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Comparison between chemically treated sisal fiber with Al_2O_3 and Fe_2O_3

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ABSTRACT : Aluminum oxide and iron oxide synthesized through sintering route. The present research work deals with ferrite and aluminum composite prepared using chemical reactions. Aluminum nitrate, ferric nitrate and ammonium chloride doped with sisal fiber has been prepared. The comparative studies of aluminum oxide and iron oxide were examined through dielectric measurement.

Keywords : Al_2O_3, Fe_2O_3, sintering method, ε, TEM

1. Introduction

Iron oxides are chemical compounds composed of iron and oxygen. All together, there are sixteen known iron oxides and oxyhydroxides. [1] Iron (III) oxide or ferric oxide is the inorganic compound with the formula Fe_2O_3. The steelmaking by-products such as dust and mill scale, very rich in iron (≈ 72% Fe), are currently produced in large quantities and represent a potential of almost 5 million tons in the world [2]. Powder metallurgy comprises a set of processes of forming having for common denominator a raw material in a powder form. The reduced iron powder is the most widely used material in powder metallurgy industry.

In the oxides of iron a-Fe_20_3 is the most stable compound. For the non-existence of Fe++ ions a-Fe_20_3 has higher electrical resistivity than other oxides of iron such as Fe_30_4, FeO, and ferrites. It has been reported, however, that at the temperature above 1200°C there is the possibility of the appearance of Fe++ ions in a-Fe_20_3. When the oxides contain ferrous ions, the hopping of electrons between ferrous and ferric ions gives rise to higher conductivity. Thus for samples possessing both the conductive and less-conductive phases the Maxwell-Wagner interfacial polarizations are observed. With the surface modified by the use of mild reducing condition of sintering Hirbon reported the interfacial polarization in the sintered compacts of a-Fe_20_3. On the other hand, in a-Fe_20_3 containing other ions of different valences such as Ti" ions polarizations due to permanent dipoles of Fe++_Fe+++ induced by Ti+4 ions were observed at very low temperature [3-7]

Aluminum oxides are chemical compounds composed of aluminum and oxygen. All together, there are sixteen known aluminum oxides and oxyhydroxides. [8-9] Aluminum (III) oxide or aluminum oxide is the inorganic compound with the formula Al_2O_3. Powder metallurgy comprises a set of processes of forming having for common denominator a raw material in a powder form. The reduced aluminum powder is the most widely used material in powder metallurgy industry.$LiAlO_2$ has various crystalline structures such as α- $LiAlO_2$, β-$LiAlO_2$, γ-$LiAlO_2$, Layered $LiAlO_2$, Corrugated $LiAlO_2$, Goethite type $LiAlO_2$ etc. The crystalline structure of $LiAlO_2$

depends mainly on the preparation methods. Many researches prepared $LiAlO_2$ with different structures. V.R. Galakhov et al. prepared α-$LiAlO_2$ with Fm-3m space group by using solid state reaction and M. Tabuchi et al. prepared α-$LiAlO_2$ with Fm3m space group by hydrothermal synthesis [10-12].

In the oxides of aluminum a-Al_20_3 is the most stable compound. For the non-existence of Al++ ions a-Al_20_3 has higher electrical resistivity than other oxides of aluminum such as Al_30_4, AlO, Al_2O_3. It has been reported, however, that at the temperature above 1200°C there is the possibility of the appearance of Al++ ions in a-Al_20_3. When the oxides contain Aluminum ions, the hopping of electrons aluminum ions gives rise to higher conductivity. Thus for samples possessing both the conductive and less-conductive phases the Maxwell-Wagner interfacial polarizations are observed. With the surface modified by the use of mild reducing condition of sintering Hirbon reported the interfacial polarization in the sintered compacts of a-Al_20_3. On the other hand, in a-Al_20_3 containing other ions of different valences such as Ti" ions polarizations due to permanent dipoles of Al++_Al+++ induced by Ti+4 ions were observed at very low temperature [3-7]

2. Material and method

2.1Chemical treatment of fiber

Ferric Nitrate (Fe $(NO_3)_3.9H_2O$) and ammonium chloride (NH_4Cl) was taken in the ratio 10:4 in 500 *ml* of distilled water. The mixture was stirred till a homogenous solution was obtained. In this mixture 10g of sisal fiber was added and then 1:1 solution of NH_4OH (liquid ammonia) was added to it then left the solution for one hour. Again the mixture thus obtained was dried and then annealed in muffle furnace at 1000°C and kept it at that temperature for 15 min. repeat this process for getting Al_2O_3 using Aluminum Nitrate (Al $(NO_3)_3.9H_2O$) in place for ferric nitrate.

The reaction may take place in this way
2Fe $(NO_3)_3.9H_2O$ +3NH_4Cl+3NH_4OH+ Fiber→ **Fe_2O_3: fiber** + 6NH_4NO_3 + 3HOCl + 18H_2O

The reaction may take place in this way
2Al $(NO_3)_3.9H_2O$ +3NH_4Cl+3NH_4OH+ Fiber→ **Al_2O_3: fiber** + 6NH_4NO_3 + 3HOCl + 18H_2O

When ferric nitrate reacts with ammonium chloride and ammonium hydroxide along with sisal fiber at 1000°C with ammonium nitrate and HO-Cl (hypoclorous acid) decomposed at such high temperature and only **ferric oxide** is left and similar for aluminum oxide.

2.2 Nature and structure of sample after firing

The both of material formed was found to in **solid crystals** in physical appearance. They are appeared in powder form and Fe_2O_3 in **Cole** in color but the Al_2O_3 sample in **white** in color.

3. Result and discussion

Due to increasing interest in the dielectric properties of oxides, dielectric measurements of iron oxide thin film are carried out. The room temperature performance of the dielectric constant with frequency is shown in Figures below. The electrical properties of the insulating material Fe_2O_3 composite were measured by impedance analyzer these dielectric measurement of Fe_2O_3 composite doped with sisal fiber shown in fig 1 and 2. In the Fig 1 represents the graph between frequency and ε for ferric oxide and fig 2 shows the comparison between frequency and ε for aluminum oxide.

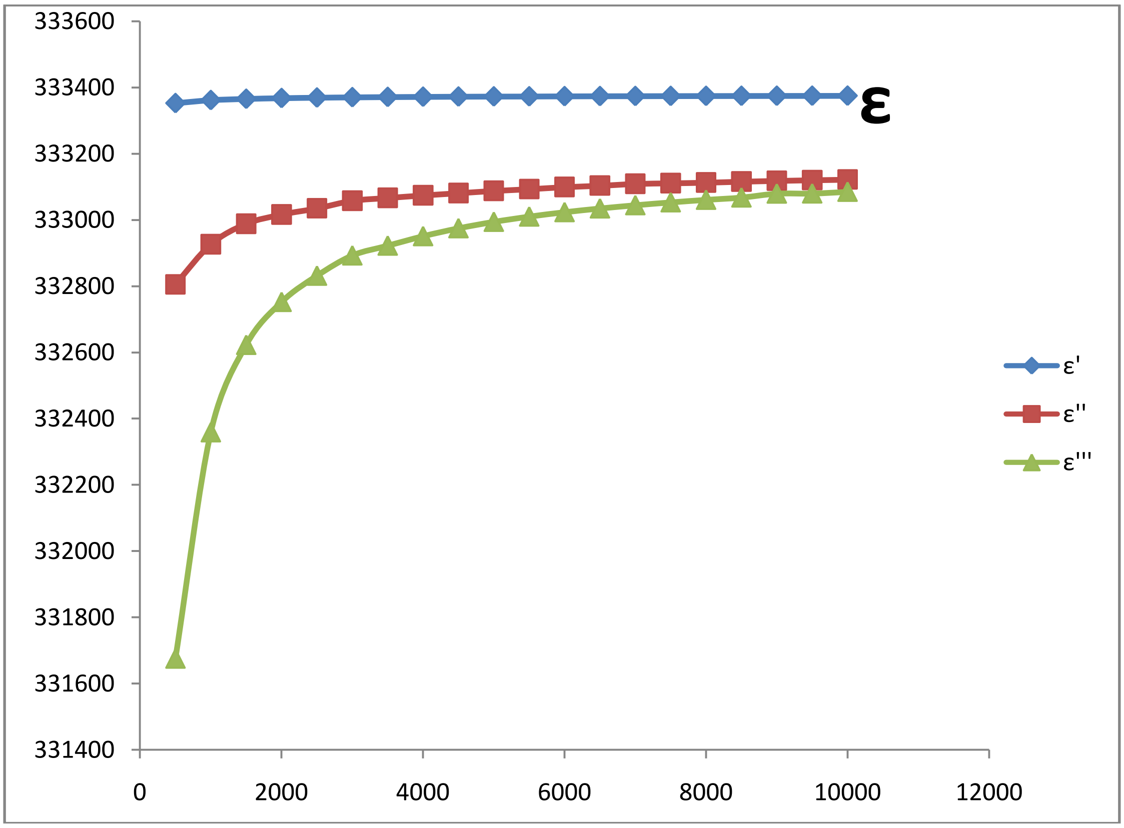

Fig 1

In fig 1 & 2 is a comparison plot of variation of dielectric constant with different frequency at constant temperature shows an increase considerably with increase in frequency. It was observed from these figures that the dielectric constant increases continuously with increase in frequency for all the samples, followed with a frequency independent behavior. This is a normal behavior for high density, fine chemically homogeneous ferromagnetic material.

The dielectric properties of ferrites are dependent upon several factors, including the method of preparation, chemical composition and grain structure, size and natural fiber (sisal fiber) and it's contains cellulose, hemicelluloses, lignin and pectin. The observed dielectric behavior of our samples may also be due to the particle size effect and is also in concurrence with observation made by other Investigators [13-14]. The effect of electrical homogeneity fine grain structure and shape of the ferrite samples affect the dielectric properties [15]. It also presence the α impurities contributes towards the change in dielectric properties. The dielectric constant of 25-30 at 10 KHz and an increasing trend is observed at higher frequencies. This is normal behavior for high density fine chemically homogenous ferromagnetic material [16].

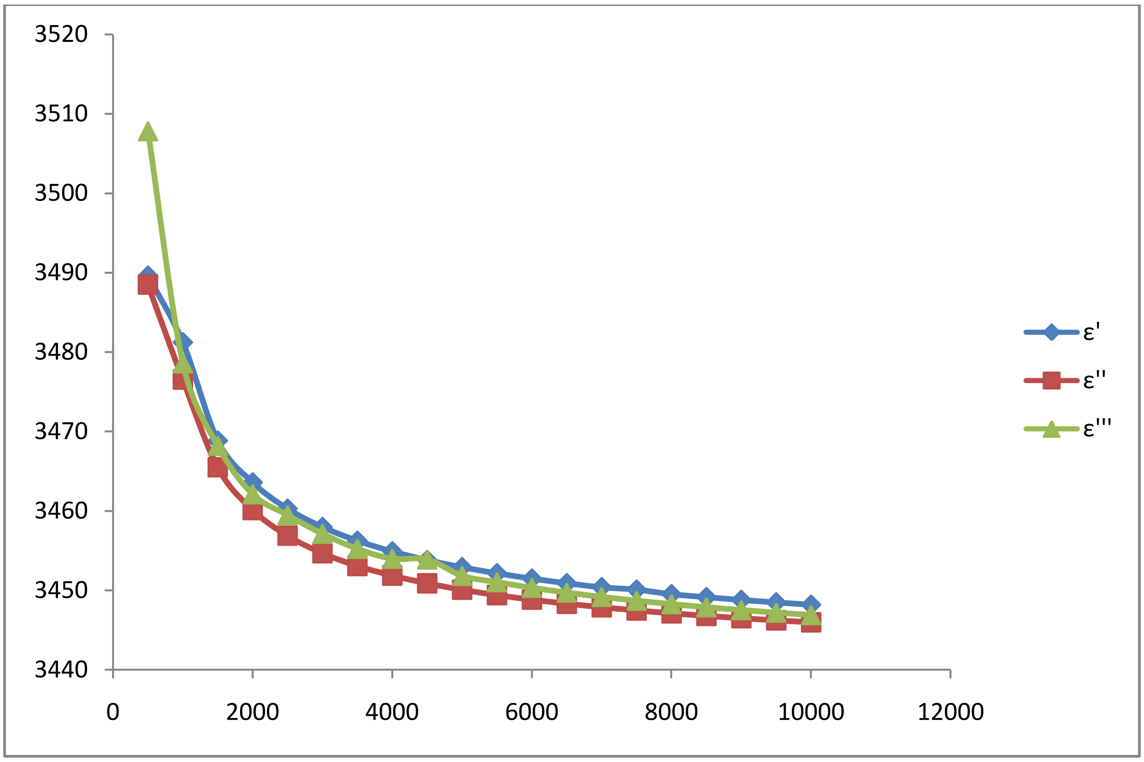

Fig 2

The electronic exchange between ferrous and ferric ions didn't follow the path of frequency which is externally applied alternating beyond the critical frequency value. The dielectric behavior of samples which shown in graphs is because of its particle size effect [17]. If ε remains unaffected with increase in temperature then frequency also remains the same, which means that the local carriers are immobile. It is understood that the local charge carriers are immobile and the thermal activation in negligible. This further suggests that the samples possess a high chemical homogeneity and fine grain distribution. The presence of moisture, cellulose, hemicelluloses, lignin and pectin is also indicated by the electrical conductivity results. It is reported the presence of moisture plays an important role in the formation and stabilization of Fe_2O_3 [18]. While in fig 2 is a comparison plot of variation of dielectric constant with different frequency at constant temperature shows an increase considerably with increase in frequency. It was observed from these figures that the dielectric constant increases continuously with increase in frequency for all the samples, followed with a frequency independent behavior.

The dielectric properties of Alumina are dependent upon several factors, including the method of preparation, chemical composition and grain structure, size and natural fiber (sisal fiber) and it's contains cellulose, hemicelluloses, lignin and pectin. The observed dielectric behavior of our samples may also be due to the particle size effect and is also in concurrence with observation made by other Investigators [13-14]. The effect of electrical homogeneity fine grain structure and shape of the Alumina samples affect the dielectric properties [15]. It also presence the α impurities contributes towards the change in dielectric properties. The dielectric constant of 25-30 at 10 KHz and an increasing trend is observed at higher frequencies. This is normal behavior for high density fine chemically homogenous material [16].

The electronic exchange between Ammonium and aluminum ions didn't follow the path of frequency which is externally applied alternating beyond the critical frequency value. The dielectric behavior of samples which shown in graphs is because of its particle size effect [17]. If ε remains unaffected with increase in temperature then frequency also remains the same, which means that the local carriers are immobile. It is understood that the local charge carriers are immobile and the thermal activation in negligible. This further suggests that the samples possess a high chemical homogeneity and fine grain distribution. The presence of moisture, cellulose, hemicelluloses, lignin and pectin is also indicated by the electrical conductivity results. It is reported the presence of moisture plays an important role in the formation and stabilization of Al_2O_3 [18].

It is observed that the dielectric constant is increased rapidly at higher frequencies and showed almost frequency. The bulk polarization of the sample results from the presence of electrodes, which do not allow transfer of the charge species into the external circuit. At higher temperatures, is observed to be increased and it might be due to migration of the iron ion. The behavior of the dielectric permittivity with frequency is related to the applied field, which assists electron hopping between two different sites of the sample. At higher frequency region, the charge carriers will no longer be able to rotate sufficiently rapidly, so their oscillation will begin to lay behind this field resulting in a decrease of dielectric permittivity, Generally, the relaxation phenomena in dielectric materials are associated with frequency dependent orientation polarization[19]. From all figures, it is clear that the dielectric constant increases abruptly at higher frequencies and shows slight variation at higher frequencies, showing the dispersion of dielectric constant at lower frequencies due to the charge transport relaxation time. The dielectric constant increases because of doping. The large value of the dielectric constant is associated with inhomogeneous dielectric structure viz. impurities, structure and pores. The dielectric behavior shows the electronic polarizability at higher frequencies due to space charge polarization [19].

TEM

Natural sisal fiber consists of proton H+ molecule in its composition and deprotonation process is also occurred in natural fiber. Deprotonation is the removal of a proton (H+) from a molecule in table 1. Deprotonation of the radical cation is a major pathway and the proton removal decreases positive charge in the molecule and an increases negative charge. Deprotonation usually occurs from the donation of electrons or acceptance of the proton using a base, which forms its conjugate acid. Fig 3 (a) (b) and (c) shows the non linear and non uniform dispersed iron oxide particles of 141.24nm and the agglomerated fiber containing 30.31-30.90mn size particles of iron oxide. The samples appear highly strained as seen in fig 3 (a) (b) and (c). The presence of dislocation loops is clearly seen. It is possible that the strain present in the sintered samples has a direct effect on the dielectric loss.

Table 1

Name	Symbol	Classification	A	B
Cellulose	$C_6H_{10}O_5$	C-H Deprotonation	C	H
Hemicelluloses	$C_5H_{10}O_5$	C-H Deprotonation	C	H
Lignin	$C_{13}H_{34}O_{11}$	C-H Deprotonation	C	H

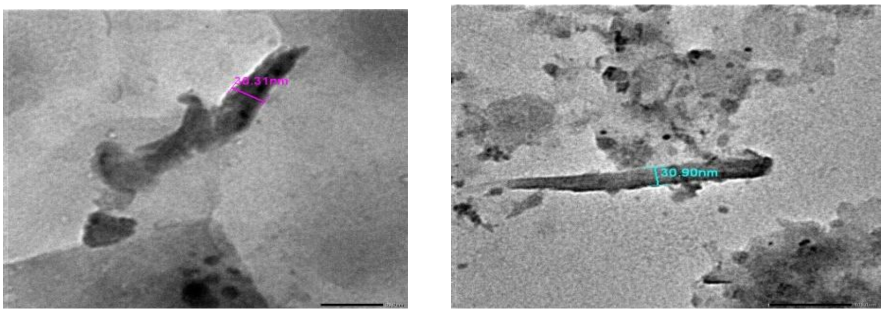

Fig 3 (a), (b)

Conclusion

The ferric and aluminum oxide synthesized by the sintering method has shown deprotonation behavior. The effect of chemical homogeneity, fine grain structure, particle size and shape of the ferrite and alumina samples are understood to affect the properties of dielectric behavior. H releases from deprotonation process which is responsible for conduction and it is shown in TEM. The effect of chemical homogeneity, fine grain structure, particle size and shape of the ferrite and alumina samples are understood to affect the properties of dielectric behavior. The presence of moisture, cellulose, hemicelluloses, lignin and pectin also contribute towards changes in dielectric properties.

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