ABSTRACT

Ceramics are generally described as inorganic, non-metallic solids that are prepared from powdered materials, fabricated into products through the application of heat. They display such characteristic properties, as hardness, low electrical conductivity and brittleness. Ceramic materials are being developed for mechanical, electrical, electronic, magnetic, chemical and nuclear applications. These applications dictate the use of chemically pure starting materials of fine and narrow particle size distribution, excellent phase homogeneity and desired microstructure of the sintered compacts. The raw materials for such applications are generally produced by chemical processing routes. Alumina with its unique combination of thermal and chemical stability and high elastic modulus is one of the potential materials for high-tech applications. Its mechanical properties can be further improved either by employing appropriate processing methods at the powder synthesis stage, or by admitting selected additives at the forming stage to enhance sinter density, and also to obtain a tailor-made microstructure of the compacts. Many researchers studied the microwave region dielectric properties of alumina, derived from the Bayer process. The low frequency dielectric properties of alumina derived from the combustion process and alumina derived from diaspore minerals have not been reported by previous investigators.

Many researchers have conducted studies on the dielectric properties of Alumina electronic applications, and some ceramic materials with lower dielectric constants than that of alumina, such as glass-ceramics, mullite and cordierite. However, when compared with the current well-developed alumina substrate, there are drawbacks in these new materials, such as new and complicated manufacturing processes, potential reliability problems, and relatively expensive materials. Therefore, the acceptance of the
alumina material by the microelectronic industry needs more research. The objective of this study is, to develop a new alumina based substrate material with a low dielectric constant, so that the signal propagation delay in the substrate can be reduced. The goal is to lower the \( \varepsilon_r \) value from the current 10 to about 5, without sacrificing other properties necessary for advanced substrate applications.

In the present investigation, an attempt is made to prepare Alumina in three different processing routes. The different types of Alumina for these studies were prepared from the conventional commercial Bayer process, non conventional combustion process, and from natural mineral diaspore.

A fine powder of conventional Bayer Process Alumina (BPA), is prepared from the commercially available BPA powder, was wet milled with iso-propanol for 10 hrs in a high energy ball mill, with tungsten carbide balls for size reduction and uniformity.

The non-conventional Combustion Derived Alumina (CDA) was synthesized by the combustion technique. In this process, the aluminium nitrate and glycene were dissolved in a required amount of Millipore water and mixed thoroughly to ensure a molecular level mixing to form a homogeneous solution. The mixture solution was transferred to a Pyrex glass dish and later introduced into a microwave oven to undergo decomposition in the microwave field of 2.45 GHz. Subsequently, these powders were calcined at 800, 900, 1000 and 1200°C at a heating rate of approximately 10°C/min., during 2 hrs of soaking time. The increasing molar ratio of Glycene / Nitrate was found to be in favour of \( \gamma \)- to \( \alpha \)-\( \text{Al}_2\text{O}_3 \) phase transition, whereas the precursor with Oxidiser / Fuel = 0.71 at 900°C yielded a relatively well dispersed ultrafine \( \text{Al}_2\text{O}_3 \) powder, with crystallite size of 38 nm.
As a third attempt, Diaspore Derived Alumina (DDA) was obtained from diaspore mineral. The Indian diaspore mineral selected for this study was ground to a median diameter ($D_{50}$) of less than 5.0 micron, using sodium polymethacrylate salt (molecular weights 1200 g.mol$^{-1}$) as a dispersant (0.14 wt %) with a solid content of 60 wt % by grinding for 8 hrs in a high energy ball mill. For the conversion of the ground diaspore into a stable phase alumina, the material was calcined at a temperature of 425°C to 450°C.

In order to improve the sintering process of these Alumina, various sinter additives such as Magnesium Oxide (MgO), rare earth elements such as Praseodymium oxide ($\text{Pr}_6\text{O}_{11}$), Gadolinium oxide ($\text{Gd}_2\text{O}_3$) and Bomehite were selected in different molar ratios and labelled into various batches. The sintering temperature was highly influenced by the incorporation of these additives, because of the formation of a liquid-phase at the intermediate sintering stage; depending on the level of addition, final microstructures with controlled grain growth or grain refinement were obtained, with greatly improved mechanical properties at room temperature. The sintering was carried out both conventionally, and through microwave energy sources. The dielectric properties such as dielectric constant, dielectric loss and resistivity of the samples, were measured by the LCR meter as a function of room temperature (28 ± 5°C).

The $\varepsilon_r$ increased with increasing sintering temperature and saturated at 4.9 to 9.83, for the Bayer process alumina batches of various molar additions of magnesium, praseodymium and gadolinium oxide at the frequency of 1 MHz, for the conventional and microwave sintered samples. This study confirmed that Combustion derived and Diaspore derived alumina can possibly be used in low frequency dielectric applications (300 Hz to 3 MHz), and the observed dielectric constant was 2 to 3.
Chapter 1 reviews the alumina material, its crystal structure, morphology, importance and applications. Chapter 2 describes the sample preparation techniques and characterization methods, used for analyzing the electrical and mechanical properties. Chapter 3 deals with the preparation of alumina ceramics from the Bayer process alumina powders, and detailed studies of the electrical and mechanical properties.

Chapter 4 gives the synthesis of aluminium oxide, using glycene as fuel by the combustion method. The powder characterisation of TGA, DTA, FTIR, XRD and TEM were used, and the electrical and mechanical properties discussed. Chapter 5 gives the systematic study of the preparation of alumina from Diaspore earth minerals by the mechano-milling process, and explains the development, characterization and the electrical and mechanical properties of Alumina. Chapter 6 gives the summary and conclusion, and scope for future work.