Low loss $MgAl_2O_4$ ceramics for terahertz windows

S.V. Egorov[1], A.A. Sorokin[1], I.E. Ilyakov[1], B.V. Shishkin[1], V.V. Parshin[1], S.S.Balabanov[2], A.V.Belyaev[2]

[1]Institute of Applied Physics RAS, Nizhny Novgorod, Russia
[2] G.G. Devyatykh Institute of Chemistry of High-Purity Substances RAS, Nizhny Novgorod, Russia

This paper describes fabrication of polycrystalline magnesium aluminate spinel by microwave sintering and hot pressing and investigation of its dielectric properties in the terahertz frequency range. The significant interest in this material is associated with its high performance properties including the impact strength and the microhardness, the possibility to operate at high temperatures and to withstand exposure to radiation. In addition, using an appropriate fabrication method the spinel can be made optically transparent. Along with that it has been shown that $MgAl_2O_4$ ceramics obtained by microwave sintering of high-purity ultrafine powders has a sufficiently high transparency in the millimeter-wave range (the dielectric loss tangent $\tan\delta < 6.0\cdot10^{-4}$ at a frequency of 200 GHz) [1]. Such ceramics can be used both in microelectronics, for example, for the manufacture of microwave ceramic resonators, and in high-power electronics – to create output windows in high-power vacuum devices of the millimeter-wave range.

In general, the processing route of low-loss ceramics fabrication follows the common practice of ceramic technology, viz. powder production, compaction and sintering. A special attention is paid to the purity and dispersity of the synthesized powder materials. In our case the initial powders were prepared by the sol-gel method. This method of synthesis and deep purification makes it possible to obtain high-purity, nanosized powders. We used two approaches for the ceramics consolidation in this work: microwave sintering and hot pressing of the powder compacts. The absence of heating elements in the microwave furnace ensures pure vacuum conditions, which makes it possible to obtain ceramics with low dielectric losses. It should be noted that one of the problems of spinel sintering is a considerable loss of magnesium (Mg) during long-term exposure to high temperature in vacuum. The capability of achieving high heating rates inherent in microwave processing allows retaining the stoichiometry of the spinel. As for hot pressing, this method provides additional densification of the sintered material and results in achieving high transparency in the visible and near-infrared range. However, the hot pressing method of spinel consolidation requires introduction of sintering additives, such as lithium fluoride (LiF), which can affect the dielectric characteristics of the final material.

The microwave sintering of the compacts was carried out in a gyrotron-based system operating in CW mode at a frequency of 24 GHz with a variable output power up to 6 kW. Hot pressing of the compacts was carried out using a uniaxial press at a temperature of 1600 °C and a pressure of 35 MPa.

To study the dielectric properties, high-density ceramic samples were produced with a diameter of 25 mm and a thickness of 2 mm. The samples fabricated by microwave sintering were either pure spinel or 1 % Y_2O_3-doped. The samples produced by uniaxial hot pressing with the LiF sintering additives were optically transparent. The dielectric characteristics were studied in the frequency range 0.05÷0.3 THz using a spectrometer based on an open high-quality factor Fabry –Perot resonator, and in the range 0.6 ÷3.3 THz using the method of terahertz time-domain spectroscopy. The results have shown that the refractive index of the samples varies weakly from 2.85 to 2.92 in the studied frequency range (Fig. 1). The refractive index of optically transparent ceramics is slightly higher than that of opaque samples (fabricated by microwave sintering). This fact may be associated with a higher density of optically transparent materials.

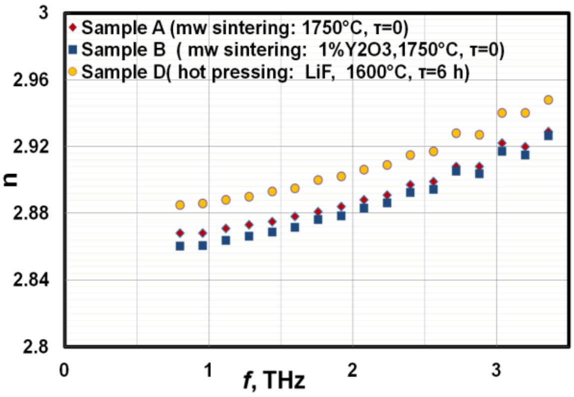

Fig. 1. The frequency dependencies of the refractive index of $MgAl_2O_4$ ceramics

The frequency dependencies of the dielectric losses of the ceramics are presented in Fig. 2. It can be seen that the value of $\tan\delta$ increases significantly from $1.5\cdot10^{-4}$ to 10^{-2} within the frequency range from 0.05 to 3.3 THz. The pure material has the lowest value of the dielectric losses. The presence of sintering additives in the ceramics slightly increases the electromagnetic absorption in the material. A comparative analysis has shown that the dielectric losses of the spinel in the terahertz frequency range are comparable in magnitude to the losses of sapphire [2] and pure polycrystalline aluminum oxide (Fig. 2) [3], while they are lower than those of the boron nitride ceramics [4].

In conclusion, the results of this study have demonstrated that pure $MgAl_2O_4$ ceramics obtained by microwave sintering exhibits relatively low dielectric losses in the terahertz frequency range and can be used, in particular, for the fabrication of output windows of terahertz vacuum electronic devices. The ceramics obtained by hot pressing is also transparent in the visible and near-infrared range and can be used for the fabrication of multispectral protective windows.

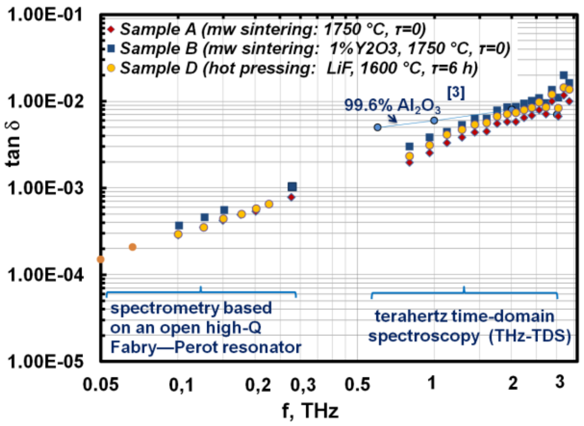

Fig. 2. The frequency dependencies of the dielectric loss tangent of $MgAl_2O_4$ ceramics

Acknowledgment

The study of the microwave sintering of MgAl2O4 ceramics was supported by Russian Foundation for Basic Research, grant No. 16-08-00736. The study of the terahertz dielectric properties was supported by the Russian Foundation for Basic Research and the Government of the Nizhny Novgorod region, grant No. 18-42-520015.

References

1. *Egorov, S.V., Bykov, Yu.V., Eremeev, A. G., et.al.* Millimeter-wavelength radiation used to sinter radiotransparent $MgAl_2O_4$ ceramics // Radiophysics and Quantum Electronics 2017, V. 59, No. 8–9, P. 690-697.
2. *Grischkowsky, D., Keiding, S.,van Exter, M. and Fattinger, Ch.* Far-infrared time-domain spectroscopy with terahertz beams of dielectrics and semiconductors // J. of Opt. Soc. Am. B 1990, V. 7, No. 10, P. 2006–2015.
3. *Rajab, K. Z., Naftaly, M., Linfield, H., et.al*. Broadband dielectric characterization of aluminum oxide (Al_2O_3) // J. Micro. and Elect. Pack. 2008. V 5, P. 101–106.
4. *Naftaly, M., Greenslade, P. J., Miles, R. E., Evans, D.* Low loss nitride ceramics for terahertz windows // Optical Materials 2009. V. 31, P. 1575–1577