Opinion

Dielectric properties of selected glass and glass-ceramics produced by plasma spraying

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Ctibor P,[1] Sedláček J,[2] Neufuss K[1]
[1]Department of Materials Engineering, Institute of Plasma, Physics Academy of Sciences of the Czech Republic, Czech Republic
[2]Department of Electrotechnology, Czech Technical University, Czech Republic

Correspondence: Ctibor P, Materials Engineering Department, Institute of Plasma, Physics Academy of Sciences of the Czech Republic, Za Slovankou 3, 182 21 Praha 8, Czech Republic, Email ctibo@ipp.cas.cz

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Selected silicate materials were sprayed by plasma in the past and characterized from the standpint of their sprayability, chemical composition and crystallographic phases, structure and mechanical properties as well as thermal stability. Present work is concentrated on dielectric properties of these thick films. Synthetic mullite, mullite mixture with glass, and pure glass were produced using the water-stabilized plasma system (WSP®). The deposits were striped-out, ground and polished to produce planparallel plates, i.e. flat samples with a smooth surface. These samples serve in principle like monoblock capacitors. All of them were tested in the alternating electric field at low voltage to examine their capacity and loss tangent in the frequency range between 100 Hz and 1 MHz. Relative permittivity was calculated based on the measured capacity. It is shown that the relative permitivity and loss tangent of plasma-sprayed glass and glass-ceramics is similarly stable as for bulk polycrystalline dielectric materials of the same composition. The family of silicon oxide based ceramics and glass is often used in electrical industry, particularly as insulators. Sintered silicate materials exhibit extraordinary volume resistivity and small dielectric losses during a wide range electric loading. These two parameters are substantially connected together and they also correspond to the material microstructural characteristics like crystallinity, grain size and porosity. At the Institute of Plasma Physics (IPP) great variety of silicates was tested from the point of view of ability to be successfully elaborated by the high feed-rate water-stabilized plasma spray gun.

Mullite (chemical formula $3Al_2O_3$-$2SiO_2$) is a refractory compound undergoing no transformation in solid state.[1] Mullite exhibits extraordinary stability of dimensions in course of thermal cycling,[2] but its application in electrical industry is mentioned in the literature as limited to mechanically supporting parts because of its high loss tangent.[3] Such a character of this material in the form of polycrystalline bulk is joint with its fine grains, low and uniformly distributed closed porosity and also because of relatively low content of the glassy (amorphous) phase. Conventional furnace processes – i.e. sintering or manufacturing of glass-ceramics[1,2] provide mullite with the above described microstructures. All materials were obtained in the form of already sintered tablets or frit of glass having industrial purity. After crushing and sieving, powder fractions suitable for plasma spraying were prepared. Mullite-glass mixture was obtained by mechanical admixing of 15vol. % of glass into mullite powder. Crucial spray parameters of this system – feeding distance and spray distance – were optimized before sample deposition. Optimum preheating temperature of the substrate was found as well. As substrates coupons made of stainless steel (AISI 316) were used. The powder was fed into the plasma jet by compressed air, deposited thickness was about 2mm. The deposits were then stripped from the substrate at cooling, thanks to thermal expansion mismatch, to form self-supporting flat samples. These ceramic samples were then ground from each side to form planar plates with a smooth surface. Such plates represent monoblock capacitors, 10x10x1mm in size. A thin films of aluminum as the contacts were sputtered in reduced pressure ($2x10^{-3}$ Pa) on the machined surfaces of the monoblock ceramics. Dielectric measurements were performed at the CTU in Prague, Faculty of Electrical Engineering, Dept. of electrotechnology. The electric field was applied perpendicular to the sample surface (in other words parallel to the plasma spray direction). Capacity was measured in the frequency range between 200 Hz and 1 MHz with help of the programmable LCR-meter (PM 6306, Fluke, USA). The frequency step was exponential on the whole studied range. Test signal voltage was adjusted to 1V AC, the stabilized LCR-meter was equipped with a "micrometric capacitor" fixture. Relative permittivity ε_r was calculated based on measured capacities and specimen dimensions. This same LCR-meter was used for the loss tangent measurement. Loss tangent values tg δ were recorded at the same frequencies as capacity. Five specimens of each material were analyzed and averages calculated. Prediction of the permittivity of a two-component system, i.e. mullite and glass, was performed according to three types of Lichtenecker rules (for example - Lichtenecker logarithmic formula: log $\varepsilon_v = v_{i.}$ log $\varepsilon_i + v_e$. log $\varepsilon_{e;}$ v_i is the volume of ceramics, ε_i is its relative permittivity -so called 'intrinsic permittivity'-, v_e the total volume of glass, based on feedstock powder mixing, and ε_e the relative permittivity of the glass). Looyenga rule was also applied for entire frequency range and compared with the experiment. The two-component system (ceramics and glass) without voids is considered to be homogeneous mixture and should have the values of calculated permittivity between the values of its components.[4,5]

Microstructure of the coatings was studied by means of optical microscopy using micrographs of polished cross sections. Micrographs were taken via CCD camera and processed using the image analysis (IA) software. Ten images of microstructures, taken from various areas of a cross section for each sample, were analyzed. X-ray fluorescence analysis (XRF analyzer XR–200, Link, UK) was used to evaluate presence of elements in the glass. Electron probe microanalysis was used for confirmation of these data on selected points of cross sections. Phase analysis was done by X-ray diffraction (XRD, diffractometer D 500, Siemens, Germany) using the filtered Cu-K_α radiation in the diffraction angle interval 5° - 90° 2θ. Dielectric properties were as follows: The value of mullite is higher than those usually reported for bulk mullite electroceramics ($\varepsilon_r = 4$ [3]), glass permittivity is even higher then reported for similar composition of glass, i.e. ε_r between 9 and 11. Loss tangent of plasma deposits exhibits certain decrease with increasing frequency, as is it also typical for a bulk ceramics.[6] The values are in general in the same frames as for bulks (sintered mullite tg $\delta = 0.016$,[3] bulk glass tg $\delta = 0.015 – 0.04$). A variation of structural features was found in the sprayed samples. Mullite is after spraying predominantly amorphous with traces of crystalline mullite and gamma alumina. Presence of SiO_2, B_2O_3, Na_2O, Li_2O and CaO was observed in glass. Cracking plays a role in the case of mullite while pores, rather large and located near the interface with the substrate, are typical for glass samples. Boiling of deposited glass due to the high heat influx from the spraying gun should be responsible for it. The structure of the mixture of both materials is intermediate and multicomponent. The complicated heterogeneous structure is probably the cause of less stable permittivity, with values lower than predicted by calculations. Studied materials are rather novel in plasma spray research. In the other hand, there exists long experience with utilization of their dielectric properties in the form of a bulk. We can conclude that they could be successfully used as dielectrics in the plasma sprayed form - comparably with the pollycrystalline sintered parts. Moreover, this ability is here firstly demonstrated on samples prepared by this particular water-stabilized plasma gun. Plasma spraying broadens the shape and size variability of electroceramic production.

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Conflicts of interest

The authors declare that there is no conflict of interest.

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