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Analysis and reoperation of the magnetic permeability spectra of textured composite based on Z-type hexaferrite by using Cramers-Kronig relations

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Abstract. The frequency dependences of the magnetic permeability of textured composite based on $\text{Ba}_3\text{Co}_{2,4}\text{Fe}_{23,2}\text{O}_{41}$ ferrite are given. The magnetic permeability spectra were obtained by coaxial method in the frequency range 0.01–18 GHz. The expert judgement on the spectra was made by using Cramers – Kronig relations. It was shown, that the Cramers – Kronig relations can be used to correct magnetic permeability measurements by reciprocal recalculation of the frequency dependences of the real and imaginary parts.

1. Introduction

Field of materials science, additive technology and polymer engineering actively develop at the present time. New technologies are based on the use of composite materials. Composite materials are materials made from two or more ingredient materials with significantly different physical or chemical properties, that when combined, produce a material with characteristics different from the individual components. The separate components remain separate and clear within the finished structure. The new material may be preferred for many reasons: common examples include materials which are stronger, lighter or less expensive when compared to traditional materials [1]. Pure powdered materials are not suitable for use in electronics equipment so they should be used as filler in the composite materials. Composite microwave materials are widely used due to their actively interaction with electromagnetic radiation.

Electromagnetic (EM) characterization of materials is a pre-requisite step before their use in microwave applications: anechoic rooms, mobile phones, wireless communications, radar, etc. EM characteristics of the materials should know for the microwave electronics applications [2-4].

In this article composite, based on hexagonal ferrite, was studied. The fillers of composites were powders of hexaferrite $\text{Ba}_3\text{Co}_{2,4}\text{Ti}_{0,4}\text{Fe}_{23,2}\text{O}_{41}$. Epoxy resin was used for production of experimental samples. EM characteristics of materials are measured by a coaxial method and LCR method. Agilent's E4980A precision LCR meter was used to measure of resistance and inductance of experimental toroid samples. The imaginary permeability spectra of the samples were investigated at frequency range from 0.01 till 18.0 GHz using a universal spectrometer based on a P4M-18 MICRAN scalar network analyzer.

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2. Materials and Methods

2.1. Nomenclature

Standard ceramic techniques were used for the fillers creation. The initial materials for the synthesis were barium oxide BaO powders, cobalt (II) oxide CoO, titan oxide TiO₂ and iron (III) oxide Fe₂O₃. Prior to synthesis, the oxides powders were dried for 3 h at temperature of 200 °C. After that powders were weighed according to a ratio.



Powders used for sintering experiments were placed for 4 h in a vibration ball mill. The ball-to-powder mass ratio was 5:1. Powders were die-pressed at about 1000 atm. Solid samples were heated up in 10–15 min to the sintering temperature (1150 °C), fired for 4 h and were cooled to the room temperature. After that samples were crushed. Powders were placed for 35 min in a vibration mill. Powders were die-pressed repeatedly at about 1000 atm. Solid samples fired in oxygen for 6 h 1200 °C finally. After that samples were placed once again. The powders were sifted by separator. The sizes of powders don't exceed 80 microns.

For production of experimental samples the polymer was used. It was epoxy resin EDP-20 by OKACHIM (Russia). In the liquid state its viscosity is known to be small. This allows a filler to be moved easily. Polyethylene-polyamine was used as the epoxy resin curing agent.

Samples were prepared as follows: hexagonal ferrite powder and epoxy resin in mass proportions of 66:34, respectively, are weighed in the desired proportions on scales Shimadzu AUX – 320 (accuracy 0.5 mg). Components of the composite were placed in the container and mixed for 15 minutes. Obtained mixture was placed in the form as washer. The mixture with ferrite is polymerized in constant magnetic field by special device for texture magnetic polymer materials [4]. It consists of a permanent magnet with the size of a field 0.5 kOe and the electric motor connected to a source of constant tension of 3V. To manufacture texture test samples, the process of polymerization was carried out for 5 h at the room temperature with the electric motor running. The final polymerization was carried out within 24 hours at the room temperature. The samples had the following sizes: thickness of 1.05 mm, an outer diameter of 7 mm and an inner diameter of 3,04 mm. The sample density was determined by weighing in a Shimadzu AUX-320 electronic balance. It was found to be around 2.45 g/cm³.

2.2. Measuring equipment

Agilent's E4980A precision LCR meter was used to measure of resistance and inductance of experimental samples. The initial magnetic permeability was calculated by formulas [5]:

$$\mu' = \frac{2\pi r_{mean} L}{w^2 S}, \quad (1)$$

$$\mu'' = \frac{r_{mean} R}{w^2 S f} \quad (2)$$

where L is inductance, R is resistance, r_{mean} is the mean radius of the coaxial sample, S is the sectional area of the sample, f is the resonance frequency, w – is the number winding turns.

For microwave characterization of samples we have used a measurement procedure based on the coaxial techniques. P4M-48 MICRAN was used to measure of the module of the reflectivity and the transmission. Coaxial cell is selected due to the electromagnetic field interacts with sample as well as in free space.

The electromagnetic (EM) parameters (ϵ' , ϵ'' , μ' , μ'') were calculated by the materials measurement software. So, the imaginary permeability was calculated by formulas [5]:

$$\mu'' = \frac{15(1 - K_r)}{\pi f d (1 + K_r)} \quad (3)$$

where d is the thickness of sample, K_r is the reflectivity.

2.3. Reoperation of the magnetic permeability spectra

It is well known that the wider the frequency range of measurements, the more enough information is provided by Cramers – Kronig relations. The Cramers – Kronig relations are actually for the response functions of arbitrary linear systems.

The authors [7] have obtained formulas (4) for reoperation of the permeabilities:

$$\begin{aligned} \mu'(f) &= I + \frac{1}{\pi} \sum_{i=1}^N \left\{ 2(\mu''_{i+1} - \mu''_i) + k_i f \ln \left| \frac{(f_{i+1} - f)(f_i + f)}{(f_{i+1} + f)(f_i - f)} \right| + d_i \ln \left| \frac{f_{i+1}^2 - f^2}{f_i^2 - f^2} \right| \right\}, \\ \mu''(f) &= -\frac{1}{\pi} \sum_{i=1}^N \left\{ k_i f \ln \left| \frac{f_{i+1}^2 - f^2}{f_i^2 - f^2} \right| + d_i \ln \left| \frac{(f_{i+1} - f)(f_i + f)}{(f_{i+1} + f)(f_i - f)} \right| \right\}. \end{aligned} \quad (4)$$

The results of calculations by the relations (4) involve an error related to the boundedness of the frequency interval in which approximation is performed. It is necessary to take advantage of the analytic properties of the function $\mu^*(f)$: (a) $\mu''(f)$ tends to zero as $f \rightarrow 0$ and $f \rightarrow \infty$ and the area bounded by the curve $\mu''(f)$ (it is proportional to the saturation magnetization) is finite and (b) $\mu'(f)$ is bounded: as $f \rightarrow 0$, $\mu'(f)$ tends to μ_0 , the static initial permeability, which is determined by independent experiments (it is also proportional to the saturation magnetization), and $\mu'(f) \rightarrow 1$ as $f \rightarrow \infty$.

3. Results and discussion

At first, resistance R and inductance L were measured. Initial magnetic permeability was calculated by formulas (1) and (2). The equations linking the experimentally measured coefficients of transmission and reflection of electromagnetic waves with the values of the magnetic permeability are described in paper [6]. Initial magnetic permeability and imaginary part of magnetic permeability allow reoperation the spectrum of the real part of magnetic permeability with the help of the Cramers–Kronig relations [7]. The spectrum of the magnetic permeability of composite material is shown in figure 1.

As shown in figure 1, the real parts of the permeability decrease from 4.9 to 1.0 rel. units with increasing frequency from 0.1 to 18 GHz. The frequency dependences of the imaginary part of the permeability have two maximum. First maximum is region around 1 GHz. It is treated by the authors of [8] to be caused by the processes of displacement of domain boundaries. Second maximum is region around 4 GHz. It is considered to be related to natural ferrimagnetic resonance [8].

Note, that reflection coefficient was calculated according to the obtained values of permeability then compared with the experimentally measured and found that the permittivity of the composite material is equal to $\epsilon' = 6,7$ rel. units, $\epsilon'' = 0,6$ rel. units.

4. Conclusions

The Z-type hexaferrites were synthesized using a ceramic technique. The composite has been prepared using epoxy as a matrix with 65 wt% the Z-type hexaferrite fillers concentration. Electromagnetic characteristics are measured in range of 0.01 – 18.0 GHz. It is show, that there are two maximum on permeability spectra. They are the processes of displacement of domain boundaries and the natural ferrimagnetic resonance.

Analysis of the spectra by using the Cramers – Kronig relations enables one to select the most reliable experimental results.

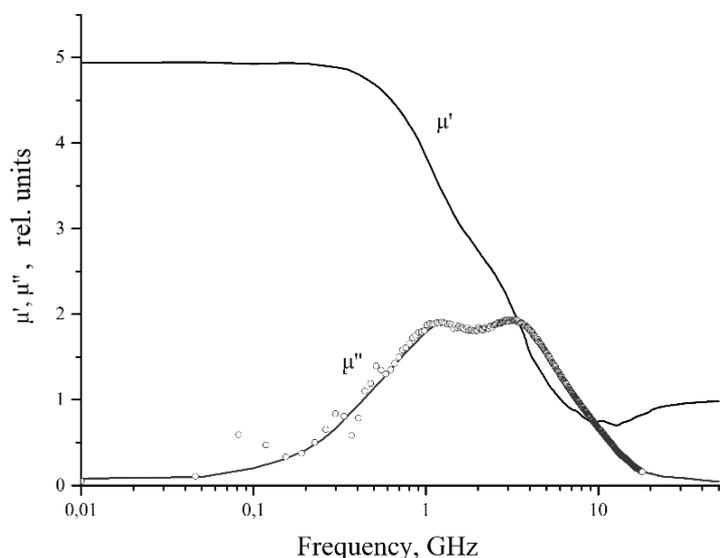


Fig.1 – The spectrum of the magnetic permeability of composite

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