SYNTHESIS AND CHARACTERIZATION OF DOPED AND UNDOPED ZIRCON BASED CERAMICS

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Homogeneous and fine sized starting powders of both undoped and doped zircon (Fe, Y, Fe and Al co-doped) were synthesized by the sol-gel technique following controlled hydrolysis and condensation steps. The powders were sintered for different temperatures and their structural characterization was carried out using wide range of characterization techniques such as XRD, NMR, SEM and TEM.

The densities and zircon yields of both doped and undoped samples have found to increase with temperature reaching a maximum at 1500°C. They show a slight dissociation beyond this temperature. A significant reduction in the starting temperature of zircon formation was observed in the Fe and co-doped sample from 1400°C to 1225°C and 1215°C respectively revealing the contribution of the dopants in zircon formation.

The microstructures of both doped and undoped samples revealed they are almost fully dense. The dopants are present in the grain boundaries in all the samples indicating no substantial solid solution formation with zircon. The Fe and Al co-doped sample is superior to all the other samples, showing promising microstructures without microcracking with fully densified zircon and high zircon yield. Almost single phase zircon was obtained at 1400°C. TEM micrographs reveal that a crystalline grain boundary phase containing some Fe and Al. This may be advantageous when high temperature creep resistance is a concern.

Another set of samples were prepared with the conventional solid state sintering method and compared with the sol-gel samples. Undoped and Fe doped samples have silica in the form of both crystalline and amorphous phases whereas Y doped sample has complex microstructure with multiphases due to yttrium. Dopants are present in the grain boundaries without substituting into the zircon sub lattice. A significant feature observed in the sol-gel samples is the absence of cristobalite phase which contributes towards crack formation in the sintered bodies.

Finally the electrical properties of both sets of samples were discussed. It was revealed that there is a transient current present due to some initial mobility within the samples, which masks the true conductivity values. Therefore the measured conductivity data could be due to the transient current combined with grain boundary conduction. Also it was suggested that doping does not help in oxygen vacancy migration due to the high activation energy of oxygen migration.