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The Study of Correlation Between Physical, Dielectric and Electrical Properties of Mixed Metal Oxide

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Abstract: *It has been observed that the semiconducting metal oxides and / mixed metal oxides show changes in the electrical properties with sensitivity to humidity and / or various gases .Therefore attention of the research workers in solid state physics has been attracted towards the semiconducting oxide environmental sensors due to the growing concern towards the pollution control . Iron titanium oxides have received considerable attention as possible electrode materials for photo electrolysis of water. Also they are of interest to geologists, being the primary ceramics of rock magnetism.*
Fe2TiO5 has attracted attention due to its thermal expansion anisotropy and spontaneous microcracking. Electrical properties as a function of temperature and relaxation spectra are discussed in this paper. At temperatures 10000C and 12500C samples of Fe2TiO5 using rutile and anatase titanium oxide are sintered. Ceramic technique is used to synthesize these samples. The XRD and FTIR techniques are used for the confirmation of pseudobrookite phase of the samples. All the samples have orthorhombic structure. Relaxation spectra showed the space-charge, which is higher for higher sintering temperature for the sample prepared from rutile TiO2. The sample prepared from anatase TiO2 has lower dielectric and electric properties at low frequency (1kHz). The parameters such as dielectric constant, dielectric loss and resitivity are discussed and analysed on the basis of structural changes.

Key Words: Mixed metal oxide, Iron titanate, Relaxation spectra, order parameter

1. INTRODUCTION

A stable phase of Fe2TiO5 is formed by solid-state reaction between Fe2O3 and TiO2 [1]. This compound exhibits magnetic spin glass behaviour [2]. The short range antiferro- magnetic order is rendered by Ti layers and above 650 K it is destroyed [3,4]. Also for thermal, thermodynamic and crystallographic properties Fe2TiO5 has been investigated [5,6,7]. Orthorhombic or monoclinic phases are observed in this compound [8]. The random distribution of Fe3+ and Ti4+ ions in M1 and M2 sites which are octahedral makes this compound electrically an n-type semiconductor [9]. Many factors such as the sintering temperature, allotropes and purity of the starting materials used to synthesize Fe2TiO5 are likely to affect the cation distribution, randomization of this distribution, distortion and therefore the physical properties. TiO2 has three different allotropes, (i) anatase, (ii) rutile and (iii) brookite [10]. The rutile form is stable at all temperatures and pressures up to 60 k bar whereas other two forms are metastable. On heating irreversible transformation from anatase to rutile takes place and the enthalpy is as low from -1.3 to -6.0 kJ / mol [10]. Kinetically, at room the transformation of the anatase into the rutile does not occur and required temperature varies, it depends on many factors such as atmosphere, purity of material, stoichiometry, particle size , area of the surface, surface defects [11].

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II. EXPERIMENTAL

The A. R. grade α-Fe2O3, rutileTiO2 fine powders were throughly mixed and mixture was first presintered at 9500C for 24 hours. The presinterd mixture was homogenized in a mortar and then sintered at 10000C for 24 hours. By similar method another mixture of α-Fe2O3, anatase TiO2 was prepared . Finally the mixtures are sintered at 12500C for 24 hours. The samples are labeled as [FTR]1000, [FTR] and [FTA]. By X-ray diffraction these samples are analysed . LCR-Q meter was used at frequency 1 kHz to measure a.c. resistivity, Dielectric constant (K') and dielectric loss (K") within the range of temperature 300K-850K. Also, LCR-Z meter was used to measure K' and K" at room temperature in the frequency range 10 kHz-1000 kHz.

III. RESULTS AND DISCUSSION

3.1. ELECTRICAL PROPERTIES: The room temperature d. c. resistivities (ρd.c) which correspond to the grain boundary are much larger than their corresponding a. c. resistivities (ρa.c) implying the insulating nature and the presence of microcracks on the grain boundary (Table 1). The room temperature d. c. resistivities (ρd.c) which correspond to the grain boundary are much larger than their corresponding a. c. resistivities (ρa.c) implying the insulating nature and the presence of microcracks on the grain boundary (Table 1).

Table 1: Inhomogeneity, Space charge, Loop area of ρa.c hysteresis, Room temperature

a. c. and d. c. resistivities, , Transition temperatures, Activation energies and Band gap.

Sample	Inhomo-geneity	Space charge K'1kHz - K'1MHz	Loop Area of ρac hysteresis Sq. Units	ρac (1kHz) KΩm	ρdc KΩm	Transition temperature(K)		Activation Energy (eV)			Band gap (eV) (430K-525K)
						T1	T2	Ea1 (335K-430K)	Ea2 (430K-525K)	Ea3 (525K-850K)	
[FTA]	-0.0038	791	17	849	1040	400	710	0.22	0.45	0.80	0.90
[FTR]1000	-0.012	357	2.71	700	5835	335	525	0.25	0.69	0.40	1.38
[FTR]	-0.002	3331	1.17	5.5	6565	400	710	0.14	0.38	0.73	0.76

The very small values of a. c. resistivities (ρa.c) highlight the presence of large carrier concentration inside the grain. There is a strong correlation between a. c. resitivity (ρa.c) and space charge as well as inhomogeneity. Also the (ρa.c) is the most for the anatase sample [FTA] 1250 which has the least space charge contribution. Therefore, it is concluded that the a. c. resistivity (ρa.c) corresponds mainly to the space charge.

3.2. Measurement of a. c. resistivity (1 kHz) with temperature :

The variation of a. c. resitivity with temperature in the range 300 to 850 K for the samples is measured at 1 kHz frequency. The log (ρa.c) Vs 1/T plots in the Figures 1,2 and 3 correspond to [FTR] 1000, [FTR] and [FTA] respectively. The two temperatures T1 (335-400 K) and T2 (525-710 K) attributed to extrinsic-intrinsic and antiferro-paramagnetic transition [4] are shown in the curve. Table1 shows three distinct activation energies as (Ea1), (Ea2) and (Ea3) corresponding to the three temperature regions. Ea1 (335K-430K) occurs near room temperature and likely to correspond to the extrinsic conduction from impurities by polaron hopping [9,16].

Ea2 (430K-525K), therefore corresponds to the intrinsic conduction giving a band gap of the order of 1 -1.4 eV. This agrees well with the earlier reports [9,16, 17]. There is a third activation energy Ea3 (525K-850K). The transition from Ea2 to Ea3 occurs (Table 1) round a temperature T2 (525-710 K) at which antiferromagnetic ordering breaks [4]. Therefore, this activation energy corresponds to the paramagnetic samples.

It is Interesting to note that [FTR]1000 exhibits thermal antihysteresis in the log (ρa.c) Vs 1/T plot. Its cation distribution implies the possibility of the coexistence of two species namely fully disordered and fully (normal/ inverse) ordered adjacent to one another, which may activate the tunneling effect [18] giving rise to antihysteresis. It is also interesting to note that Ea3 < Ea2 for this sample (Table1). The loop area of thermal hysteresis of a. c. resistivity (ρa.c) is the largest for the sample [FTA]. The magnitudes of loop area corresponding to the dielectric and resistivity

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hystereses follow the same trend. Therefore, it is concluded that the origin of both the dielectric and resistivity hystereses lies in the presence of microcracks.

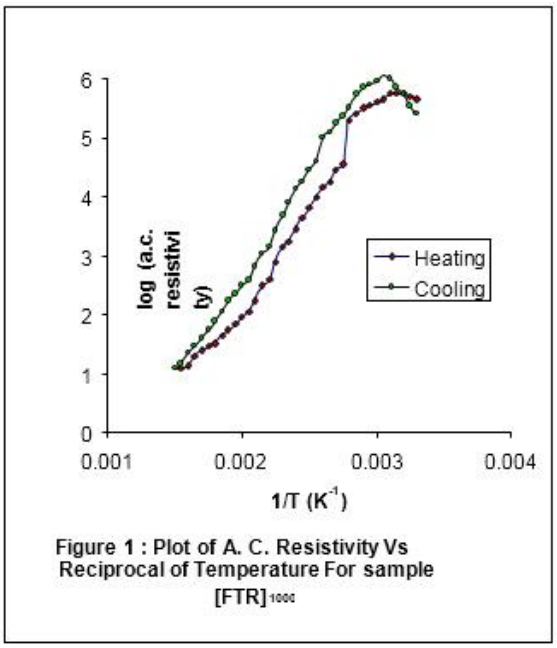


Figure 1 : Plot of A. C. Resistivity Vs Reciprocal of Temperature For sample [FTR]1000

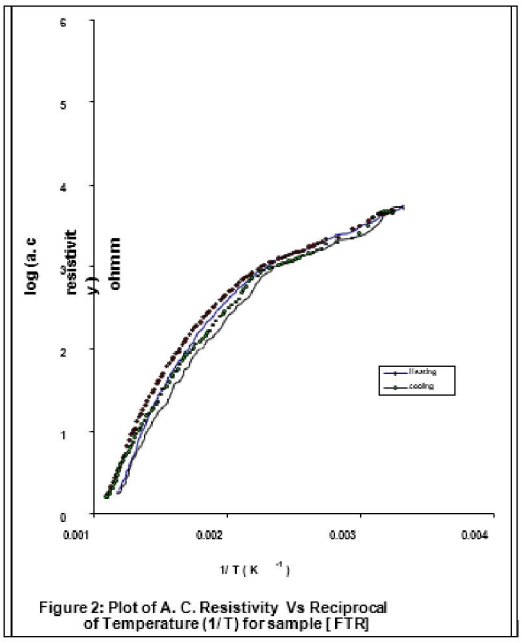


Figure 2: Plot of A. C. Resistivity Vs Reciprocal of Temperature (1/T) for sample [FTR]

3.3.: The Correlation between Structural, Dielectric and Electrical Parameters:

A very interesting picture emerges out in the case of Fe2TiO5. The Fe3+ always prefers a lower i. e. tetrahedral symmetry. At the same time it prefers an octahedral bonding. A Fe2TiO5 has two octahedral sites and therefore Fe3+ is capable of entering into any one of these sites. However, due to its preference for tetrahedral symmetry it distorts the octahedral site. The two octahedral sites of Fe2TiO5 being adjacent will have the opposing effect. Therefore, an order parameter is defined as,

λ = Fe3+ on M2 site - Fe3+ on M1 site.

when Fe3+ on M2 site = 1.5 and Fe3+ on M1 site = 0.5, the structure is fully symmetric (λo =1).

Therefore for inverse ordered cation distribution $\lambda' = (\lambda - \lambda o)2$

Therefore, the order parameter corresponding to the ordered cation distribution

[Ti1] [Fe2] O5 is $\lambda = 1$.

Similarly, corresponding to the inverse ordered cation distribution [Fe1] [Fe1 Ti1] O5 is $\lambda' = 1$.

Table 2: The correlation between structural, Dielectric and Electrical parameters.

Sample	Order Parameter λ'	Space Charge	Inhomo-geneity	Loop area of K' hysteresis (Sq. Units)	Loop area of ρa.c hysteresis (Sq. Units)	ρa.c (KΩm)	ρd.c (KΩm)
[FTA]	0.04	791	-0.0038	4.00	17.00	849	1040
[FTR]1000	0.14	357	-0.012	3.83	2.71	700	5835
[FTR]	0.31	3331	-0.002	3.02	1.17	5.5	6565

It is clear from the Table 2 that as the order parameter (λ') increases there is lowering of tetrahedral symmetry which results into increase in d. c. resistivity (ρd.c) and space charge and decrease in a. c. resistivity (ρa.c), loop area of K' hysteresis, loop area of ρa.c hysteresis and inhomogeneity. A strong correlation between ρa.c and space charge shows that the conduction is mainly due to the space charge.

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IV. CONCLUSIONS

The comparison of sample [FTR] with sample [FTR]1000 shows that, the Debye particle size and densities increase with the increase in the sintering temperature and inhomogeneity, porosity decrease. In short, growth of material improves with the sintering temperature. The higher sintering temperature also causes more Fe3+ to go to M1 site lowering its symmetry.

At higher sintering temperature (1250oC) the dielectric constant has increased about nine times. It is also observed that dielectric loss has increased about three times.

When [FTR] is compared with anatase sample [FTA], it is observed that the use of anatase increases inhomogeneity, porosity, Debye particle size, room temperature a. c. resistivity and the dipolar contribution at high frequencies and decreases densities. The cation distribution shows that in [FTA] Fe3+ prefers M2 site lowering its symmetry, which results into the increase in volume.

The sample [FTR]1000 exhibits thermal antihysteresis in the log (ρa.c) Vs 1/T plot. Its cation distribution implies the possibility of the coexistence of two species namely fully disordered and fully (normal/ inverse) ordered adjacent to one another, which may activate the tunneling effect giving rise to antihysteresis. It is interesting to note that Ea3 < Ea2 for this sample.

The loop area of thermal hysteresis of resistivity (ρa.c) is the largest in case of the sample [FTA]. It is observed that as the order parameter (λ') increases there is lowering of tetrahedral symmetry which results into increase in d. c. resistivity (ρd.c), space charge and decrease in a. c. resistivity (ρa.c), loop area of K' and ρa.c hysteresis and inhomogeneity. A strong correlation between ρa.c and space charge shows that the conduction is mainly due to the space charge. The magnitudes of loop area corresponding to the dielectric and resistivity hysteresis follow the same trend. Therefore, it is concluded that the origin of both the dielectric and resistivity hysteresis lies in the presence of microcracks and the sintering temperature reduces microcracking whereas the use of anatase widens microcracks.

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