

Dielectric spectroscopy on alumina-epoxy composites towards the quantification of filler-particulate microstructure effects

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Abstract

Cylindrical ingots of epoxy composites containing alumina particulates with different microstructures were prepared and their dielectric properties were spatially investigated by the capacitance method for frequencies varying between 20 Hz and 1 MHz. Nano-porous (90 Å), or non-porous alumina particulates, with sizes varying from 63 to 200 μm, were mixed as composite fillers at various contents (0.1-5% wt). Sample-wafers were extracted from the cylindrical ingots to investigate any porosity induced effects on particulate mixing, dispersibility, precipitation, and relate the filler content to the electrical response observed at various depths (Fig. 1).

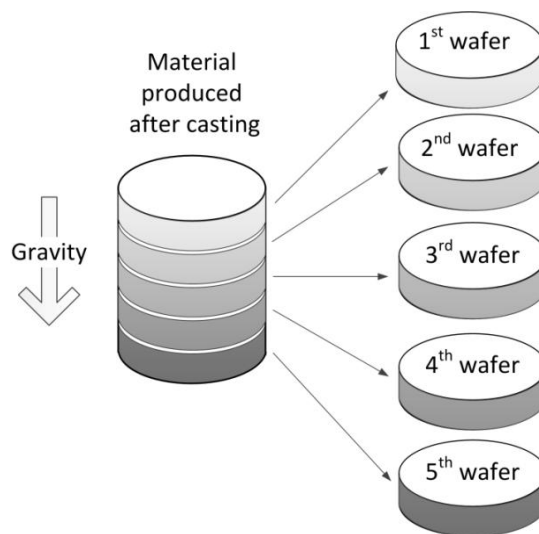


Fig. 1: Typical ingot structure and sample wafer extraction

According to the experimental results, the nano-pore effects dominate and reduce the effective relative dielectric constant compared to the neat resin values, while for the non-porous alumina fillers the effective relative dielectric constant increases with the filler content (Fig. 2).

The induced changes on corresponding dissipation factor values are insignificant. The dehydration effect caused by the ultrasound waves during mixing extracts trapped water molecules from the nano-pores and their presence affects polymerization, and therefore dielectric and electrical response of the composites. Finally, the presence of nano-pores in the alumina particulates generates particle stratification effects and the filler concentration increases near the top layers of the molded material (Fig. 3).

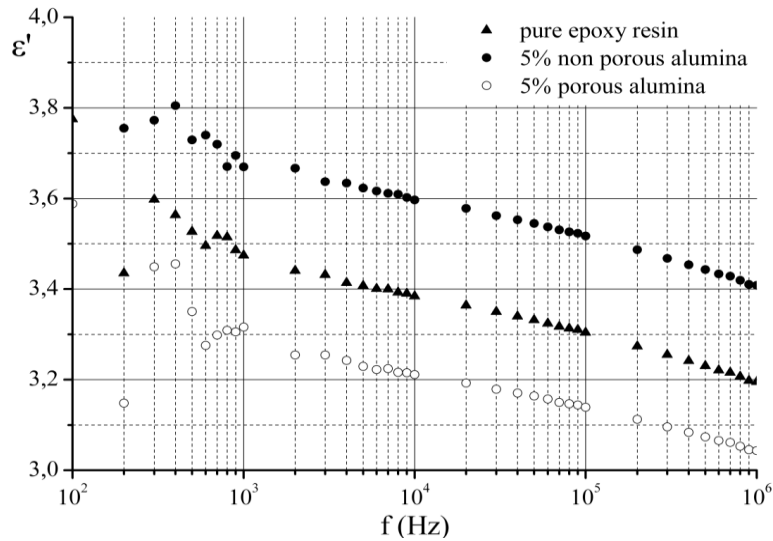


Fig.2: Induced changes on relative dielectric constant values of epoxy resins, by the non-porous and porous Al_2O_3 micro-fillers contained at equal concentrations (wt).

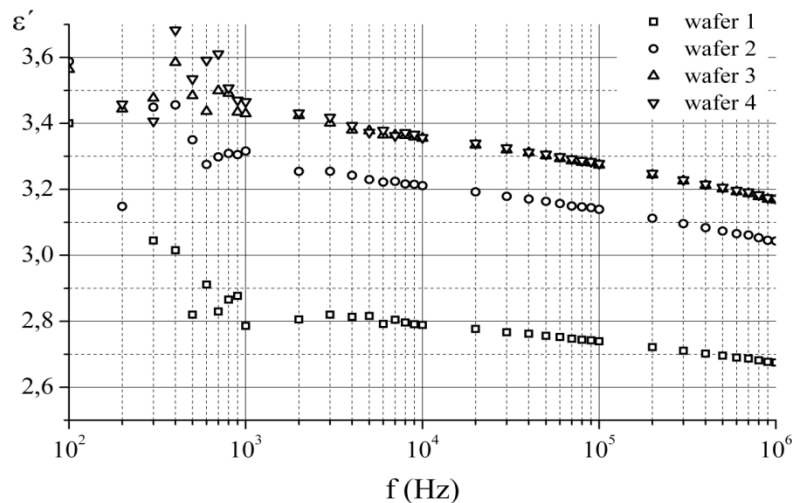


Fig. 3: Stratification effects investigated by the dielectric properties of epoxy resins, doped by porous Al_2O_3 fillers at 5% (wt) concentrations. Sample level positions in the initially produced cylindrical ingot are marked by the wafer numbers (1=top, 4=bottom).

The scope of this work is to investigate possible dispersion effects of the micro particle filler as they may affect the homogeneity and therefore the electrical response of laboratory produced composite resins. Irregular dispersion phenomena of the filler in the matrix of the epoxy resin might be induced during the hardening process of the resin either by: precipitation effects due to gravitational forces, agglomeration of the particle fillers, or by electrostatic and chemical interactions between the dispersed filler particles and the liquid resin during the polymerization process

Conclusions

Permittivity characterization may be utilized to investigate dispersibility and stratification effects of filler particles in the matrix of epoxy resins after the casting. The dehydration of the filler particles will have to be applied systematically, especially when ultrasound

process is used for mixing filler particles in the liquid resin (prior to hardening process). The nano-pore filler particles alter drastically the permittivity properties compared to the non-porous particles of the same entity. Nano-pore alumina fillers provide interesting dielectric properties on epoxy resin composites as they reduce the permittivity without increasing the dissipation factor of the composite. This may aid quest towards low-k and low-loss insulating materials, frequently used to eliminate cross-talk between current carrying parallel conductors in electronic circuits.

References

- S.S. Vaisakh, M. Hassanzadeh, R. Metz, S. Ramakrishnan, D. Chappelle, J.D. Sudhaa, S. Ananthakumar, “Effect of nano/micro-mixed ceramic fillers on the dielectric and thermal properties of epoxy polymer composites”, *Polym. Adv. Technol.*, Vol 25 (2), pp. 240–248, 2014.
- M. Kurimoto, H. Okubo, K. Kato, M. Hanai, Y. Hoshina, M. Takei, N. Hayakawa, “Permittivity Characteristics of Epoxy/Alumina Nanocomposite with High Particle Dispersibility by Combining Ultrasonic Wave and Centrifugal Force”, *IEEE Transactions on Dielectrics and Electrical Insulation*, Vol. 17, (4), pp. 1268 – 1275, 2010.
- S. Singha, M. J. Thomas, “Permittivity and tan delta characteristics of epoxy nanocomposites in the frequency range of 1 MHz – 1 GHz”, *IEEE Transactions on Dielectrics and Electrical Insulation* Vol. 15, (1), pp. 2-11, 2008.
- S. Singha and M. J. Thomas: “Dielectric Properties of Epoxy Nanocomposites”, *IEEE Transactions on Dielectrics and Electrical Insulation*, Vol. 15, (1), pp. 12-23, 2008.
- H. Zhao, R. K.Y. Li, “Effect of water absorption on the mechanical and dielectric properties of nano-alumina filled epoxy nanocomposites”, *Composites: Part A* Vol. 39, (4), pp 602-611, 2008.
- Yasmin, J. L. Abot, I. M. Daniel, “Processing of clay/epoxy nanocomposites by shear mixing”, *Scripta Materialia* Vol. 49, (1), pp. 81–86, 2003.
- T. Tanaka, “Dielectric Nanocomposites with Insulating Properties”, *IEEE Trans. Dielectr. Electr. Insul.*, Vol. 12, (5), pp. 914-928, 2005.
- La Mantia F P, Schifani R and Acierno D 1983, *J. Applied Polymer Science* 28 3075-80
- L. Ramajo, M. Reboledo and M. Castro, “Dielectric response and relaxation phenomena in composites of epoxy resin with BaTiO₃ particles”, *Composites: Part A*, Vol. 36, pp. 1267-1274, 2005.
- Y.Yao, S.Liu, W.Zhang, “Regeneration of Silica Gel Using Ultrasonic Under Low Temperatures”, *Energy & Fuels*, vol.23, no.1, p457-463, 2009.