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Connectivity and Surface Microstructure of PZT-SiO_2 Ceramic Glass Composites

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Abstract

PZT ceramic of morphotropic phase boundary (MPB) compositions was prepared by a conventional oxide method and its composite with glassy SiO_2 was prepared. The Scanning electron micrograph of the composite samples were recorded to exactly determine the connectivity and surface microstructure of the sample. In the present study the microscope viz. XL30, Philips Co., USA**,** was used. The SEM photographs recorded give the crystallite size as less than 1.2 µm. The dominance of glass matrix with higher percentage of silica is visible in the micrographs.

Keywords: Composite, Electroceramic Materials.

Introduction

The markets and material technologies for ceramics used as insulators, substrates, packages, capacitors, resistors, semiconductors, piezoelectric devices, and superconductors have shown an outstanding growth during the last three decades[1]. For each type of material, market factor, segmentation and trends are strongly connected with technology and developments. The electronic ceramics industry is a highly technological one characterised by rapid innovation and constant changes. Working with a wide variety of functionally different materials, the electronic ceramic industry is still in the process of strong expansion and redirection. It interfaces directly with the electronic industry through the widespread use of ceramic components as an integral part of electronic devices and packages[2]. Several of the electronic ceramic market segments have matured, yet they are still growing at a steady pace.

Electronic ceramics provide basic components as well as support a variety of electronic products including computers, industrial controllers, consumer automotive devices and digital switches. They can be also used as active components, such as semiconductors to control voltage and electrical currents, and as passive components, such as capacitors and resistors to control electrical currents or voltages, or electromechanical applications[3], such as ferrite magnets or piezoelectric devices. The significance of electroceramic materials can be gauged with respect to the present demand of electroceramics in the global market and projected demand[4] in the future. One needs to maintain a day to day updated database on the consumption and market demands of the ceramics in question. The commercial interest generated for a particular type of advanced ceramic has to be gauged and the research, development as well as manufacture has to be planned accordingly.

In the present study we had tried to develop a ceramic composite with silica glass as a matrix and a piezoelectric ceramic viz. Lead Zirconate Titanate[5] (PZT) dispersed in the matrix. It is expected that this composite can lead to integration of silicon technology (semiconductors) with ceramic technology through an interface viz. silica glass.

Preparation of the Samples

To characterise the material we need to prepare the samples of the composite at different proportions. So as to find out the appropriate ratio at which the material exhibits the optimum figures of merit. The method of preparing the samples is mentioned graphically using a flow diagram below. Here the preparation of PZT ceramic is omitted since standardised procedures were followed[6]. Different proportions of the PZT: Glass were prepared viz. 100% PZT, 5% silica glass, 10% silica, 15%, 20%, 25% and 30%. Higher percentages of glass were ignored, because a preliminary study carried out with high glass contents in the composites led to a large reduction in piezoelectric and dielectric properties of the composites so obtained.

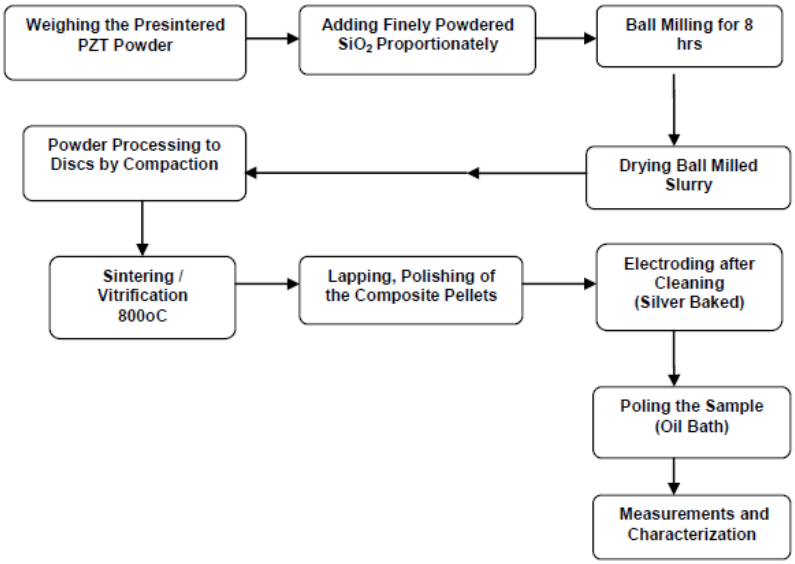

Fig.1: Preparation of PZT : SiO_2 Ceramic Glass Composites

Experimental

The studied ceramics of morphotropic phase boundary (MPB)[7] compositions of the general formula: $Pb(Zr_{0.54}Ti_{0.46})O_3$ were prepared by a conventional ceramic mixing method. PbO, ZrO_2, and TiO_2 (Fluka chemika) of the required amounts were carefully weighed and mixed by wet ball milling for 24 hours using zirconia balls and acetone as media. The mixture powder was dried at 100^{o}C for 12 hours. After drying, the mixture powder was calcined in a closed alumina cup at 800^{o}C for 4 hours at a constant heating rate of 300^{o}C/h. The calcined powder was then ground in a mortar and pestle to crush agglomerates to pass through a 100 mesh sieve and was filled in a covered alumina crucible, a $Pb(Zr_{0.54}Ti_{0.46})O_3$ atmosphere powder were placed on the top, bottom and in the vicinity of the crucible in order to maintain stoichiometries as closely to the nominal compositions as possible. Moisture contents and binder were removed by heating the sealed alumina crucible at 500^{o}C for 1 hour at the constant heating rate of 300^{o}C/h. Then, the lightly compacted powder in three sealed alumina crucibles, were sintered for 1hour each. Sintering temperature was set at 1200^{o}C. The samples were characterized for weight loss and sintered density. X-ray powder diffraction (XRD) analysis was carried out at room temperature to determine phase structure and lattice parameters. SiO_2 powder sieved using 100 mesh sieve were added to the sintered PZT powder. Calculated amounts of SiO_2 powders were added to the prepared ceramic (i.e. 5, 10, 15, 20, 25, and 30% by weight of PZT) and ball milled for 4 hrs. The mixture now forms the raw material for Ceramic Glass composite. The ball milled powder is again sieved and compacted in a hardened steel die. An isostatic pressure of 150 MPa is applied to make a pellet of 13mm diameter. The green pellets are then loaded into a furnace and heated to about 800^{o}C and maintained at that temperature for an hour after which it is cooled down to normal temperature within 2 hours. It is expected that sintering of the samples have not taking place while vitrification of the glass phase has resulted. It is important that no solid state reaction between PZT and Glass (SiO_2) should take place. A surface sheen on the pellets implies the composite has been formed. The composite pellets are now ready for characterisation.

The scanning electron microscope is used to determine the average crystallite size and the surface morphology[8]. It gives information about the grain evolution and grain size. It also gives the information about the inter-granular and the intra-granular pores and the distribution of grains in the composite samples. The surface under study may or may not be polished and etched, but it must be electrically conductive; a very thin metallic surface coating must be applied to nonconductive materials. Magnifications ranging from 10 to in excess of 50,000 diameters are possible, as are also very great depths-of-field. Accessory equipment permits qualitative and semi quantitative analysis of the elemental composition of very localized surface areas. In the present study the microscope viz. XL30, Philips Co., USA**,** was used for characterisation. The instrument was used under high vacuum conditions. Surface viewing consisted of securing the samples (surface side parallel to the viewing stub) directly to the carbon coated tape. After the sample stubs were mounted, samples were coated with gold (Denton Desk II sputtering system, 40 mA, 30 s). Samples were loaded onto an SEM mount and screwed in the sample holder within the SEM. Vacuum was applied to the instrument and imaging commenced at 10- 15 kV once 1.4 x 10^{-5} mm Hg pressure was attained in the SEM.

Results and Discussions

The Scanning electron micrographs of the composite samples were recorded to exactly determine the connectivity of the samples. It is very important to know the connectivity because different connectivities lead to different end results for the same phase ratios. We had assumed the material to possess a 0-3 connectivity and the SEMs shall give a direct insight into the material. The SEMs of Silica and

PZT in its pristine forms were first recorded, and then the subsequent composites were viewed in the light of the pristine phases. It can be seen that we had tried to maintain the magnification and the wavelength of the electron beam to be uniform. Yet in some cases to enhance the feel of the microstructure we had to change the magnification and wavelength. At some places due to precipitation of Silica from the matrix (at high silica ratios) the grains could not be properly resolved, even after the depth of the field was enhanced to a maximum. Secondly in an attempt to keep the magnification constant (within a particular range) some details may be found missing. But for all practical purposes and elucidation of the microstructure the method followed was accurate.

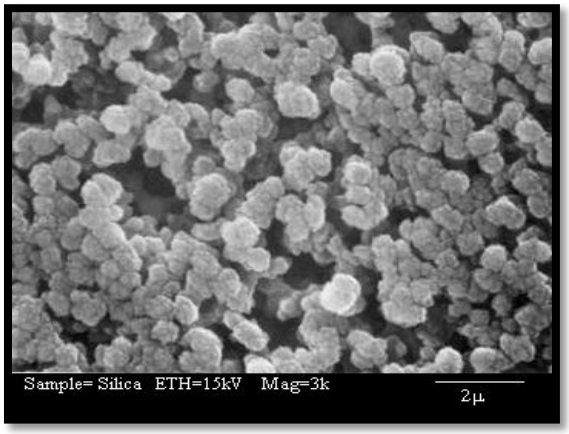

Fig. 2: Scanning Electron Micrograph of Silica Bowder

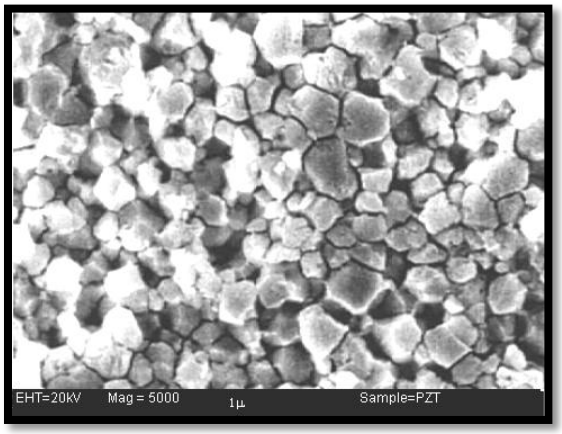

Fig. 3: Scanning Electron Micrograph of PZT Ceramic Sintered at 1200^{o}C

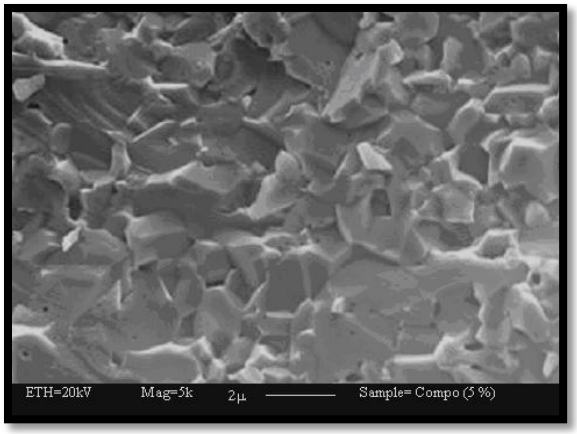

Fig.4: Scanning Electron Micrograph of PZT: Silica 5% composite

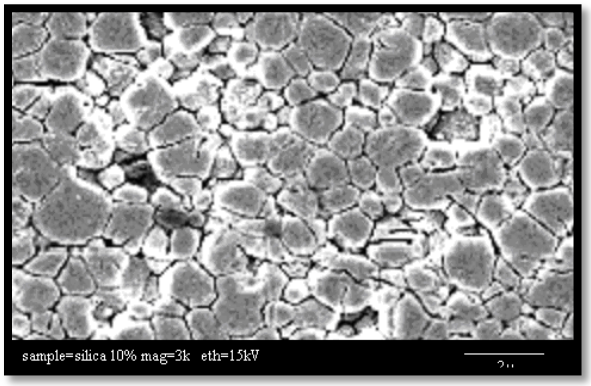

Fig. 5: Scanning Electron Micrograph of PZT : Silica 10% Composite

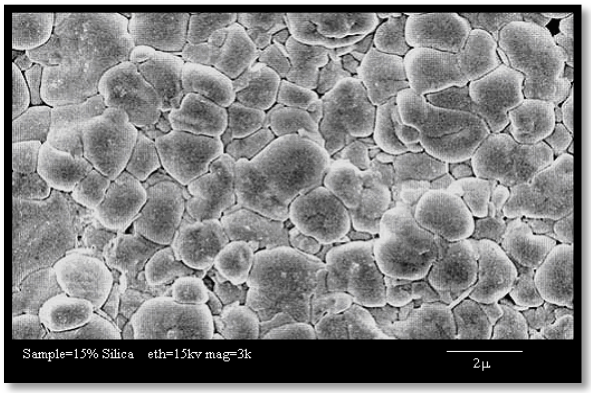

Fig. 6: Scanning Electron Micrograph of PZT : Silica 15% Composite.

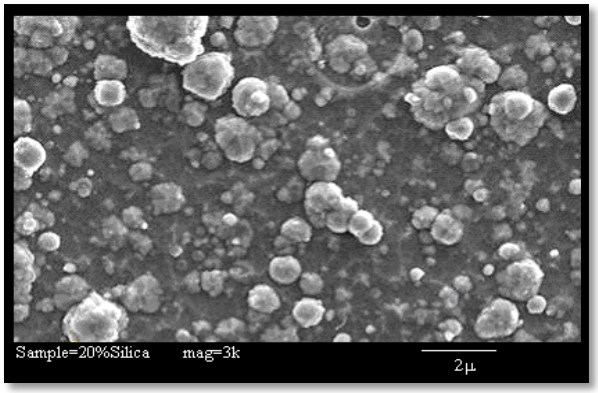

Fig. 7: Scanning Electron Micrograph of PZT : Silica 20% Composite.

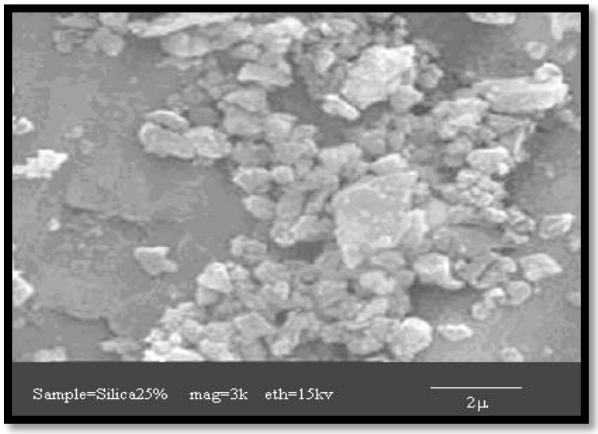

Fig. 8 : Scanning Electron Micrograph of PZT : Silica 25% Composite

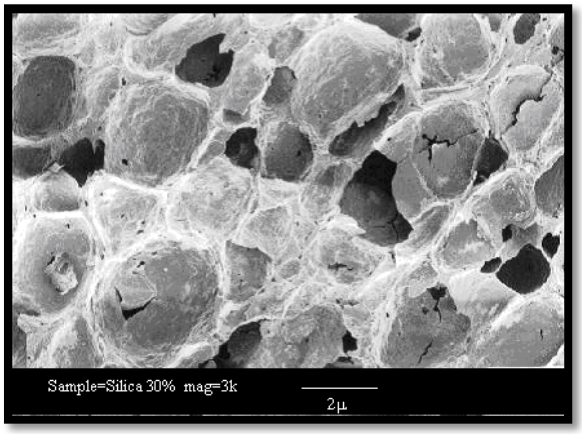

Fig. 9: Scanning Electron Micrograph of PZT: Silica 30% Composite

Conclusions

The SEM photographs recorded give the crystallite size as less than 1.2 μm. In SEM only the surface morphology can be known in detail and the rest of the picture is extrapolated from the data[9]. The most striking feature which is revealed from the SEM data figure 2 to 9 is the dominance of glass matrix with higher percentage of silica which is visible in the micrographs.

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