SUPERPLASTICALLY FOAMING METHOD TO MAKE CLOSED PORES INCLUSIVE ALUMINA BASED CERAMICS

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Introduction

Low permittivity filler or substrate is needed to reduce the signal delay accompanied by the development of highly integrated electric device operated at higher frequency. We have already fabricated alumina based porous ceramics utilizing our innovated superplastically foaming method. In this method, pores expand utilizing the superplastic deformation driven by the gas pressure evolved from the foaming agent previously inserted[1,2].

We employed 3 mol% of yttria stabilized zirconia (3YSZ) as additives which has been usually used to facilitate superplastic property of alumina. However, the permittivity of 3YSZ (27) is larger that of alumina (8.5), lowring the permittivity with pore introduction was harmed by additive with such dielectric property.

In the present study, we report the successful fabrication of superplastically foamed porous alumina ceramics, at which low permittivity magnesium aluminate spinel was co-dispersed with small amount of zirconia powder.

Experimental

Alumina (AKP-30; Sumitomo Chemical, Tokyo, Japan) was used as main matrix starting powder. As superplasticity facilitating dispersoids, 3 mol% of yttria stabilized zirconia (3YSZ), zirconia without dopant (0YZ), or magnesium aluminate spinel (spinel) was added. As additives to the alumina matrix powder, 3YSZ (TZ-3Y, Tosho, Tokyo, Japan), 0YZ (TZ-0Y, Tosho, Tokyo, Japan), or magnesium aluminate spinel (Spinel) were tested. The Spinel was obtained through a solid-state reaction between alumina and magnesia powders (MO-V20P, average grain size: 0.2 µm; Ube Materials Industries, Yamaguchi, Japan) previously at 1200°C for 4 h and identified by x-ray diffraction (XRD) as a single-phase spinel.

Composite granules composed of foaming agent β-SiC surrounded by alumina based powder were prepared. Based on the results for previously described macroscopic single foam, compositions giving porosity over 30% were tested. After compaction followed by sintering, the superplastically foamed porous ceramics containing only closed pores were fabricated.

Fully densified ceramics using the same matrix materials, with porosity controlled by compaction pressure, were also fabricated using the same temperature regime, to act as reference materials.

The apparent density was measured with by Archimedes method using water as the medium. Cross sections were observed by optical or scanning electron microscopy (S-4300, Hitachi, Tokyo, Japan). Crystalline phases formed were identified by X-ray diffraction (XRD) (Multiflex, Rigaku, Tokyo, Japan).

Silver paste was painted on the top and bottom faces of a disc specimen and heat treated at ~ 300 °C for 1 h to form parallel electrodes with a diameter of 10 mm. The AC electric capacitance at 100 Hz was measured with an impedance analyzer (HP4192A, Palo Alto, USA) and the dielectric constant was determined from the capacitance, sample thickness, and electrode area.

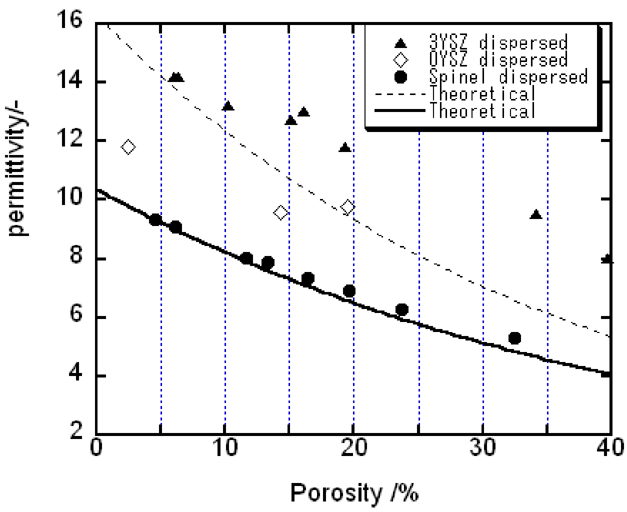

Fig.1 Porosity dependence of dielectric constant for superplastically foamed ceramics dispersed with spinel or 0YSZ.

The dependence of the dielectric constant on the ambient humidity was evaluated using two different methods. One method modeled the effects of condensation occurring on a cooled capacitor exposed to a humid atmosphere. Samples were submerged in distilled water for 10 min., then the surface water was removed and the capacitance measured. The other method aimed to determine the time dependence of the capacitance change upon abruptly changing the capacitor's environment from a dry to a humid atmosphere. The sudden change to a humid atmospheric environment was brought about by bubbling air through water and introducing the damp air stream into a glass tube containing the electrode sample.

Results and Discussion

Figure 1 illustrates the relationship between the porosity (ρ) and the dielectric constant (k) in the superplastically foamed porous ceramics formed from composite granules with different constituent matrix. The dielectric constant decreased monotonically with porosity. Figure 1 also shows two theoretical plots, one based on the logarithmic mixture rule and the other on having the pore/matrix aligned parallel to the longitudinal direction. For the latter case, k decreased linearly with the porosity.

Figure 2 illustrates the change in dielectric constant before and after submersion in water for superplastically foamed, conventional porous, and fully densified ceramics. Into the matrix, spinel was dispersed in both porous and fully densified ceramics because they enhanced superplasticity maintaining the permittivity. The degree of porosity for superplastically foamed and

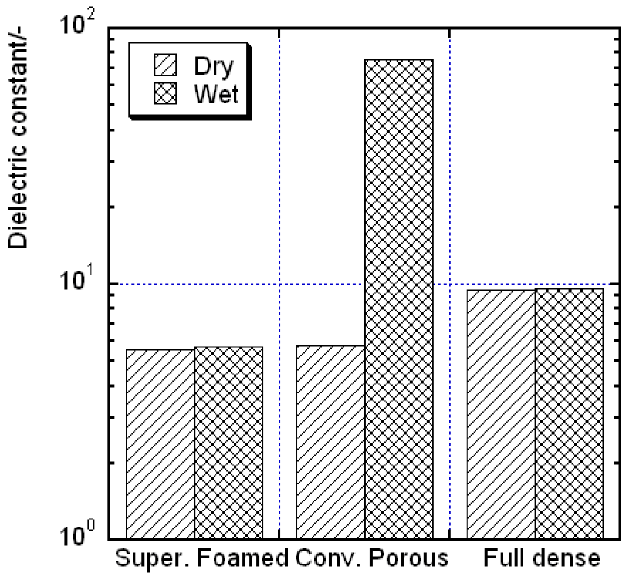

Fig.2 Dielectric constant of alumina based ceramics with dry and wet conditions

conventional porous ceramics was around 30 %. The dielectric constant for the three types of porous ceramics was approximately 5.5, or about half of that of fully densified ceramics, irrespective of the fabrication method used.

After submerged into water, the dielectric constant of conventional porous ceramics soared by more than one order of magnitude while its increase for dense one was marginal. Since the pores in the conventional porous ceramics with porosity over 30% should be open nature, residual water increased the dielectric constant substantially. Such porous ceramics, even possesses lowered dielectric constant, is not suitable for practical use because its value is apt to soar under sweating condition.

In our superplastically porous ceramics, on the other hand, change in dielectric constant before and after water submersion was only negligible, similar to full dense one. Since the introduced pores were all closed nature, no water infiltrated into the inner pores, resulting in identical dependence to that for dense one

Conclusion

Alumina based closed pore dominated substrates have been fabricated by using our innovated superplastically foaming method. Co-dispersing of magnesium aluminate spinel (Spinel) and zirconia without dopant (0YZ) provided adequate superplastisity to alumina matrix, while individual dispersing of spinel or zirconia resulted in only small deformation. The dielectric constant of superplastically foamed alumina decreased monotonically with porosity. Introduced pores were all closed nature at least up to 30% of porosity, at that porosity the permittivity became half as that of full dense one. Affect of the swetting water or ambient humidity have been proved to be identical to those of full dense one, which is favorable for semiconductor substrate.

References

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