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TECHNOLOGICAL ASPECTS OF STRENGTHENING RADIO-TRANSPARENT CORDIERITE GLASS-CERAMICS FOR ROCKET TECHNOLOGY

Assoc. Prof. Oleksandra Amelina, PhD

Prof. Olersandr Zaichuk, DcS

Department of Chemical Technologies of Ceramics, Glass and Construction Materials,
Ukrainian State University of Science and Technologies, Ukraine

E-mail: zaychuk_av@ukr.net

Prof. Tsvetan Dimitrov, PhD,

Department of Chemistry, Food and Biotechnology

"Angel Kanchev" University of Ruse, Razgrad Branch,

E-mail: tz_dimitrow@abv.bg

***Abstract:** The article is devoted to the technological aspects of strengthening cordierite ceramics used as nose fairings in rocket equipment. The strengthening was performed by ion exchange in a barium salt melt. The influence of technological factors on the main properties of the strengthened cordierite ceramics for rocket applications has been determined.*

***Keywords:** Cordierite ceramics, Strengthening, Ion exchange.*

INTRODUCTION

The absence of domestic production of missile armaments in Ukraine necessitates the search for technical solutions for the development and manufacturing of missiles of various classes. One of the key components of a missile or any aerial vehicle is the nose radome, which is typically made of ceramic or glass-ceramic materials. Radomes must meet a number of requirements, primarily radio transparency in the radio-frequency range to ensure undistorted signal transmission, which directly affects target-seeking accuracy. In addition, a combination of physical and mechanical properties determines the operational reliability of the radome. Among them, zero water absorption provides high erosion resistance. Thermal stability under rapid heating is ensured by a low thermal coefficient of linear expansion of the ceramic. Low dielectric constants (dielectric permittivity and dielectric loss) determine the high radio transparency of the components (Zanotto, E.D., 2010).

Among the materials used for manufacturing radomes, the most widely known are quartz ceramics and lithium aluminosilicate ceramics, which are used in the seeker heads of guided missiles (air-to-air missiles, anti-radiation missiles, new-generation anti-tank guided missiles). The advantages of quartz ceramics include high thermal shock resistance and low density. However, their mechanical strength is insufficient, and the high porosity of the products requires an additional moisture-proof coating (Sebastian, M.T., 2015). Lithium aluminosilicate ceramics—with spodumene or eucryptite as the primary crystalline phase—are characterized by high mechanical strength and thermal stability. Their drawback is the reduced dielectric performance associated with the presence of lithium oxide in their composition (Zaichuk, A., 2020). Ceramics containing α -cordierite as the main crystalline phase fully meet the requirements for radome materials; however, their mechanical properties can be further improved using ion-exchange treatment.

The authors of (Beall, G.H., 2016) investigated the effect of ion exchange on the mechanical properties of glass-ceramics of various aluminosilicate systems and demonstrated the mechanisms of surface ion substitution inherent to glasses and glass-ceramics. In another study (Lu, J., 2021), materials

with enhanced strength characteristics were obtained by ion-exchange surface treatment of lithium aluminosilicate glass-ceramics in molten potassium nitrate.

Thus, the search for advanced ceramic materials for manufacturing radomes—materials that satisfy a complex set of dielectric, physical-mechanical, and operational requirements, as well as allow technological improvements—is an important and practical engineering challenge.

EXPOSITION

Methods of Investigation

Samples of cordierite-based ceramic materials were produced by slip casting into gypsum molds. The slip was prepared by co-milling, in the wet state, an eutectic glass of the $\text{MgO-Al}_2\text{O}_3\text{-SiO}_2$ system together with enriched kaolin grade Zref-1, magnesium hydroxide, and technical alumina grade G-0, in proportions calculated to ensure complete chemical incorporation of the glass components into cordierite. Firing of the ceramics was carried out in a stepped regime with a maximum temperature of 1350 °C and a holding time of 1 h. The samples were cooled together with the furnace. After firing, the samples were machined to standard dimensions $d = h$.

Surface ion strengthening was performed in molten barium nitrate at 750 °C for 5, 10, and 15 h.

To remove residual nitrate salts from the sample surface, thermal treatment was carried out at 900 °C for 2 h.

Mechanical strength was measured on samples with dimensions $d = h = 10$ mm using a PSU-10 press. Density was determined on samples of the same dimensions by the hydrostatic weighing method. The thermal coefficient of linear expansion (CTE) was measured using a quartz dilatometer on beam-shaped samples of dimensions $5 \times 5 \times 50$ mm.

FTIR spectra of the investigated materials were obtained using a Shimadzu IRSpirit-X spectrometer equipped with an ATR attachment (QATR-S, Specac).

Results and Discussion

The main idea behind increasing the mechanical strength of cordierite ceramics was to generate compressive stresses in the surface layers by substituting magnesium ions with barium ions. For this purpose, samples of cordierite ceramics were held in molten barium nitrate for 5, 10, and 15 hours. To ensure complete removal of nitrate residues, the samples were subsequently re-fired at 900 °C for 2 hours.

For all cordierite ceramic samples, compressive strength, density, and the thermal coefficient of linear expansion (CTE) were determined. The results of the compressive strength measurements are presented in Fig. 1.

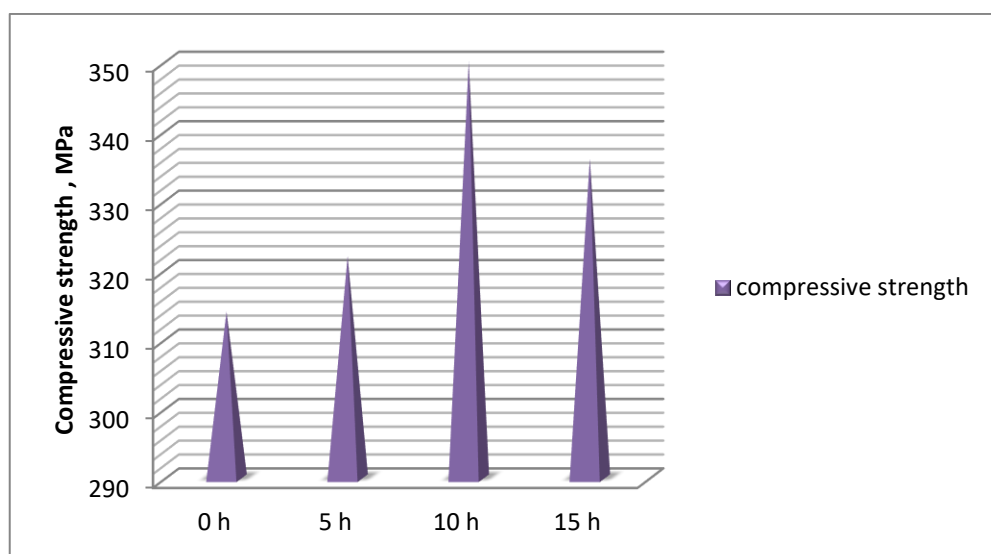


Fig. 1 Effect of holding time in the barium nitrate melt on the compressive strength of cordierite ceramics

A clear trend toward increasing compressive strength up to 350 MPa is observed in Fig. 1 for samples held in the melt for 10 hours. Further exposure up to 15 hours leads to a decrease in strength values to 337 MPa.

The density of the cordierite ceramics remains almost unchanged at 2.37–2.38 g/cm³, and the CTE of the investigated ceramics remains stable at $14 \times 10^{-7} \text{ }^{\circ}\text{C}^{-1}$.

Figure 2 presents the results of FTIR spectroscopy in the range of 400–1600 cm⁻¹ for cordierite ceramic samples subjected to ion exchange in molten barium nitrate. The obtained results demonstrate a positive effect of magnesium–barium ion exchange in the surface layers on the mechanical strength of cordierite ceramics. An increase in compressive strength from 314 to 350 MPa is observed for samples treated for 10 hours in the melt. Further prolongation of the ion-exchange treatment results in reduced compressive strength. The strengthening procedure has no significant influence on the thermal coefficient of linear expansion or density.

The FTIR spectra of the treated ceramics recorded in the 400–1600 cm⁻¹ range indicate that the exposure of the samples to molten barium nitrate does not substantially alter the primary structural units of cordierite ceramics. The spectra of the investigated samples exhibit the characteristic features of α -cordierite and demonstrate several key absorption bands (Harrati A., 2022). Absorption bands with maxima at 948 and 1146 cm⁻¹ arise from the asymmetric and symmetric stretching vibrations of Si–O–Si bonds in SiO₄ tetrahedra. Stretching modes of Al–O bonds correspond to the bands at 948, 676, 618, and 574 cm⁻¹. The band with a maximum at 766 cm⁻¹, appearing as a somewhat indistinct shoulder compared to reference α -cordierite, corresponds to the stretching vibrations of MgO–O.

The absorption band at 904 cm⁻¹ is associated with deformation vibrations of B–O–B linkages in BO₃ triangles and appears due to the absence of the corresponding band in the reference α -cordierite spectrum.

Overall, the FTIR spectra indicate that immersing the ceramic samples in molten barium nitrate does not significantly modify the fundamental structural units of cordierite ceramics. The developed strengthening method can therefore be successfully applied to improve the operational performance of radio-transparent ceramic radomes for aerospace applications.

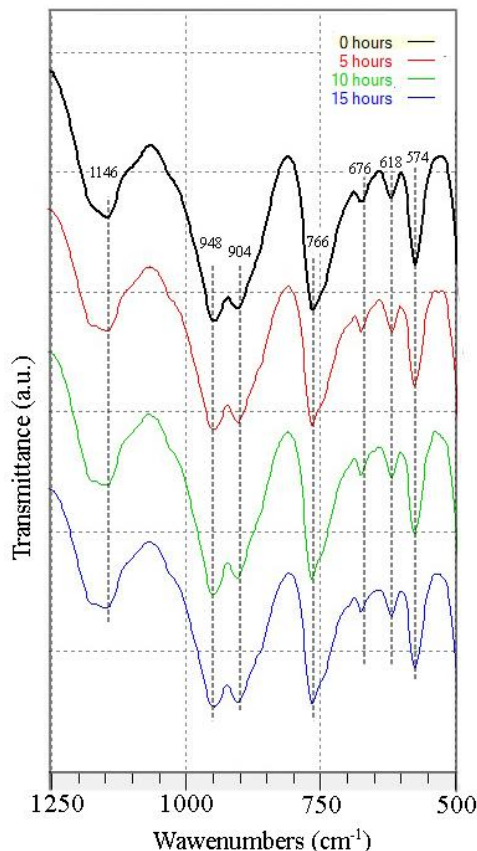


Fig. 2 Effect of holding time in the barium nitrate melt on the FTIR spectra glasses of cordierite ceramics

CONCLUSION

The conducted investigations confirmed a positive effect of magnesium–barium ion exchange in the surface layers on the mechanical strength of cordierite ceramics. An increase in compressive strength from 314 to 350 MPa is observed for samples treated in the molten salt for 10 hours. Further extension of the ion-exchange duration leads to a decrease in compressive strength. The strengthening treatment has no significant influence on the thermal coefficient of linear expansion or on the density of the ceramics.

The FTIR spectra of the treated ceramics, recorded in the 400–1600 cm⁻¹ range, indicate that exposure of the samples to molten barium nitrate does not substantially alter the primary structural units of cordierite.

The developed strengthening method can therefore be successfully applied to improve the operational and aerodynamic performance of radio-transparent ceramic radomes for aerospace applications.

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