

Microwave Characterization and Performance Assessment of Polymer-Magnetic Nanocomposites using Transmission-Line Based Microwave Resonators

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Abstract

This paper presents the synthesis and microwave characterization of superparamagnetic polymer nanocomposites with uniformly dispersed 8nm Fe₃O₄ nanoparticles for RF and microwave applications. Microstrip linear resonators (MLR) with modified substrate structures have been designed at microwave frequencies (2.5GHz) and employed to characterize the changes in the intrinsic properties of the polymer nanocomposites. In addition, frequency characteristics of the resonator have been examined under ambient conditions (i.e., 24°C and 1Atm of pressure) while applying external magnetic fields up to 0.6 Tesla. Frequency tunability of 37 MHz (1.5%) and unprecedented improvement in the loaded quality factor Q_{Loaded} from 13 to 70 (5.4X increase) have been observed, with 13.7dB of reduction in the insertion loss from 24.2dB to 10.5dB.

Key words: Frequency Tunability, Magnetic Nanoparticles, Polymer Nanocomposites, Transmission Line Resonators, Q factor.

I. INTRODUCTION

In recent years, the employment of polymer materials with embedded nanoparticles as a synthesized microwave substrate has attracted tremendous attentions from both academia and industries due to their multifunctionalities and ease of manufacturing. Particularly, conducting polymer nanocomposites have been identified as an effective material for microwave absorption and EMI shielding [1][2]. Nevertheless, those magnetic composites usually have intrinsic high loss at microwave frequencies, which are undesirable for RF and microwave applications. The successful recent demonstration of superparamagnetic polymer nanocomposites, with uniformly dispersed 8nm Fe₃O₄ magnetic nanoparticles by our research team, should result in a new class of low-loss microwave substrates with unprecedented tunability. Moreover, this technology offers a new approach for fabrication of high performance RF and microwave devices that are inaccessible based on conventional magnetic materials.

This study reports a viable potential application of using a superparamagnetic composites material incorporated in a PCB substrate to make tunable RF and microwave devices. In this paper, a microstrip linear resonator (MRL) with a modified substrate is designed

and constructed in order to investigate the change in the effective permeability and the losses in the presence of an externally applied magnetic field. Also, as a consequence of the field-induced variation in these properties, some notable effects in the resonance frequency, Q factor and insertion loss will be explored.

II. DESIGN AND FABRICATION

The MRL is a half wave resonator that consists of a simple microstrip transmission line which is interrupted at two different points.

In this device, the length l_r of the designed central microstrip equals to an integer multiple of the guided wavelength. Consequently, this dimension is adjusted to set the resonance frequency. The geometry of the microstrip linear resonator fabricated over modified Rogers's 6010 PCB substrate with embedded magnetic polymer nanocomposites is shown in Figure 1.

Obviously, the resonance frequency of the microstrip linear resonator also relies heavily on the effective material properties of the substrate given by:

$$f_r = \frac{v_p}{\lambda_g} = \frac{c}{\lambda_g \sqrt{\epsilon_{\text{eff}} \times \mu_{\text{eff}}}}, \text{ where } \lambda_g = \frac{2l_r}{n} \quad (1)$$

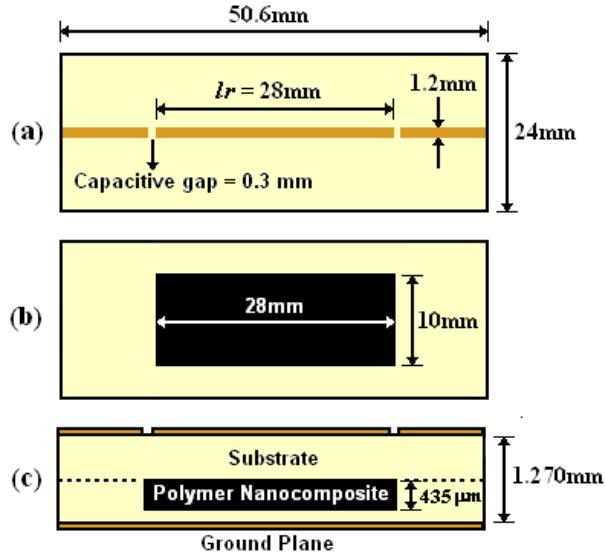


Figure 1: Layout of the MLR resonator with the key dimensions specified. (a) Top view of the first layer. (b) Top view of the second layer. (c) Cross-sectional view of the device.

The MLR was designed and simulated using Advanced Design System (ADS) 2009 from Agilent Technologies. Also, 3D EM finite element simulations were performed using Ansoft HFSS v11.1, through which the different design parameters of the resonator were optimized. In order to avoid impedance mismatch with testing instruments, the characteristic impedance of the microstrips at input/output feed lines were designed to be 50Ω . The MLR device was fabricated in a class-1000 Cleanroom facility, using a typical photolithography process followed by wet etching of the copper clads to pattern the microstrip lines.

The resonator-based test fixture is constructed by bonding two PCB laminates (with $\epsilon_r = 10.2$, $\tan\delta = 0.0023$ and thickness = $635\mu\text{m}$) together. As shown in Figure 1(a) and Figure 2 (a), the first laminate hosts the microstrip lines of the MLR structure. The copper layer (ground plane) from the back side of the first laminate was completely removed. As shown in Figure 1(b) and Figure 2(b), the copper clad was stripped from the top of the second laminate followed by formation of a $435\mu\text{m}$ -deep rectangular cavity with a total area of 280mm^2 . This cavity is properly aligned with the central microstrip line of the MLR device patterned on the front-side of the first laminate. In addition, the ground plane of the MLR is retained underneath the second laminate.

The polymer nanocomposites is thereafter deposited into the micromachined cavity using a syringe and followed by baking in a vacuum oven at 90°C for 4 hours for hardening (curing) purpose.

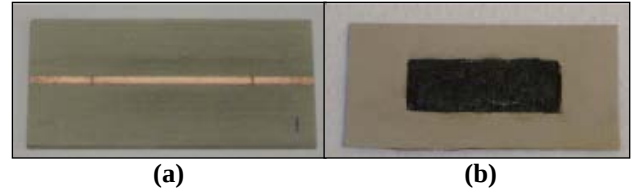


Figure 2: Configuration of the fabricated MLR resonator. (a) Top-view photo of the first laminate with the microstrip linear resonator. (b) Top-view photo of the second laminate with polymer nanocomposite filled cavity.

After the curing process, deviations in the resonance frequency, insertion loss and quality factor of the MLR are evaluated under externally applied magnetic field, in order to determine the field susceptibility and frequency tunability of the device due to the field-induced variation of the effective microwave properties of the deposited nanocomposite layer.

III. SYNTHESIS OF POLYMER NANOCOMPOSITES AND MAGNETIC CHARACTERIZATION

$8\text{nm Fe}_3\text{O}_4$ or CoFe_2O_4 nanoparticles (NPs) coated with oleylamine and oleic acid were synthesized following a standard chemical co-precipitation procedure. The low-loss polymer, courtesy of Rogers Corporation, was thereafter dissolved in hexane together with calculated amount of surfactant-coated nanoparticles to obtain the polymer nanocomposites with uniform dispersion of NPs at desired concentration [3].

The surfactant plays a dual role in this process. First of all, it completely encapsulates and isolates individual particles and thus weakening the magnetic exchange interactions between them. Secondly, the choice of surfactant is also important to enhance the binding between the macromolecular chains of the polymer with the individual nanoparticles, which would prevent the particle diffusion during the formation of the polymer nanocomposite thus effectively suppressing the tendency of agglomeration [4].

This chemical process is important because our goal is to retain the superparamagnetic properties of both individual magnetic nanoparticle and entire macroscopic polymer nanocomposite material. Superparamagnetism would eliminate hysteretic response in the M-H characteristics which has direct implication in reducing the magnetic losses as well as eddy current losses for the benefit of low-loss microwave applications [3].

Figure 3 shows the TEM image of chemically synthesized, surfactant coated cobalt ferrite nanoparticles dispersed in the polymer provided by

Rogers Corporation and spin coated as 20 μm -thick films on glass substrates or microwave calibration boards. As seen in the Figure 3, excellent dispersion is achieved and nanoparticles do not form clusters. The inset in the top right corner of the same figure shows a classic example of agglomeration if the correct chemical processing is not done during the polymer film coating process [3].

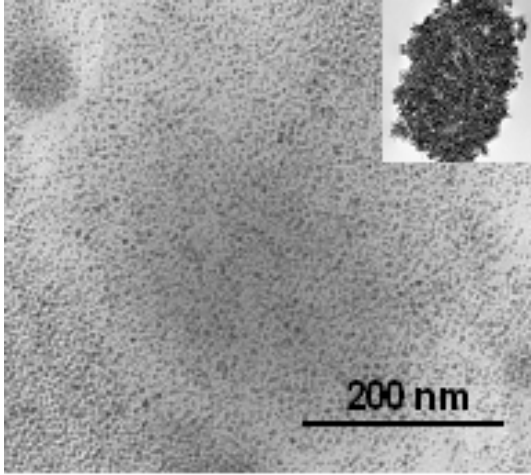


Figure 3: Transmission electron microscope image of CoFe_2O_4 nanoparticles (mean size ~ 8 nm) uniformly dispersed in Rogers polymer. Inset in upper right corner is a typical example of particle agglomeration without the chemical processing.

To confirm if indeed the individual nanomagnetic properties are retained for polymer nanocomposite film coated over a large area, one has to measure the magnetic hysteresis loops (M-H curves) and verify if true superparamagnetic response is obtained. The magnetization measurements were done in a commercial Physical Property Measurement System (PPMS) from Quantum Design equipped with a superconducting magnet.

Figure 4 presents measured M-H loops for the CoFe_2O_4 nanoparticles only and the same nanoparticles in two different concentrations loaded into the polymer matrix. The data show excellent saturation magnetization characteristics and zero coercivity as well as zero remanence at room temperature which is the characteristic signatures of superparamagnetism. A large coercivity of several kOe opens up at 10K (not shown in this paper) well within the magnetically blocked state of the nanoparticles. As mentioned earlier, superparamagnetism (or lack of coercivity) is desirable for low-loss microwave magnetic materials [3]. Thus the polymer nanocomposites developed in this work is well-suited for implementation of many tunable and low-loss RF and microwave devices.

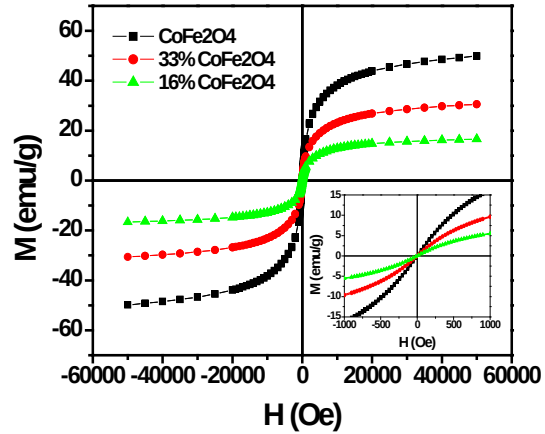


Figure 4: M-H curves for as-synthesized cobalt ferrite nanoparticles and two different concentrations (by wt %) of the nanoparticles dispersed inside the polymer films. Inset shows the expanded view of the low field region clearly indicating lack of coercivity or remanence in the materials.

IV. MEASUREMENT SETUP

The two-port device measurement setup consists of several important components. A fix electromagnet (Model 7500w from Alpha Scientific), which provides a maximum field strength of 0.88 Tesla with 1 inch separation between the two poles. A DC Power supply (Model LB-705-FM from Lambda Scientific), which provides the necessary current and voltage to operate the electromagnet from zero up to its maximum field strength with excellent accuracy. And finally, the enhanced vector network analyzer (Model E5071B from Agilent Technologies), which enables two-port S-parameters measurement between 300KHz and 8.5GHz. All the aforementioned key parts are properly calibrated and connected as shown in Figure 5 and Figure 6.



Figure 5: Two-port microwave test setup with well-calibrated external magnetic field applied by the electromagnet

Non-magnetic SMA connectors and coaxial cables were employed to avoid movements in the connections as well as undesirable EM interference during the measurements.

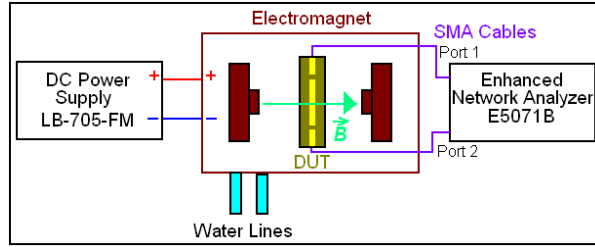


Figure 6: Schematic for the two-port microwave measurement setup with the arrangement of the major equipments specified.

The system was calibrated using SOLT (Short, Open, Load and Thru) calibration method, in order to de-embed the effects and losses introduced by the cables and connectors and thus setting the reference planes for the measurement at the two terminals of the MLR resonator.

The frequency characteristic of the microstrip linear resonator was first measured using the E5071B enhanced vector network analyzer in absence of DC magnetic field. Thereafter, the device under test (DUT) was then placed between the poles of electromagnet, as shown in Figure 7, to study the effect of the externally applied DC magnetic field on the resonance frequency, the insertion loss and the quality factor of the device with built-in nanocomposites.

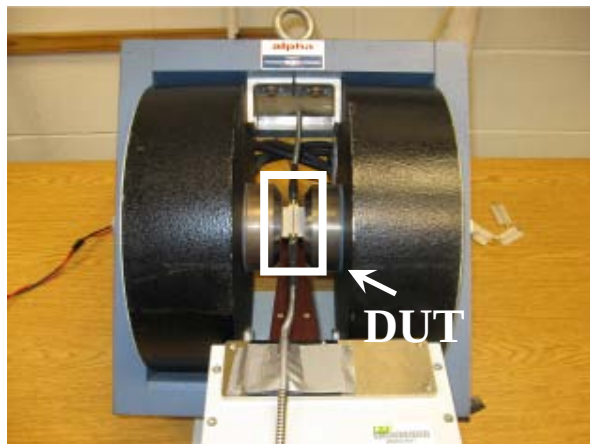


Figure 7: The MLR resonator (DUT) is placed between poles of the electromagnet and connected to the vector network analyzer through non-magnetic SMA connectors and cables

V. EXPERIMENTAL RESULTS

Microstrip linear resonators prepared on LiMnTi polycrystalline ferrite substrate have been examined under low-level biasing magnetic fields by R. Pourush, *et al.*. Measured frequency tunability of 17% and degradation in the Q factor from 51 to 38 (25.49%) were reported [5]. Also, it was observed that as the strength of the applied DC magnetic field increases, the loss increases and hence the value of the quality factor drops [5].

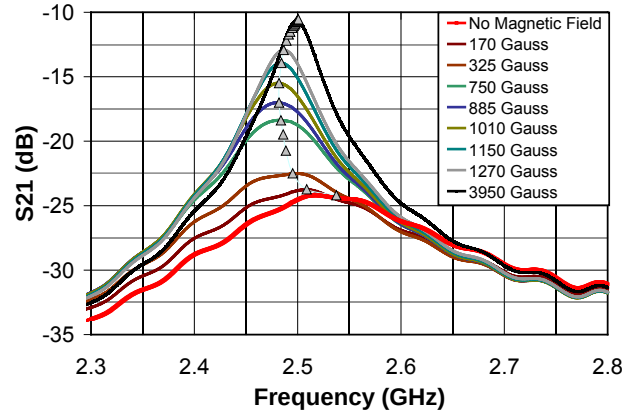


Figure 8: Measured transmission characteristics (S_{21}) of the MLR with embedded nanocomposites.

Figure 8 presents the measured transmission (i.e., S_{21}) of the microstrip linear resonator device under externally applied DC magnetic field of various strengths. As shown, a resonance frequency of 2.5375GHz along with an insertion loss of 24.2 dB and a measured loaded Q of 13 is observed in the absence of magnetic field. As the strength of externally applied DC magnetic field is varied from zero to 6000 Gauss, its impacts on the measured resonance frequency, the insertion loss and the quality factor of the MLR resonator is investigated. During the entire test, a deviation of 37 MHz in the resonance frequency was observed (from 2.5375 GHz to 2.4801 GHz). The influence of the DC magnetic field on the resonance frequency of the device is shown in the Figure 9. This external field was transversely applied to the plane of the resonator substrate.

Figure 10 and Figure 11 show the dependency of the maximum transmitted power at the resonance frequency and the loaded Q on the strength of the external magnetic field, respectively. Particularly, before an external DC magnetic field is applied, a low transmission featured by a high insertion loss of 24.2dB along with the lowest loaded Q factor of 13 has been observed.

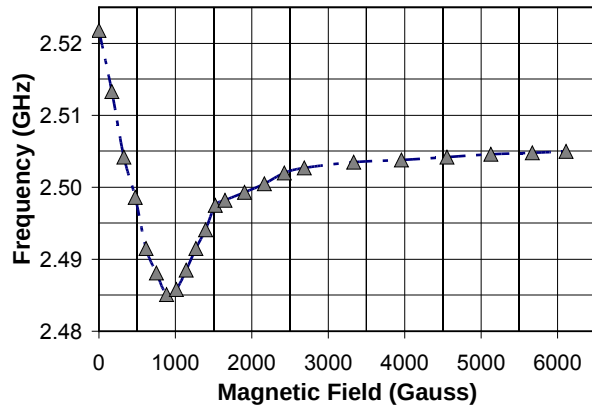


Figure 9: Variation of the resonance frequency vs. externally applied DC magnetic field

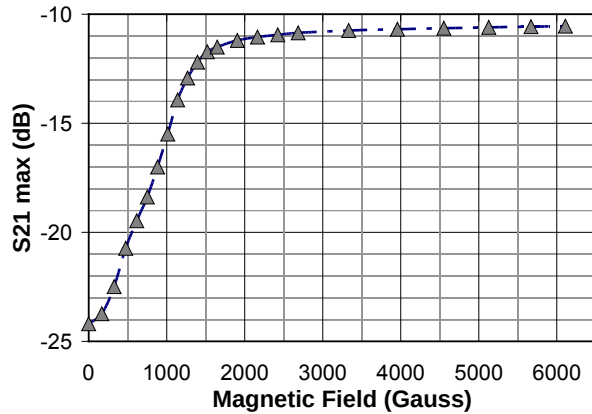


Figure 10: Dependence of transmission property S_{21} (maximum transmitted power at the resonance frequency) to the externally applied DC magnetic biasing field.

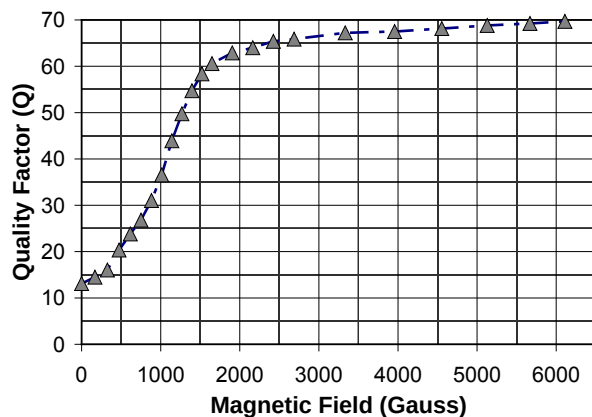


Figure 11: Dependence of the loaded Q factor to the external applied DC magnetic biasing field

As the strength of the external magnetic biasing field is increased, the loss decreases and the Q factor

increases until the effect of the external field saturates. Under an external field of 0.6 Tesla, the maximum loaded Q factor of 70 is demonstrated along the lowest insertion loss of 10.5dB. It is therefore clearly observed that the losses decrease and the maximum transmitted power and the loaded Q factor increased when the applied external magnetic field is strengthened.

VI. CONCLUSIONS

Polymer nanocomposites with 8nm Fe_3O_4 nanoparticles have demonstrated great potential for implementation of tunable microwave devices. Such superparamagnetic materials have shown great promise as either the core material or the building block for the low-loss and tunable microwave substrates. Particularly, such materials can be employed to construct a sandwiched multi-layer laminate or to form different tridimensional structures and shapes, which enable designers to tailor the effective electrical and microwave properties of the substrates under the influence of external magnetic fields. One of the novelties of such material resides in its superparamagnetic properties at microwave frequencies. Consequently, no additional losses are added to the system because of the lack of coercivity or remanence in the material.

Measured frequency tunability of 37 MHz (1.5%) and unprecedented enhancement of the quality factor from 13 to 70 (5.4 X improvement) are achieved with an externally applied DC magnetic field less than 0.6 Tesla.

The observed variations in the resonance frequency, insertion loss and quality factor of the fabricated device clearly indicate the great susceptibility of the device to DC magnetic-field. Undoubtedly, this nanocomposite material shows fascinating properties that has never been reported and should be further explored for microwave device applications.

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