Investigation of the Microwave Dielectric Properties of Cu based Nanocomposites

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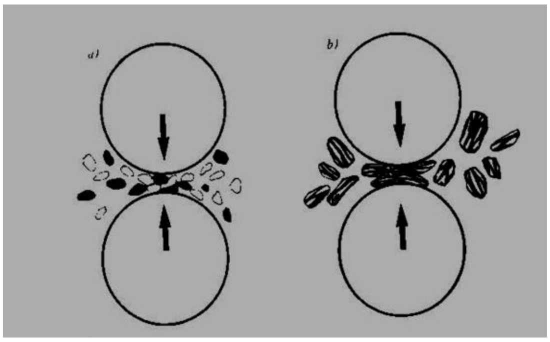
- **Purpose**

An EMI shielding material is a material able to restrict radiated or conducted electromagnetic energy over a wide frequency range. Nowadays, materials engineering suggests the use of nanostructured materials to obtain successful solutions because they follow the requirements of lightweight, ease processing and effectiveness. Polymer nanocomposites are a class of nanostructured materials adopted as an issue for the remove of electromagnetic interferences. Throughout this work, we looked over the structural and the microwave dielectric properties of skinny slabs of epoxy resin reinforced by nanocristalline Cu powders. By nanocrystalline powders, we mean materials with grain size below 100 nm. In such materials, the critical microstructural feature that determines properties and performance is the grain size. The grain size, in turn, depends upon the processing method used. The choice of nanocrystalline Cu powders is justified by the availability of copper powder and its skin-deep characteristic when exposed to microwaves.

- **Experimental Section**

Nanocrystalline Cu powders were obtained by mechanical milling. It is a severe plastic deformation technique, which allow metal powders to be nanostructured by repeated cold-welding, fracturing and re-welding in high- energy milling process. This process can induce material defects (dislocations and/or two-dimensional defects) that lead to the grain size refinement and packages of nanodefects. High-energy mechanical milling was accomplished using a planetary ball mill (RETSCH PM400). To carry out the experience, a milling velocity of 200 rpm were respected, 18 hardened steel balls were loaded with 10g of elemental Cu powder in a vial and all handling were performed in a glove compartment under protective atmosphere of Argon. Small samples of powder were retained after 3 h, 12 h, 33 h and 58 h milling durations for structural and microwave characterization. Obtained powders were subject to an X-Ray Diffraction (XRD) analysis using an X'PERT PRO MPD PANalytical diffractmeter in order to investigate their structural properties after milling. Grain size refinement, lattice parameter changes and accumulated defects were explored in the range of [10^o-90^o] Bragg angles with a step of 0.026^o using CuKα radiation.

For microwave characterization, thin slabs of 1 mm thickness were shaped by solid solution dispersion technique. The bulk samples were subject to an experiment of two-port S parameters measurement in a rectangular waveguide (R120) in association with a vectorial network analyser (Keysight Technologies N5222A).



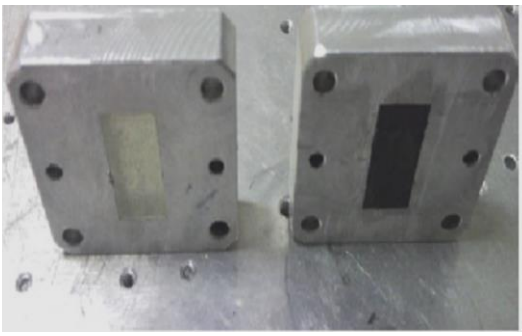


Fig.1. Fracture- welding mechanism during powders milling.

Fig.2. Nanocomposite sample within Rectangular Waveguide.

- **Results and Discussion Section**

  1. Structural Characterization

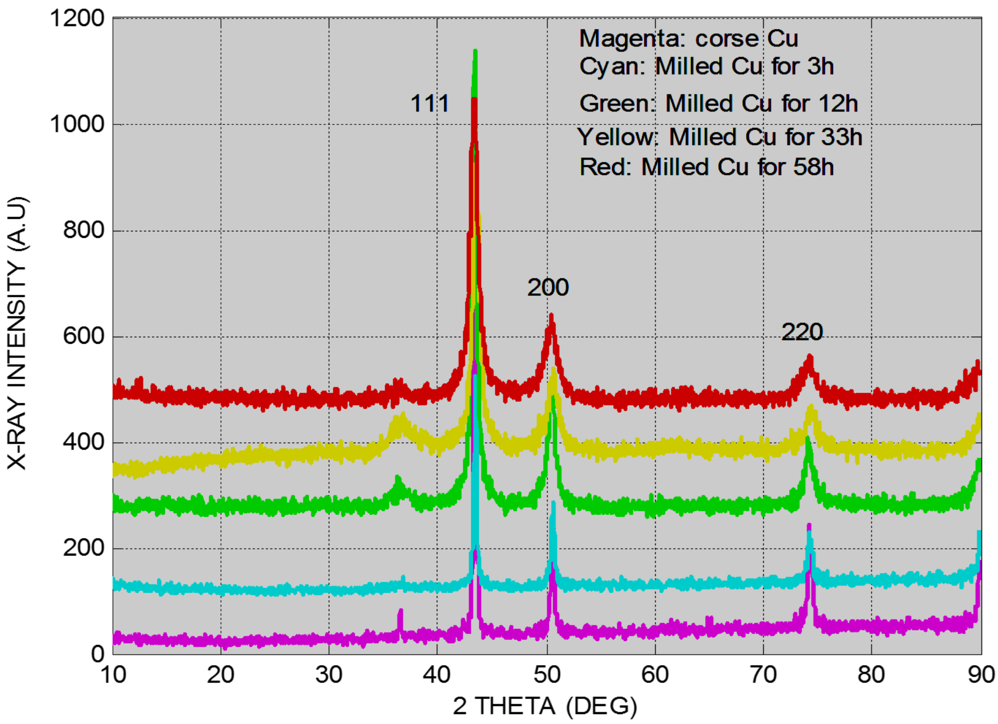


Fig.3. XRD Spectra of coarse and milled Cu powders.

Coarse Cu powder shows XRD peaks at 43.462°, 50.549°, 74.321° corresponding to the planes (111), (200) and (220) in the fcc crystalline structure of Cu. XRD spectra analysis is conducted related to the observed changes surrounding the named peaks after milling. The Cu peaks remain consistent after milling process. XRD lines move slightly and widen as the milling process continues. Additionally, their intensity decreases indicating that milling releases little to no energy, providing evidence that fcc crystalline structure remains intact.

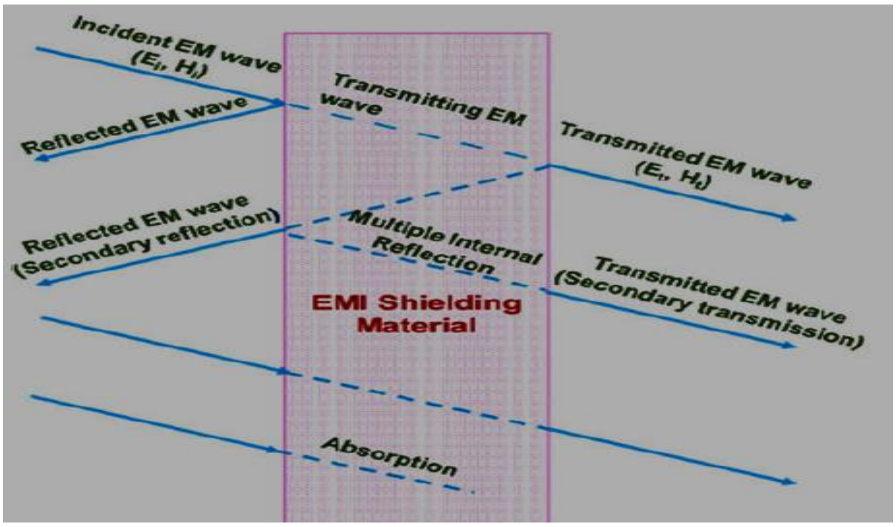
According to Williamsons Hall method, we calculate the structural parameters in terms of average grain size D and residual defects fraction ε for the coarse and the milled Cu powders:

$$D = \frac{K\lambda}{\beta_{hkl} cos\theta} \qquad \varepsilon = \frac{\beta_{hkl}}{4tan\theta}$$

With K is a shape factor, λ is the wave length of the diffractmeter's radiation, θ is the half Bragg angle, and β_{hkl}: Full width at half maximum (FWHM). We can deduce that the reduction of the grain size is rewarded by the increase of defects fraction, which allow to a disordered cubic structure of the Cu crystalline structure after milling. The enlargement of the XRD peaks in Fig.3 also proves this.

2. Microwave Characterization

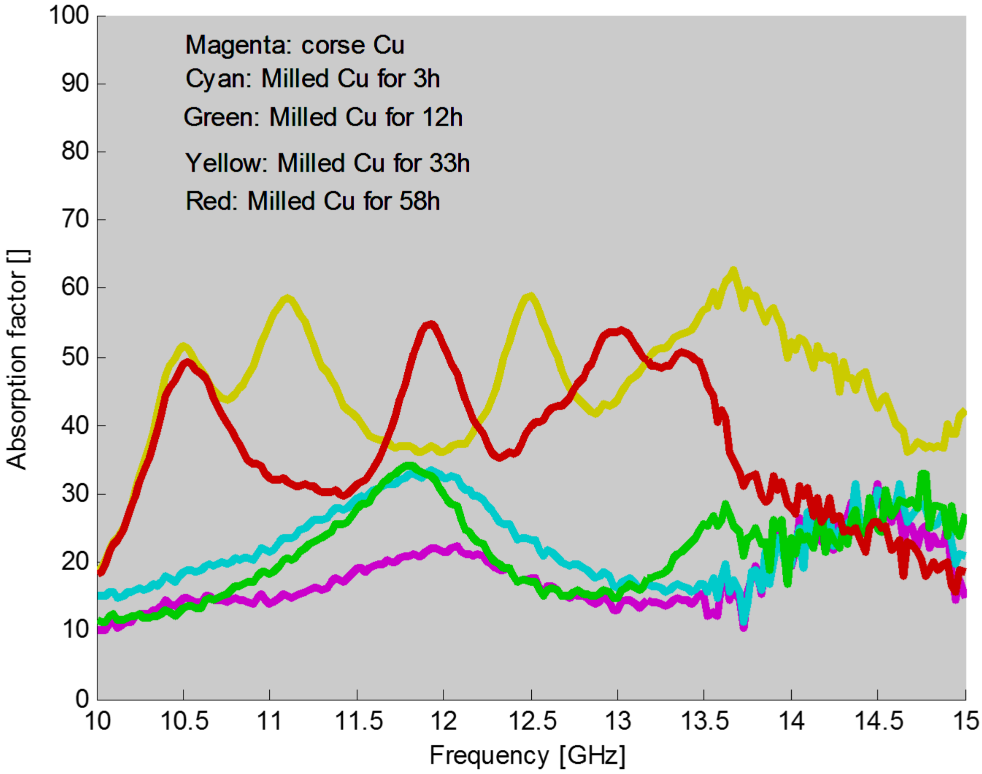
For an EMI shielding material, the total EMI shielding effectiveness is contributing from three mechanisms, namely absorption, reflection and multiple-internal reflections. Experimentally, the shielding effectiveness is obtained from complex scattering parameters (S parameters) of a network analyser. We measured the complex scattering parameters S of the elaborated thin slabs via the reflection/ transmission technique. The involved metallic wave-guide of $19.05 \times 9.525\ mm^2$ section is a structure able to propagate microwaves avers the band of [9.84 - 15] GHz. The fundamental concept of the reflection/ transmission technique is to sandwich the sample test between two waveguide sections and to carry out a two port scattering measures via the VNA.



The absorption factor is a microwave parameter calculated using the squared scattering parameters S_{11} and S_{21}:

$$\boldsymbol{A} = \mathbf{100}(|\boldsymbol{S}_{\mathbf{22}}|^2 - |\boldsymbol{S}_{\mathbf{12}}|^2)$$

The absorption factor informs about the induced losses within the material under the action of the incident electromagnetic field. We observe that the coarse Cu based nanocomposite shows the lowest absorption factor. Due to the dispersion of milled Cu powders with ultra-fine grains in the resin epoxy matrix, the absorption factor spectra move towards higher percentages with a reaching more than 60%. The dispersion of the milled Cu powders of different milling durations emphasizes the dissipation phenomenon; this behaviour is linked to the structural properties of the milled powders: large specific surface area induced after grain size refinement enhances the dissipation mechanisms of the electromagnetic field.



The computation of complex dielectric permittivity followed the noniterative approach proposed by A.H. Boughriet. Dielectric permittivity spectra exhibit the effect of the structural refinement and the resulting morphology on the microwave absorbing properties.