
CHAPTER - III

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ELECTRICAL CONDUCTIVITY OF $Mg_xZn_{(1-x)}Fe_2O_4$ FERRITES

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INTRODUCTION

Magnesium ferrite and allied compounds have found widespread applications in microwave devices. They may indeed be counted among the most versatile of the ferrites. This is largely due to the comparatively low magnetic and dielectric losses obtainable and in particular, to the high resistivities which can be obtained by selection of suitable minor additives and use of appropriate firing procedures. Among the binary ferrites that include Magnesium ferrite as one of the major components, Magnesium-Zinc ferrite has been the most thoroughly examined. This work was largely motivated by a desire to corroborate and study the ramifications of Neel's theory (1).

In this chapter conduction in oxides and ferrites is briefly discussed. The explanation of the results on the temperature dependent measurement of resistivity of the Mg-Zn ferrite sintered for two different times at the same temperature is presented.

3.1 CONDUCTION MECHANISM

The metal oxides have predominant ionic bonding character. The electrical conductivity is mainly described by the electrons with 3d like wave functions the outer electronic configuration of O^{2-} being stable. The satisfactory conclusions of the eigen values of these functions remain the problem of great difficulty in solid state physics. Therefore the realistic energy level scheme must take help of the simplified theoretical models based on empirical rules. As far as the electrical properties of oxides are concerned the models are successful only in providing the semiquantitative explanations of experimentally established results.

Energy level schemes in solids are on the basis of any one of the two initial postulates, viz. the nearly free electron model and tight bonding approximation. In free electron model, the electrons are assumed to move throughout the solid and small perturbation is supposed to be due to interaction of particles. In the latter, it is assumed that the solid consists of an array of atoms and interaction may be treated as a small perturbations of the properties of the free atoms.

The 3d levels in the first transition elements are systematically filled from Sc to Ni. In the crystalline field of solid, the levels are split into a triplet and doublet. The triplet states lie below

doublet states in oxides with rocksalt structure. These oxides are then expected to be metallic conductors at least above the antiferromagnetic disorder temperature (2). Below the Neel temperature exchange process could give rise to further splitting of 3d band. The oxides behave as semiconductors at all temperatures and have intrinsic activation energies far in excess of those which would be accounted for by exchange splitting. It is evident from this that descriptions in terms of conventional band theory fail to account for the electrical transport properties. The failure has its origin in the low mobility of electrons in 3d band.

3.2 ELECTRICAL CONDUCTION THROUGH SOLIDS

There are different types of electrical conductors; metallic, ionic and semiconductors. Good conduction is generally associated with the metals. Semiconductors are of considerable interest because of certain unique and useful characteristics. In many respects, the semiconductors resemble the ionic conductors. The electrical charge transport occurs by migration of charge carriers in the solid state material. The charge carriers though free in metals do not move freely in semiconducting substances, but suffer some resistance during their migration.



All solids contain electrons, according to band theory the electrons in the crystal are arranged in allowed energy bands separated by forbidden bands, these bands are called band gaps or the energy gaps represented as Eg. Generally, in semiconductors the charge transport occurs due to the migration of an electron from valence band to conduction band.

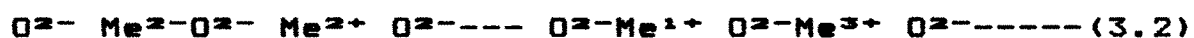
At room temperature the conduction band is partly filled with electrons. The completely empty or completely filled bands have no capability of conducting electricity. This is the consequence of Pauli's exclusion principle. In insulators the band gap is very wide and thermal electrons are unable to cross the potential barrier. There is narrow band gap for semiconductors, imparting partial conductivity to these materials.

3.3 CONDUCTION IN OXIDES

The majority of the transition metal oxides are semiconductors and their electrical conductivity may be described by an equation

$$\sigma = A/T^{(n-1)} \exp(-B/KT) \text{ ----- (3.1)}$$

where A and B (1 or 2) are constants. The activation energy can have values up to 1.5 eV, though much smaller values are encountered at lower temperatures. For an oxide of composition MeO, the activation of a conduction electron may be represented by



The energy needed for the relation of ion pairs Me^{3+} and Me^{1+} corresponds to a gap in the density of states. The following terms are considered to be contributing to the activation energy which arises from an electron correlation effect.

- I The difference between ionization energy and the electron affinity of the free Me^{2+} ion.
- II The difference in Madelung energy of the two configurations shown in eqn. (3.2).
- III The difference in crystal field stabilization of the above configurations.
- IV Energies of the polarization of the surrounding crystal lattice.

The terms I to IV are of important significance depending upon the conditions that prevail during conduction.

3.4 CONDUCTION IN FERRITES

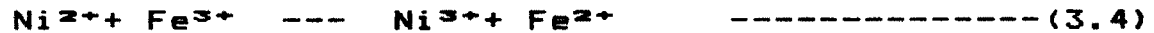
The electrical conductivity of a ferrite depends very markedly upon the amount of iron present in the lattice in the ferrous state. A high concentration of ferrous ions leads to high electrical conductivity. Thus in ferrous ferrite the resistivity is of the order 10^{-2} or 10^{-3} ohm cm, while in a nickel - zinc ferrite it increased from about 10^3 ohm cm to about its thousand folds, when the content of iron in ferrous state was increased systematically (3). In certain nickel and magnesium ferrites resistivities as high as 10^{11} ohm cm have been recorded (4).

According to the explanation given by Verwey and De Boer (5) both Fe^{2+} and Fe^{3+} ions are present at the same octahedral B site in spinel structure. The conduction takes place when the electrons move from divalent iron ions to trivalent iron ions. Since conduction in ferrites is assumed to be related to d-electrons, the direct electron motion between Fe^{2+} ions (and between M^{2+} ions) can be neglected. The charge carriers in spinel can be introduced by forming mixed system in the primary ferrite Fe_3O_4 . In a stoichiometric compound the ferrous ions are created by thermal activation according to the reaction



where the B denotes octahedral sites and M is a

divalent metal ion. E_g is the energy required to transfer the electron M^{2+}_B to Fe^{3+}_B . As an example, the presence of nickel on octahedral site of the spinel favours a conduction mechanism as



which can explain the predominant conduction mechanism in the nickel-zinc ferrite (6).

A mechanism of conduction can be proposed as long as ions of the same parent atom having different valence states are found in crystallographically equivalent positions in the lattice. Thus the extra electron on a ferrous ion requires little energy to move to a similarly situated adjacent ferrite ion. The valence states of the two ions are interchanged. Under influence of an electric field, extra electrons can be considered to constitute the conduction current, jumping from one iron ion to the next. Since the materials are semiconductors, one may expect their resistivity to decrease with increase in temperature.

3.5 DEPENDENCE OF RESISTIVITY ON DIFFERENT FACTORS

As in case of many other semiconductors the resistivity of ferrites is very susceptible to temperature changes.

The charge transport takes place by thermally activated hopping involving interactions with phonons. From this consideration the temperature variation of resistivity can be represented by equation in the form of an exponential law,

$$\rho = C \exp (+ \Delta E/KT) \text{ -----(3.5)}$$

C is constant which may be temperature dependent in diffusion conduction process. K is the Boltzmann constant, T is absolute temperature and ΔE activation energy. In ferrite constant, C can be identified to a different ρ_0 , which is not dependent on temperature. We can put the above equation in the form

$$\rho = \rho_0 \exp (+ \Delta E/KT) \text{ -----(3.6)}$$

Komar and Klivshin (7) showed that the graph of $\log \rho$ versus $1/T$ is linear and the break in linearity appears at Curie temperature. The slope of the straight line gives $\Delta E/K$ from which energy of activation can be calculated.

The conductivity can be suppressed by the uses of impurities when the corresponding partially filled levels are situated above half way over the band gap. The E_g values for Mn is smaller than for Ni and Co. This is in agreement with the decrease in conductivity that was obtained by Van Uitert(6) by substitution of a few mole percent Mn for the divalent ions in Ni and Co ferrite. The variation in dc resistivity for various dopant concentration of indium in a manganese zinc ferrite is observed. The indium ions are observed to occupy both tetrahedral and octahedral sites (8). In same ferrite, Rao and his coworkers observed the effect

of Cr impurity on the dc resistivity (9).

The presence of ferrous (Fe^{2+}) ions reduces magnetostriction but it is highly undesirable in microwave ferrite applications (10). Snoek (11) pointed out that the presence of ferrous ions can be reduced strongly by taking iron less than the stoichiometric ratio in the composition. Van Uitert showed that an excess of iron above stoichiometry and high firing temperature both cause remarkable reduction in resistivity.

3.6 HOPPING OF POLARONS IN FERRITES

In the lattice of spinel ferrites there exists the electrostatic crystal field due to the presence of ions. There is Coulomb interaction between the charge carriers and the crystal field. As the charge carriers move during the conduction process the ions get partially dragged along with it. Such a displacement of ions leads to the polarization of the surrounding region. Thus the carriers get situated at the centre of the polarization potential well. If the well is sufficiently deep the carrier is trapped at the lattice site and conduction is achieved by thermal activation. The combination of potential strain and the electron is

described as 'polaron'. It has been concluded that for ferrites the conduction is determined by thermally activated hopping of polarons (12). The charge transport by polaron from site to site is more commonly known as "polaron hopping".

3.7 ENERGY OF ACTIVATION

Band gap is defined as the difference in energy between the lowest point of conduction band and highest point in valance band. This viewpoint is true provided the semiconducting material is perfectly pure and is in the form of a single crystal. This is seldom the case for oxide semiconductors such as polycrystalline ferrites. Besides there may be present some impurities giving rise to trap levels and localized states(13). The presence of pores provides insulating path to the conduction electron. Magnetization states also affect the conduction mechanism. There is additional splitting of 3d band and the Bloch type wave function are not appropriate for the description of electrons, which are fully localized on specific cations. The conventional band theory fails to give satisfactory account of conduction properties of ferrites. So that the idea of band gap which is often used for semiconductors is augmented by the concept of energy of activation in case of oxide semiconductors.

It is necessary to imagine some discrete conducting states rather than conduction band and valence band. Some minimum energy is needed for an electron to jump from one state to the other as there exists certain potential well within the two. The conduction cannot occur unless the carriers are able to cross the potential well and for this purpose they need to be activated by some external agency such as thermal energy. According to kinetic theory of heat, the energy is imparted to the particles and if this is just sufficient, the conduction can take place. This is called as 'thermal activation' and the minimum energy needed for the current carriers to go to conducting state may be defined as energy of activation. Activation can also be effected by using other forms of energies such as optical wave energy or by applying electrical or magnetic fields.

3.8 EXPERIMENTAL TECHNIQUE

The d.c. electrical conductivity in the present case was measured by the two probe method on compressed and sintered pellets of about 1 cm diameter and 0.2 cm in thickness, with the experimental set up shown in figs. 3.1 and 3.2. The measurements were carried out

with the help of a special conductivity cell fabricated in the workshop. The cell consists of two brass cylindrical rods fitted in two porcelain discs. The fitting screws were used to keep the assembly of discs and rods firmly. The screws provided on the rods were used for external contact with the help of silver leads, insulated from each other by ceramic beads. To have the good ohmic contact, the pellets were polished and silvered on opposite faces and sandwiched between the rods. The entire assembly was kept inside the temperature regulated furnace. The calibrated chromel-alumel thermocouple was used to measure the temperature of pellet accurately by keeping its tip close to the sample. The actual experimental set up is shown in photograph of Fig. 3.1. The d.c. voltage of about 2 volts was applied across the pellet through transistorized power supply unit (T.P.S.U.), whereas thermo emf was measured with digital voltmeter and the current with electronic multimeter.

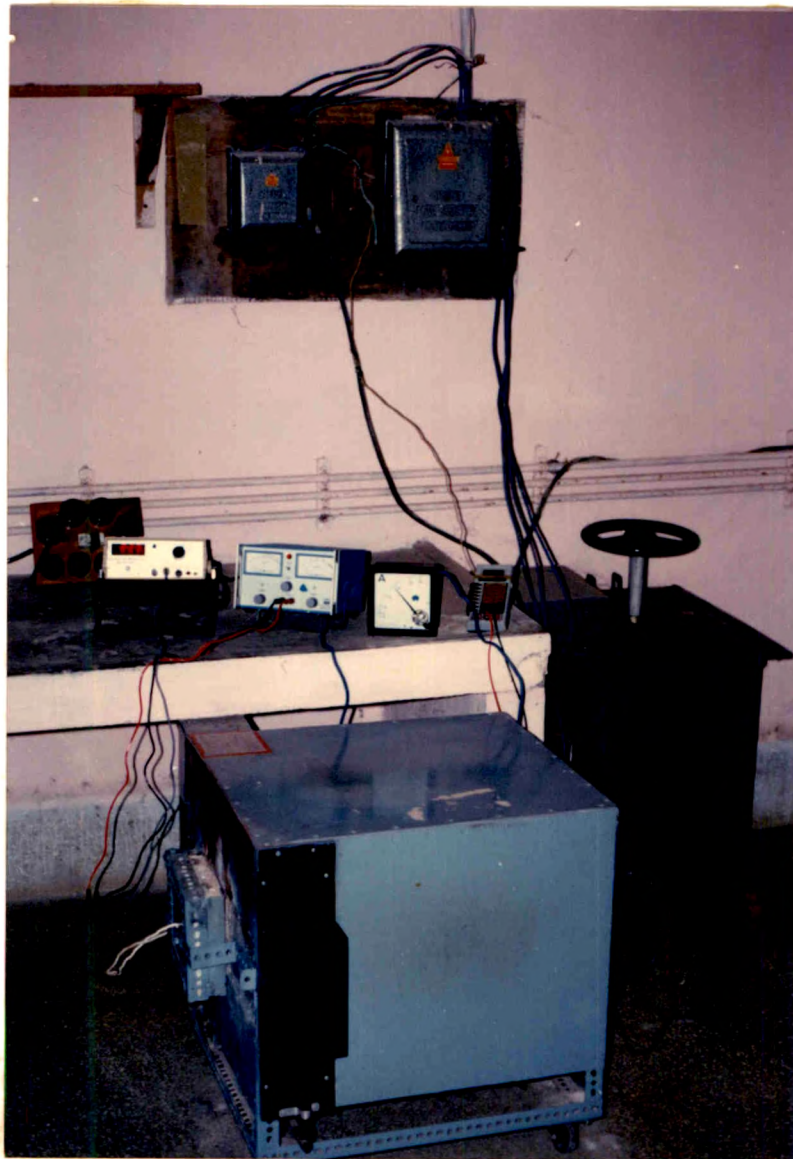
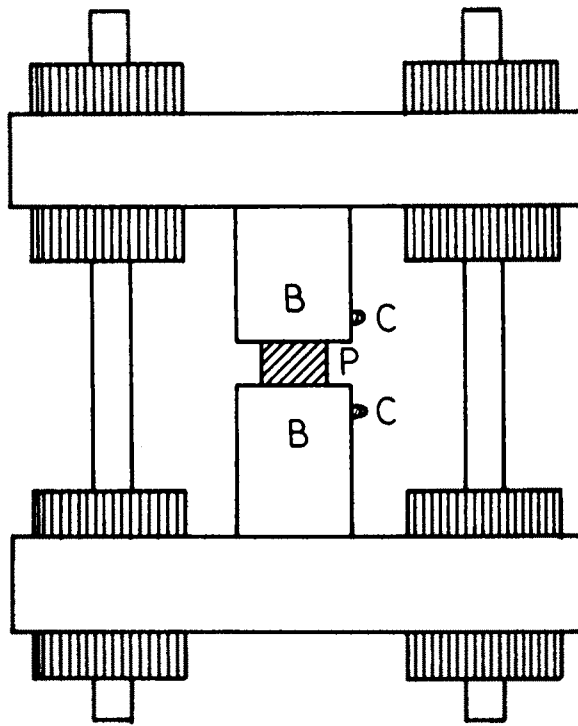


FIG.3.1- EXPERIMENTAL SET-UP FOR D.C. ELECTRICAL CONDUCTIVITY MEASUREMENT.



P - Pellet
 B - Brass electrode
 C - Connection lead

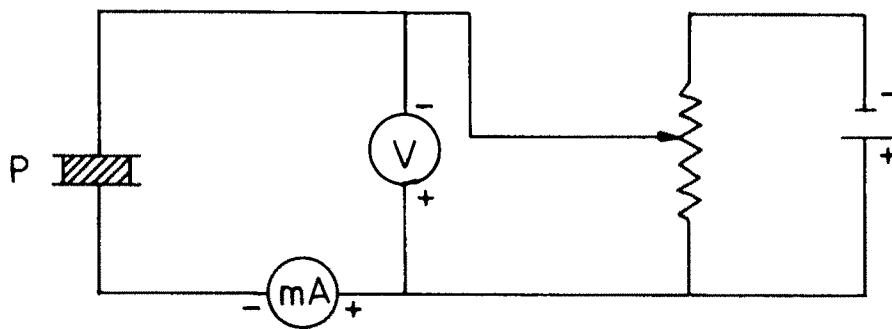


Fig.3.2 Sample holder and circuit diagram for electrical resistivity

The temperature of the furnace was gradually increased from room temperature to 750° C and corresponding current was measured for every change of 1 millivolt in thermo emf for each sample. The resistance of the pellet was calculated by Ohm's law

$$R = V/I \text{ -----(3.7)}$$

where V = voltage across the pellet in volts
I = current through the pellet in amps.

Resistivity was calculated by the formula,

$$\rho = R.A/h \text{ -----(3.8)}$$

where A = area of cross section of a pellet.
h = height of a pellet.

3.9 RESULTS AND DISCUSSION

The electrical resistivity measurements of $M_{0.8}Zn_{1-x}Fe_2O_4$ samples were made from room temperature to 750° C. The plots of $\log \rho$ versus $10^3/T$ are shown in figs. 3.3a to 3.4e for samples sintered at 1100° C for 15 hours and 30 hours respectively. In general the resistivity curves corresponding to samples fired at 1100° C for 15 hours lie above the lines for the same

samples sintered at 1100° C for 30 hours. The slope of each straight line (except for Zn content = 0.8 and 1.0) changes at a particular value of temperature. This value of temperature has been identified as the Curie temperature as explained below. It is observed that the temperature variation of resistivity obeys the relation (3.6).

Activation energies corresponding to para and ferrimagnetic regions (to high and low temperature regions respectively) have been calculated from the slopes of the lines and are tabulated in table 3.1. The activation energy for conduction in paramagnetic region is found to be higher than the activation energy for conduction in ferrimagnetic region. The observed Curie temperatures from $\log \rho$ versus $10^3/T$ plots can be found in table 3.1. The Curie temperatures agree reasonably well with the values reported earlier (14).

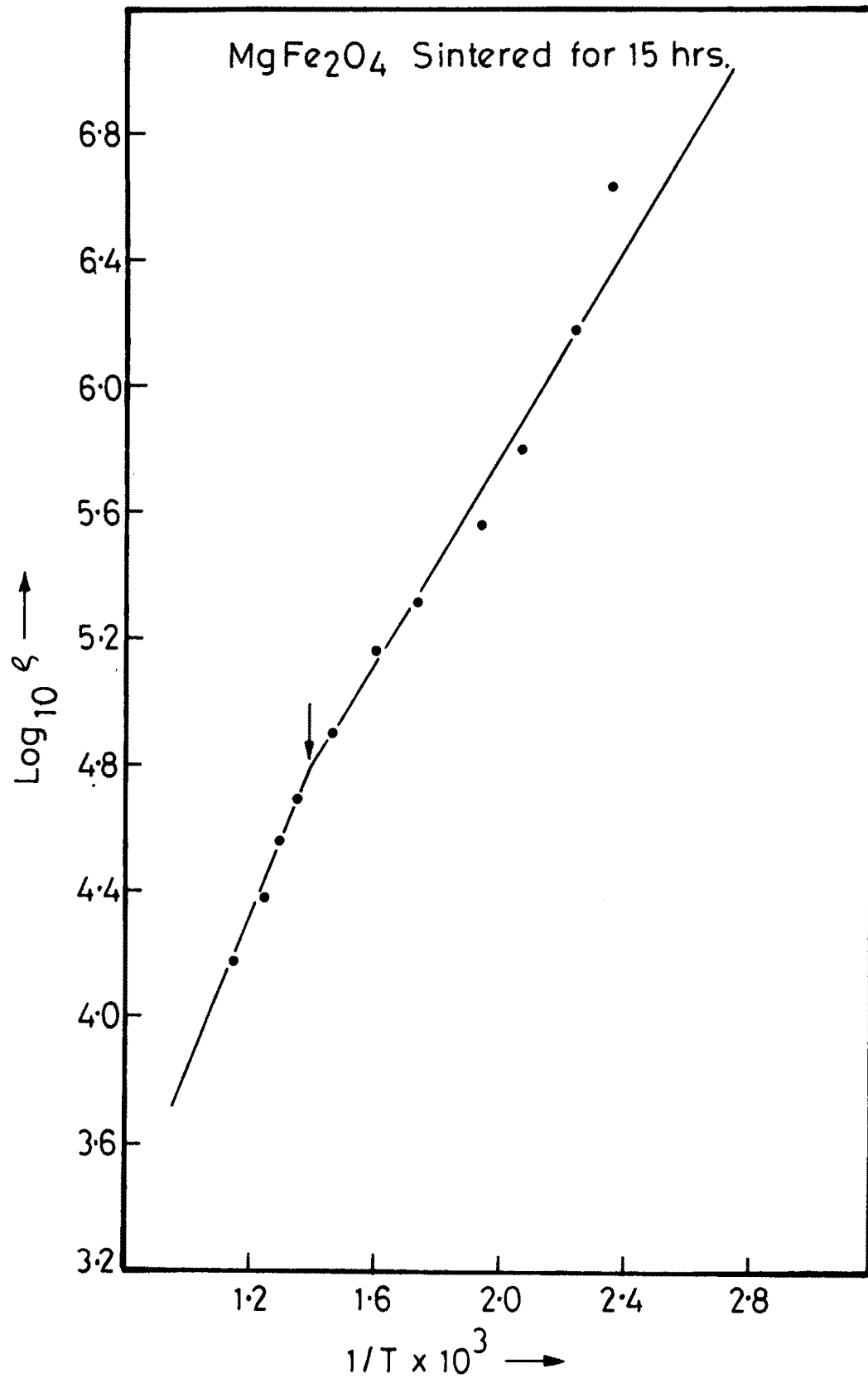


Fig.— 3.3 a

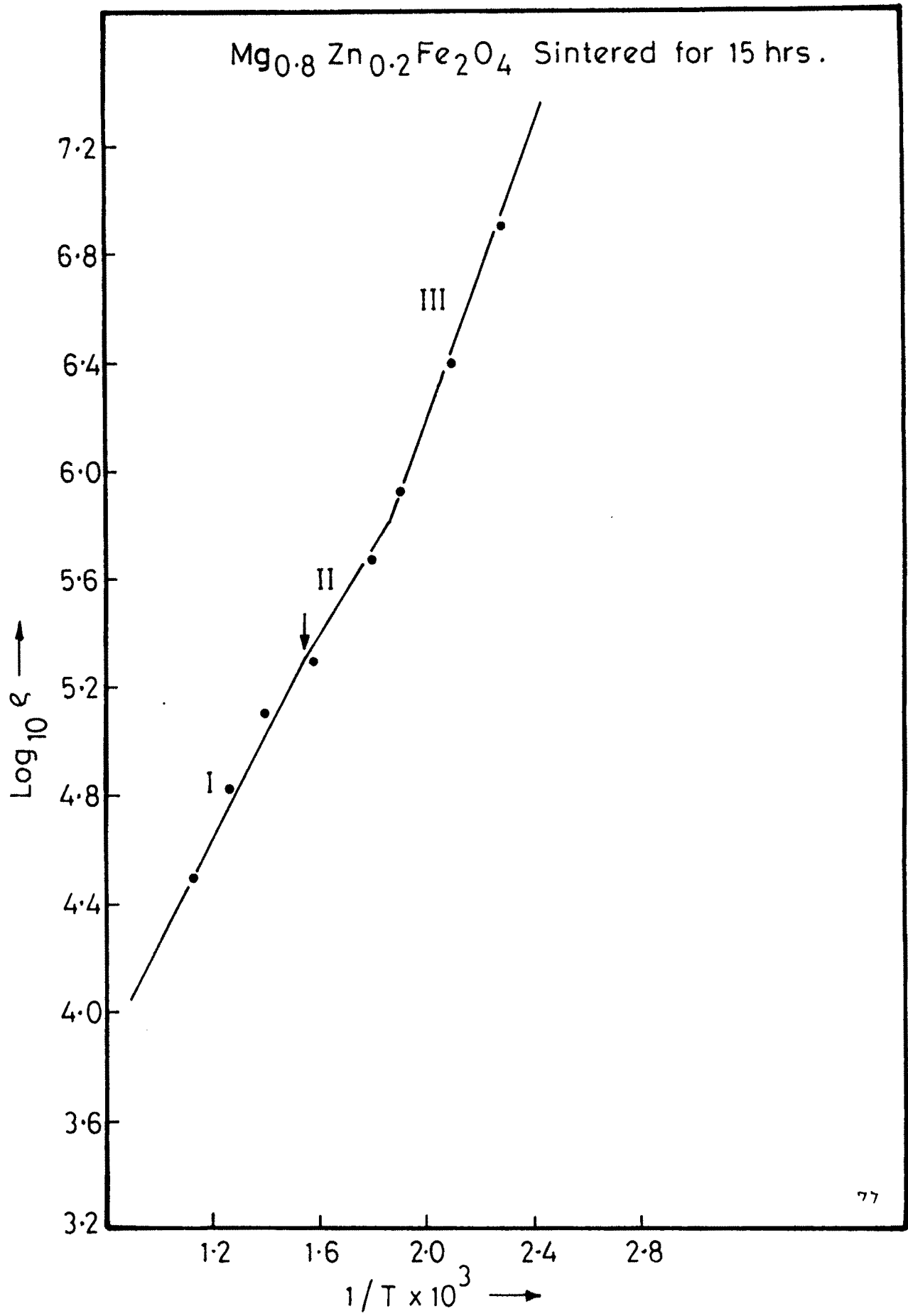


Fig.-3.3 b

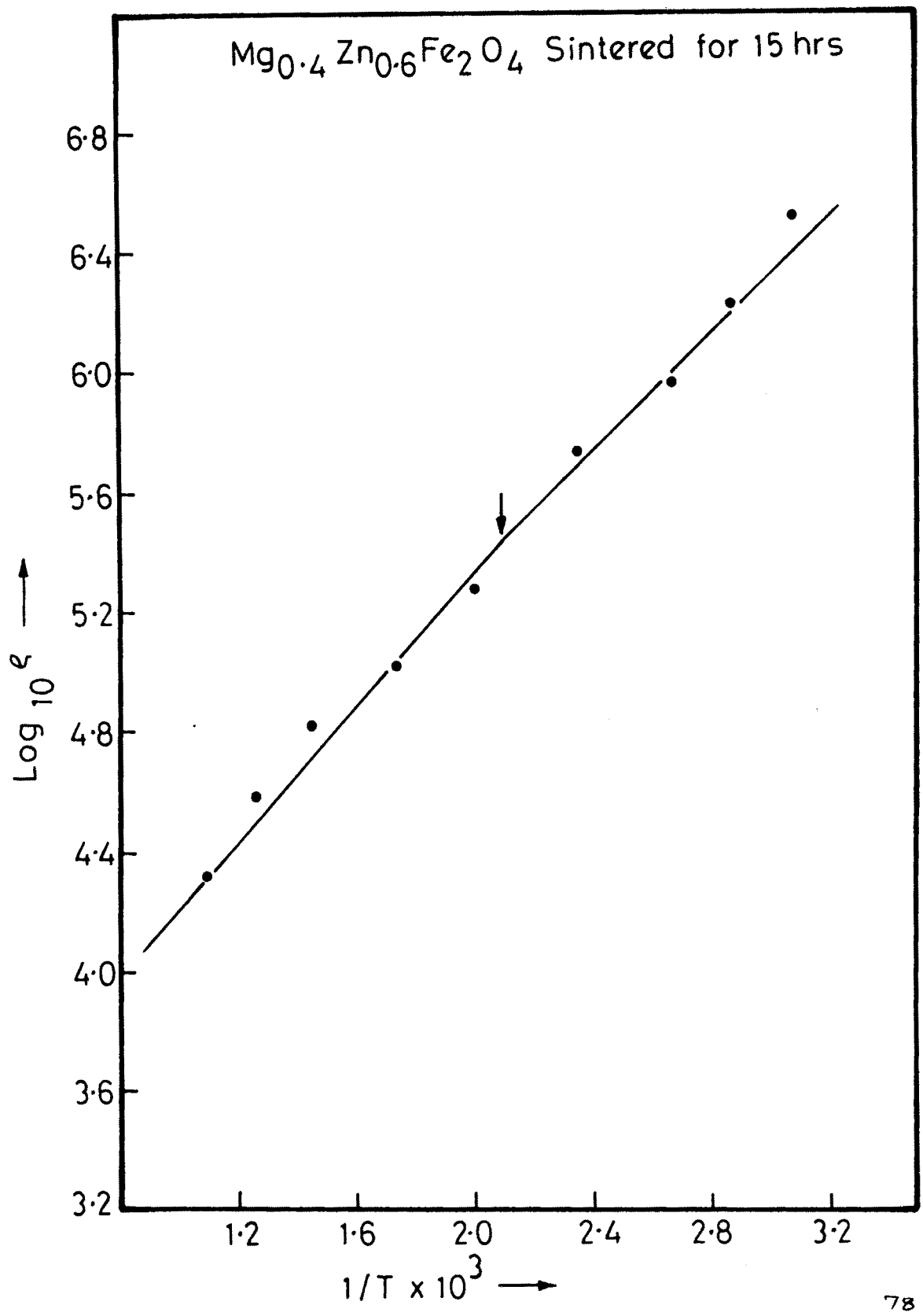


Fig.- 3.3c

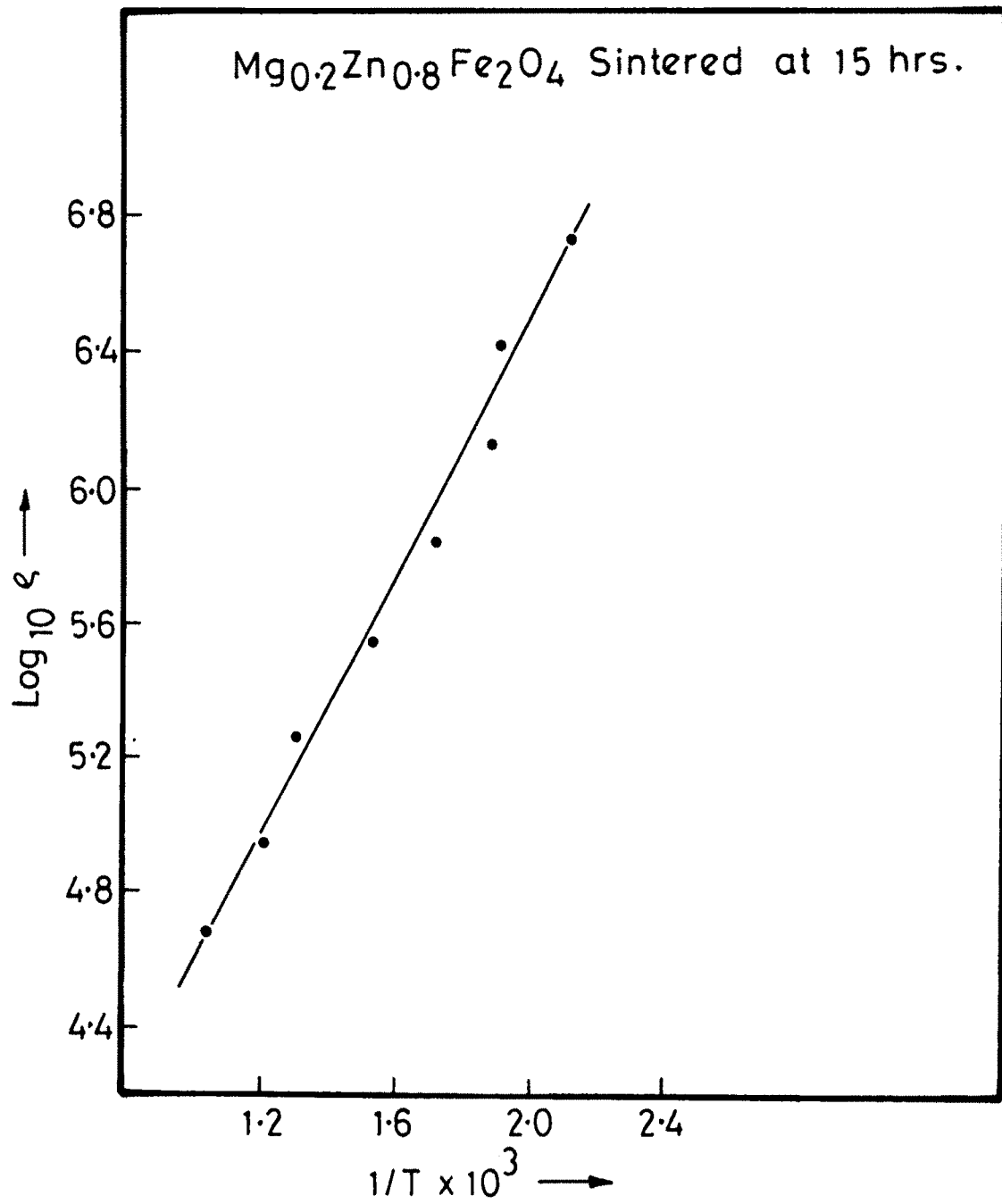


Fig.-3.3d

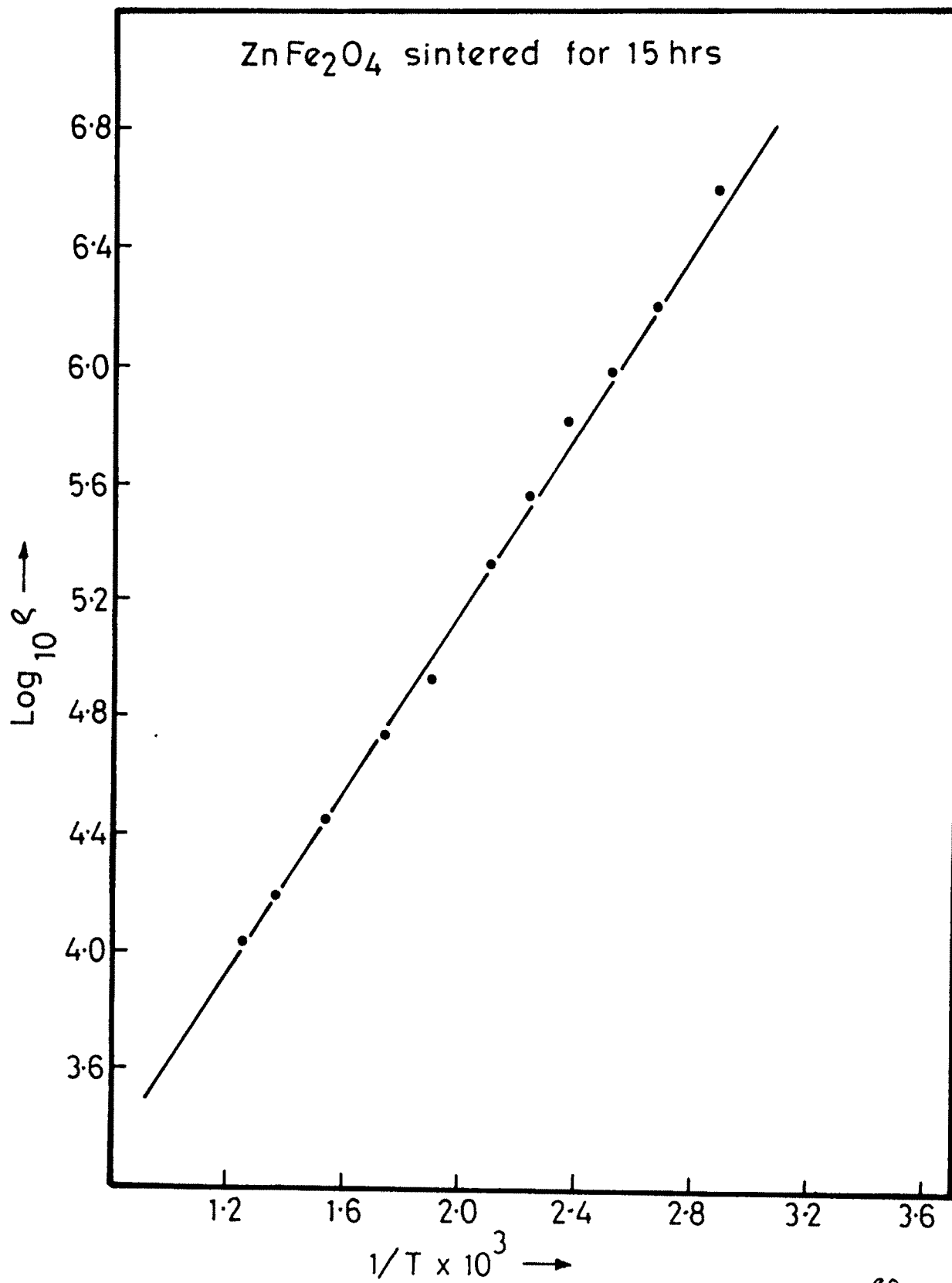


Fig. - 3.3 e

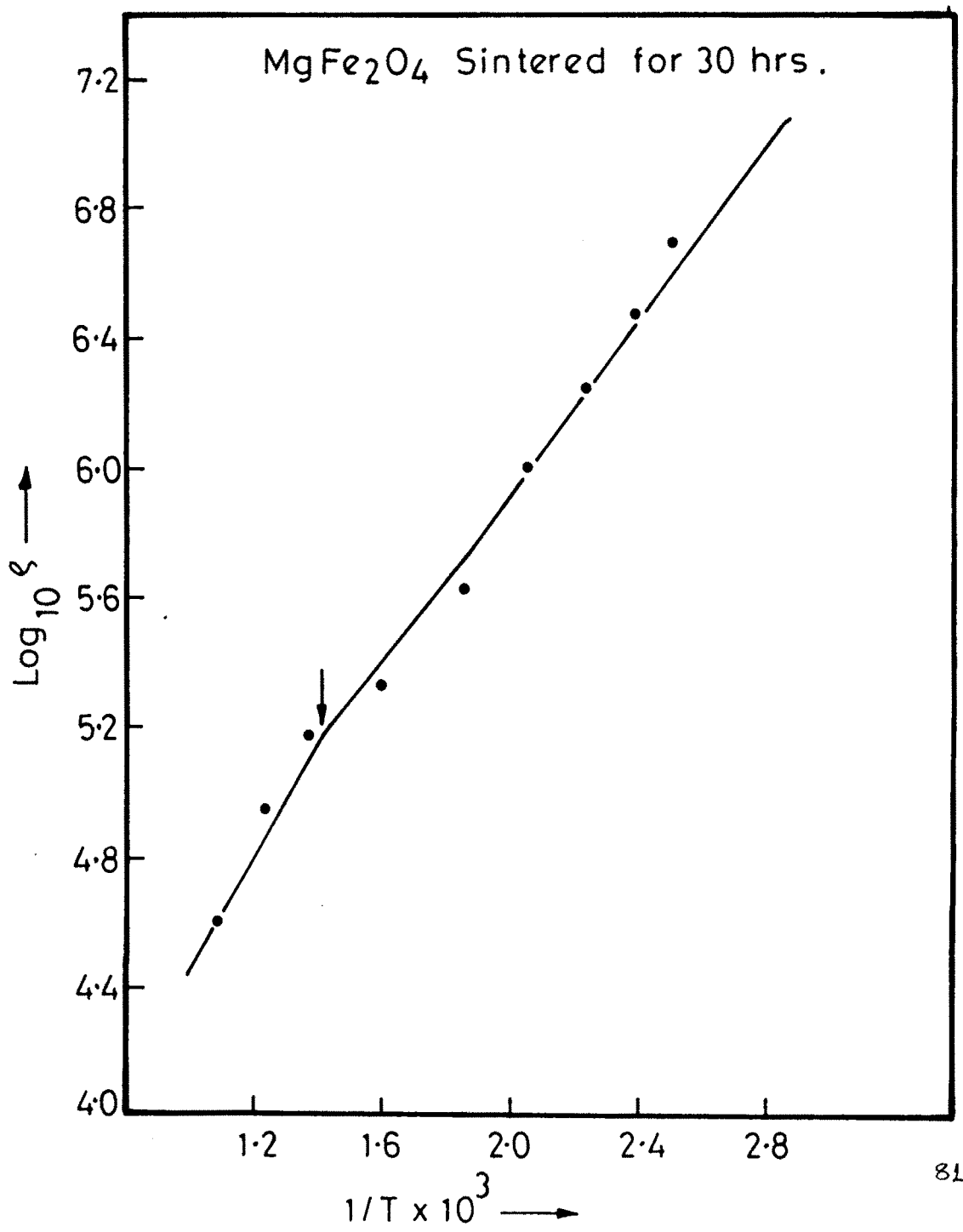


Fig.-3.4 a

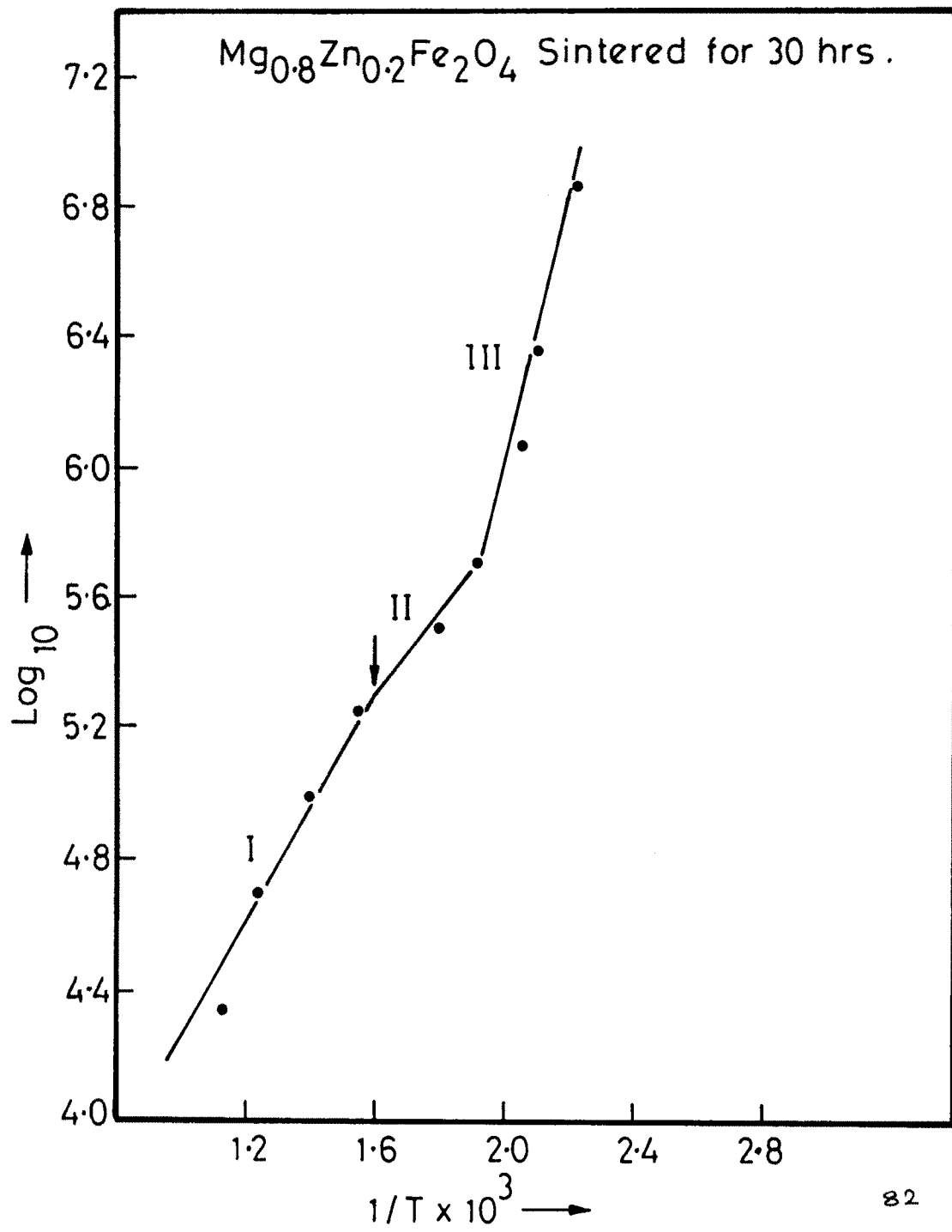


Fig.—3.4b

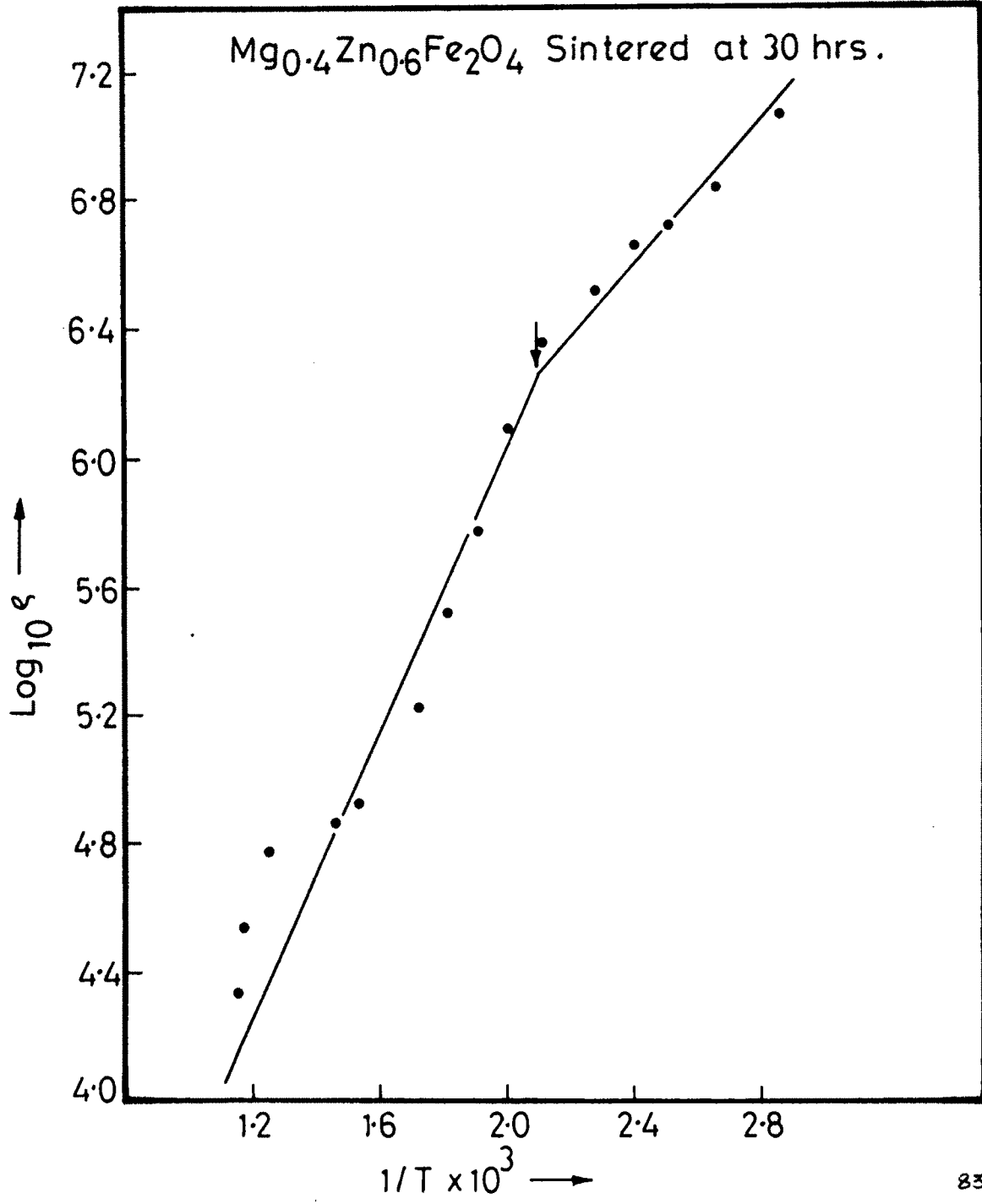


Fig.— 3.4c

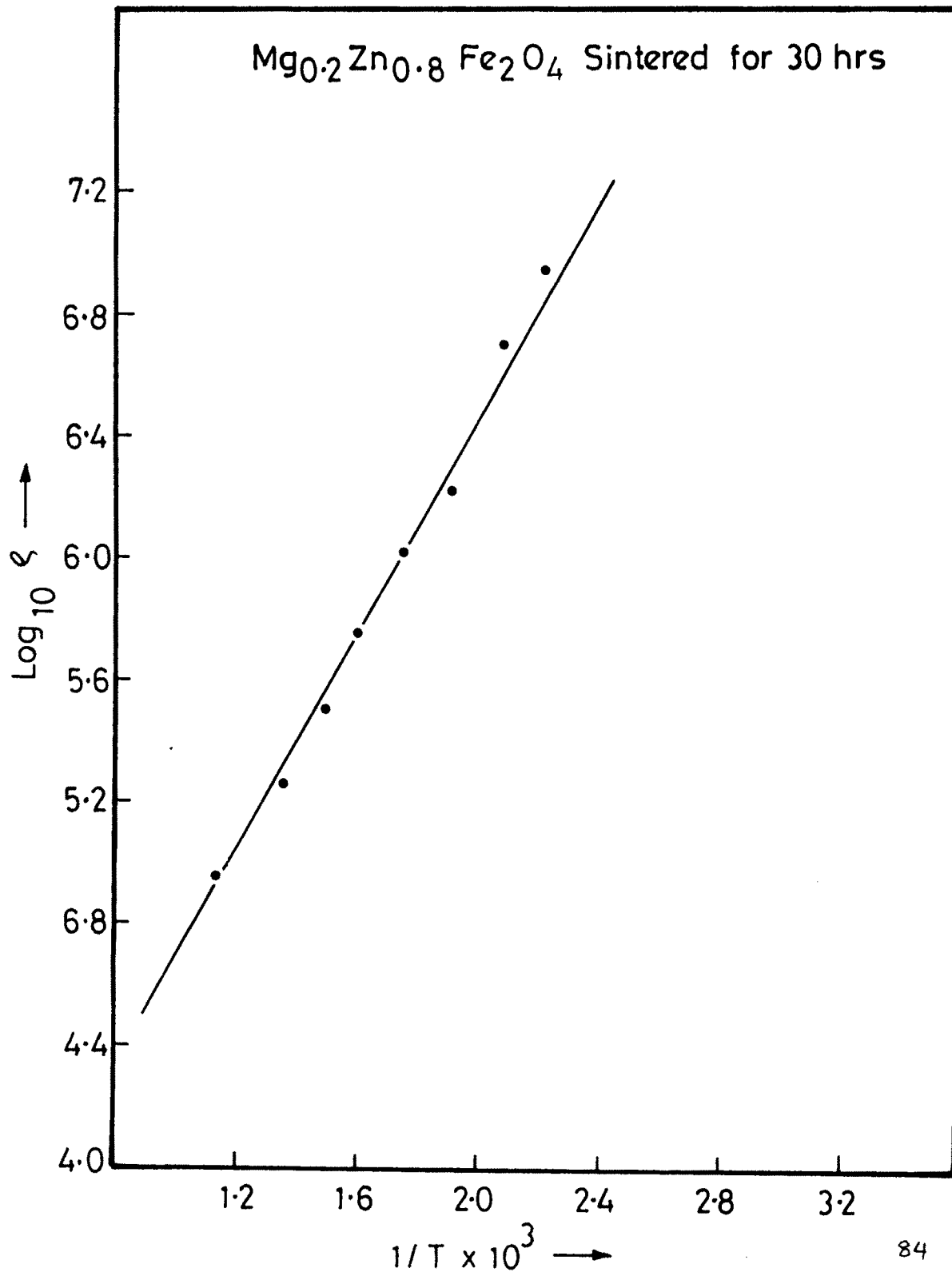


Fig.—3.4d

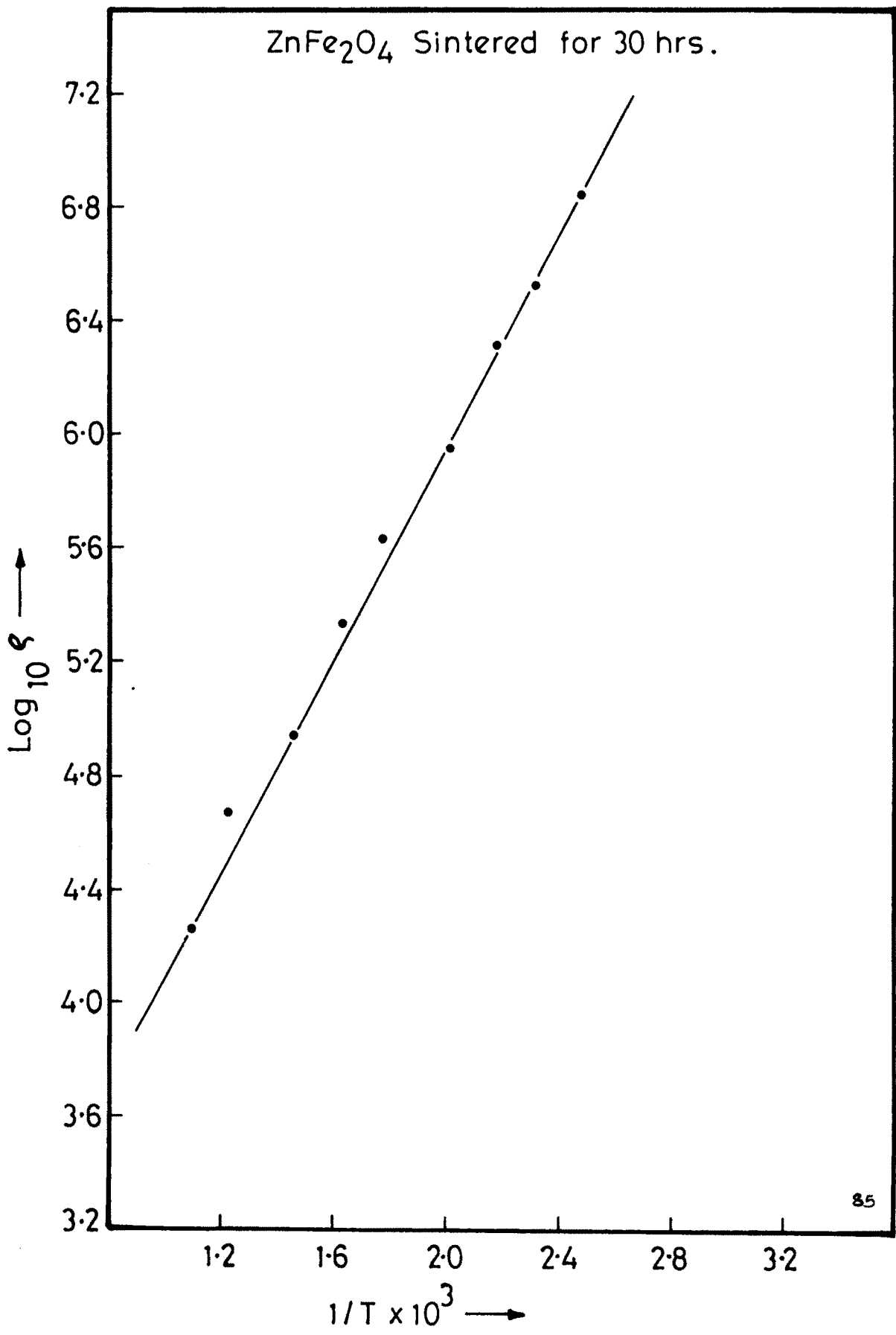


Fig.—3.4 e

TABLE - 3.1

VALUES OF E, CURIE TEMPERATURES FOR
 $Mg_xZn_{1-x}Fe_2O_4$ FERRITES SINTERED AT 1100°C
 FOR 15 & 30 HOURS

HISTORY	SAMPLES	E Values in eV		Curie Temperature in °C (from \log_{10} vs $1/T \times 10^3$ graphs)
		Para Region	Ferri Region	
	MgFe ₂ O ₄	0.4636	0.3246	441
Sintered	Mg _{0.8} Zn _{0.2} Fe ₂ O ₄	0.3974	0.5228	368
for	Mg _{0.4} Zn _{0.6} Fe ₂ O ₄	0.2243	0.2025	198
15 hours	Mg _{0.2} Zn _{0.8} Fe ₂ O ₄	-	-	-
	ZnFe ₂ O ₄	-	-	-
	MgFe ₂ O ₄	0.3397	0.2649	426
Sintered	Mg _{0.8} Zn _{0.2} Fe ₂ O ₄	0.3477	0.5960	352
for	Mg _{0.4} Zn _{0.6} Fe ₂ O ₄	0.4636	0.3974	181
30 hours	Mg _{0.2} Zn _{0.8} Fe ₂ O ₄	-	-	-
	ZnFe ₂ O ₄	-	-	-

The observed discontinuities in the $\log \zeta$ versus $10^3/T$ have been attributed to several reasons. It has been shown theoretically by Irkhin et al (15) that the change in the slope of straight line should occur while passing through the Curie point. This point separates the upper ferromagnetic region from the lower paramagnetic region, as the spin - spin interaction vanishes due to thermal vibrations. Similar results have also been reported by Komar and Klivshin (7) in case of several other ferrites and they have stated that the change in the activation energy of conduction at a particular temperature has some relation with the ferromagnetic Curie temperature of ferrites. Such a proof for the evidence of a relation between magnetic ordering and conductivity also comes from our results. The breaks in the conductivity plots may be also due to some other reasons like conduction mechanism (16). Recently reported Ghani's (17) work on conductivity of Ni - Zn ferrites has shown more than one break in the conductivity profiles. They have attributed conduction mechanism in the first region to the presence of impurities, in the second region to the phase transition from tetragonal to cubic form and in the third region to magnetic ordering change.

Apart from other things the conductivity in ferrites has been connected with the presence of ions with variable valency (18). These ions are randomly distributed over the crystallographically equivalent positions. The conductivity in the case ferrous ferrite has been explained on the basis of the presence of Fe^{2+} and Fe^{3+} ions on B sites. The electron can have a transition from Fe^{2+} to Fe^{3+} within the octahedral position without causing much change in the energy of

the system. The activation energy required for usual electron hopping is of the order of 0.2 eV or less. Since the activation energies in the present case are in excess of 0.2 eV, it suggests that the conductivity mechanism in these ferrites is governed by hopping of polarons.

In the ferrites having the spinel structure the B-B distances are smaller than A-A and A-B distances. Even then B-B distance is more than the sum of ionic radii of the cations involved indicating a little or no overlap between d-d wavefunction of ions on adjacent octahedral sites. This gives rise to a situation where electrons are not free to move through the crystal but remain fixed on B sites necessitating a hopping process. The result of the conduction by hopping process is large effective mass and low mobility of current carriers. The temperature dependence of electrical conductivity in such a case involves less temperature dependent concentration of carriers and mostly associated with the temperature dependent mobility. The mechanism of the hopping process itself involves occasional excitation by lattice vibration of the carriers with high degree of probability. Heikes and Johnston (16) have given the mobility hopping process as

$$\mu = ed^3/IT \exp (-q/KT) \text{-----} (3.9)$$

Thus mobility which is a central factor deciding the activation energy depends on the phonon spectrum of the crystal on one hand and shows local variations due to local surroundings on the other. In solids with the large coupling constants and narrow conduction band small polarons formation is more probable.

In oxides of iron group of metals the overlap of 3-d wave functions between neighboring metal ions is relatively small. There is a strong experimental proof for the existence of small polarons and hopping processes (18, 19). The energy levels have been calculated only after making a number of simplifying assumptions and using suitable approximations(18). Klinger (19) has explained the conduction mechanism in magnetite like solids, using two phase polaron model. He has concluded that at low temperature the conduction is via thermally activated motion of strongly correlated polarons and at high temperature via weakly activated hopping motion or a non-activated Brownian like tunneling motion of polarons. The activation energy in the paramagnetic-region is generally found to be more than that for ferrimagnetic region. This can be attributed to the effect of magnetic ordering in the conduction process (20). According to strict theoretical consideration the anomalous changes in the activation energy occur. In fig.3.5, the compositional variation of T_c values for our samples is shown. It is seen that the T_c values show decrease with the addition of Zinc in the ferrites. Since, $MgFe_2O_4$ is a partially inverse ferrite, the change in the T_c values in the samples indicates that there may be some cation migration from B site to A site, leading to the observed reduction in T_c values.

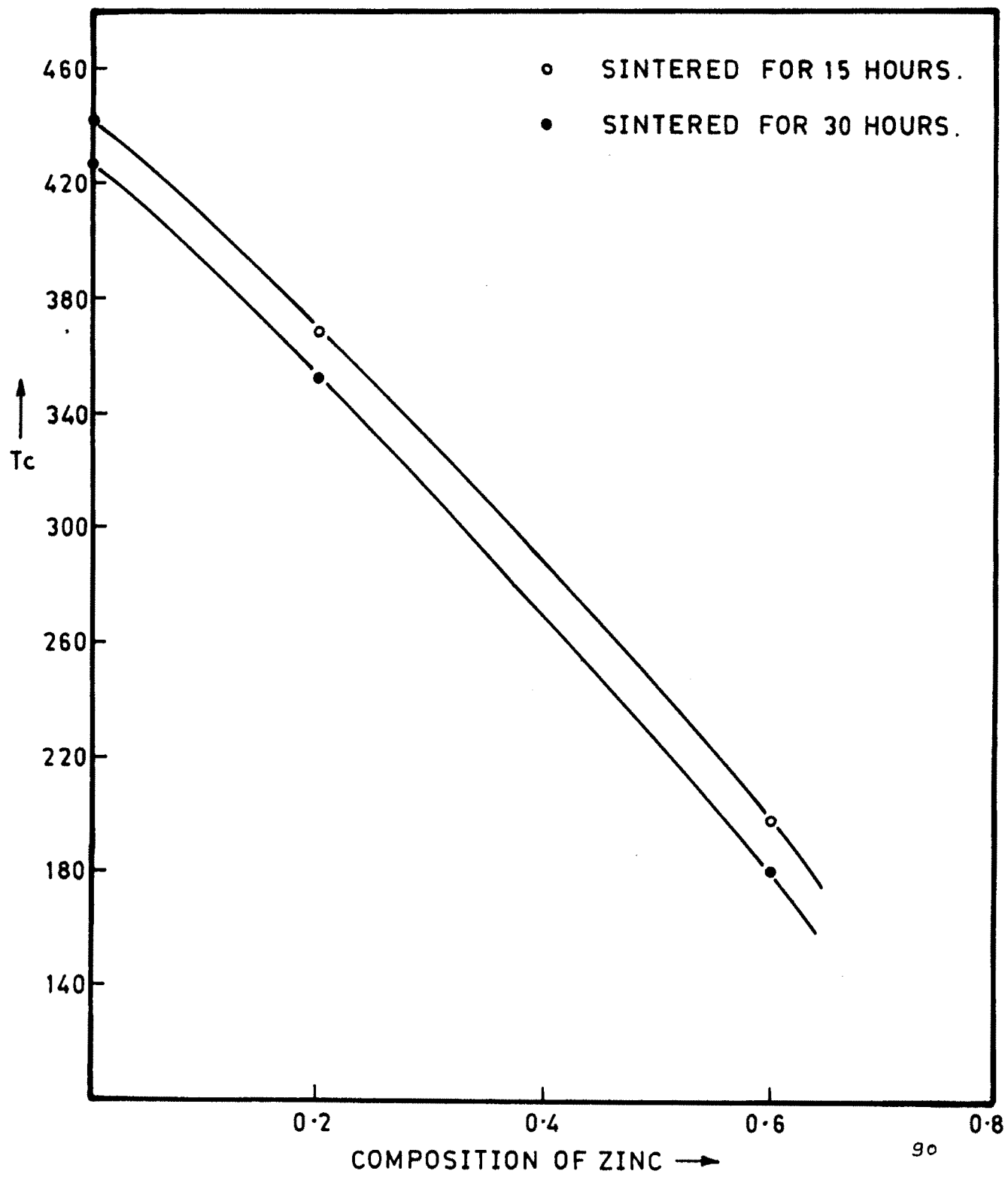


FIG. 3.5

The phenomenon of conductivity of ferrites poses many complexities. Therefore the interpretation of D-C resistivity in polycrystalline materials becomes rather difficult, due to the presence of grain boundaries giving rise to additional scattering. The cation or anion vacancies may be responsible for acceptor or donor levels and number of charge carriers may remain uncertain in a ferrite sample, especially when it has undergone a unique heat treatment. This presents additional complexities from the point of view of interpretation of d.c. conductivity data. Besides, the anion or cation vacancies may also contribute a component of ionic conductivity to the dc conductivity of the samples(16). Various conduction mechanisms based on different models for the donor levels have been given by different workers (21, 22).

In all cases, curves for samples sintered at 1100° C for 30 hours lie necessarily below the curves for samples sintered at 1100° C for 15 hours. This fact clearly reveals that the resistivity decreases when the samples are sintered for longer sintering times. In table 3.1 the comparison of activation energies at two sintering temperatures for each composition is shown. There is very little difference in transition temperature in two sintering cases and these two are very nearly equal to Curie temperature. However the general trends of the plot is that these transition temperature shift to the lower temperature side when sintering temperature is increases. The porosities are calculated at these two different sintering procedures. It can be clearly seen that the values of porosity are also reduced for longer sintering times.

The above results can also be explained on the basis of

microstructural changes brought up by sintering conditions. 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, permeability, eddy current losses etc. are predominantly governed by heat treatment. Besides methods of preparation, the conducting properties depend upon the presence of impurities as in the case of all other semiconductors.

Porosity increases the resistivity markedly. The presence of pores filled by air may provide insulating path to the electrons giving rise to high resistivity. Oxygen stoichiometry also plays a leading role. An oxygen ion vacancy will contribute two extra electrons to 3d band. If these extra charges are detached from their vacancies by thermal agitation n type of conductivity is induced in oxygen deficient and p-type conductivity in oxygen excess materials (23). Heat treatment during the preparation process changes the electrical properties of polycrystalline ferrites due to rapid decrease in oxygen dissociation pressure (24). This leads to the formation of small amounts of divalent ions during firing and the ferrite posses high conductivity.

The grain size of individual crystallites influence the conduction due to increased number of grain to grain contacts (25). Contact surface area inside the granular ferrite matrix thus effectively increases considerably. According to elementary law of electricity the resistance is inversely proportional to cross sectional area of the conductor and hence the resultant resistance of matrix may decrease. In turn,

the activation energy falls down. During cooling process while sintering under oxygen atmosphere, i.e. air, it is possible that the films of high resistivity may form over the grain boundaries. The oxygen gets locked inside the pores and while cooling it oxidizes divalent ions which are lying near the grain surface producing very thin film of oxide around the grain. Besides, the presence of air gaps between the grain leads to the formation of inhomogeneous dielectric structure (26). This may affect both d.c. and a.c. conductivity of ferrites considerably and thus the conduction mechanism through ferrites may much depend upon porosity. It is observed that the porosity is high at lower sintering temperature. The pore density decreases due to densification at higher temperature (27). There is further reduction of resistance due to loss of number of pores in polycrystalline matrix of grains in ferrites which are sintered at high temperature. More systematic study is needed on these extrinsic factors and their effect on properties of ferrites.

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