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Dielectric-ferrite film heterostructures for magnetic field controlled resonance microwave components

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ABSTRACT

An investigation of the composite "α-Al_2O_3 dielectric resonator-thick ferrite film" heterostructures magnetic field tunable microwave properties has been conducted. Thick high-density high-quality $NiFe_2O_4$ spinel and M-type hexaferrite $BaFe_{12}O_{19}$ films were deposited on the surface of the dielectric by tape-casting technique. Specific organic suspensions for ferrite films synthesis were developed; optimal conditions for pre-heat treatment and annealing have been defined. It was found, that magnetic field has a profound impact on microwave transmission characteristic of composite resonator, including peak absorption level and unloaded *Q*-factor. Both effects were attributed to increase of the magnetic part of the composite resonator internal losses at frequencies close to ferromagnetic resonance. Since qualitatively similar results were obtained for both cm-wave (with nickel ferrite) and mm-wave (with barium hexaferrite) resonators, the proposed method of electronic control over dielectric resonator properties can be successfully utilized in a very broad frequency range, basically, from few GHz to more than 100 GHz.

1. Introduction

Current trends in mobile communications, wireless technologies, satellite television and radiolocation require improvement of the existing UHF components and advancement into new frequency bands [1–3]. Stripline and dielectric resonator based UHF filters are the key elements of the modern transceiver equipment [4–8]. Besides indisputable advantages of such devices (like high quality factor and temperature stability), the lack of dynamical tuning of their characteristics (such as operating frequency, *Q*-factor, insertion losses, etc.) is their crucial drawback, since utilization of electronically tunable components can greatly increase the functionality of microwave devices. Thus, development of tunable devices on the basis of high-*Q* dielectric resonators (DR) is an important and urgent task.

In general, electronic control could be realized if device includes ferroelectric [9], semiconductor [10] or ferrite [11] constituent. This is, in essence, the practical realization of the composite materials concept. In such structures tuning is fulfilled due to the either electric field dependence of added component dielectric constant, conductance and/or capacitance or, as in the last case, due to its high frequency magnetic permeability adjustment with magnetic field. Other tuning techniques, with the assistance of electromechanical actuators, MEMS [10,12] or DR temperature change [13] are also known.

In the first two of abovementioned cases, an undesirable degradation of resonator quality factor and thermostability usually take place, therefore incorporation of ferrite component with tunable permeability seems more advantageous since it allows for purely electronic control over composite resonator electrodynamics characteristics without noticeable deterioration of the *Q*-factor.

In the present paper, we considered the electronic control of RF properties of composite heterostructure, comprising of polycrystalline α-Al_2O_3 bulk DR with a layer of ferrimagnetic material. While tuning mechanism is similar with that, utilized in [11], we exploited thick-film tape-casting ferrite component instead of a bulk ferrite, which, as expected, should decrease mass and dimensions of composite structure, and also make it compatible with planar technology. Large area of contact between ferrite and dielectric in such heterostructure favors substantial penetration of DR main mode high-frequency electromagnetic field into magnetic film, resulting in noticeable impact of ferrite permeability on the

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DR resonance mode even for marginal relative volume of the ferrite component. At the same time, small ferrite volume could not lead to substantial degradation of DR eigen-oscillations *Q*-factor, providing that the bias magnetic field is far from resonant value. Such resonators with electronically controlled electrodynamic characteristics can find application in reconfigurable matched filters [14] with dynamic modification of the filter pulse response characteristic in accordance to the spectrum of incoming signal [15]. Besides, as it was demonstrated earlier, two-layered ferrite-dielectric structures can operate as a magnetic field tunable mm-wave band isolator [16].

For the ferrite constituent of composite resonator we selected such traditional microwave ferrites as $NiFe_2O_4$ with spinel structure and M-type barium hexaferrite ($BaFe_{12}O_{19}$). The former is widely used in lower part of microwave frequencies, whereas the latter one (due to the large magnetocrystalline uniaxial anisotropy field) is utilized in millimeter wavelengths, at frequencies above 50 GHz. Hence, this choice of materials allowed us to investigate the suggested composite resonator design in a very broad frequency range.

It is known [17], that in order to get a desired parameters of ferrite microwave devices, a relatively thick film (from few tenths to few hundreds microns) must be utilized. One of the promising techniques for thick crystalline films deposition is a tape-casting [18,19]. It allows to obtain a high-density film and provides capability for film thickness variation during the deposition.

For the manufacturing of a two-layer "α-Al_2O_3 dielectric resonator-ferrite film" structures the film should have high density and homogeneity [20]. The suspension for the thick films deposition by tape-casting method consists of two main components: an inorganic powder precursor and an organic component. Therefore, the production of thick high-density films by tape-casting method is primarily dependent on both the microstructure of the powder precursor (particle size and agglomeration) and the nature of organic components. Also, an important key point during the synthesis of uniform thick films is its annealing conditions (prebaking and final annealing temperatures). Therefore, the development of the main suspension components (powder precursor and organic component) and determination of annealing conditions are important.

The aim of this work is to develop the high-density high-quality nickel ferrite spinel $NiFe_2O_4$ and M-type barium hexaferrite $BaFe_{12}O_{19}$ thick films on the surface of the α-Al_2O_3 bulk dielectric resonator and to produce electronically tunable microwave resonance components for cm- to mm-wave frequency bands on the basis of these two-layer composite structures.

2. Experimental part

2.1. Synthesis of powder precursors

The starting powder precursors for the synthesis of spinel $NiFe_2O_4$ thick films were obtained by the precipitation from non-aqueous solutions according to the method described in [21]. $Fe(NO_3)_3{\cdot}6H_2O$, $Ni(NO_3)_2{\cdot}9H_2O$ (analytical grade), NaOH (purity 97%), diethylene glycol (DEG, purity 99%) and oleic acid (pure OLA) were used as initial reagents. Nanoparticles of $NiFe_2O_4$ were obtained by the heat treatment of prepared solution in the temperature range 200–220 °C (60 h).

The starting powder precursors for the synthesis of M-type barium hexaferrite (BHF) thick films were obtained by the sequential precipitation of barium carbonate on pre-precipitated iron (III) hydroxide from aqueous solutions of their salts in the stochiometric ratio corresponding to the formula $BaFe_{12}O_{19}$. A 1 M solutions of an analytically pure $Fe(NO_3)_3$ and $BaCl_2$ were used. Iron hydroxide was precipitated from an aqueous ammonia solution (pH 4.3) and barium carbonate with an ammonia-carbonate (pH 9) precipitant. Nanoparticles of BHF were obtained by the heat treatment of precipitates at T = 1000°C. This method of M-type BHF nanoparticles synthesis was described in details in [22].

Samples were investigated by X-ray phase analysis (XPA) and full-profile X-ray phase analysis on a DRON-4 diffractometer (Cu Kα radiation). The size of powder particles was estimated from the broadening of the XRD peaks. Assuming the Gaussian shape of the diffraction peak, the linear line broadening β was calculated from the formula: $\beta = \sqrt{B^2 - b^2}$, where *B* is the total linear broadening of the line and *b* is the instrumental broadening. The particles size was calculated from the Scherrer formula: $D = 0.9\lambda/(\beta_{hkl}\cos\theta_{hkl})$. The size and morphology of powder particles have been determined using a SELMI transmission electron microscope (TEM)—125 K at 100 kV of accelerating voltage. Size distribution was obtained from the analysis of TEM images with the use of image tool 3 and Originpro 8.5 Sr1 software packages. When calculating particles size distribution, tem images were analyzed according to the procedure described by Peddis et al. [23].

2.2. Synthesis of spinel $NiFe_2O_4$ and M-type $BaFe_{12}O_{19}$ nanocrystalline thick films

The suspension for film deposition consists of the powder precursor and the organic component. The organic component was a mixture of plasticizer (dibutyl phthalate), dispersant, binder (polymethyl methacrylate, PMMA) and solvents (isopropanol and acetylacetone). Dibutyl phosphate and a mixture of dibutyl phosphate and tannin were used as dispersants. The suspension consists of the precursor powder, isopropanol, dibutyl phosphate, acetylacetone, PMMA and dibutyl phthalate in the ratio of 30%, 22%, 4%, 30%, 9% and 5% of the total weight of suspension component, respectively. The prepared suspension was stirred in a planetary mill (mixing speed 300 rpm, 4 cycles for 30 min).

Thermogravimetric and differential thermal analysis (TGA/DTA) of the obtained suspensions were carried out. The experiments were conducted in dynamic air or oxygen atmospheres, 100 mL/min, in the *T* = 20–1000 °C temperature range, using a SDT Q600 V8.1 Build 99 equipment.

Then, the obtained suspensions were deposited by the type-casting method (DEPOSITION RATE WAS 0.1 MM/S) on α-Al_2O_3 dielectric resonators.

The prebaking of the thick films was carried out at slow heating and thermal shock conditions at 500 °C, using a heating rate of 30 °C/min. The films underwent final annealing in a conventional furnace at a heating rate of 60 °C/h. Temperature ranges were *T* = 800–1100 °C and *T* = 1000–1300 °C for $NiFe_2O_4$ and M-type $BaFe_{12}O_{19}$ thick films, respectively.

Synthesized films were characterized by X-ray diffraction analysis (XRD) on X'pert powder PAN Analytical diffractometer with Cu Kα radiation and Bragg–Brentano geometry. For the phase characterization, the JCPDS database was used.

The micrographs of the films were obtained using field emission gun scanning electron microscope (JSM 6510LV, Jeol, Japan).

2.3. Transmission characteristics of the composite "$\boldsymbol{\alpha}$-Al_2O_3-thick ferrite film" resonators

2.3.1. Composite resonator "$\boldsymbol{\alpha}$-Al_2O_3-nickel ferrite"

Investigation of the composite dielectric-ferrite resonator properties in centimeter wave band were conducted using custom made measuring cell. The cell was comprised from a microstrip transmission line section, placed inside a shielding metallic case. The microstrip transmission line was manufactured from a 0.01-in.