

Magnetically Controlled Nanocomposite for Microwave Elements

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Abstract — Multiphase microwave dielectrics with a high electric Q -factor and controlled thermal stability of electric properties on the base of barium polytitanates were developed. A nature of a Q -factor increase in multiphase systems is determined. Composite resonant elements on a basis of multiphase materials with a high Q -factor and a ferrimagnetic film were developed. It was shown that composite resonators are characterized by high Q -factor and an opportunity to tune the resonance frequency with the magnetic field, which allows their use in various tunable microwave devices.

Keywords — microwave dielectrics, dielectric Q -factor, composite resonant elements, magnetic film, non-reciprocity of electrophysical properties.

I. INTRODUCTION

In the microwave filters, solid state generators, antennas for modern radiolocation systems, wireless and satellite communications the dielectric resonators (DR) based on materials with low dielectric losses $\tan\delta$ (hence high Q -factor) at the microwave range and a high temperature stability of electrophysical properties are extensively used as the element base [1-5]. Usually they are single-phase systems since appearance of other phases causes a rising of the dielectric losses. For now different approaches to develop the materials with high dielectric permeability, Q -factor and low temperature coefficient of dielectric constant on a base of single-phase systems, that find practical applications, has been proposed. But DRs manufactured on the basis of such materials are insensitive to external electromagnetic fields, what doesn't allow to tune their properties. Furthermore, a value of thermal stability coefficient for single-phase systems is fixed, what doesn't always allow compensating the temperature instability of microwave circuits. For practical applications, materials with different values of temperature stability and a certain coefficient of dielectric constant temperature stability (τ_e) are often needed. All this factors limit the area of practical use of DRs based on single-phase microwave dielectrics. That is why it is important to search the ways to create both microwave materials, for which it is possible to realize the fine control of thermal stability by changing chemical composition, and resonant elements with a resonance frequency controlled by external electromagnetic field.

It is known, that dielectric materials on a basis of BaTi_4O_9 are characterized by relatively high values of dielectric constant ($\epsilon = 36$), Q -factor ($Q_f \approx 35000-40000$ GHz), and also by temperature stability (temperature coefficient of dielectric constant is $\tau_e = -(3-4) \cdot 10^{-5} \text{ K}^{-1}$) [6]. It was shown by us that an addition of oxides of a number of metals (in particular zinc oxide) allows to influence on temperature stability of BaTi_4O_9 electrophysical properties and to increase the Q -factor value. However, the nature of this effect was not clarified.

It is known too, that one of the ways to develop the tunable DRs is by combining the non-magnetic microwave resonator with high Q -factor and a sensitive to the external magnetic field (but characterized by significant dielectric losses) ferromagnetic film, applied to the end face of the resonator [7].

Although ferrites are not record-breaking in terms of magnetic parameters value among the magnetic materials, due to their high resistivity they can be used at the microwave frequencies. Earlier we developed composite resonators using $\alpha\text{-Al}_2\text{O}_3$, that is characterized by the value of dielectric constant $\epsilon \sim 10$, and nickel ferrite NiFe_2O_4 [8]. It was shown that obtained composite elements are able to change their properties, this is necessary for practical use. At the same time higher values of dielectric constant for BaTi_4O_9 ($\epsilon \sim 36$) allow to decrease dimensions of resonator, i.e. to decide the task of microminiaturization.

As the ferromagnetic material for a ferromagnetic film synthesis we chose nanocrystalline powders of nickel-zinc ferrites $\text{Ni}_{1-x}\text{Zn}_x\text{Fe}_2\text{O}_4$, magnetically soft materials with the high saturation magnetization, effective magnetic permeability, low coercive force [9].

Therefore the aim of this investigation is to develop and clarify the nature of a high- Q multiphase microwave dielectrics, based on barium tetratitanate and zinc oxide; explore the possibility to manage their electrophysical properties temperature stability, and also design the composite resonant microwave elements on the basis of multiphase dielectric material with high Q -factor and ferrimagnetic film of nickel-zinc ferrite, which resonance frequency can be tuned by external magnetic field.

II. EXPERIMENTAL DETAILS

A. Synthesis of barium tetratitanate BaTi_4O_9 – ZnO

Barium tetratitanate was obtained by a solid state reactions synthesis method. As the initial reagents were used BaCO_3 , TiO_2 and ZnO of puriss. spec. qualification. At the first stage a finely dispersed powder of BaTi_4O_9 was synthesized. At the second stage to the BaTi_4O_9 powder different amounts of ZnO were adding at the temperature of 900°C for 3 hours. Sintering of ceramics samples was carrying out at the temperatures of 1320 – 1340°C for 2 hours.

B. Synthesis of nanoscaled particles of solid solutions of nickel-zinc ferrites $\text{Ni}_{1-x}\text{Zn}_x\text{Fe}_2\text{O}_4$ with the spinel structure

The particles of nickel-zinc ferrites solid solutions were synthesized by the sequential precipitation of hydroxides method similar to work [10]. As the initial reagents the aqueous solutions of iron $\text{Fe}(\text{NO}_3)_3$, nickel $\text{Ni}(\text{NO}_3)_2$ and zinc $\text{Zn}(\text{NO}_3)_2$ nitrates were used and a 1M solution of sodium hydroxide was used as the precipitator. After the full precipitation of all the components the suspension was brought to the boil and being boiled for 1 hour. The precipitate was filtered from the mother solution and washed on a filter with bidistilled water. The final product was sintered in a muffle furnace at the temperature of 800°C .

C. Applying of films of nickel-zinc ferrite $\text{Ni}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$ with the spinel structure to a dielectric resonator

For the applying of ferrite films were used suspensions consisted of a powder precursor of the nickel-zinc ferrite and an organic component. As the organic component was used a photopolymer (ultraviolet glue PermaBOND UV630). Films of ferrite were polymerized in the absence of magnetic field and at the magnetic field of $H = 2900$ E, directed along the normal and in the parallel to the resonator axis, similar to the work [9].

D. Investigation of electrophysical properties of composite resonant elements

Absorption spectra of manufactured composite resonators were measured with Agilent N5230A PNA vector analyzer. Investigations of composite resonators properties were conducted at the centimeter waves range (X-band) using a measuring cell in a form of a X-band waveguide with the area of section of 23×10 mm². Investigations were conducted similar to the ones in the works [8, 10].

III. RESULTS AND DISCUSSIONS

On the fig. 1 we present the XRD pattern of synthesized BaTi_4O_9 and pattern of two-phase sample on a basis of BaTi_4O_9 –ZnO. It is seen, that when ZnO is introduced an additional phase appears. The additional phase that appears in the system BaTi_4O_9 –ZnO was identified as $\text{Ba}_3\text{Zn}_7\text{Ti}_{12}\text{O}_{34}$ ($\text{BaZn}_{2/3}\text{Ti}_4\text{O}_{11/3}$) [11].

Later the phase diagram of the BaO–ZnO–TiO₂ system study, made by authors of [12], showed that the additional phase in the system BaTi_4O_9 –ZnO is probably matches the phase $\text{BaZn}_2\text{Ti}_4\text{O}_{11}$. Therefore, $\text{BaZn}_2\text{Ti}_4\text{O}_{11}$ materials were synthesized by the solid state reactions method, the XRD pattern of this material is presented on fig. 1.

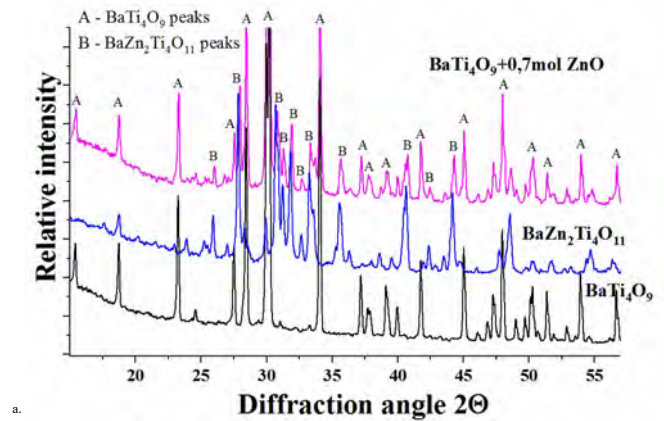
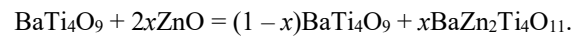


Fig. 1. XRD patterns of ceramics samples of BaTi_4O_9 , BaTi_4O_9 – ZnO, and ceramics with the $\text{BaZn}_2\text{Ti}_4\text{O}_{11}$ additional phase.

It can be seen from the fig. 1, that peaks appearing in the BaTi_4O_9 –ZnO system and peaks of the additional phase $\text{BaZn}_2\text{Ti}_4\text{O}_{11}$ matches. An interaction between barium tetratitanate and zinc oxide can be described by a reaction:



Regardless of an amount of zinc oxide entered in barium tetratitanate the BaTi_4O_9 –ZnO system consists of BaTi_4O_9 and $\text{BaZn}_2\text{Ti}_4\text{O}_{11}$ phases only. It is worth nothing a number of interesting regularities that appears after the introduction of ZnO in BaTi_4O_9 : a) if the temperature coefficient of dielectric constant at microwave frequencies is in the range of $-(3-4) \cdot 10^{-5} \text{ K}^{-1}$, then the introduction of ZnO can change τ_e from negative to positive values; b) at the appearance of the additional phase $\text{BaZn}_2\text{Ti}_4\text{O}_{11}$ electric Q -factor increases, what is unexpected, because usually the additional phases presence in a microwave dielectric causes additional losses (Q -factor decreases). The investigation of $\text{BaZn}_2\text{Ti}_4\text{O}_{11}$ electrophysical properties showed that unlike the BaTi_4O_9 phase the $\text{BaZn}_2\text{Ti}_4\text{O}_{11}$ phase is characterized by a positive τ_e . Therefore, at the appearance of $\text{BaZn}_2\text{Ti}_4\text{O}_{11}$ phase, which is characterized by a positive value of τ_e , there is an effect of thermal compensation and temperature stability of electrophysical properties improves.

It was shown too, that the $\text{BaZn}_2\text{Ti}_4\text{O}_{11}$ phase is characterized by the higher value of Q -factor compared with the main phase (BaTi_4O_9). Thus, the appearance of the $\text{BaZn}_2\text{Ti}_4\text{O}_{11}$ phase in the system BaTi_4O_9 – ZnO is accompanied by the improvement in the electrophysical properties thermal stability and Q -factor increase, what is not typical for microwave dielectrics.

On the basis of materials of the BaTi_4O_9 – ZnO system the high-quality dielectric microwave resonators were developed, that can be used in a design of various devices (microwave filters, solid state generators, etc.). However, their resonance frequency can't be influenced by the external electromagnetic field, what significantly limits their potential applications.

Theoretical calculations of an interaction of electromagnetic field with the composite DR, comprising high- Q cylindrical dielectric resonator based on the BaTi_4O_9 – ZnO system and the film of ferromagnetic material applied to the end face of it showed an opportunity of resonance frequency change under the influence of external permanent magnetic field.

For determination of thin ferrite film influence on characteristics of composite DR the theory of perturbations could be applied to the Maxwell's equations. We take the fields distribution for a dielectric resonator without a ferrite film as the unperturbed state (state 1), and the perturbed state (state 2) would be the field distribution for a resonator with ferrite layer. Then initial equations for calculations are

$$\begin{cases} \nabla \times \mathbf{E}_1 = -j\omega_1 \mu_1 \mathbf{H}_1, & \nabla \times \mathbf{E}_2 = -j\omega_2 \mu_2 \mathbf{H}_2, \\ \nabla \times \mathbf{H}_1 = j\omega_1 \varepsilon_1 \mathbf{E}_1, & \nabla \times \mathbf{H}_2 = j\omega_2 \varepsilon_2 \mathbf{E}_2, \end{cases} \quad (1.1)$$

where \mathbf{E}_i and \mathbf{H}_i – electric and magnetic field, ω_i – angular resonance frequency, μ_i and ε_i – magnetic and dielectric permeability. Lower index designates perturbed and unperturbed condition respectively.

Then it can be proven that a change of complex frequency of resonator after the perturbation is given by an equation

$$\frac{\omega_2 - \omega_1}{\omega_1} \approx - \frac{\int_{V_s} (\Delta \varepsilon \mathbf{E}_1^* \cdot \mathbf{E}_2 + \Delta \mu \mathbf{H}_1^* \cdot \mathbf{H}_2) dV}{2 \int_{V_c} \Delta \varepsilon \mathbf{E}_1^* \cdot \mathbf{E}_2 dV}, \quad (2)$$

where $\Delta \varepsilon$, $\Delta \mu$ – difference of DR and ferrite parameters, V_s – volume of ferrite sample, V_c – volume of a non-magnetic constituent.

If an inserted ferrite sample is homogenous and much smaller in volume than the main volume of resonator the electromagnetic field outside a resonator does not change ($\mathbf{E}_1 = \mathbf{E}_2$, $\mathbf{H}_1 = \mathbf{H}_2$). Then using known equations for the fields of unperturbed mode for a pure dielectric resonator, we can obtain an analytical expression that describes a relative frequency shift [13].

In the work [14] it was shown that for the effective excitation of FMR in a composite resonator located inside a waveguide the eigen modes of dielectric $E_{\pm 11\delta}$ are most effective and analytical expressions for a resonance frequency shift under an action of field for those dielectric modes were obtained:

$$\frac{\Delta f_r}{f_{r0}} = -(\chi' \pm 0.837 \chi_a') \frac{V_s}{V_c}, \quad (3)$$

where χ' , χ_a' – real parts of the magnetic susceptibility tensor components, Δf_r – a shift of a composite resonator resonance frequency, f_{r0} – a resonance frequency of a composite resonator in the absence of biasing DC magnetic field. Different signs in (3) mean different polarization states of unperturbed mode of resonator, sign “+” corresponds to the eigen mode of $E_{+11\delta}$ type with the right circular polarization, sign “–” corresponds to the $E_{-11\delta}$ mode with the left circular polarization.

Also, in the work [14] it was shown, that the biasing magnetic field H can trigger a change of Q -factor of a resonance in a composite structure and therefore a change of resonance mode linewidth and losses. An expression for a Q -factor of a composite resonance element under an action of external magnetic field:

$$\Delta(1/2Q) = (\chi'' \pm 0.837 \chi_a'') V_s K / V_c, \quad (4)$$

where Q is a Q -factor of a composite structure, $K \approx 1$ is a dimensionless coefficient. Unlike the expression (3), this formula contains imaginary parts of magnetic susceptibility tensor components. So, if a dielectric part doesn't make significant energy losses [13] a significant Q -factor and a resonance linewidth of corresponding mode change, controlled by magnetic field, is expected.

The components of the magnetic susceptibility are the functions of an internal field $H_B = \gamma(H_0 - 4\pi M)$, where M is a magnetization of a ferrite, moreover, all the components of the magnetic susceptibility χ' , χ'' , χ_a' , χ_a'' are proportional to magnetization. So it comes out that a frequency shift $\Delta f_r / f_{r0}$ is proportional to the ratio of volumes V_s / V_c of constituents and a magnetization of a ferrite. Due to a dependency of the microwave magnetic susceptibility on magnetic field and the ferromagnetic resonance (FMR) influence this shift is controlled by a change of a magnetic field value H_0 .

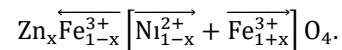
We believe that it is possible to find the optimal ratio between sizes volumes of DR and ferrimagnetic film, which will provide on the one hand a relatively high electric Q -factor of a composite resonator and on the other hand a change of resonance frequency due to a magnetic field influence.

Another consequence of the ferrite influence on properties of a composite resonator is a non-reciprocity of electrophysical properties in relation to a direction of electromagnetic field passage through a structure (isolation). A value of an isolation (R) for a pure ferrite resonator according to [15] equals to the square of a relation of a frequency of operation to a FMR linewidth in a ferrite

$$R_{\max} \approx \left(\frac{4\omega}{\gamma_{\text{eff}} \Delta H} \right)^2, \quad (5)$$

where γ_{eff} is a gyromagnetic ratio of a sample (effective). Non-reciprocity of characteristics is conditioned by the properties of a ferrite only and dielectric just strengthens an interaction of a ferrite constituent with an electromagnetic wave travelling in a waveguide, therefore it can be assumed that the expression remains the same on the qualitative level.

Solid solutions of Ni-Zn ferrites with a high saturation magnetization were chosen for the magnetic phase. It is stipulated by the fact that solid solutions form a structure of the reverse spinel that can be presented in the following way [16, 17]:



If you denote the magnetic moment of the ion Ni^{2+} as m then the magnetization of a one ferrite molecule in the x range of 0-0.5 with the good precision equals to

$$M_s = 10x + (1-x)m. \quad (6)$$

In this case the maximal magnetization equals to the magnetization of an iron ion, namely 5 Bohr magnetons (μ_B) [16].

The synthesis of solid solutions of nickel-zinc ferrites $\text{Ni}_{1-x}\text{Zn}_x\text{Fe}_2\text{O}_4$ was carried out by the precipitation from aqueous solutions method. The study of magnetic properties showed, that the largest magnetization value of the particles take place at $x = 0.5$, the average size of particles was 50 nm (fig. 2).

Method of film applying to a dielectric resonator and a choice of the optimal film thickness (near 100 μm) were determined in the work [8].

The study in the microwave range (10 GHz) showed that the samples of BaTi_4O_9 ceramics are characterized by the value of the dielectric constant of 36, the value of Q of 35000-40000 and the temperature coefficient $\tau_e = -3 \cdot 10^{-5} \text{ K}^{-1}$. After the introduction of zinc oxide the value of the dielectric constant slightly decreases ($\epsilon = 34$), the Q -factor increases ($Q = 70000$) and the thermal stability improves ($\tau_e \sim 10^{-6} \text{ K}^{-1}$). Properties of $\text{BaZn}_2\text{Ti}_4\text{O}_{11}$ were investigated in the microwave range to clarify the nature of zinc oxide influence on properties of BaTi_4O_9 . It was shown that $\text{BaZn}_2\text{Ti}_4\text{O}_{11}$ is characterized by the parameters $\epsilon \sim 20$, $\tau_e = +2 \cdot 10^{-5} \text{ K}^{-1}$ and $Q \geq 9000$ at the frequency near to 10 GHz. This data indicate that after the introduction of zinc oxide into barium tetratitanate the ($\text{BaZn}_2\text{Ti}_4\text{O}_{11}$) phase with a higher Q -factor, compared to BaTi_4O_9 , and with a positive τ_e appears. This change provides the Q -factor increase for 2-phase materials and the increase of properties thermal stability due to the effect of volumetric thermal compensation. Moreover, changing the concentration of ZnO in the $\text{BaTi}_4\text{O}_9 - \text{ZnO}$ system we can change the value of τ_e from negative to positive values.

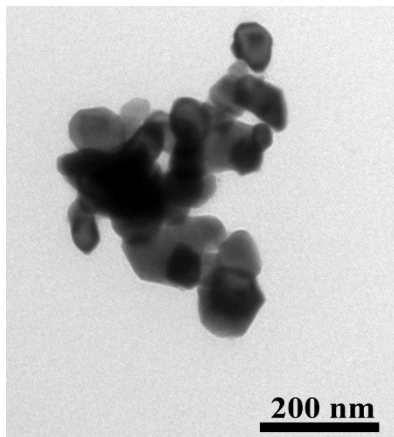


Fig. 2. TEM microphotographs of $\text{Ni}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$ particles.

It is important to note that a ferrimagnetic film applied to the end face of a resonator make a moderate impact on the Q -factor of it. At the same time the value of frequency shift due to the influence of magnetic field in the range of 0-3500 Oe reached the value of 71 MHz at the frequency around 11 GHz (fig. 3).

The investigation of composite resonant elements in the waveguide have shown that elements have a strong non-reciprocity of electrophysical properties, in particular the difference of losses for “direct” and “reverse” directions of passing wave reached $\sim 50 \text{ dB}$. This fact indicates that such resonators can be used to develop microwave devices with magnetic field controlled parameters. However, although high

values of the isolation were reached the “direct” losses in some cases reached the value of 10 dB, at the same time for practical applications this value must not be higher than 1.5 dB. Therefore, the simulation of the composite resonant element used as a non-reciprocal field controlled constituent in a microwave waveguide was made using the software ANSYS HFSS (fig. 4) to determine the possibility to obtain necessary characteristics. The results of the simulation showed that it is possible to reach the value of “direct” losses of 0.8 dB for the $H_{01\delta}$ mode, at the same time the reverse losses were 34 dB. As it can be seen from the fig. 4 due to the influence of the dielectric resonator the magnetic field concentrates inside and near the resonator and the strength of field in the location of ferrite film is in approximately 8 times higher comparatively to the rest of waveguide. Thus, a dielectric resonator increases the effectiveness of the interaction between a ferrite film and the electromagnetic field of waveguide mode and also strengthens non-reciprocity of properties.

As the simulation showed, the optimal position of a composite resonator in a waveguide under given conditions is near the thin wall of a waveguide. In summary, if a high-quality ferrite film is applied to a DR it is indeed possible to reach the high values of non-reciprocity in resonant elements and low values of energy losses for waves passing in the appropriate direction.

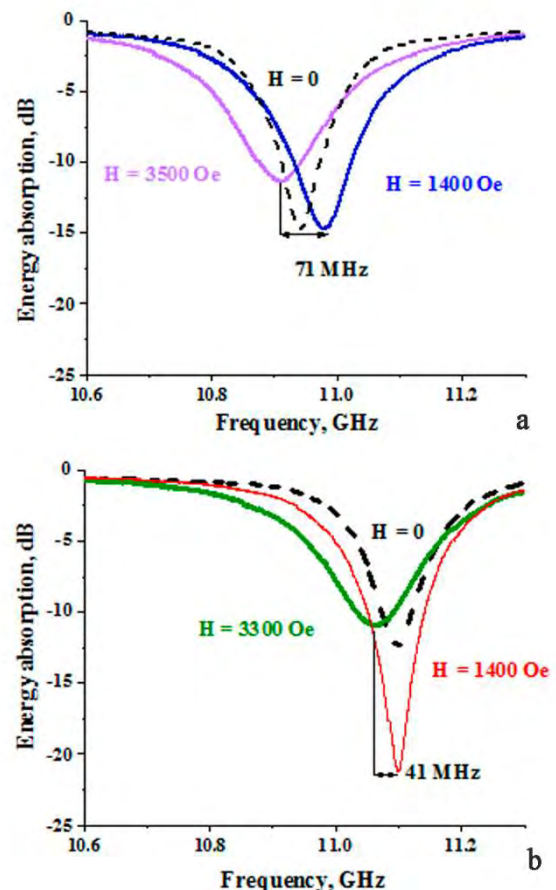


Fig. 3. Resonance frequencies shifts on the energy absorption spectra for the composite resonators with ferrite films polymerized by the normal magnetic field (a) and the parallel magnetic field (b) relative to the resonator axis.

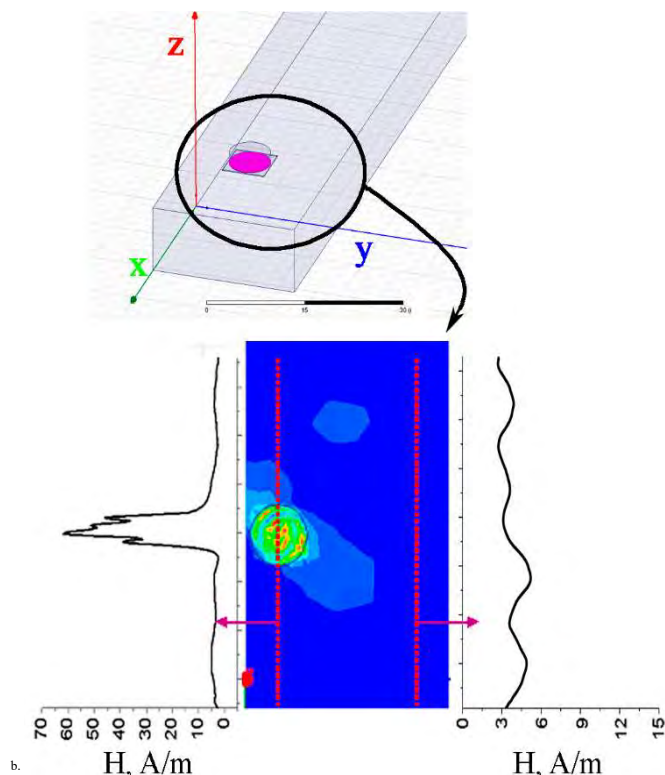


Fig. 4. The image of the X-band waveguide with the composite resonator geometry for the simulation where the purple area is the ferrite film (fig. a); the projection of the magnetic field strength distribution on the plane passing through the ferrite film (fig. b, middle) and field distribution along symmetrically located lines passing through the ferrite film (fig. b, left) and through the empty volume of the waveguide (fig. b., right).

IV. CONCLUSION

The possibility of the creation of multiphase high- Q microwave dielectrics is demonstrated. On the example of the $\text{BaTi}_4\text{O}_9\text{--ZnO}$ system it was shown that the increase of the Q -factor in multiphase systems can occur due to the presence of additional phase, which is characterized by a higher Q -factor comparing to the main phase. The improvement of the thermal stability of electrophysical properties of multiphase systems occurs due to the volumetric thermocompensation effect. Composite dielectric resonators on the basis of high- Q materials and ferrimagnetic field with the resonance frequency, controlled by the magnetic field, are developed.

Obtained composite resonant elements can be used in non-reciprocal components of waveguide and microstrip lines of microwave technics, cascades of filters, X-band isolators, field controlled superheterodyne transmitters etc.

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