

PHASE COMPOSITION OF CERAMICS OBTAINED BY APPLYING ULTRASONIC VIBRATIONS

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Rapid development of electronics requires using materials of high quality. Research on the development of inorganic dielectrics with desired properties has become increasingly important. The important factor in process of obtaining material is the possibility of formation of a required crystal structure, in particular, using ultrasonic mechanoactivation.

BaAl₂Si₂O₈ shows complex polymorphism and crystallizes in monoclinic, orthorhombic and hexagonal structures [1-3]. The physical properties of celsian depend on its crystal structure. For example, a compound with the monoclinic structure is thermodynamically stable in the temperature range of 20-1590°C, whereas the hexagonal phase of celsian is stable up to 1760°C [2].

Low thermal shock resistance that related to the structural transformation of the α - hexagonal modification into the β- hexagonal modification in the 280-320°C temperature range appears to be a drawback of hexacelsian [4].

Monoclinic form does not undergo polymorphic transformations and is characterized by high dielectric and mechanical properties.

The aim of this work is to study the influence of ultrasonic mechanoactivation on crystal structure of BaAl₂Si₂O₈.

BaAl₂Si₂O was obtained by using the oxides of BaCO₃, Al₂O₃, SiO₂ as the initial components in the ratio of 1:1:2. The synthesis was carried out in alundum crucibles in air by solid-state reaction at temperatures of 1300-1450°C, the time of synthesis was 2 h. The synthesized powders were wet-ground in ethanol. The coupling agent (e.g., PVA glue) was added to some parts of synthesized powder after which that then pressed at 100 MPa into pellets. The sintering was carried out at temperatures from 1440°C to 1520°C for 2 h in air atmosphere.

Another part of the powder was ultrasonically treated at normal atmospheric pressure for 0.5-1.5 h using ultrasonic generator UZG 1-1 of 1 kW power and magnetostrictive transducer PMS 1-1. Then the obtained material was pressed at 100 MPa into pellets and sintered at temperatures from 1250°C to 1350°C for 2 h in air atmosphere. The phase composition of the obtained material after processes of synthesis, ultrasonic mechanoactivation and sintering was determined by X-ray diffraction monochromatized CuK_α radiation in the angle range of 20-65°. Dielectric measurements of the samples were made at 100 kHz with an E7-8 bridge in the temperature range of 20-350°C. Open porosity was studied using the optical microscope Olympus GX 41. Processing of the experimental data was performed using the software Autoscan 2500 Studio.

According to X-ray diffraction studies ceramic of hexagonal BaAl₂Si₂O is obtained after synthesis (the presence of the monoclinic modification is recorded only against a background (Fig.1)).

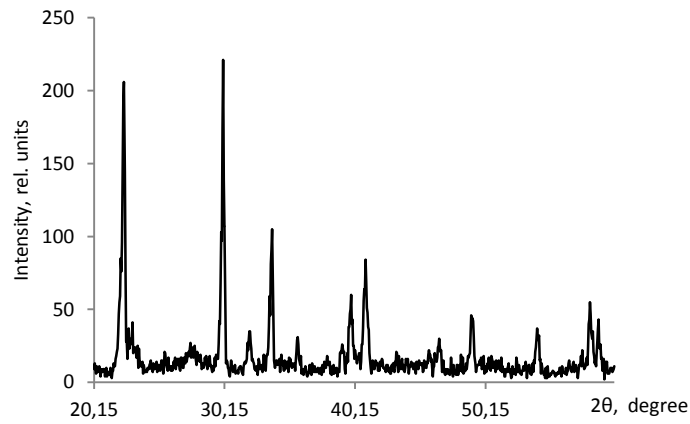


Fig. 1. X-ray diffraction pattern of ceramic samples of $\text{BaAl}_2\text{Si}_2\text{O}$ after synthesis.

The analysis of X-ray diffraction patterns shows that after sintering the crystal structure is similar to the initial for the samples obtained from the synthesized material without mechanoactivation (Fig.1).

It is found that the crystal structure of hexagonal modification was formed at synthesis temperatures of 1300–1450°C. For single-phase ceramic samples of hexagonal $\text{BaAl}_2\text{Si}_2\text{O}$ the temperatures of synthesis and sintering must be 1450°C and 1500°C, respectively.

The study of the samples obtained after mechanoactivation (UVs grinding) and subsequent sintering shows that the influence of ultrasonic vibrations (UVs) for 0.5 h (Fig. 2a) leads to a monoclinic modification. The influence of UVs leads to the increase of crystal structure of the monoclinic modification (Fig. 2b)

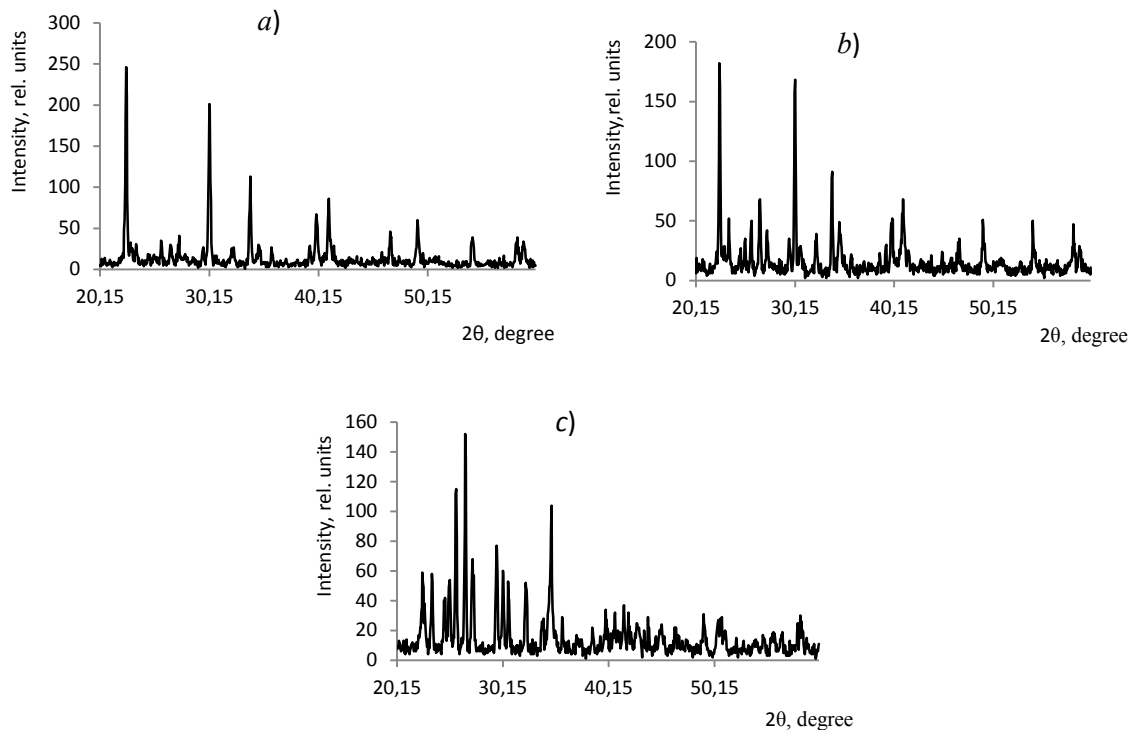


Fig. 2. X-ray diffraction pattern of ceramic sample of hexagonal $\text{BaAl}_2\text{Si}_2\text{O}$ after ultrasonic treatment and subsequent heat treatment: a) UVs treatment for 0.5 h; b) UVs treatment for 1h; c) UVs treatment for 1.5 h.

Further increase in ultrasonic exposure time for 1.5 h results in the formation of monoclinic crystal structure (Fig. 2c).

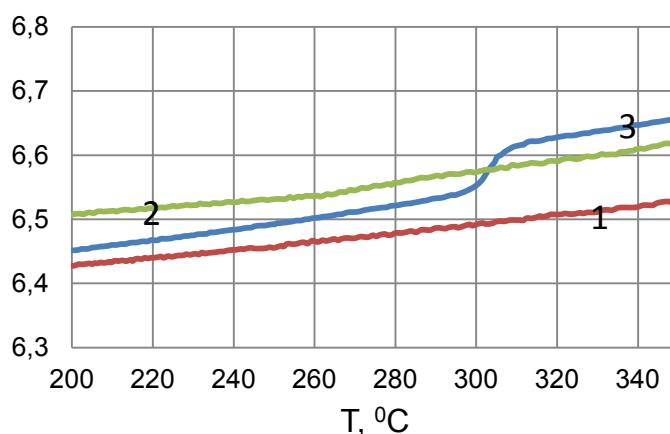


Fig. 3. Temperature dependencies of permittivity of the samples with different modifications of crystal structure: 1 – monoclinic, 2 – mixture of phases, 3 – hexagonal.

The temperature dependence of the dielectric constant of the samples with different modifications of crystal structure shows that in the temperature range from 20°C to 290°C the dielectric behavior does not depend on the modification of crystal structure.

Structural transformations of α - hexagonal into β hexagonal modifications for ceramic samples of $\text{BaAl}_2\text{Si}_2\text{O}$ occurs in the temperature range of 280-320°C that is coherent with the results of DTA analysis [5], where the transition temperature is corresponded to 312°C. Monoclinic form has not polymorphic transformations. The phase transition for the two-phase samples is absent (Fig. 3).

The studies have shown that hexagonal $\text{BaAl}_2\text{Si}_2\text{O}$ is synthesized at temperatures of 1300 - 1450°C. Subsequent ultrasonic treatment of the synthesized $\text{BaAl}_2\text{Si}_2\text{O}$ stimulates polymorphic transformation, i.e. by selecting the ultrasonic treatment regime it is possible to obtain a material of the given modification of crystal structure. Thus, an increase in processing time of UVs up to 1.5 h leads to the formation of the monoclinic-phase structure.

It has been determined that the values of electrical parameters of the sample $\text{BaAl}_2\text{Si}_2\text{O}$ do not depend on the modification of crystal structure. Ceramic materials have low porosity, high Q-factor and have dielectric parameters allowing using these materials for ceramic resonators, and other microwave devices.

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