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Effect of a Temperature Mode of Radiation-thermal Sintering the Structure and Magnetic Properties of Mn-Zn-ferrites

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2000NM Mn-Zn-ferrites have been produced by radiation-thermal sintering (RTS). We have studied the effect of RTS on the electromagnetic properties of ferrites. X-ray diffraction have been used to investigate general aspects of phase transformations during the radiation-thermal sintering of green compacts.

Keywords: Ferrite ceramic, Radiation-thermal sintering, Soft-magnetic ferrites, Microstructure.

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1. INTRODUCTION

Soft-magnetic ferrites are widely used in radioelectronics and instrumentation, high density magnetic recording, data storage, drug delivery. Spinel Mn-Zn-ferrite is one of the most important in a practical sense the magnetic oxides [1].

At present industrial production of of ferrites is carried out using the classical ceramic technology [1]. The product of desired shape is compressed from ferrite powder synthesized from a mixture of starting ferrite components and granulated with binder. Samples were subjected sintered in a furnace at temperatures from 900 to 1500 °C in air or a special gas atmosphere. The main drawback of traditional technology is a high energy intensity and duration. In recent years, a growing interest of researchers is a method of radiation-thermal sintering (RTS) – a method comprising heating a mixture of the original components or compacts by high-energy electron beams without the involvement of third-party sources of heat. Advantages PTS are to energy saving, a significant reduction in processing time, the absence of contact of the heated body and a heater heating, the material homogeneity throughout the volume, improved microstructure improved functional properties [2].

In this paper, the technology of the RTS was used for the manufacture of polycrystalline ferrite ceramic material $MnZnFe_2O_4$. The magnetic and dielectric properties and microstructure of ferrite were investigated.

2. EXPERIMENTAL SECTION

The raw material for to produce the method of RTS ceramic material $MnZnFe_2O_4$ used mixtures of the starting oxides Fe_2O_3, MnO, ZnO analytical grade brand. Oxides milled in a vibratory mill M-200 for 2 hours. The obtained mixture was calcined in a furnace with a rotating-tube "Granula" at a temperature of 960 °C for 5 hours to obtain of producing a ferrite powder. The synthesized powder was pulverized for 2 hours in the M-200 vibratory mill. Before milling, bismuth oxide mechanically activated in an APF-3 planetary mill was added to the synthesized powder in order to activate the sintering process. To the milled mixture was added a binder in the form of a 10 % polyvinyl alcohol solution (10 wt %) and triethanolammonium citrate (0.1-0.4 wt %). The mixture was then granulated by rubbing it through 0.500 and 0.315 mm sieves. Triethanolammonium citrate was added to the binder as a surface-active substance (SAS) in order to raise the density of green compact. The granulated powder was pressed at 200 MPa into ring green compact dimensions $D = 16$ mm, $d = 7$ mm, $h = 6$ mm. After drying to a relative humidity below 0.5 wt %, the green compacts were placed in specially designed RTS cell and exposed to fast electrons (electron accelerator ILU-6, electron energy 2,5 MeV, the operating frequency of the resonator 117 MHz, the maximum pulse current of beam 450 mA, the pulse repetition frequency to 50 Hz, beam current pulse duration 0.5 ms). The temperature of the samples during heat treatment was monitored with a Pt/Pt-Rh thermocouple. The special software used to control the rate of heating and cooling, with the possibility of exposure to a predetermined temperature for a total processing time. To prevent the thermocouple signal from being distorted by the electron beam, a third, platinum electrode was used, with one end welded to the working junction and the other grounded. During the processing of different batches of samples were heated to a temperature of 1000 °C, 1100 °C, 1200 °C and 1300 °C respectively and maintained at the desired temperature for 60 minutes. As a result, we've got 5 ring samples sintered at different temperatures.

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The density of the samples was determined by measuring their mass and volume. The measurement of the magnetic characteristics of soft magnetic material ring samples was determined by using measurement apparatus MK-3E by a ballistic method. The constant magnetic field is 800 A/m. The measurements were performed at room temperature of 25 °C.

The elemental composition of all the samples was determined by electron probe X-ray microanalysis on a JEOL JSM 7800F (Japan) equipped with a PGT IMIX image analysis system (accelerating voltage 20 kV, counting time per data point 90 s, analyses at three or four points and averaging of the data).

The phase composition of bulk samples was determined by X-ray diffraction on DRON-3M (CuKα radiation, graphite monochromator) and Rigaku Geigerflex diffractometers. In the latter case, the X-ray source used was an iron anode X-ray tube (working current 25 mA, voltage 25 kV, X-ray wavelength 0.193728 nm). In intensity data collection, we used an Mn filter. The X-ray beam was focused in Bragg-Brentano geometry with two Soller slits. All the measurements were made at room temperature.

3. RESULTS AND DISCUSSION

Fig. 1 show a typical X-ray diffraction pattern of 2000NM green compact, and Fig. 2 show a typical X-ray diffraction pattern of the 2000NM ferrite prepared by RTS.

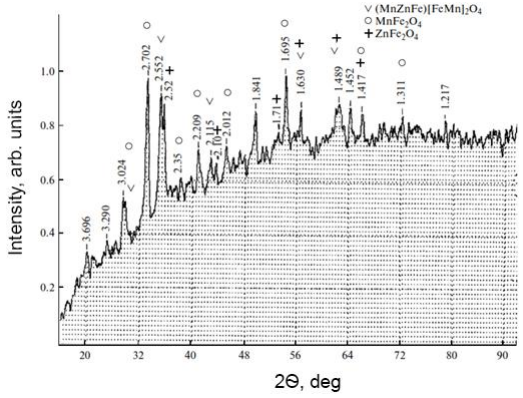

Fig. 1 – Typical X-ray diffraction pattern of a 2000NM green compact

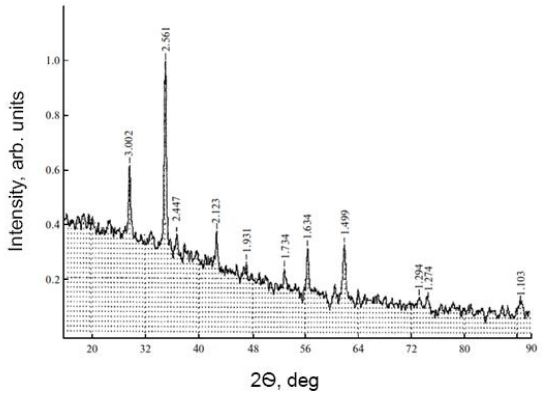

Fig. 2 – Typical X-ray diffraction pattern of a sample 2000NM ferrite obtained by RTS at 1300 °C (single phase (MnZnFe)$[FeMn]_2O_4$)

As seen in Fig. 1, the green compact contains three spinel phase: (MnZnFe)$[FeMn]_2O_4$, $MnFe_2O_4$ and $ZnFe_2O_4$. This is attributable to the decomposition of the ferrite phase after the synthesis in the rotating-tube furnace during cooling in air between 800 and 600 °C. RTS is accompanied by phase transformation, which lead to the formation of a Mn-Zn spinel ferrite, whereas the above three phases disappear (Figs. 2, 3). To prevent decomposition of the Mn-Zn ferrite phase after RTS, cooling from 800 to 600 °C was carried out in an inert atmosphere (nitrogen).

Fig. 3 shows a typical SEM image of the sample marks 2000NM ferrite sintered by radiation-thermal method for 1 hour at temperature 1000 °C.

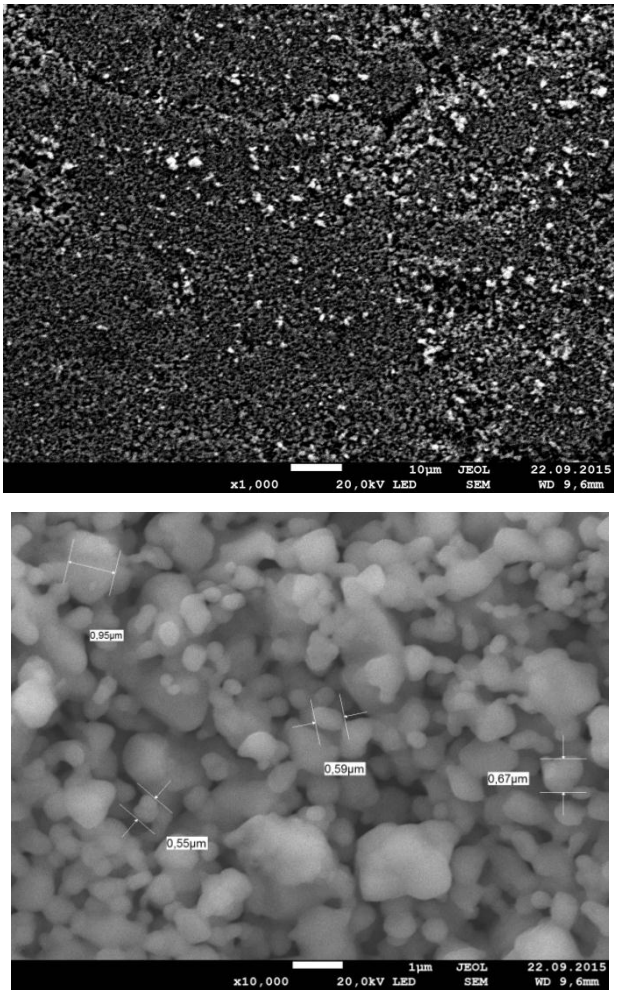

Fig. 3 – SEM image of the sample Mn-Zn-ferrite obtained by RTS at 1000 °C

Fig. 4 shows a typical SEM image of the sample marks 2000NM ferrite sintered by radiation-thermal method for 1 hour at temperature 1300 °C.

It is known that most electromagnetic ferrites parameters (permeability, induction, coercive force, hysteresis loss and eddy currents, etc.) is structurally sensitive, i.e. depend on the structure of the sintered article [2]. Structure of sintered body as a dispersion system is determined by the size and number of pores, nonmetallic inclusions, grain size and shape. It is formed during sintering and largely determined by the quality and structure of the green compact obtained by molding.

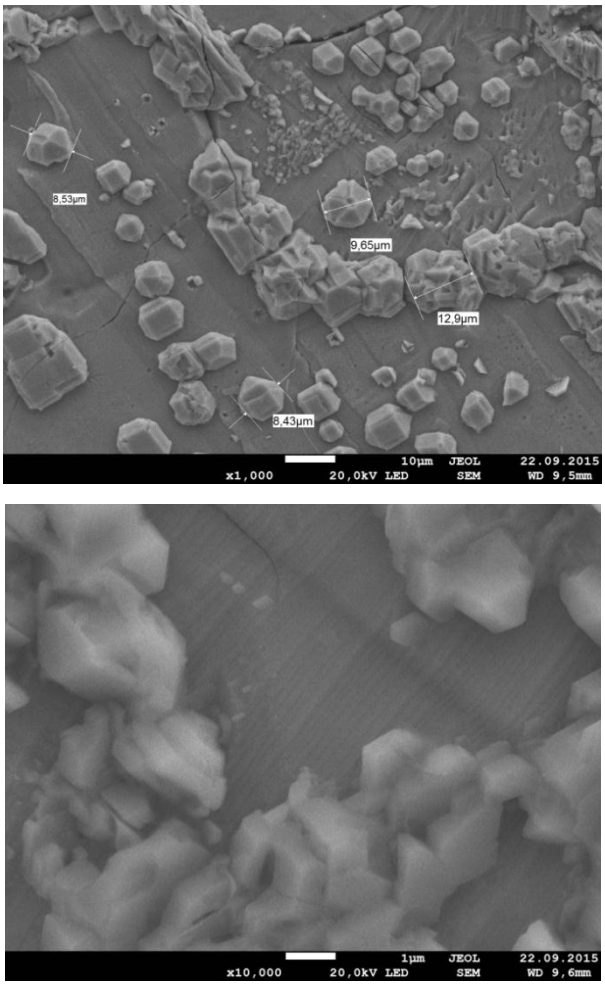

Fig. 4 – SEM image of the sample Mn-Zn-ferrite obtained by RTS at 1300 °C

Microstructure parameters also determine the shape of the hysteresis loop. In particular, Mn-Zn-ferrite with relatively fine grains are typically "tilted" hysteresis loop, and Mn-Zn-ferrite with coarser grains are of a conventional S-shaped, which defines the field of use. The first ferrites have a low residual induction, they are used as the reel hubs filtering circuit connection, while the latter have a large value of the residual induction and smaller hysteresis loss is used in power transformers with a working frequency of 25 kHz. Fig. 6 shows the hysteresis loops at different temperatures radiation heat sintering. We see that sintering at 1300 °C, the hysteresis loop has an

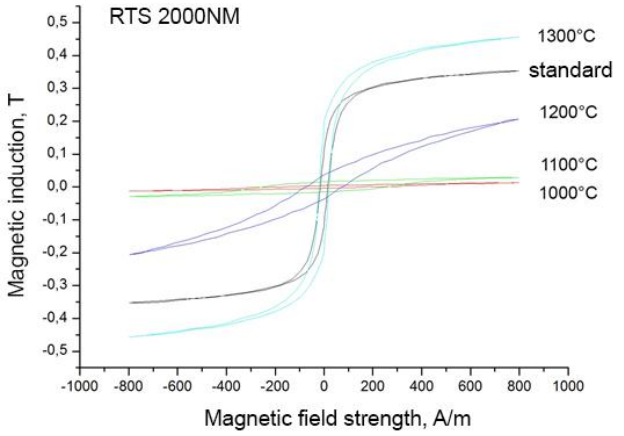

Fig. 5 – The hysteresis loops sample of Mn-Zn-ferrite obtained by RTS

S-shape, whereas the sintering at 1000 °C have a "tilted" loop.

Different temperature modes of radiation-thermal sintering influence the magnetic characteristics of Mn-Zn-ferrite brand 2000NM. Most best results were obtained at 1300 °C. This can be seen when viewing the hysteresis loop (Fig. 5) and in the study of the magnetic characteristics of the sintered samples (Table 1). The standard sample is marked with the classic ceramic technology.

4. CONCLUSIONS

Studies have confirmed the effectiveness of radiation and thermal sintering of Mn-Zn-ferrite brand 2000NM to work in strong fields.

Studies of the electromagnetic characteristics of the samples sintered Mn-Zn-ferrites showed most best results are obtained by sintering 1300 °C for 1 hour in a beam of accelerated electrons. It is caused by the difference in the grain sizes and shapes, and the difference in pore size and number of samples sintered at 1000 °C and 1300 °C.

Radiation-thermal sintering ferrite ceramic can be an alternative technology produce soft magnetic ferrite ceramic against ceramic classic. Demonstrate the economic benefits of such technology [4].

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Table 1 – Magnetic characteristics of the Mn-Zn-ferrites brand 2000NM

Characteristics	The sintering temperature of radiation-thermal method				Classical method
	1000 °C	1100 °C	1200 °C	1300 °C	standard
The initial magnetic permeability	2.7	–	2070	538	2000
Maximum magnetic permeability	14.3	–	255	3728	3500
Coercive force by induction H_c (A/m)	187	253	76	21	24
Residual magnetic induction, B_r (T)	0.004	0.02	0.04	0.2	0.13
Rectangular loop	0.30	0.53	0.18	0.44	0.32

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