COMPOSITE HIGH-Q MICROWAVE ELEMENTS WITH RESONANCE FREQUENCY CONTROLLED BY SPINEL-TYPE FILM

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Technologies are moving away from the use of monomaterial components in favor of more functional composites capable of changing properties under external influences. Earlier we produced composite resonant elements based on high-Q dielectrics and ferrite films, capable of changing the resonant frequency and peak absorption under the influence of a magnetic field. Microwave dielectric materials based on non-magnetic substances exhibit a high Q-factor but lack nonlinearity of characteristics. On the other hand, magnetic materials demonstrate nonlinearity but possess a low Q-factor compared with non-magnetic dielectrics. Thus, the creation of composite elements that combine a high Q-factor with the ability to modify characteristics under the influence of an external magnetic field appears intriguing.

Although the further improvement of dielectric materials is difficult, it would be justified to work on improving the properties of the ferrite films. In order to acquire films of exceptional quality with non-linear characteristics, the development of high-quality dense materials becomes crucial. Therefore, an important technical challenge is to develop a synthesis method that yields weakly agglomerated small nanoparticles.

In industrial settings, ferrites are typically synthesized through solid-phase methods, which provide mechanical homogeneity but lack chemical homogeneity at the molecular level. Moreover, this synthesis approach requires high processing temperatures. To increase the chemical homogeneity of the synthesized materials, precipitation from aqueous solutions is often used due to the large volumes of the product obtained. This method involves the use of aqueous solutions containing salts and a precipitant. During precipitation, amorphous precipitates form, which are challenging to wash and filter from the mother liquor. Subsequently, these precipitates can lead to the formation of strongly agglomerated products during heat treatment.

Among all existing methods, thermal decomposition can be singled out. Decomposition at high temperatures gives the system enough thermal energy to anneal defects, which leads to the formation of almost monodisperse small particles.

This study is aimed at obtaining small and loosely agglomerated nanoparticles of zinc-nickel ferrite using the stearate decomposition method. A comparison will be made between the temperature of nanoparticle formation during the synthesis of stearate decomposition and the co-precipitation deposition from aqueous solutions. The influence of the synthesis method on the magnetization properties will also be investigated. Zinc-nickel ferrites were chosen due to the high saturation magnetization and low coercive force, which will allow to increase the changes in the properties of the composite resonators under the influence of the field and reduce the losses due to remagnetization.

$Zn_xNi_{1-x}Fe_2O_4$ materials were synthesized by stearate decomposition method. The solutions of $Ni(NO_3)_2$, $Zn(NO_3)_2$, and $Fe(NO_3)_3$ were used as initial reagents. The precipitant was an aqueous-ethanol solution of sodium stearate. After precipitation of all the components,

the suspension was heated to 80°C for 1 hour. The precipitate obtained was filtered off from the mother liquor and washed on the filter. The resulting product was dried in an oven in a cuvette at the temperature of 110–120 °C. The final product was obtained after heat treatment of the precipitate in a muffle furnace with an automatic program control unit in an air atmosphere at temperatures of 600 °C, 700 °C, 800 °C, and 850 °C for two hours.

With increasing air temperature, the metal stearates decompose, resulting in the formation of metal oxides and carbon dioxide. It was observed that the formation of the single-phase product $Zn_xNi_{1-x}Fe_2O_4$ occurs at temperatures above 450 °C. The XRD diffraction patterns obtained after the 450 °C heat treatment display diffraction peaks corresponding to $Zn_{0.4}Ni_{0.6}Fe_2O_4$ with a spinel structure, without any other observable diffraction lines. The sharp peaks indicate well-crystallized grains following sintering at 600 °C. Consequently, the stearate decomposition synthesis of $Zn_xNi_{1-x}Fe_2O_4$ particles enables a reduction in the treatment temperatures (by 200 °C compared to co-precipitated ferrites).

It was discovered that the distribution of cations in the sub-lattice is random. Fe^{3+} ions in the tetrahedral position exhibit a less pronounced ultrafine field and isomeric shift compared to Fe^{3+} ions in the octahedral field of ligands. The Fe^{3+} ion has a higher spin compared to Zn^{2+}. Consequently, as the concentration of Zn increases, the ultrafine values decrease due to the presence of Zn as the nearest neighbor of Fe. The magnetic properties of powders with varying compositions, synthesized at 800°C (and at 600°C for stearate decomposition synthesis), were determined using a vibrating magnetometer. All samples exhibit magnetically soft behavior. The magnetic properties undergo changes with increasing Zn concentration. The highest saturation magnetization value was observed for the $Zn_{0.4}Ni_{0.6}Fe_2O_4$ ferrite synthesized via the stearate decomposition method after heat treatment at 800°C. The particles in the obtained powders exhibit high saturation magnetization (M_s) of 45.6 Am^2/kg for stearate decomposition synthesis and M_s = 44.8 Am^2/kg for co-precipitated samples. The coercive force (H_c) is low, with values of 1.3 kA/m for stearate decomposition synthesis and 3 kA/m for co-precipitated samples. Although the characteristics of particles synthesized from stearate decomposition appear slightly better, the difference in magnetization falls within the margin of error.

The study of the microstructure of the synthesized zinc-nickel spinel films was carried out on a scanning electron microscope SEC miniSEM SNE 4500MB. To estimate the density of the films from the data of electron microscopy, the surface degree of filling was calculated, for which the image was binarized, an adequate threshold for identification and the ratio of the film area to the total area were determined. Films produced by employing particles derived from stearate decomposition displayed higher density compared to films utilizing particles obtained through the coprecipitation method.

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