SYNTHESIS AND CHARACTERIZATION OF ZIRCONIA DOPED ELECTRICAL PORCELAIN INSULATOR

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Abstract - *Now a day the proper power distribution is the important task that must get done in a proper manner. As far as power handling capability of insulator is concern it becomes challenging when the insulator fails to provide required value and bearable when subjected to the high valued signal Voltage or Surge). In this journal we have examined the properties of a basic building block i.e Kaolin (Al$_2$Si$_2$O$_5$(OH)$_4$), Feldspar (potassium) KAlSi$_3$O$_8$ and Quartz (SiO$_2$) of insulator along with the help of Zirconium oxide as a doping agent. In the whole process we first analyze the base composition and then compare with the Zirconium Oxide (zirconia) doped sample and reach to the conclusion. The techniques that we have used are X- Ray Diffraction, Scanning electron microscope, Bending Strength, Apparent Porosity, bulk density and Dielectric Strength that depend on the phase of Zirconium Oxide. Another aspect of this experiment is to make a sample so that layers of SiO$_2$ that we are using on MOS devices like MOSFET, SCR etc. In order to make the device more compact, SiO$_2$ layer can't be reduce by <1nm if it get reduce then it can raise the problem of tunneling current and it increases exponentially as the thickness of the layer decreases so in order to solve the problem (more compactness) this material can be useful.*

Key Words: **Porcelain Insulators, Alumina, Zirconia, Devices insulating layer, Feldspar, Kaolin.**

1. INTRODUCTION

Insulator is the one which comprises of the specialty to block a certain signal and not allow to pass through it. Electrical Porcelain Insulator playing a vital role in the modern power distribution system [2]. The basic building block of this Insulator is clay that is Silica, Kaolin and feldspar [3]. The kaolin provides the plasticity behaviour Silica is responsible for mechanical strength and feldspar is for the flux which can let the material to attain the behaviour at lower temperature instead to go at the higher one. Now a day high strength electrical porcelain insulator becomes a need as far as transmission lines (OHE cable insulator) and power distribution industry is concern [2].

Mostly the porcelain insulator are having high bending strength but still they got damage because of lightening with thundering or surge exertion on it[1]. In this experiment we

are using Zirconium oxide [8] as a doping agent which has compared with the base composition of the insulator material.

The another aspect of this experiment is to use the qualities of Zirconium oxide[4] i.e. high strength and stability at high temperature and also high dielectric strength as compared to SiO$_2$ layer on the gate terminal of Metal oxide Semiconductor. The recent research also focused to develop the other oxides material that has more dielectric constant
The properties of insulating material are:
1.   It must have high mechanical strength.
2.   It must have very high dielectric constant in order to withstand very high value signal [2].
3.   It must have high insulation resistance to hinder the path of current that goes to the earth and cause to damage the intelligence of the signal.
4.   It physical as well as electrical properties must not get vary when subjected to high temperature.
The reasons that can cause qualities of insulator degrade are as follows:
1.   Because of vandalism damage can cause to an insulator so it should be strong enough to bear the shock.
2.   Due to the erosion its can get degrade.
3.   Explosive shattering can also be the big reason for failing of insulator.
4.   Mostly porcelain insulator have a risk of internal puncture so flash under is also a major reason to degrade its behaviour.
To analyse the sample we have gone through XRD (X-Ray Diffraction), Scanning Electron microscope (SEM), Bending Strength porosity and density tests and with the help of XRD we will analyse and compare the phase obtained in the Zirconium oxide containing sample in the form of pallets. On the basis of phase we conclude with the specific properties of that particular phase (Mullite) [11]. As far as electrical properties are concern by the help of XRD analysis we can also comment on the dielectric behaviour of the sample by comparing it on the basis of phase.
Zirconium oxide that gives the value of dielectric constant in between 20 to 25 that is relatively high as compared to SiO$_2$ [13]used on the gate terminal of MOS [6]. The region between 600° C to 1300°C is the important zone in order to transform the clay into a rigid substance so therefore we sintered it at 1200°C. Zirconium oxide has three structural phases. [3] these are:

a.   Monoclinic phase
b.   Tetragonal phase
c.   Cubic phase

The monoclinic phase is thermodynamically stable below 1127 °C, around 1127°C there is a transition occurs to the tetragonal structure, which is a slightly distorted version of the cubic structure and is stable up to 2297°C and the melting temperature at 2707°C [7]. In the Monoclinic phase there are two non-equivalent oxygen sites with coordination numbers of 3, while all the Zirconium oxide atoms are equivalent and have a coordination of 7. In this experiment our work is to analyse the form of Zirconium by XRD graph because previous experimental and theoretical work has concluded that the cubic and the tetragonal phase has much larger static dielectric responses as compared to the monoclinic phase because of strong anisotropy in the tetragonal phase make it capable to become high dielectric response phase in addition to tetragonal cubic also possesses the same.

In the dielectric material when an external electric field is exerted then the local field (E_{local}) where dipoles are present formed and that depolarizes the applied Electric field $E_{applied}$ by acting oppositely so the charges produced on the surface gives a macroscopic electric field. Dipole-Dipole interaction also produced electric field.

Now the relation begins:

$E_{macro} = E_{applied} + E_{depolar}$.

i.e. $E_{local} = E_{macro}$

The above equation makes the material favourable for insulating properties as applied field is equal to the depolarizing field so they nullify each other.

Mullite ($3Al_2O_3 \bullet 2SiO_2$)[10] is a phase which comprises of alumno-silicates and has become a contender of high-temperature structural ceramic. The contribution of Mullite comes as far as traditional and advanced ceramic is concern. It plays vital role in insulating properties structural view of the phase can be easily identified by needle like structure in scanning electron microscope analysis. It has stable crystalline phase in alumno-silicate at normal atm. in room at high temperature. Mullite [11] also possesses good thermal stability (low thermal conductivity), good chemical stability. Because of distribution of glassy phases by impurity ingredients along grain boundary or forming isolated gap will influence the strength (mechanical) response at high temperature Experiment results suggests mullite phase in XRD analysis after get match with JCPDS software.

1.1 Experimental

1.1.1 Selection of material

For plasticity:- In order to have plasticity property in the sample or the product Kaolin is more preferred than any one because kaolin is itself a binder so no additive or binder we need to use.

For Flux: - By the use of Feldspar[3] we can regulate the melting temperature so the property which comes at higher temperature we get it here or we can easily anticipate the fact i.e. it reduces the vitrification temperature.

For Quartz: - Quartz provides the refractory crystalline phase or skeleton contributing to the mechanical strength of the body.

1.1.2 Sample Preparation

Table -1: % constituent in the experiment

Sample name	Kaolin (%)	Feldspar (%)	Quartz (%)	Doping Agent (ZrO₂) (%)
1	50	25	25	0
2	50	25	20	5
3	50	25	15	10

2. EXPERIMENTAL PROCEDURE

Firstly the materials get collected that is Kaolin, Feldspar and Silica these are the base materials for the Porcelain Insulator. The material gets converted into a fine powdered form to get mix with the other materials. With the help of mortar and mixing equipment it gets mixed in the homogenous form (of all three). After the completion of mixing let them all in motorized Sieve Shaker and convert to 45μ.

After obtaining the desired size of the material, pallets are formed by the tool steel mold for testing purpose which is done after sintering and before sintering in each case. After the testing of base composition we will dope Zirconium oxide in it and replace the Silica by some calculated percentage. With the increase in the content of Zirconium oxide the content of Silica will reduce and thus with the help of the results we will conclude the properties that we are getting with the addition of Zirconium oxide. Mechanical tests are Bending Strength, Bulk Density, Apparent Porosity and Dielectric Strength [9]. With the help of XRD we will find the phases of the material, SEM provides the micro structure of the material to get the pictorial view of each sample in this way we have performed the experiment.
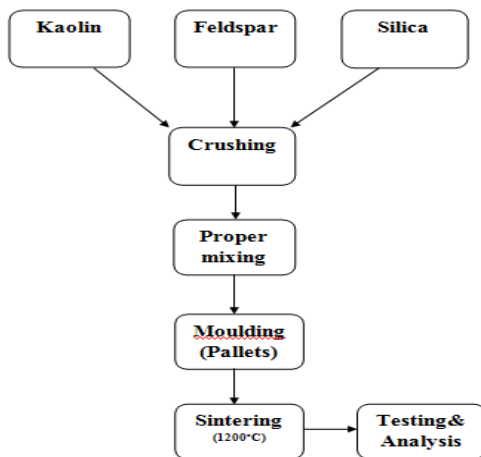
Chart -1: Flow Chart for testing.

3. RESULTS AND DISCUSSION

This investigation is based on the use of Zirconium oxide as a doping agent to analyze insulating properties of sample and there will be comparison of pure (base Composition) and ZrO_2 doped sample.

3.1 Linear shrinkage and Weight loss

Table -2: Linear shrinkage and Weight loss:

Name of sample	% Length change	% Breadth change	% Width change	Weight loss%
1	18	0.67	1.9	6
2	5.87	5.9	9.08	6.03
3	5.33	6	9.9	6.45
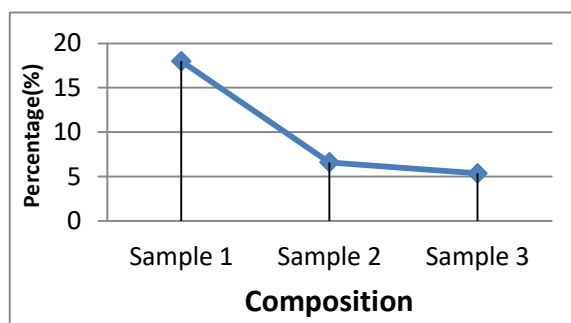
Fig -1: Shrinkage comparison of samples.

When subjected to 1200oC increase in the shrinkage because material becomes denser has been observed. As shown in the above figures.
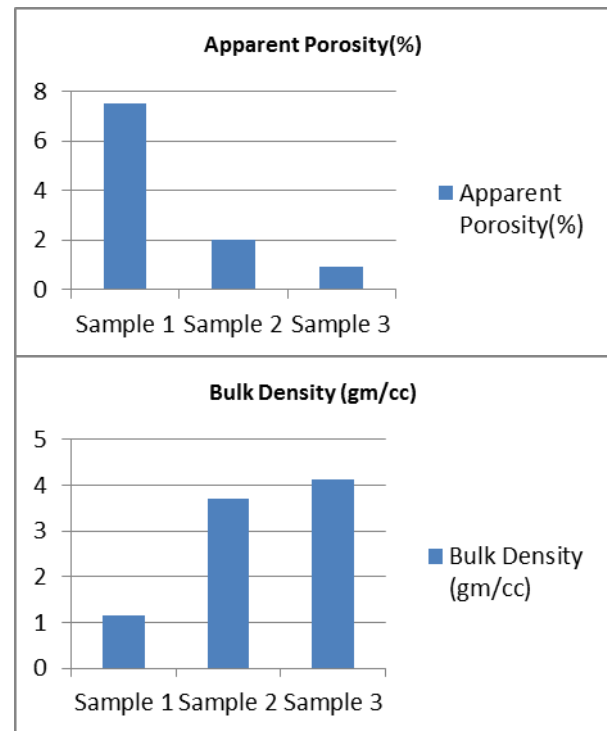
3.2 Bulk Density and Apparent Porosity

Fig -2 Apparent porosity and Bulk Density comparison
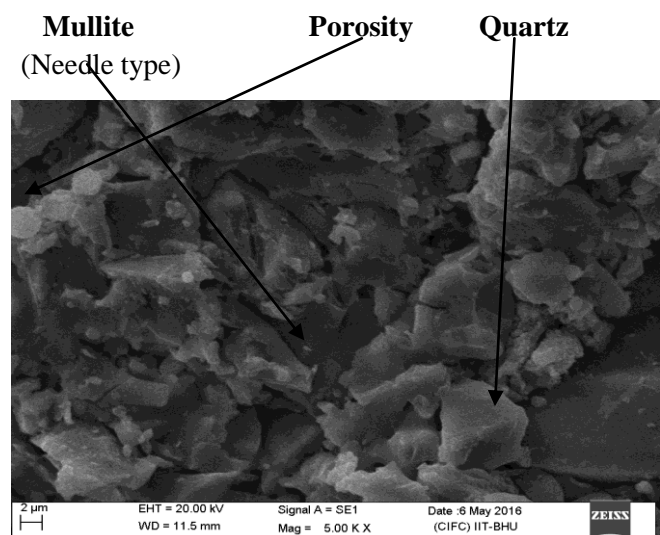
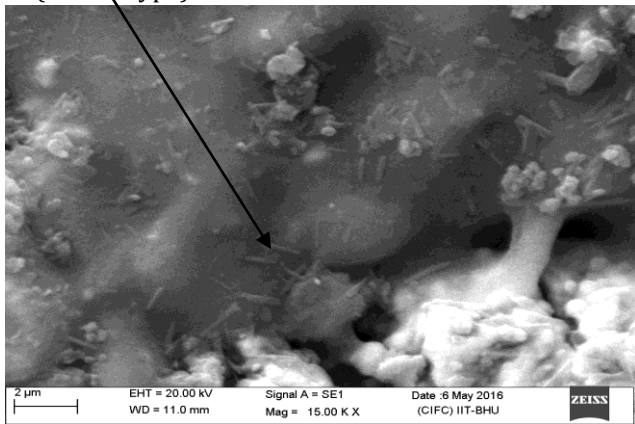### 3.2 Scanning Electron Microscope:

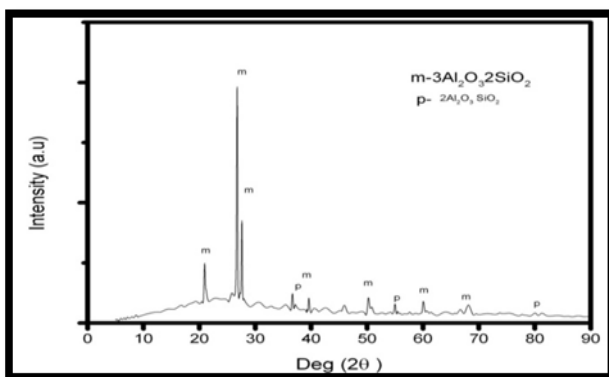**Fig -3(a)**: SEM view of Base composition
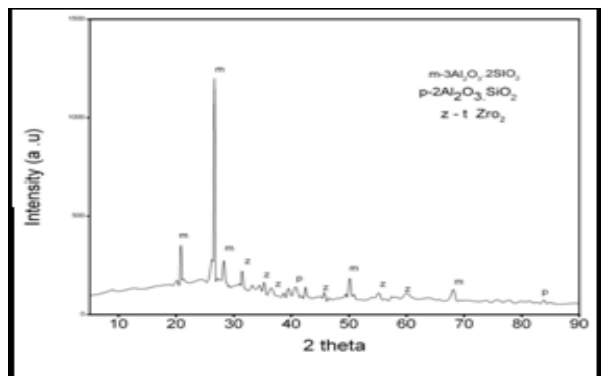
Mullite
(Needle type)

Fig -3(b): SEM view of Zirconium Oxide doped

As a result of the SEM we can say that the phase combination that we are getting as the content of Zirconium oxide get increases and as a result of that we are able to see the sharp image of the needles that refers the Mullite phase (desired)

3.3 X-Ray diffraction analysis:

Fig -4(a): X-ray Diffraction of Base composition

Fig -4(b): X-ray diffraction of sample doped with Zirconium oxide

The movement we add Zirconium oxide (by some percent) in the composition it comes into the picture in the form of the small peaks and the highest peak shows here mullite (desired) after matching with the JCPDS data base software. As a result of which here we have ensured insulating properties as well as the strength of the material.

Zirconium oxide peaks are also present in the form of tetragonal phase which is one of the phases of zirconia possessing appreciable dielectric strength

3.4 Bending Strength:
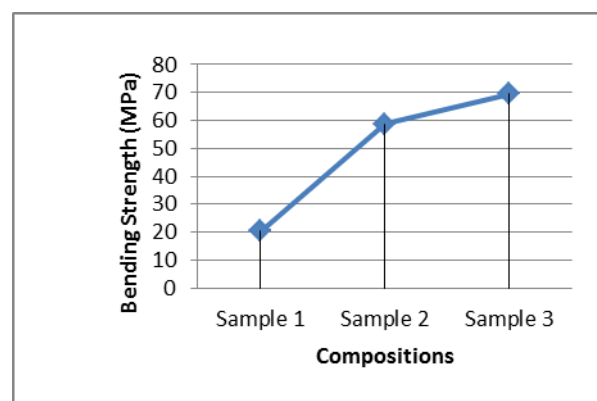
Fig -5: Bending Strength comparison of all Samples.

3.4 Dielectric Strength:

According to the Lattice Dielectric Properties of Zirconia that the structural parameters, including all the 3 phases in which tetragonal and cubic have much larger static dielectric response than the monoclinic phase[4,5]. So here with the help of experimental analysis we come to know that zirconium oxide that has present here is of Tetragonal phase which is useful for better dielectric responses and the expected value are in the range of 35- 50KV/cm by some literature based theoretical and practical calculations[14].

4. CONCLUSIONS

We have the pure composition which comprises of the basic material like Kaolin, Feldspar, Quartz. And here we use Zirconium oxide as a doping agent in order to avoid the conduction capability and the mechanical strength of an insulator which is suitable for the transistor amplification of a signal, Protecting the device from the burn out condition, maintain the intelligence of the signal at the output end, Withstanding of the material wherever required etc.
Following conclusion that has been observed:
1.   Linear shrinkage increases on sintering.

2.   X-ray diffraction confirmed that mullite phase developed.

3.   S.E.M. also confirmed needle type structure of mullite phase.

4.   Bulk density increase by increasing Zirconium oxide content.

5.   Dielectric property also get increase because of using Zirconium oxide in tetragonal phase form which is the best for having good dielectric strength as we saw in the XRD analysis.

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