

CHAPTER - III

ELECTRICAL CONDUCTIVITY OF Mg-Co FERRITES

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INTRODUCTION

The ferrites are oxide magnetic materials, having semiconducting properties. They possess wide range of resistivities, from 10^{-2} ohm-cm in Fe_3O_4 to 10^{11} ohm-cm in magnesium ferrites [1]. The electrical conductivity studies provide information about the usefulness of ferrites in electronics industries and computer technology. The high resistivity and permeability of magnesium based ferrites demands for high frequency applications. The charge transport mechanism in ferrites can be investigated with the help of conductivity, thermoelectric power and magneto resistance studies. The rate of conduction governs the dielectric and magnetic behaviour of ferrites. It has thus created considerable interest in the electrical conductivity and the frequency dependent dielectric behaviour of ferrites [2,3,4].

In polycrystalline ferrites, the electrical properties such as, high resistivity, low eddy current losses etc., are highly influenced by the heat treated conditions. Also, the presence of impurities affects the conductivity, determining the semiconducting nature of ferrites.

In this chapter the studies on resistivity of $Mg_{1-x}Co_x$ Fe_2O_4 ($x = 0, 0.4, 1$) samples; sintered at $900^\circ C$ for 20 and 30 hours have been reported. The details of conduction mechanism are included and the results are discussed on their basis.

3.1 ELECTRICAL CONDUCTION IN SOLIDS

The study of electrical conduction in solids is synonymous with the study of electrons in solids. A crystalline solid consists of a regular array of atoms, in continual state of vibration. Electrical charge transport occurs by the migration of charge carriers from one region of solid to another. In any free atom the number of positive charges equals the number of negative charges hence solid is normally electrically neutral. Conductivity depends upon number of charge carriers which allows the classification of solid state materials as metals, semiconductors and insulators. The charge carriers are free to move in metals, but do not move freely in semiconductors, because of some resistance during their migration. Earlier, the conductivity in solids was explained by assuming, the free electron model by Drude and Lorentz in 1900. They considered metal as a box filled with non-interacting electrons. According to recently accepted band theory, the electrons in crystals are arranged in allowed energy bands. These allowed energy bands are separated by the regions of energy for which no wave like

electron orbits exist. These are called as forbidden bands or band gaps. The band in which valence electrons are present is called valence band. It consists of discrete energy levels. The highest point in the valence band is called valence band edge. The higher energy band in which conduction electrons are present is called conduction band. The lower point in conduction band is called as conduction band edge. Band gap is the energy difference between the conduction band edge and valence band edge.

For conduction to occur, the conduction band should be partially filled by the electrons. If it is completely empty or completely filled conduction does not take place. This is a consequence of Pauli's exclusion principle, according to which no two electrons in an atom can occupy one quantum state because of variations in quantum numbers. There is no possibility for the electron to change over the state, hence conduction is impossible.

Based on the width of band gap all solid materials are classified into three heads as, metals, semiconductors and insulators. In metals the conduction band overlap the valence band hence no band gap exists. Therefore, a small driving force or potential difference is sufficient to transfer the charge carriers. In semiconductors there is a narrow band gap imparting partial conductivity to these materials. In case of

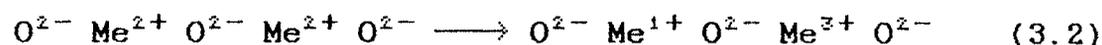
insulators large band gap is observed, hence it is impossible to impart conduction to these materials by any means such as heat treatment or increasing potential etc.

3.2 ELECTRICAL CONDUCTIONS IN OXIDES

In majority, the 'd' block element oxides are semiconductors. The electrical conductivity of these oxides are represented by the equation,

$$\frac{A}{T^{B-1}} \exp\left[\frac{-B}{KT}\right] \quad (3.1)$$

where A and B are constants. The activation energy ranges, upto 1.5 ev and very much smaller values are encountered at sufficiently low temperature. For an oxide of composition MeO, the activation of a conduction electron may be represented by,



Before activation

After activation

where Me = Transition metal ion.

The energy needed for the relation of Me^{3+} and Me^{1+} ion pairs corresponds to the energy gap in the density of states. On the basis of electron correlation, one has to consider the following terms for the contribution to activation energy.

- i) The difference between ionization energy and electron affinity of the free Me^{2+} ion.

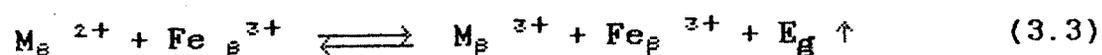
- ii) The difference in Madelung energy of the two configurations represented by equation (3.2).
- iii) The difference in stabilization of crystal field of above configuration.
- iv) The polarization energy of the surrounding crystal lattice.

The polarization term (iv) affects significantly mobility of charge carriers (electrons or holes) which are bound to impurity centres. The first factor may decide the resistivity of polycrystalline samples, but the purity of the starting chemicals and the thermal history of preparation give variation to this contribution at different time. Activation energy depends on all above factors.

3.3 CONDUCTION IN FERRITES

The spinel lattice is formed by large number of oxygen ions and small number of divalent metal cations are situated on oxygen tetrahedra and octahedra. The bond formation in ferrite compounds is almost of ionic character. The conductivity in ferrite is governed by 3d-like wave function. According to Verwey and De Boer [5] both Fe^{2+} and Fe^{3+} ions are present at the same octahedral B-site in spinel structure, conduction takes place when the electrons move from divalent iron ions to trivalent iron ions. The direct motion of electrons between Fe^{2+} can be neglected as

conduction is assumed to be due to e^- electrons. In a stoichiometric compound the ferrous ions created by the thermal activation, according to relation,



The index β denotes the octahedral site and 'M' is a divalent metal ions such as Ni, Mn, Co etc. 'Eg' is the energy required to transfer the electron from M_{β}^{2+} to Fe_{β}^{3+} . As an example, in case of $NiFe_2O_4$ the presence of nickel on octahedral site favours the conduction mechanism [1] as,



This explains the conduction mechanism in $NiFe_2O_4$.

3.4 CONDUCTION BY POLARONS

In a crystal lattice, a moving charge interacts with the charges of ions. Hence the atoms of the lattice create a local deformation or the strain in the lattice. This deformation or strain of the lattice tends to follow the electrons, when electron moves through the lattice. Due to this the effective mass of the electrons increases. Heavy ion cores are set into motion as long as the electron moves forward. In other words, electron partially drags the ions along with it. The strong Coulomb interaction in ionic crystals such as ferrites exhibits this effect. The combination of the electron and the strain lattice field is

called as "polaron" [6]. The strength of lattice interaction is measured by a coupling constant defined as,

$$\frac{1}{2} \alpha = \frac{\text{Deformation Energy}}{h \omega}$$

where ' ω ' is the longitudinal phonon frequency. Lattice vibration is of elastic nature due to the thermal energy. Phonon is nothing but the quantized energy of lattice vibration, similar to that of photon of electromagnetic wave. The value of coupling constant ' α ' is high in ionic crystals and low in covalent crystals. When α is higher, polaron is called as "larger polaron" and smaller value of α helps to refer polaron as "smaller polaron". The electrons associated with a large polaron moves freely in a conduction band but the electrons associated with small polaron get trapped inside the single ion and hence spend more time. Due to high temperature treatment the electron moves from site to site by thermally activated hopping but at low temperature, the electron tunnels slowly through the crystals. Holes or electrons may get trapped by including a local deformation of lattice. Crystal polarization and band edge degeneracy cause this effect.

3.5 SCATTERING OF CHARGE CARRIERS

The charge migration through the crystal is difficult due to the presence of electrostatic fields, impurities, electrical strains, thermal vibrations, degeneracy of band edges. They distort the path of conduction electrons during

migration. It is usually called as scattering of charge carriers. It imparts resistance for charge motion.

During thermal treatment, the atoms in lattice are pulled apart and lattice gets somewhat expanded. The displaced atoms thus alter the electric potential of their neighbouring atoms. The effect of application of external electric field is changing the magnitude and direction of the moving particle's momentum. This effect is described as thermal scattering or lattice scattering [7]. In extrinsic semiconductors, presence of doner or acceptor impurities alters the concentration of stray electrons or holes. In such a case, electron bound to impurities the radius of the order of few lattice spacing so that appreciable deflection of a few moving charge carriers will occur at distances much grater than the lattice space. The impurity scattering depends on the temperature. When both electrons or holes present inside the semiconducting material, the conduction electrons and holes present inside the semiconducting material, deflect each other during their cross over or transit. Thus scattering takes place.

3.6 FACTORS CONTRIBUTING TO RESISTIVITY OF FERRITES

The stoichiometry of ferrite compound affects the resistivity. The electrical resistivity of ferrites depends markedly upon amount of iron present in the lattice of ferrous state. If concentration of ferrous ions is higher, resistivity ceases. Reduction of resistivity also occurs when sintering

temperature is higher. The conductivity also depends upon cation distribution, presence of impurities, chemical valence etc. Van Uitert (1950) showed that high resistivity ferrites can be prepared by,

a) avoiding excess iron in the lattice or
b) by adding small quantities of magnesium in nickel ferrites or cobalt in magnesium ferrites. Due to addition of such ions the formation of Mg^{+2} or Ni^{+2} is inhibited. In mixed ferrites the conduction mechanism gets diluted in both the sets of lattice sites. For example, in case of Mg-Co ferrites magnesium enters octahedral sites. The partial substitution of cobalt for magnesium dilutes the conduction that occurs through the tetrahedral sites. The conduction can be divided as p-type conduction and n-type conduction. When the cation of high resistivity material is substituted by a cation, that tends to stay in lower valence state it leads to the p-type conduction. On the other hand if cation is substituted by cation which stays in higher valence state, it forms n-type conduction [8].

As far as the type of conduction in Mg-Co ferrite system is concerned it is observed that the $MgFe_2O_4$ is n-type semiconductor and $CoFe_2O_4$ is p-type semiconductor. The resistivity of mixed system is thus due to electron-hole compensation [9].

3.7 HOPPING OF POLARONS IN FERRITES

The presence of ions in spinel crystal structure produces the electrostatic crystal field inside the lattice. Hence there is always Coulomb interaction between charge carriers such as electrons or holes and the ionic crystal field of this lattice. Since during the conduction process the transporting electrons pulled the ions along with them, ultimately ions get displaced from their mean positions. This displacement of ions leads to the polarization of the surrounding region. A polarization well is formed inside which charge carrier get situated at the centre. If the well is sufficiently deep the carrier is trapped at the lattice site and it can be activated by the thermal treatment. Generally the combination of potential strain and electron is described as "polaron". The charge from one site to other is transported via polarons in ferrites and called as "polaron hopping". The relation between conductivity, mobility and charge concentration using polaron hopping mechanism is given by,

$$\sigma = e (n_e \mu_e + n_h \mu_h) \quad (3.5)$$

σ is the conductivity

n_e is the concentration of electrons

n_h is the concentration of holes

e is the electronic charge

μ_e is the mobility of electrons

μ_h is the mobility of holes

3.8 ENERGY OF ACTIVATION

Energy of activation is defined as the minimum energy required for electron to jump from valence band to conduction band. This view point is mostly useful in case of pure single crystalline semiconducting materials. But in case of polycrystalline materials, presence of impurities may give rise to trap levels and localized states [10]. Impurities such as pores provide insulating path to conduction electrons. Magnetization states also affect the conduction mechanism. Hence conventional band theory is insufficient to give full explanation of the conduction properties of ferrites. Therefore, instead of idea of "band gap" which is used for semiconductors, a new concept of "energy of activation" is introduced in case of oxide semiconductors. For this, some discrete conducting states separated by forbidden nonconducting states is to be considered instead of conduction band and valence band. In order to transfer the electron from lower state to higher state, the carriers should be able to cross the potential barrier after activation by some external agency. Activation energy is thus the minimum energies needed for an electron jump from one state to the higher state.

3.9 TEMPERATURE DEPENDENCE OF ELECTRICAL RESISTIVITY

As in semiconductors, the electrical resistivity of ferrites is sensitive to temperature changes. In order to diffuse the carriers from one conduction state to another it must exceed certain minimum energy barrier, ΔE . According to Boltzmann statistics, the thermal energy of the carriers to pass over the barrier height ΔE , varies exponentially with temperature. Due to thermal vibration, phonons are constantly created and the electron jumps between the pairs of the states by absorbing or emitting phonons alternately. The charge transport takes place via thermally activated phonons. Using these considerations the temperature variation of resistivity can be represented by the equation as,

$$\rho = C \exp \left[\frac{\Delta E}{KT} \right] \quad (3.6)$$

Where 'C' is the temperature dependent constant in conduction by diffusion process, K is the Boltzmann constant,

T is the absolute temperature and

ΔE is the activation energy.

In ferrites, conduction constant C can be replaced by a temperature independent constant ρ_0 . Hence equation (3.6) becomes

$$\rho = \rho_0 \exp \left[\frac{\Delta E}{KT} \right] \quad (3.7)$$

Komar and Klivshin [11] determined that the nature of the graph $\log \rho$ versus $\frac{1}{T}$ is linear and the break point appears at Curie temperature. The slope of straight line $\left[\frac{\Delta E}{K}\right]$ is used to calculate the energy of activation.

3.10 EXPERIMENTAL SET UP FOR RESISTIVITY STUDIES

The circuit diagram and experimental set up for the measurement of resistivity is shown in figs. 3.1 and 3.2 respectively. For measurement of resistivity two probe method was used. The sample holder consists of two small cylindrical brass electrodes which are fitted in the two porcelene discs. The porcelene discs are held firmly by connecting rods with the help of nuts. The sample fits between two cylindrical rods. Screws are provided on brass rods for external connection. Silver wires were used for external connections.

The ferrite sample in the form of pellet was fitted between two brass rods. For good ohmic contact the sample was polished and silver paste was applied on both sides. The entire assembly was then kept in the temperature regulated furnace. The calibrated chromel alumel thermocouple was used for the measurement of temperature.

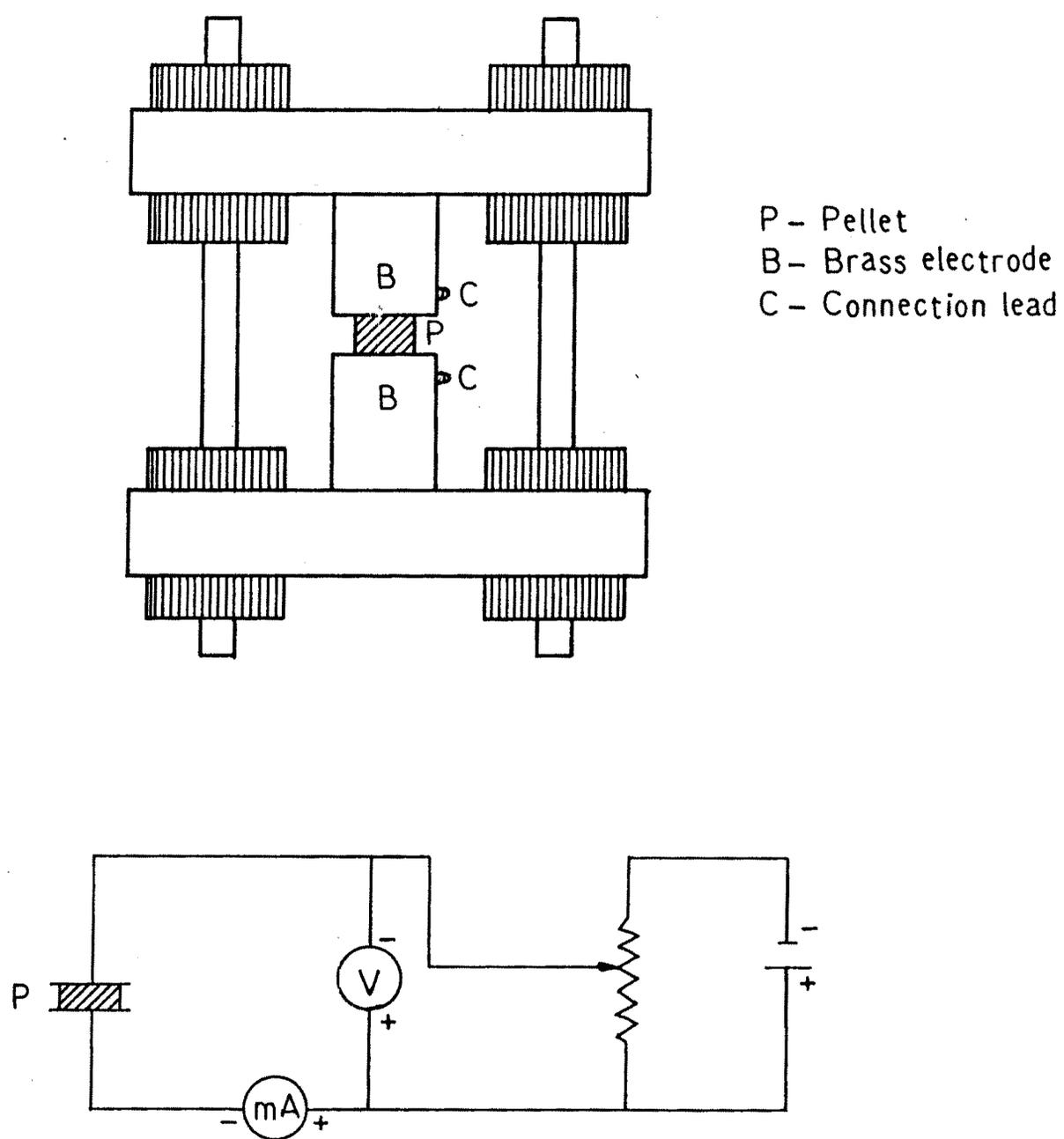


Fig.3.4 Sample holder and circuit diagram for electrical resistivity

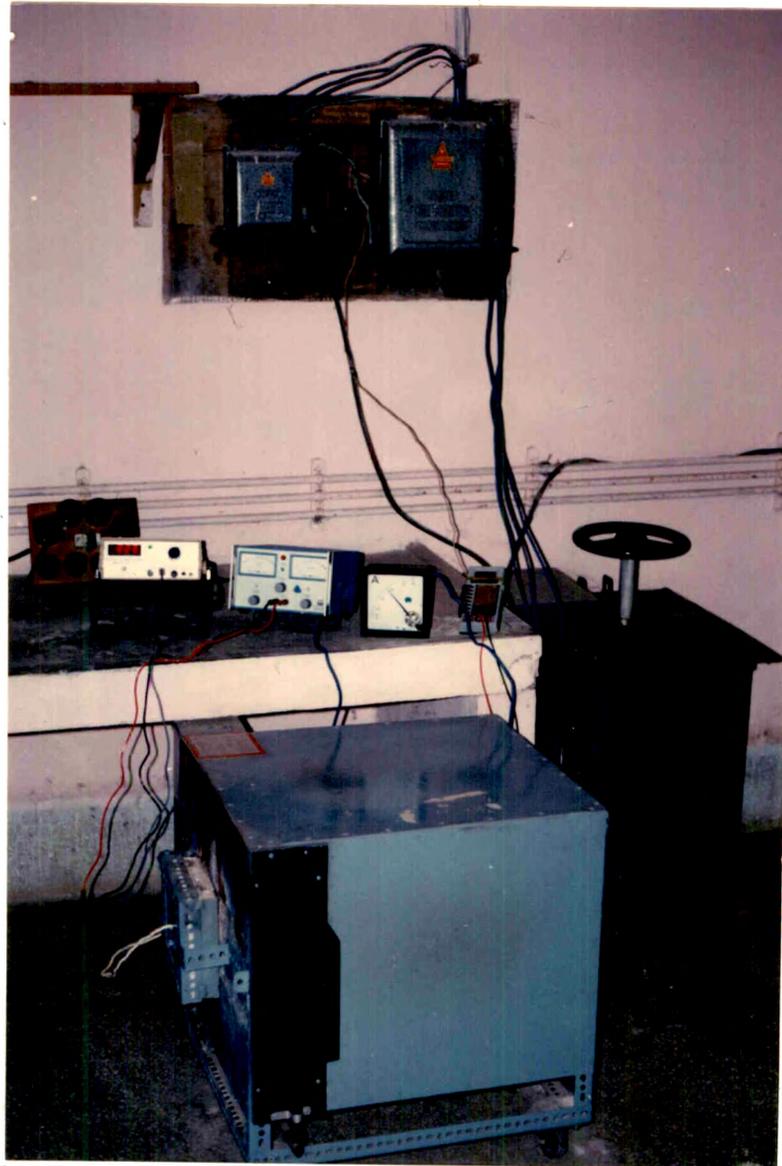


Fig.3.2 EXPERIMENTAL SET UP FOR THE MEASUREMENT OF RESISTIVITY.

3.11 MEASUREMENT OF D.C. RESISTIVITY

The d.c. resistivity of the ferrite system $Mg_{1-x} Co_x Fe_2O_4$ ($x = 0, 0.4, 1$) sintered at $900^\circ C$ for 20 hrs and 30 hrs was measured using conductivity cell. The D.C. supply of 5V was taken through the transistorized power supply unit. The thermo emf generated was measured with electronic digital multimeter. The variation of d.c. current was noted with respect to the potential difference across the sample.

The circuit used automatically eliminates the loading effect of voltmeter and reads the correct voltage across the pellet. The temperature of the furnace was gradually increased from room temperature to $650^\circ C$ and reading of current (in microampere) was noted, for every one millivolt difference in thermocouple emf. The resistance of the sample was calculated by using Ohm's law and resistivity by the relation

$$\rho = R \frac{A}{h}$$

Where, 'A' is the area of cross section and 'h' is the thickness of pellet.

3.12 RESULTS AND DISCUSSION

The electrical resistivity measurements were made on $Mg_{1-x} Co_x Fe_2O_4$ ($x = 0, 0.4, 1$) samples from room temperature to $750^\circ C$ with experimental set up discussed above. The plots



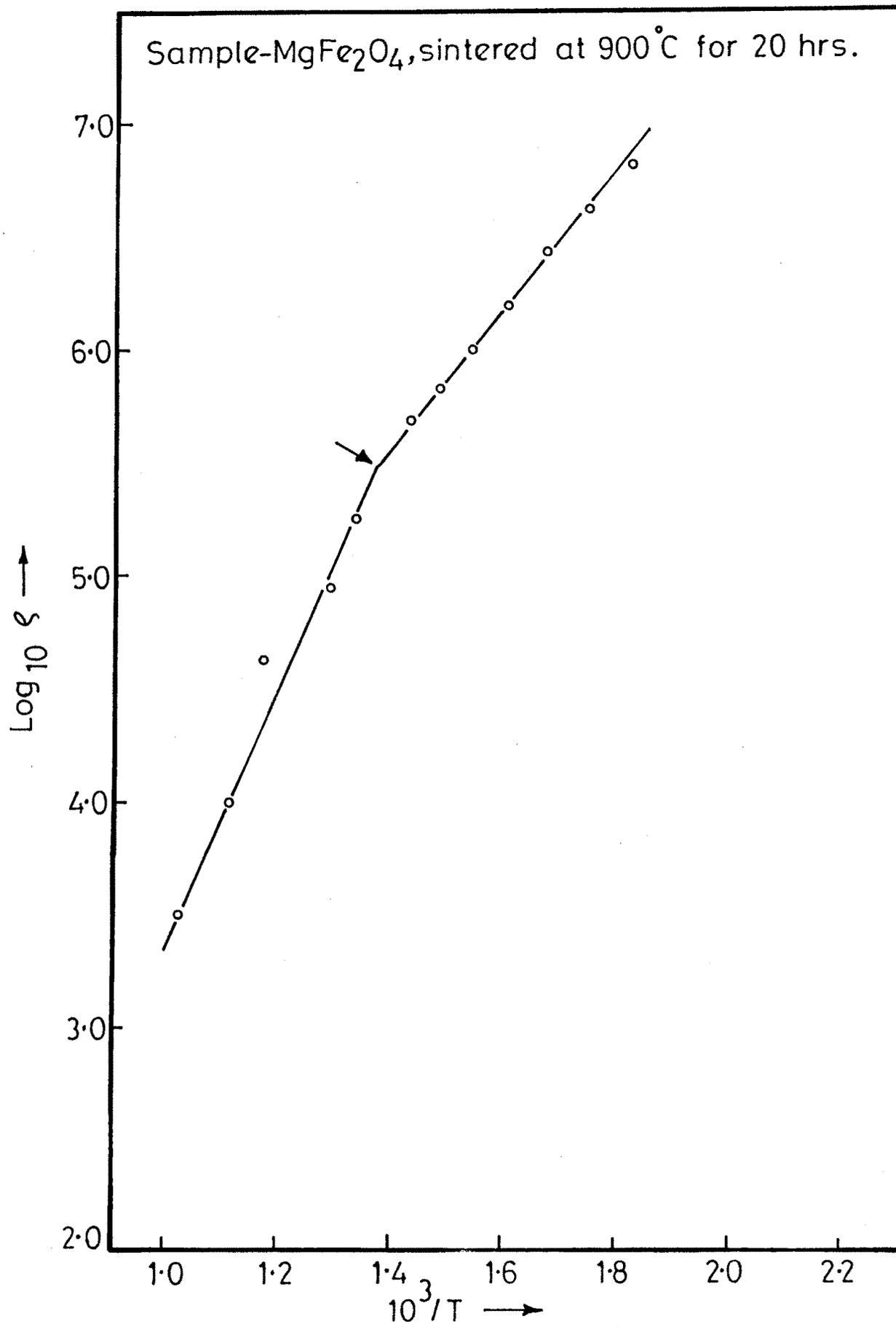


Fig.3.3- $\text{Log}_{10} \epsilon$ vs $\frac{1}{T} \times 10^3$

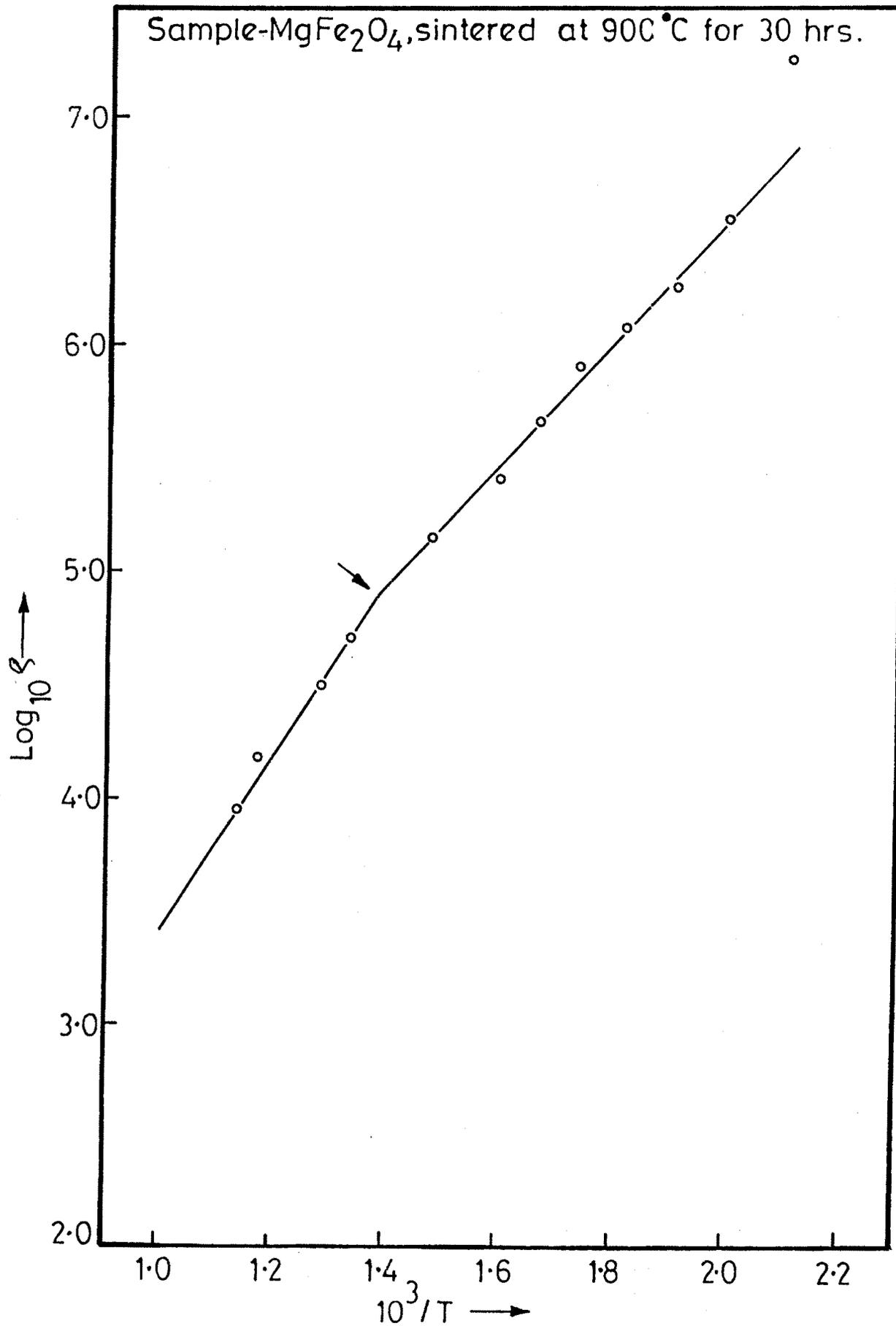


Fig 3.4- $\text{Log}_{10} \sigma$ vs $\frac{1}{T} \times 10^3$

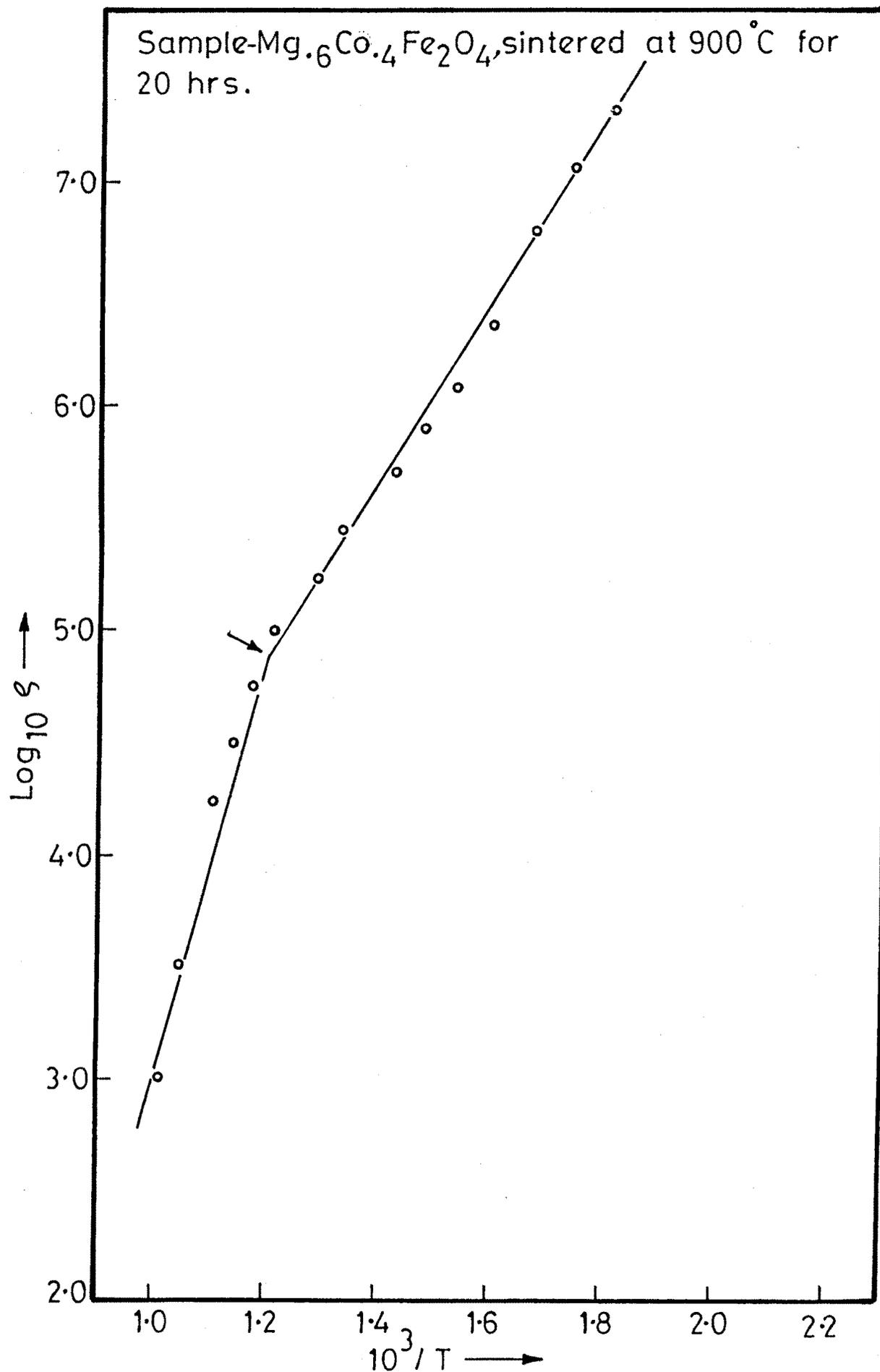


Fig.3-5- $\text{Log}_{10} \epsilon$ vs $\frac{1}{T} \times 10^3$

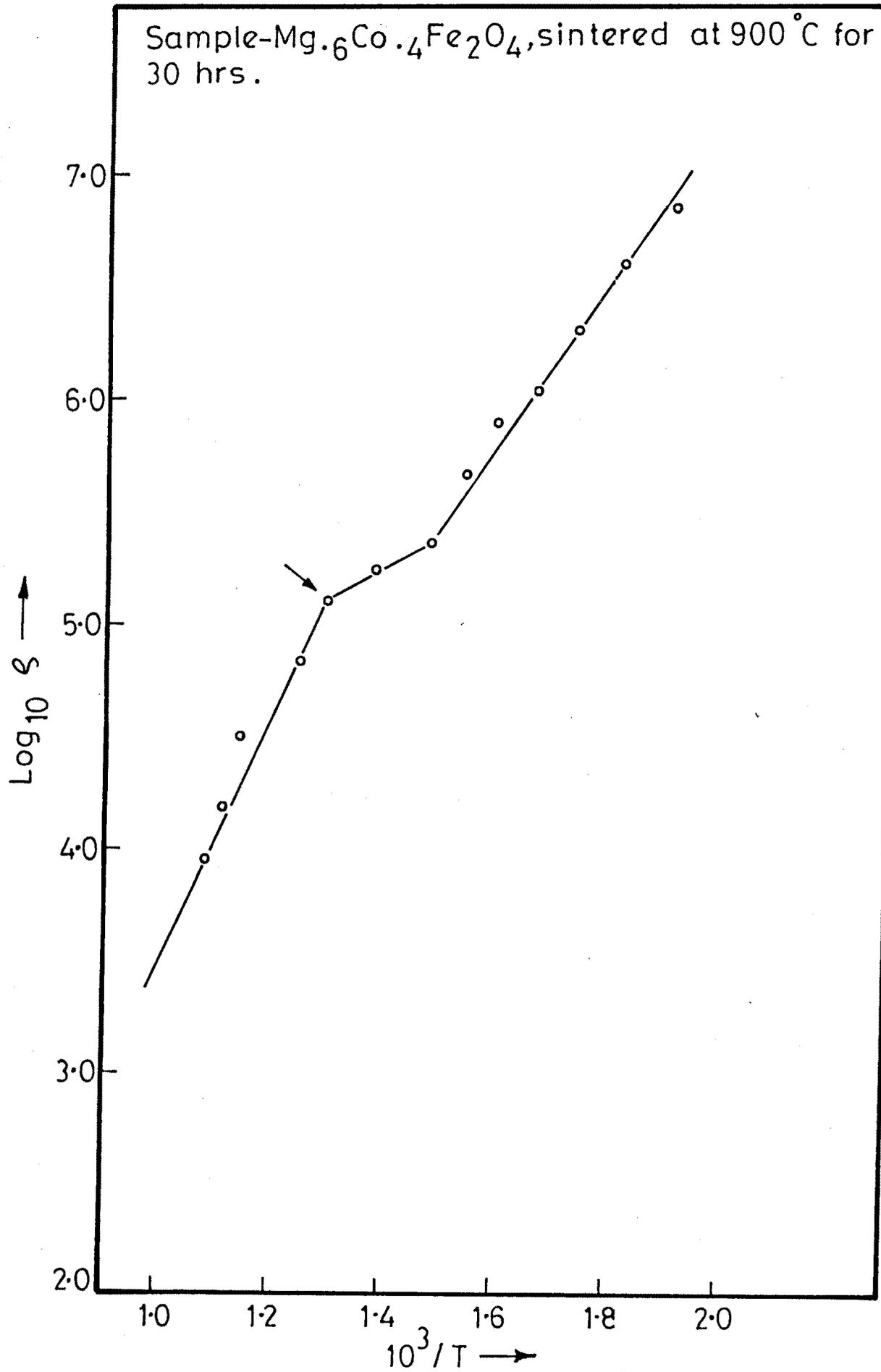
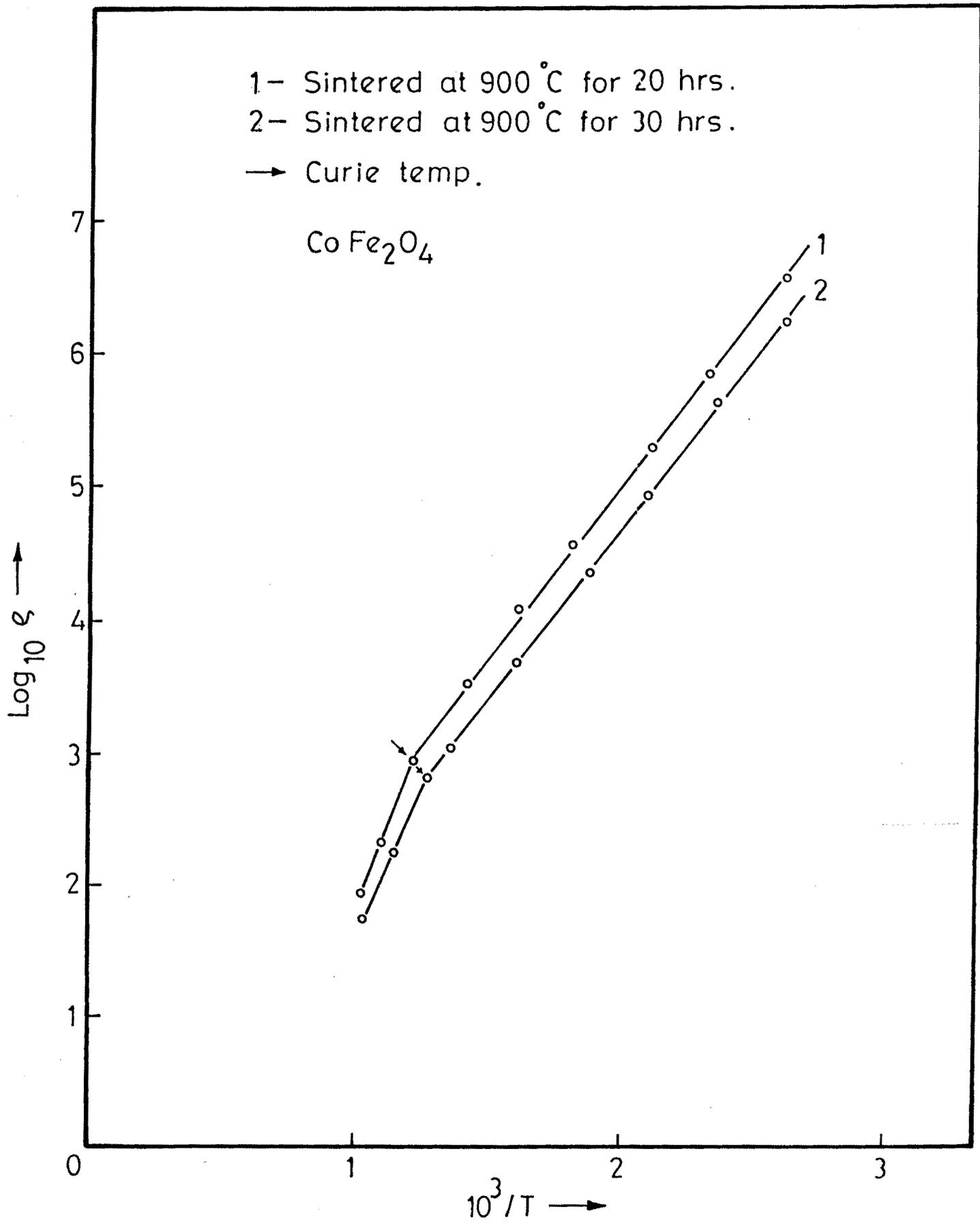


Fig.3.6-Log₁₀ ε vs $\frac{1}{T} \times 10^3$

Fig.3.7- Log₁₀ ε vs 10³/T

T A B L E - 3.1

Activation energy and Curie temperature data of Mg-Co ferrites

Sample	Sintering temperature and time		Activation energy in eV		Curie temperature in °c from $\log_{10} \rho \rightarrow 1/T \cdot 10^3$ graph
	Temp., °c	Time, hrs	paramagnetic region	ferrimagnetic region	
MgFe ₂ O ₄	900	20	1.048	0.5957	450
MgFe ₂ O ₄	900	30	0.8843	0.5886	450
Mg _{0.6} Co _{0.4} Fe ₂ O ₄	900	20	1.629	0.7502	525
Mg _{0.6} Co _{0.4} Fe ₂ O ₄	900	30	1.031	0.6645	500
CoFe ₂ O ₄	900	20	1.03	0.4824	546.7
CoFe ₂ O ₄	900	30	0.9269	0.4804	520.7

of $\log \rho$ versus $1/T \times 10^3$ are represented in figures. 3.3 to 3.7. In all the three ferrites under study, the marked change in slope of straight line is observed. It can be seen from the figures that break in plots occurs at temperature which correspond closely with the observed ferrimagnetic Curie temperatures. At this temperature, the ferrimagnetic material changes over to a paramagnetic one. The break in the curves is indicated by an arrow. The change in slope is either due to Curie temperature [11] or change in conduction mechanism [12]. In both the regions, resistivity plots obey the Arrhenius relation of temperature dependence of resistivity, viz

$$\rho = \rho_0 \exp \left[\frac{\Delta E}{KT} \right]$$

Plots of $\log \rho$ versus $1/T \times 10^3$ are linear and with rise in temperature $\log \rho$ decreases, determining semiconductor nature of the ferrites under study.

The activation energies in paramagnetic and ferrimagnetic regions are contained in table 3.1. These are calculated from the slopes of straight lines shown in figs. 3.3 to 3.7. From the table it is evident that in all the samples the activation energy in paramagnetic region is higher than that in ferrimagnetic region. The data on Curie temperatures obtained from $\log \rho$ vs $1/T$ plots is also given in table.3.1. The

observed Curie temperatures agree well with the values reported earlier [13].

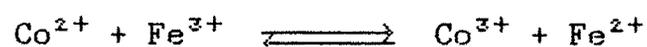
The explanation of the fact that, the change in slope of straight line occurs, while passing through the Curie point is given by Irkhin [14] et al; based on fully theoretical treatment. The spin-spin interaction between the atoms in upper ferrimagnetic region vanishes due to the thermal vibrations caused by the heat treatment. This would transform the sample from ferrimagnetic to paramagnetic state. Break point or Curie point is thus, the point of separation, between ferrimagnetic and paramagnetic region. The change in slope indicates, the change in activation energy in two regions.

Different mechanisms are available for the transition temperature, where conduction in sample shows the break. According to Komar and Klivshin [11] change in slope of the plot $\log \rho$ versus $1/T$ is because of magnetic transition. The B-B distance on the octahedral site is much larger than the sum of ionic radii of the cations. The result is that, the electrons are not free to move in the crystal but remain fixed to B-site. However, the conduction is due to the activation of states on account of change in cation valency i.e. $\text{Fe}^{2+} \rightleftharpoons \text{Fe}^{3+}$ hopping mechanism. The discontinuity is explained as ordering of Fe^{2+} and Fe^{3+} ions on octahedral B-site [15] accompanied by a small change in

crystal structure. The lowering of activation energy is contributed to the spin ordering effect [16]. In short, change in slope at Curie temperature is mainly due to spin disordering of electrons. [17]. This indicates that, there is some predominant change in conduction mechanism due to magnetic phase transition.

Conduction mechanism in ferrites is explained by Klinger [18], by using two phase polaron model. According to him, low temperature conduction is via magnetically co-related polaron while at high temperature, it is via Brownian tunneling motion of polarons. Since the polarons have high activation energy in nonmagnetic region and low activation energy in magnetic region [19]; mobility should change. Fröhlich [20] introduced quantum mechanical approach to polaron model, to calculate the actual mobility of polarons in crystals.

The presence of cobalt on octahedral sites favours the conduction mechanism of



type in cobalt magnesium ferrites. Parker et al [21] studied the electrical conductivity of NiFe_2O_4 ferrite with a small substitution of cobalt and established two valence states of cobalt.

It was pointed out by Broese Van Groenou [22] that the presence of Fe^{2+} ions among the Fe^{3+} ions gives rise to n-type

conductivity at sufficiently high temperature. This is due to the movement of extra electron on Fe^{2+} ion moving through the crystal. A similar situation exists for Co^{3+} and Mg^{3+} ions in CoFe_2O_4 and MgFe_2O_4 respectively. In this case deviating valency is higher than that originally present (Co^{2+} , Mg^{2+}) and p-type conduction is found. Thus in iron excess ferrites, Fe^{2+} content is important and conduction is due to hopping of electron from Fe^{2+} to adjacent Fe^{2+} ions leading to n-type conductivity i.e. $\text{Fe}^{3+} + e \rightleftharpoons \text{Fe}^{2+}$ with low value of activation energy. Such behaviour is found in case of MgFe_2O_4 ferrite, whereas low conductivity in CoFe_2O_4 is due to deficiency of iron leading to p-type conductivity with relatively large activation energy (Table 3.1).

The lowest conductivity in $\text{Mg}_{1-x}\text{Co}_x\text{Fe}_2\text{O}_4$ with relatively largest activation energy may be attributed to the electron-hole compensation effect [23].

The background of the general results of Curie point and activation energies in ferrites helps to discuss the results of $\text{Mg}_{1-x}\text{Co}_x\text{Fe}_2\text{O}_4$ ($x = 1.0, 0.4, 0.0$) ferrite samples sintered at 900°C for 20 hours and 30 hours respectively.

From figures 3.3 to 3.7 it is clear that the resistivity of Mg-Co ferrites decreases when sintering time is increased. This is evident from decrease in activation energy value of

each composition after higher sintering time. Reszlescu, Cucireanu, et al [24] studied the time variation of electrical conductivity in spinel ferrite samples sintered and quenched quickly. They found that electrical conductivity decreases with relaxation time for CoFe_2O_4 and increases with time for MgFe_2O_4 . The value for conductivity of CoFe_2O_4 at room temperature as found by Ramanamurthy [25] to be $\sigma = 1.97 \times 10^{-8} \text{ Ohm}^{-1} \text{ cm}^{-1}$. Reziescu et al found the σ value at $1100^\circ\text{C} = 5.7 \times 10^{-4} \text{ Ohm}^{-1} \text{ cm}^{-1}$. Hence in CoFe_2O_4 conductivity increases with temperature. Similar thermal effect of conductivity observed in MgFe_2O_4 . For MgFe_2O_4 sample $\sigma = 1.25 \times 10^{-8} \text{ ohm}^{-1}$ at 27°C and at 1100°C $\sigma = 4.65 \times 10^{-2} \text{ ohm}^{-1} \text{ cm}^{-1}$.

Reddy and Satyanarayana et al (1984) have studied the electrical properties of MgFe_2O_4 and stated that MgFe_2O_4 is an intrinsic semiconductor. It is observed in both cases because of the presence of single phase in X-ray diffraction pattern. As reported recently Mg-based ferrites can be used as negative temperature thermistors [9].

In figures 3.3 to 3.7 it is observed that the plots for samples sintered for larger sintering time lie towards lower temperature side and vice versa. This reveals the fact that with increasing sintering time resistivity decreases. This

leads to the shifting of Curie temperatures towards lower temperature side. This is due to the fact that a sample having higher grain size favour easy transition between ferri and para regions. The porosity for each sample is calculated and tabulated in Table.4.1. It is in accordance with the results mentioned above.

The variations in electrical properties occur since, the microstructural changes are brought in during sintering. Polycrystalline ferrites show higher resistivity in the presence of pores, grain boundaries and other impeding agents including substitutional and impurity ions. The electrical properties such as conductivity, eddy currents, permeability are governed by the heat treatment. The presence of impurities greatly affects the conducting properties of ferrites as in the case of semiconductors. The factors contributing to the measured electrical properties are as follows.

- i) Porosity increases the resistivity markedly, the presence of pores filled by air may provide insulating path to the electrons which give rise to high resistivity.
- ii) Oxygen stoichiometry : An oxygen vacancy contributes two extra-electrons to 3d-band. If these extra charges are detached from their vacancies by thermal agitation;

n-type conductivity is induced in oxygen deficient and p-type conductivity in oxygen excess materials [9].

iii) Heat treatment during the preparation process changes the electrical properties of polycrystalline ferrites due to rapid oxygen dissociation pressure [26]. This leads to the formation of small amount of divalent ion during firing and ferrite possesses high conductivity.

iv) The reduction of activation energy at higher temperature is also associated with the reduction in grain boundary area. The grain size of individual crystallites influences the conduction due to increased number of grain to grain contact [27]. The presence of air gap between the grains results in the formation of inhomogeneous dielectric structure [28]. This greatly affects the a.c. and d.c. conductivity of ferrites and hence conduction mechanism in ferrites is largely dependent on porosity. Therefore, smaller the porosity higher will be the conductivity.

At higher temperature densification occurs resulting in decrease of porosity, hence the resistivity ceases. Therefore, it can be emphasized that one of the factor for higher conductivity in ferrites is the increase in average grain size and removal of porosity during heat treatment.

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