GLASS MATRIX COMPOSITES WITH LEAD ZIRCONATE TITANATE PARTICLES PROCESSED BY MICROWAVE HEATING

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The possibility of toughening glass and ceramics by addition of a piezoelectric particulate secondary phase is the focus of current research. It is hypothesised that stress concentrations at the tip of an advancing crack can re-orient piezoelectric domains within the reinforcement in the direction of the stress field around the crack, thus dissipating energy which contribute to fracture toughness increment of the composite. Previous work has focussed on producing glass/piezoelectric inclusion composites by conventional sintering. This process is accompanied by extended porosity formation, as well as Pb depletion in the PZT phase. In several cases, the long processing times required to fully sinter the glass matrix lead to glass-PZT reactions and to loosing the stoichiometry of the PZT inclusions.

In this study, the novel production of new glass matrix composites reinforced with piezoelectric inclusions by using microwave heating was investigated. Specifically, lead-zirconate-titanate (PZT) particles in lead silicate and borosilicate glass matrices were considered. Mixtures of glass and PZT powders were prepared and used to fabricate powder compacts by uniaxial cold-pressing. In order to achieve densification, the compacts were subsequently heated in a single mode applicator, connected to a generator operating at the 2.45 GHz ISM frequency. The applicator is of the TE10n type and impedance matching devices are installed along the transmission line. A translating non-contact short circuit terminates the applicator, while 4 circular ports allow temperature monitoring and optical observation of the samples during the process. Temperature was monitored by means of a sapphire optical fibre, connected to a Mikron M680 pyrometer. The generator forward power was varied between 500 and 1500W, and a directional coupler allowed for reflected power measurement. The samples were placed, one per run, in an alumina fibre lining to provide the proper insulation. In order to improve the process speed, in some configuration an auxiliary absorber (SiC) was added to raise the samples temperature in the initial stages. The suitability of this solution was tested to produce dense composite samples.

Direct microwave absorption usually resulted in samples unhomegeneities and low reproducibility of the treatments, while the use of the auxiliary absorber allowed a more controllable sintering. Despite the presence of the auxiliary absorber on only one side of the samples, no shape distortion was recorded, and densification was homogenous. As expected, when the glass transition temperature of the glass matrix is reached, microwave absorption drastically increased, as well as the heating rate. The lack of a proper power control during this phase can lead to overheating and thermal runaway, having the consequences of severely deforming the samples, as well as causing the Pb depletion in the regions where temperature reached 800°C.

Sample characterisation was conducted by standard techniques such as SEM, XRD and hardness tests. For comparison, a number of samples were prepared by conventional sintering in an electric furnace. Microstructure characteristics of conventionally and microwave processed samples were compared in terms of residual porosity, homogeneity of PZT distribution in the glass matrix and quality of PZT/glass interfaces. The results are discussed on the basis of the shorter processing times required by microwave heating in comparison to conventional heating which lead to the desired preservation of the stoichiometric PZT composition of the inclusions and to minimisation of glass/PZT reactions at the matrix-inclusion interfaces. This is particularly relevant for borosilicate glass matrix composites which require a relatively high sintering temperature (720 °C) and thus cannot be fabricated by conventional sintering due to loss of Pb and change of stoichiometry of PZT particles.

Further issues regarding the optimisation of the microwave heating parameters to achieve controlled shape and dense glass/PZT composites will be discussed.