 1 – Detail B – indicating the reflection coefficient versus distance, presenting the new impedance level produced by the discontinuity.

The reflection coefficients (is defined according Eq. (A) (ANTONOVICI, 2015; COLLIN, 1992; DASCHER, 1996).

$$\rho^2 = \frac{E_r}{E_i} \qquad \text{(A)}$$

Basically, the TDR measurements are described in terms of this coefficient.

By measuring the ρ of the air assembled Z_0= 50Ω line. When $\rho \leq 0.05$ the sample is introduced under the line, consequently, the discontinuity changes accordingly ρ is different from zero and a new transmission line impedance (Z) is obtained, according Eq. (B).

$$Z = \left(\frac{1-\rho}{1+\rho}\right) \cdot Z_0 \quad \text{(B)}$$

It is well know that in free space the Z_0 is described by Eq. (C).

$$Z_0 = \sqrt{L_o / C_o} \quad \text{(C)}$$

where L_0 and C_0 are, respectively, inductance and capacitance per unity of length in free space.

Therefore, the new capacitance of the line, enclosing the sample, is described by Eq.(D).

$$Z = \sqrt{\frac{L_0}{C_0 \varepsilon_{eff}}} \quad \text{(D)}$$

Finally, the method described above determines effective permittivity ($\mathcal{E}_{eff}$), Eq. (E).

$$\sqrt{\varepsilon_{eff}} = \frac{Z_o}{Z} \quad \text{(E)}$$

The permittivity is measured as effective because a portion of the fields is in air above the substrate (Figure 1 – Detail A) and the other portion permeates the material. $\mathcal{E}_{eff}$ is a function of the material permittivity, the microstrip line width (W) and microstrip thickness (h). Therefore, the $\mathcal{E}_{eff}$ is lower than the substrate's permittivity (COLLIN, 1992). Once is calculated, the dielectric constant of the material ($\mathcal{E}_r$) can be obtained considering the dimension of the microstrip using a Schneider's Equation(SCHNEIDER, 1969), Eq.(F).

$$\varepsilon_{eff} = \frac{\varepsilon_r + 1}{2} + \frac{\varepsilon_r - 1}{2}\left(\frac{1}{F}\right) \quad \text{(F)}$$

where F is obtained according to Eq. (F)

$$F(w,h) = \sqrt{1 + 10\frac{h}{w}} \quad \text{(G)}$$

2.3 TDR Experimental Setup

The setup for TDR system consisted in a HP Model 1815B oscilloscope connected to a HP Model 1817A remote sampler via a HP 1106 tunnel diode pulse generator that produce the EM pulse. The arrangement provides the input parameter necessary for

the operation system, illustrated in Figure 1. The tunnel diode generated a pulse with rise time of 20 ps and 250 mV. A 50 Ω connector is used to join the TDR to the microstrip line through another low low-loss coaxial cable. Finally, a 50 Ω load is connected at the end of the microstrip line. The system is built to obtain Z_0 50 Ω. When the specimen is placed in the microstrip line it produces a change in the initial waveform pulse. The change is due to the reflected pulse and is employed to measure ρ and determine Z as describe in Eq. (B).

2.4 VNA Experimental Setup

To compare the TDR dielectric measurements, VNA dielectric measurements were also carried out. A two port waveguide transmission/reflection method has been employed for measurement of complex permittivity values of studied materials. The measurement set up consisted of a VNA Model Agilent N5230C PNA – L series, a X-band waveguide calibration kit, a sample holder, and a wave guide. The VNA was connected in the rectangular waveguides to measure the scattering parameters (S_{11} and S_{21}) in X-band (8.2-12.4 GHz). The rectangular samples of 0.85 mm were inserted into rectangular sample holder which matches the internal dimensions of X-band waveguide (25×13mm). The sample holder was placed between the flanges of the rectangular waveguide, connected to the two ports VNA. It was present results based on S_{11} parameter and impedance and reflection coefficient from Smith Chart. Additionally, the ε_r was calculated from scattering parameters (ASTM D5568-08) using the Agilent 85071E material measurement software.

3 I RESULTS

3.5 TDR Results

The results of permittivity from TDR technique is presented at Figure 2.

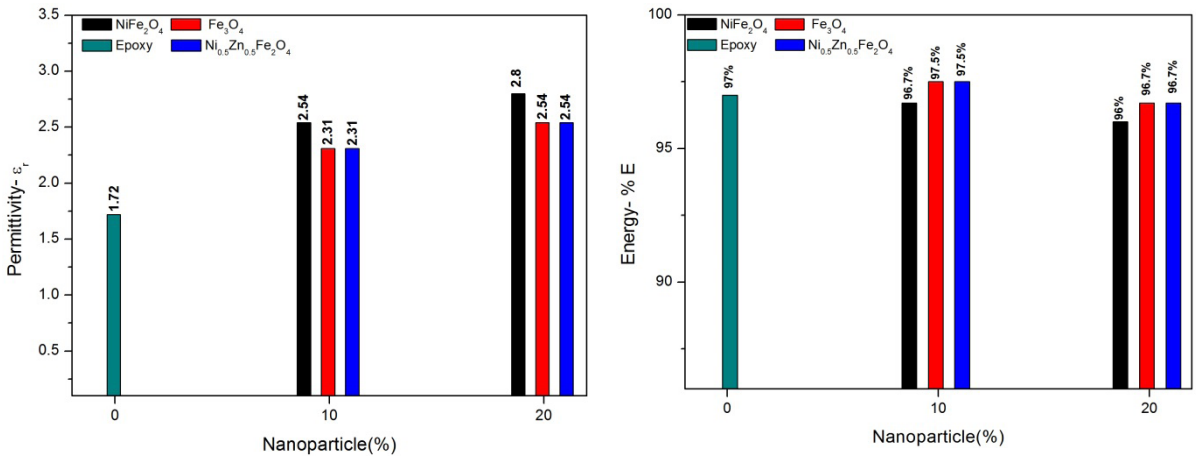

Figure 2- Results from TDR technique: (a) permittivity and (b) %E.

It's possible to see that the average permittivity of the samples is around 2.5. This variation is due to the type and concentration of nanoparticles. In **Erro! Fonte de referência não encontrada.**(b), we can see the %E calculated from reflection coefficient. It's observed that for all samples the reflection is low, and 96%, i.e., the reflected energy is around 4%.

3.6 VNA Results

The permittivity VNA results are presented in Figure 3 as a function of frequency X-Band (8.2-12.4GHz) of $NiFe_2O_4$, Fe_3O_4 and $Ni_{0.5}Zn_{0.5}Fe_2O_4$ samples.

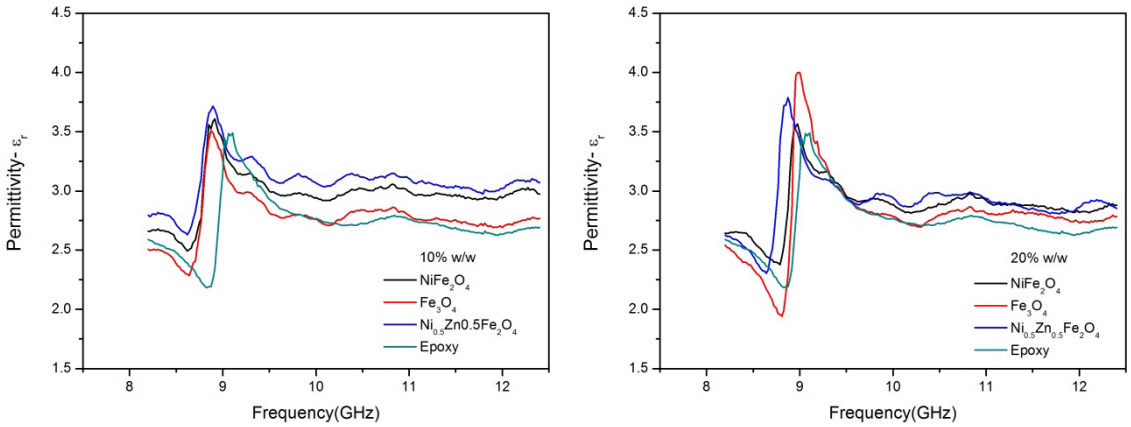

Figure 3-Permittivity VNA Results: (a) 10% w/w samples and (b) 20% w/w samples.

In Figure 3, for all samples, ε is almost constant from 9 to 12 GHz with $\mathcal{E}_r \approx 3.0$. It is observed a well-defined resonance peak presented around 8.8 GHz. At higher concentration, the peak shifted to higher frequency. At low concentration, the $\mathcal{E}_r$ of $Ni_{0.5}Zn_{0.5}Fe_20_4$ samples is slightly bigger than $NiFe_2O_4$ or Fe_3O_4. As the nanoparticle concentration increases, the sample behaviors, from 9 to 12 GHz, changes and the $\mathcal{E}_r$ of $Ni_{0.5}Zn_{0.5}Fe_20_4$ decreases to $\mathcal{E}_r \approx 2.4$.

Nevertheless, at the resonance peak increases to $\mathcal{E}_r \approx 4.0$. The energy lost (%E) is presented in **Erro! Fonte de referência não encontrada.**. %E $\mathcal{E}_r \approx$ represents the $\mathcal{E}_r$ plus the absorbed energy.

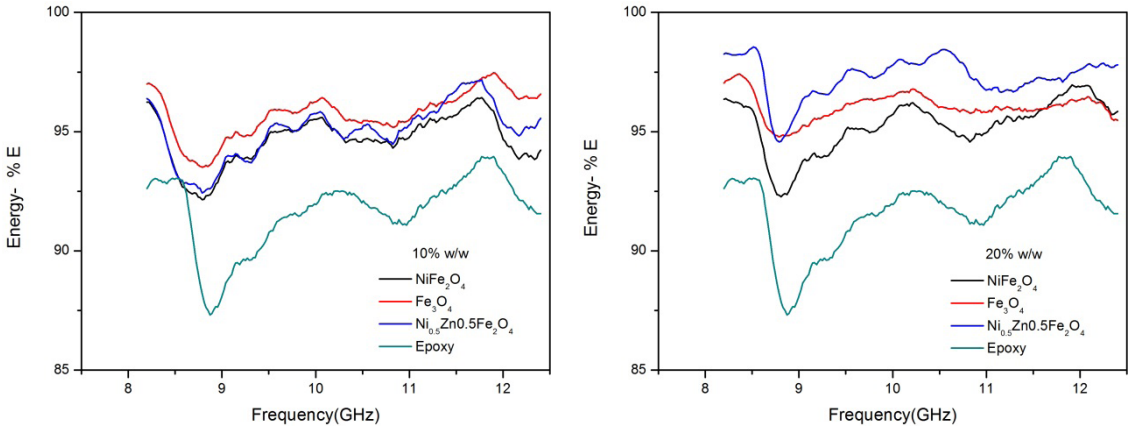

Figure 4-%E Results: (a) 10% w/w samples and (b) 20% w/w samples.

In Figure 4 is shown the $\%E \approx$ 96% for all nanoparticles. However, at 8.8 GHZ is observed the $\%E$ minimum around 92%. It was observed that as more particles are added to the epoxy, the comportment of $\mathcal{E}_r$ and %E changes, indicating the influence nanoparticles concentration on EM properties. $Ni_{0.5}Zn_{0.5}Fe_20_4$ 20% presents the best results. In Figure 3(b), the %E of nanocomposites is around 96% for Epoxy is 92% . At the resonance peak at Figure 3(b), the %E minimum is observed for nanocomposites between 92% and 94% and 87% for epoxy. As observe, the $\mathcal{E}_r$ maximum is the %E is minimum.

3.7 Results Discussion

The permittivity values measured using the VNA are ranging between 1.5 and 4 and the resulted obtained applying the TDR is around 2.5. The difference is due to the measurement mode. VNA acquires the results in a specific frequency range (from 8.2 to 12.4 GHz) in the frequency domain. The TDR measures broadband frequency in the time domain. The obtained values agree quite well with literature (HIPPEL, 1954). However, the NRW algorithm that calculated the $\mathcal{E}_r$ for VNA technique has some limitations, i.e., calibration. Due to setup and procedures, calibration technique requires data-based standards with known electrical performance and its results must be accurate to avoid errors and incoherent results. Otherwise, the acquired results are inexact (COLLIER; SKINNER, 2007).

Compared to the TDR method, calibration requires considerable time to be executed. Besides calibration errors, during the measurements, VNA setup might cause multiple reflections on the samples, reducing the accuracy. The multiple reflections occurs due to the thickness of the samples, when its thickness is less or equal to the half of incident wavelength (KIM et al., 2014; LUUKKONEN et al., 2011; PAULA; REZENDE, 2011).

When compared with conventional methods, the TDR technique might be more

efficient and precise because the pulse/step generator does not need calibration, the measurements are faster and more straightforward. Moreover, the pulse generator can eliminates the multiple reflections mismatch error (NICOLSON; ROSS, 1970)

Therefore, TDR systems is suggested to improve the accuracy of the ε_r through the ρ and it is able to evaluate quickly whether the material is an efficient absorber or not.

4 I CONCLUSION

For the permittivity and loss tangent measurements, a new approach using microstrip line was presented. Allied to an easier assembly and a straightforward calibration method, the obtained technique uses basic concepts of transmission line and time domain reflectometry. To illustrate the simplicity of the new approach, the permittivity and loss tangent also was acquired using the VNA method, which is a common time consuming measurement that demonstrates certain complexities as calibration. Comparing the results, there were no significant differences. Therefore, the time domain reflectometry method is potential tool for acquiring the electromagnetic properties of radar absorbing materials.

5 I ACKNOWLEDGEMENT

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SOBRE A ORGANIZADORA:

Marcia Regina Werner Schneider Abdala: Mestre em Engenharia de Materiais pela Universidade Federal do Rio de Janeiro, Graduada em Engenharia de Materiais pela Universidade Federal do Rio de Janeiro. Possui experiência na área de Educação a mais de 06 anos, atuando na área de gestão acadêmica como coordenadora de curso de Engenharia e Tecnologia. Das diferentes atividades desenvolvidas destaca-se a atuação como professora de ensino superior atuando em várias áreas de graduações; professora de pós-graduação *lato sensu*; avaliadora de artigos e projetos; revisora de revistas científicas; membro de bancas examinadoras de trabalhos de conclusão de cursos de graduação. Atuou como inspetora de Aviação Civil, nas áreas de infraestrutura aeroportuária e segurança operacional em uma instituição federal.

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