

TWO-COMPONENT FERRIMAGNETIC CERAMICS SYNTHESIZED IN A SOLAR FURNACE

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<https://doi.org/10.5281/zenodo.17539017>

Abstract. *The iron oxide and calcium phosphate components obtained in the required proportions are mixed and ground in a ball mill for 5-8 hours, then dehydrated and dried at 300°C, mixed with the addition of 6-10% PVS to the mass, blocked and dried in a MS-100 compaction unit with a force of 30 kN, melted in a large solar furnace, cooled with water, ground to a particle size of 20 μm, dehydrated, mixed with the addition of 6-10% PVS to the mass, molded in a MS-100 compaction unit with a force of 30 kN, dried at 300°C, fired in a selite furnace at 1280°C for 15-16 hours and cooled at 20-40°C for 4-5 hours. The results show that ferrimagnetic ceramics with the components $\text{Ca}_3(\text{PO}_4)_2$ and Fe_2O_3 form phases $\text{Fe}_{2,939}\text{O}_4$, CaFe_2O_4 , $\text{Ca}_{28,8}\text{Fe}_{3,2}(\text{PO}_4)_{21}\text{O}_{0,5}$, the particle size is less than 20 μm, the presence of Ca, P, O, Fe elements, the Curie temperature is 740 K and the resistivity is $3.4 \cdot 10^9 \text{ Ohm} \cdot \text{m}$.*

Keywords: *iron (III) oxide (Fe_2O_3), calcium phosphate ($\text{Ca}_3(\text{PO}_4)_2$), mixing, grinding, structural properties, magnetic properties, magnetite, magnetization, solar technology, flux density, resistivity, synthesis, quenching, Large Solar Furnace, cell parameters, singony, particle size, Curie temperature.*

INTRODUCTION

Ferrites are ferrimagnetic ceramics that combine high magnetic properties and high resistivity, and consequently, low losses for eddy currents, which allows them to be used in UHF and microwave fields, i.e., where metal-soft materials cannot be used anymore. This is an important advantage of ferrites over other magnetic materials. Ferrites are complex systems of iron and divalent (rarely monovalent) metal oxides, having the general formula MeOFe_2O_3 . The ions of metal oxides: Ni^{4+} , Mn^{2+} , Co^{2+} , Fe^{2+} , Zn^{2+} , Cd^{2+} , Li^+ and others give the name ferrite. For example, $\text{NiO Fe}_2\text{O}_3$ is nickel ferrite, $\text{ZnO Fe}_2\text{O}_3$ is zinc ferrite. Ferrites used in technology are also called oxyferrites. Recently, ferrites with the general formula $3\text{Me}_2\text{O}_3 \cdot 5\text{Fe}_2\text{O}_3$ (where Me is the ion of a two or three-valent metal) have been widely used.

The properties of ferrites and, accordingly, products from them strongly depend on their composition and production technology. In industry, the simplest technology is used, which consists of the sintering of oxides at high temperature; it consists of the following. Initially, a ferrite powder is prepared, consisting of burned oxides of the corresponding metals, finely ground and thoroughly mixed. A plasticizer (usually a solution of polyvinyl alcohol) is added to the powder, and products of the required shape are pressed from the resulting mass under high pressure and fired at 1100-1400 °C. During the firing process, ferrite, a solid solution of oxides, is formed. At the same time, shrinkage occurs, which can be 10-20%. It is very important that the combustion

occurs in an oxidizing environment (usually in air). The presence of even a small amount of hydrogen can cause partial reduction of oxides, which leads to an increase in magnetic losses. The resulting ferrite products are hard and brittle and do not allow for mechanical processing except for grinding and polishing [12].

The magnetic and physical properties of ferrite depend on the methodology adopted for its production [1]. Usually, ferrites are produced by firing ceramics, which includes high-temperature reactions between their constituent oxides or carbonates [2]. Chemical methods such as sol-gel [3], citrate-nitrate precursor method [4], microwave annealing [5], chemical co-precipitation method [6] and others are also used. The magnetic properties are also influenced by the stoichiometric composition of nickel-zinc ferrite $\text{Ni}_{1-x}\text{Zn}_x\text{Fe}_2\text{O}_4$ [7]. Jun Hu and Mi Yang [10] noted that the addition of CuO and V_2O_5 during the preparation of NiZn ferrite reduces the granularity of the raw material and improves the magnetic properties of the ferrite obtained at low firing temperatures in the temperature range of 930-1200 °C. Additives such as Cu [11], Mn [10], and others have been reported to affect the magnetic properties of NiX ferrite.

Ferrites have a face-centered, densely packed cubic lattice, in which oxygen ions form both tetrahedra and octahedra, which also participate in the formation of magnetic properties. In the center of the tetrahedron, there is a metal ion. If this ion is Fe^{3+} , then the material has magnetic properties, for example, nickel (NiOFe_2O_3) and manganese (MnOFe_2O_3) ferrites. If this ion is Zn^{2+} or Cd^{2+} , then magnetic properties are absent, and a non-magnetic ferrite is formed, for example, zinc (ZnOFe_2O_3) or cadmium (CdOFe_2O_3). These phenomena are explained by the fact that in ferrites, an indirect exchange interaction occurs between the magnetic moments of neighboring atoms, which leads to their antiparallel orientation. In this regard, the crystal lattice of ferrites can be represented as consisting in a magnetic ratio of two sublattices with opposite directions of magnetic moments of ions (atoms). In magnetic ferrite, the magnetization of the sublattices is not the same, therefore, a total spontaneous magnetization arises, and in non-magnetic ferrite, the total magnetization is equal to zero.

Technical ferrites are, as a rule, solid solutions of magnetic and non-magnetic ferrites. Magnetically soft ferrites primarily include two groups of ferrites: nickel-zinc and manganese-zinc, representing three-component systems $\text{NiO} - \text{ZnO} - \text{Fe}_2\text{O}_3$ and $\text{MnO} - \text{ZnO} - \text{Fe}_2\text{O}_3$. Non-magnetic ferrites are added to magnetic ones to increase magnetic permeability and reduce the coercive force. However, at the same time, the Curie temperature decreases [12].

The creation of new materials based on calcium phosphate ($\text{Ca}_3(\text{PO}_4)_2$) and iron (III) oxide (Fe_2O_3) has undergone significant changes in recent decades due to technological progress and the development of computational methods. The purpose of this work is to study the structure and properties of two-component ($\text{Ca}_3(\text{PO}_4)_2$ and Fe_2O_3) ferrimagnetic ceramics synthesized in a solar furnace with a flux density of 200 W/cm^2 [8-9].

MATERIALS AND METHODS

Solar technologies, unlike traditional ones, are characterized by a high heating rate, allowing for the production of finely dispersed homogeneous material of high chemical purity. At the same time, the ferritization process occurs simultaneously in ferrite materials. Such features determine the possibilities of creating improved ceramics, composites of intermetallic compositions, and functional-gradient materials. The stoichiometric quantities of the component, calculated in molar percentages, and the composition were subjected to wet mixing and grinding to a fineness of 63 μm in a ball mill coated with water-resistant and wear-resistant material. The mixing and grinding process takes 5-8 hours, taking into account the ratio of raw materials, water,

and wear-resistant stones of 1.0:1.2:1.2. The resulting suspension is dehydrated and dried in drying cabinets (Snol) at a temperature of 300°C. Then, the dried mass is moistened with 6-10% PVS (polyvinyl alcohol), and then formed as a block with a force of 30 kN on a MS-100 press. The blocks are dried in a drying cabinet or outdoors at room temperature. Block molds are melted in the Large Solar Furnace at a concentrated flow density of 200 W/cm², and the melted mass is cooled using the water quenching method. The grinding of the hardened cake is carried out in a ball mill until the material particles reach a size of 20 μm and then dehydrated. The resulting masses are mixed with a 6-10% PVS solution (polyvinyl alcohol), the mixture is poured into a mold and pressed on a MS-100 press with a force of 30 kN and dried at 300°C. The material is fired in a silicon furnace for 15-16 hours at a temperature of 1280°C and cooled to a temperature of 20-40°C for 4-5 hours [8-9].

Methods for studying the properties of the obtained ferrimagnetic ceramics were used: scanning electron microscope, X-ray diffraction, magnetic measurements (the method allows studying the dependence of specific magnetization and magnetic susceptibility on temperature in small amounts of substances), resistance measurement.

RESULTS

Figure 1 shows a radiograph (RF) for optimal composition. Numerous distinct peaks are observed on it, indicating the presence of a crystalline structure.

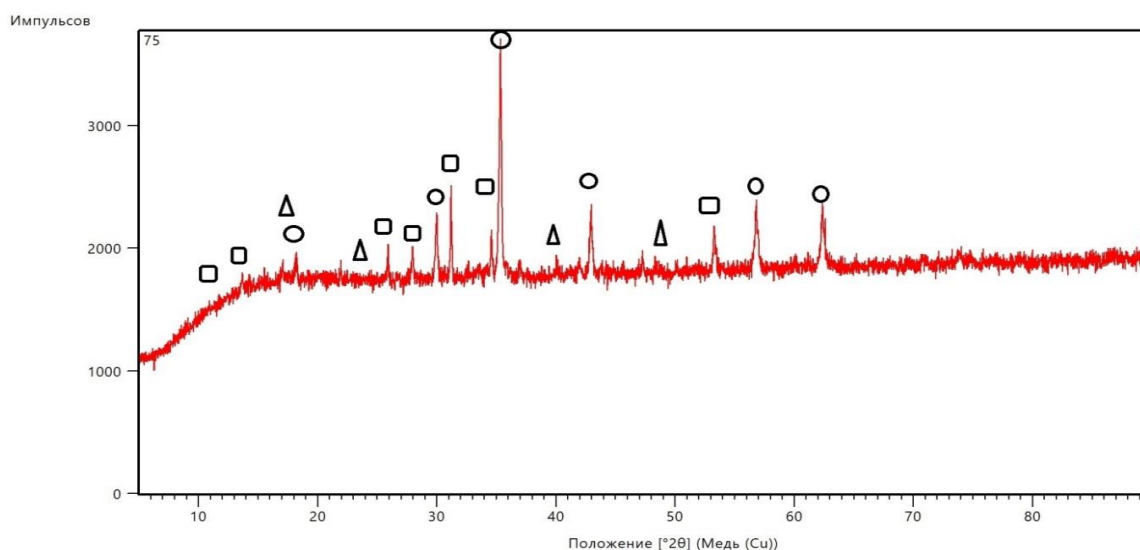


Figure 1. X-ray structural analysis graph (○ - $\text{Fe}_{2.939}\text{O}_4$, Δ - CaFe_2O_4 , □ - $\text{Ca}_{28.8}\text{Fe}_{3.2}(\text{PO}_4)_{21}\text{O}_{0.5}$).

The following phases were formed:

- magnetite (spherical), empirical and chemical formula $\text{Fe}_{2.939}\text{O}_4$, cubic crystal system, cell parameters a (Å): 10.4352, b (Å): 10.4352, c (Å): 37.4029;
- calcium and iron oxide (rectangular-triangular), empirical formula CaFe_2O_4 and chemical formula $\text{Ca}(\text{Fe}_2\text{O}_4)$, orthorhombic crystal system, cell parameters a (Å): 9.4052, b (Å): 9.4052, c (Å): 6.8813;
- calcium and iron phosphate (tetrahedral), empirical formula $\text{Ca}_{28.8}\text{Fe}_{3.2}\text{O}_{84.5}\text{P}_{21}$, chemical formula $\text{Ca}_{28.8}\text{Fe}_{3.2}(\text{PO}_4)_{21}\text{O}_{0.5}$, hexagonal syngony, cell parameters a (Å): 12.8870, b (Å): 27.2800, c (Å): 15.2190. The diffraction angle of X-rays is 2θ.

The image obtained using a scanning electron microscope is shown in Figure 2.

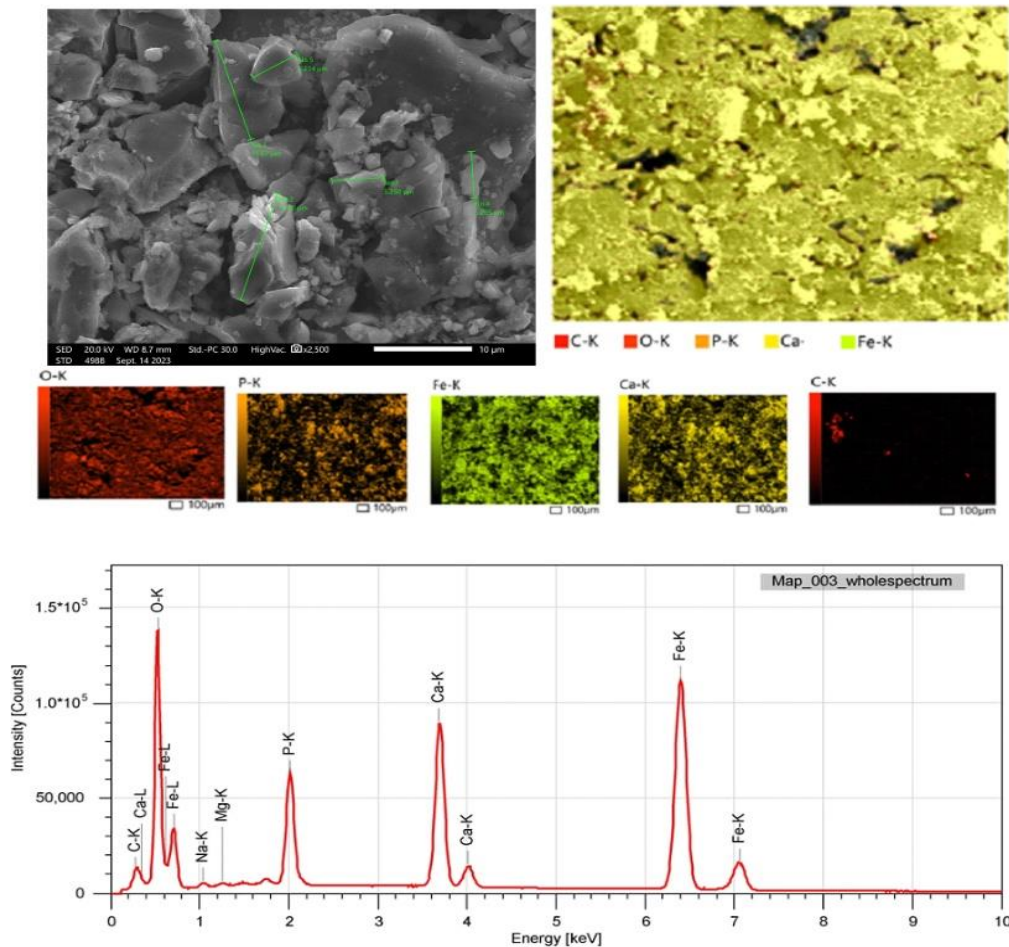


Figure 2. Scanning electron microscope graph

Increase $\times 2500$, permissible particle size $10\ \mu\text{m}$, particle size $5.250\ \mu\text{m}$, $2.414\ \mu\text{m}$, $1.167\ \mu\text{m}$, $0.825\ \mu\text{m}$, $0.734\ \mu\text{m}$, main peaks belong to P, Fe, Ca elements and have intensities from 2 to 6.4 keV. The graph shows the distribution of elements confirming the presence of O, P, Ca, and Fe. These peaks indicate the composition of the sample and its chemical environment.

Figure 3 shows an image obtained using magnetic measurements.

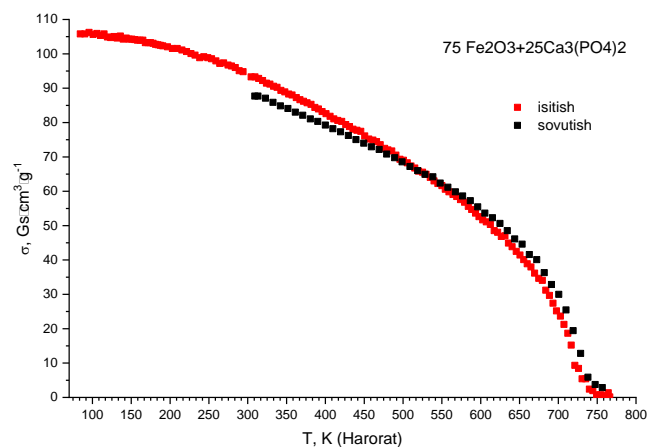


Figure 3. Magnetization-temperature dependence graph for ferrimagnetic ceramics.

If we analyze the graph of the sample in Figure 3, the magnetization is high at low temperatures and gradually decreases with increasing temperature. With an increase in temperature to 740 K, the magnetization sharply decreases and approaches 0. This corresponds to the material's

magnetization disappearance temperature, i.e., the Curie point (740 K). The maximum magnetization value is 105. Consequently, at low temperatures, the material becomes ferromagnetic, and at high temperatures, paramagnetic. The electrical resistance of ferrimagnetic ceramics was measured using the volt-ampere characteristic method.

CONCLUSION

Summarizing the above results, it can be seen that ferrimagnetic ceramics with components $\text{Ca}_3(\text{PO}_4)_2$ and Fe_2O_3 form phases $\text{Fe}_{2,939}\text{O}_4$, CaFe_2O_4 , $\text{Ca}_{28,8}\text{Fe}_{3,2}(\text{PO}_4)_{21}\text{O}_{0,5}$, the grain size is less than 20 μm , the content of Ca, P, O, Fe, the Curie temperature is 740 K, and the resistivity is $3.4 \cdot 10^9 \text{ Ohm} \cdot \text{m}$.

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