

Chapter - III

Conductivity Studies

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3.1 INTRODUCTION :

Ferrites are semiconductors by nature. The room temperature resistivity can vary from 10^{-3} ohm-cm to 10^{+9} ohm-cm.

In Fe_3O_4 low resistivity is observed due to the presence of Fe^{+2} and Fe^{+3} ions in the crystallographically equivalent sites.⁽¹⁾ Due to their high resistivity the Ferrites find preference over metals and alloys in high frequency operation, which exhibit large eddy current losses. The studies on conductivity furnish a useful data from the application point of view and also in understanding the mechanism of conduction. Conduction in Ferrites is attributed to hopping process, which obeys the law $\rho = \rho_0 \exp^{-E/KT}$ Where E is activation energy. Komar et al⁽²⁾ have observed breaks or discontinuities in $\log \rho$ Vs $1/T$ plots for many Ferrites. This discontinuity occurs near curie temperature and segments represent different conduction mechanisms. Anomalous results showing different values of resistivity were observed in Ni Ferrite⁽³⁾ Van Uietret⁽⁴⁾ has observed the marked changes in resistivities of the same sample at room temperature before and after the grinding of surfaces in Ni-Zn Ferrite.

He attributed this change to Zn which volatilizes during heat treatment and encourage the formation of Fe^{+2} ion on the surface. Due to this NiZn ferrite shows low

resistance before grinding the surface. Koops⁽⁵⁾ has studied variation of resistivity and dielectric constant with frequency. He found that the resistivity and dielectric constant fall by large amount at higher frequencies in inhomogenous materials.

The conduction mechanism in ferrites is due to exchange of electron from Fe^{+3} to Fe^{+2} in the same (octahedral lattices) lattice. This requires little energy to move an electron under the influence of electric field these extra electrons can be considered to constitute the conduction current by hopping from one site to the other. Recently Klinger⁽⁶⁾ has reviewed the conduction mechanism in ferrites. He stated that hopping of polarons is the main conduction mechanism in ferrites. He has suggested two phase mechanism for hopping of polarons.

Contributions to resistivity of materials are from cation distribution, crystal structure, porosity, grain size, impurity inclusions, chemical or oxidation states and other unknown and known scattering mechanisms.

Jefferson⁽⁷⁾ has studied the conductivity and Seebeck constant⁽⁸⁾ for Cu containing ferrites Rezlesal et al⁽⁸⁾ have measured the electrical and magnetic properties of Cu containing ferrites and obtained cation distribution. Parker⁽⁹⁾ et al have measured resistivity of non stoichiometric Zn and Ni ferrite and observed discontinuities in the resistivity plots. The observed temperatures of these discontinuities are lower and lower than the curie temperatures for more and more

iron content, Ahmad ⁽¹⁰⁾ et al have observed the discrepancies in the theoretical and observed results. They measured the temperature dependence of electrical conductivity, Hall effect and thermoelectric power of Cu-Zn Ferrites and interpreted the results on basis hopping of small radius polarons. Domain Wall resonance in Cu containing ferrites has been studied by Srinivasan ⁽¹¹⁾ and he has attributed this resonance to hopping process.

In this chapter review of the conduction mechanism in oxides and ferrites and polaron theory are briefly discussed.

The resistivity and magnetisation in iron rich ferrites is also dealt with. Measurements on temperature dependence of resistivity have been carried out. The curie temperature are also observed. The results are discussed and interpreted in terms of the present theoretical models.

3.2 CONDUCTION MECHANISM :

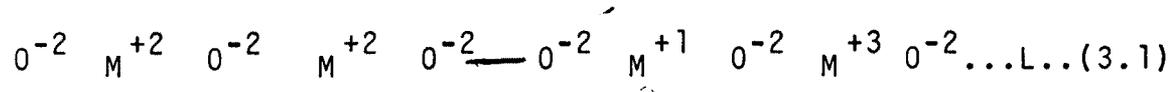
In general oxide crystal structures may be conveniently regarded in term of lattice of oxygen ions and relatively small metal ions occupying the interstices. Although the bonding in the transition metal oxides is predominantly ionic, the electrical conductivity tends to be decided by the electron with d-like wave functions and most probably the impurity states.

In the elements of first transition series, the 3d levels are being systematically filled from Sc to Ni. In the crystalline field of a solid the levels are split into a

triplet and a doublet. The triplet or t_{2g} states lie ~~below~~ below the doublet or e_g states in oxides with rock salt structure. These oxides then be expected to be metallic conductor at least above antiferromagnetic temperature. ⁽¹²⁾ Below the Neel temperature the exchange forces could give rise to further splitting of 3d band. The oxides however be have as semiconductor at all temperature and have intrinsic activation energies in excess of those which would be accounted for by exchange splitting. From this it is evident that descriptions in terms of conventional band ~~theory~~ ^{structure} fails to account for the electrical transport properties.

3.2 a CONDUCTION IN OXIDES :

For an oxide of composition M_0 the activation of a conduction electron may be represented by



where M^{+2} is transition metal ion

The energy needed for the relation of ion pairs M^{+3} and M^{-1} corresponds to a gap in the density of states.

The following terms are contributing to the activation ^{Energy} which arises from an electron correlation effect.

- i) The difference between ionisation energy and the electron affinity of the free M^{+2} ion.
- ii) The difference in modelung energy of the two configurations shown in eqn.(3.1).

iii) The difference in crystal field stabilisation of the above configuration.

iv) Energies of the polarisation of the surrounding crystal lattice.

3.2 b MIXED VALANCE AND CONTROLLED VALANCY SEMICONDUCTION :

A relatively good conductivity in ionic compounds with an appreciable concentration of metal ions in two valence states was first investigated by de Bore and Verwey (13) for magnetic Magnetite has 1/3 of metal ions ⁽¹³⁾ ~~on~~ ^{at} tetrahedral sites and 2/3 on octahedral sites. The octahedral site contain equal number of Fe⁺² and Fe⁺³ which are randomly distributed above a disorder temperature 119°K. Charge transport occurs by transfer of electrons between ~~other wise~~ ^{divalent and} trivalent iron ions. The proces differs from one represented in eqn.(3.1) and energy term (iv) polarisation effect is only present. The overlap between the 3d like wave functions of nearest neighbour cations is sufficient to give rise to almost metallic type conduction.

3.2 C CONDUCTION IN FERRITES :

This above mechanism of electron transport does not apply to other simple ferrites where all Fe ions are trivalent. Transport may then be represented by



Where E_g , the activation energy may reflect the difference between third ionisation potential of Fe⁺³ and M⁺³ ions in

ions in the solid. Jonker ⁽¹⁴⁾ has studied the ferrite $\text{CO}_x\text{Fe}_{3-x}\text{O}_4$ and predicted qualitatively the mechanism stated above.

Ionic state of the cations in a ionic crystal gives some insight into the effect of impurities on electrical properties.

In so far as the ionisation potential of O^{-2} ion is the dominant term. Substitution upto 1% or so of the cations of another element of the same valency should have little effect upon the conductivity. If the dopant has higher valency it will contribute donor centers, if it has lower valency it will provide acceptor centers.

Analogous considerations may apply to cations of mixed valency in ferrites which result from a departure from the ideal metal to oxygen ratio. An oxygen ion vacancy will contribute two extra electrons to the 3d band. If these extra charges are detached from their vacancies by thermal agitation n-type conductivity is induced in oxygen excess materials. Some oxides are very difficult to prepare homogenously with a stoichiometric anion to cation ration. Extrinsic semiconduction may then prevail throughout the temperature region amenable to electrical measurments. In such cases it may still be possible to infer the activation energy of intrinsic conduction samples from which extrinsic effects have ben supressed by introduction of compensations

impurities.

This was first demonstrated by Van Uitred⁽¹⁴⁾ in $\text{Ni Fe}_2\text{O}_4$ by replacing 1 to 2% Fe by Mn or Co. Electrons donated by oxygen vacancies will fill the impurity levels in preference to iron conduction levels. Mixed valency state will be confined to the impurity element if its local concentration exceeds twice that of oxygen vacancies. This technique is based on the assumption that impurity band conduction is absent and the allowed impurity concentrations indicate that the cation wave functions do not extend significantly beyond nearest neighbour distance in the B lattice. Electrons and holes are known to move by thermally activated hopping mechanism except for pseudometallic composition region close to magnetite.

3.3 CONDUCTIVITY

Imperfection in lattice will result in a spectrum of donor and acceptor levels within the energy gap. Total number of such levels may be very small compared to N_0 , where N_0 are metal ions and have little effect on electrical conductivity in the intrinsic region, but affects the low temperature properties. It is a general feature of semiconductors that at sufficiently low temperatures the electrical transport properties have a complicated dependence on T which is determined by structural defects and impurities.

The conductivity can then be written as

$$\sigma = e (n \mu_1 + p \mu_2) \dots\dots\dots (3.4)$$

μ_1 and μ_2 are mobilities of electrons and holes, n and p are the concentration of mobile electron and hole.

Activation energy does not provide much insight as the conduction mechanism because temperature dependent hopping mechanism and activation of electrons into the conduction levels are of the same. Form indistinguishable included in the activation energy found out experimentally.

3.4 THERMOELECTRIC POWER :

Studies on thermoelectric power provides a method to separate the contribution to activation energy E in the expression (3.5).

$$\sigma = \frac{B}{T} \exp \left(- \frac{q_1 + q_2 + E}{K_T} \right) \dots\dots\dots (3.5)$$

For the electrical conductivity assuming the schematic band structure model^(a) for the oxide semiconductors, the phenomenological relation between the peltier coefficient and Seebeck coefficient together with the equation of electrical conductivity in terms of carrier concentration and their mobility gives the relations.

$$eQ = -K \ln (N_0^1 / n) \quad \text{For n type} \dots (3.6)$$

$$eQ = +K \ln (N_0^1 / p) \quad \text{For p type} \dots\dots (3.7)$$

Such a study establishes the density of charge carriers and ~~their~~ ~~average~~ ~~mobility~~ ~~together~~
their average mobility together

with the electrical conductivity measurements.

3.5 D.C. CONDUCTIVITY CELL :

The cell for the measurement of D.C. conductivity consisted of two silver discs about 1cm in diameter and 0.5mm in thickness electrically connected to silver wires. The pellet was placed between these two silver discs and the assembly was held between two thick brass cylinders fixed to porcelain discs. These discs were tightly held by three screws symmetrically situated along their peripheries to ensure uniform pressure and good electrical contact between silver discs and the pellet.

The conductivity cell, thus prepared was placed in temperature regulated furnace. A chromel-alumel thermocouple was used along with a digital multimeter of least count 0.1 mv to measure the temperature accurately

The experimental arrangement and circuit dig. are shown in plate (A) and in Fig 3.1 respectively.

CURIE TEMPERATURE DETERMINATION :

Experimental arrangement and schematic diagram for determination of curie temperature is shown in plate B & Fig(3.2) . For the determination of curie temperature the ferrite sample was kept in a copper pan suspended with the help of a steel spring in a vertical muffle furnace, the

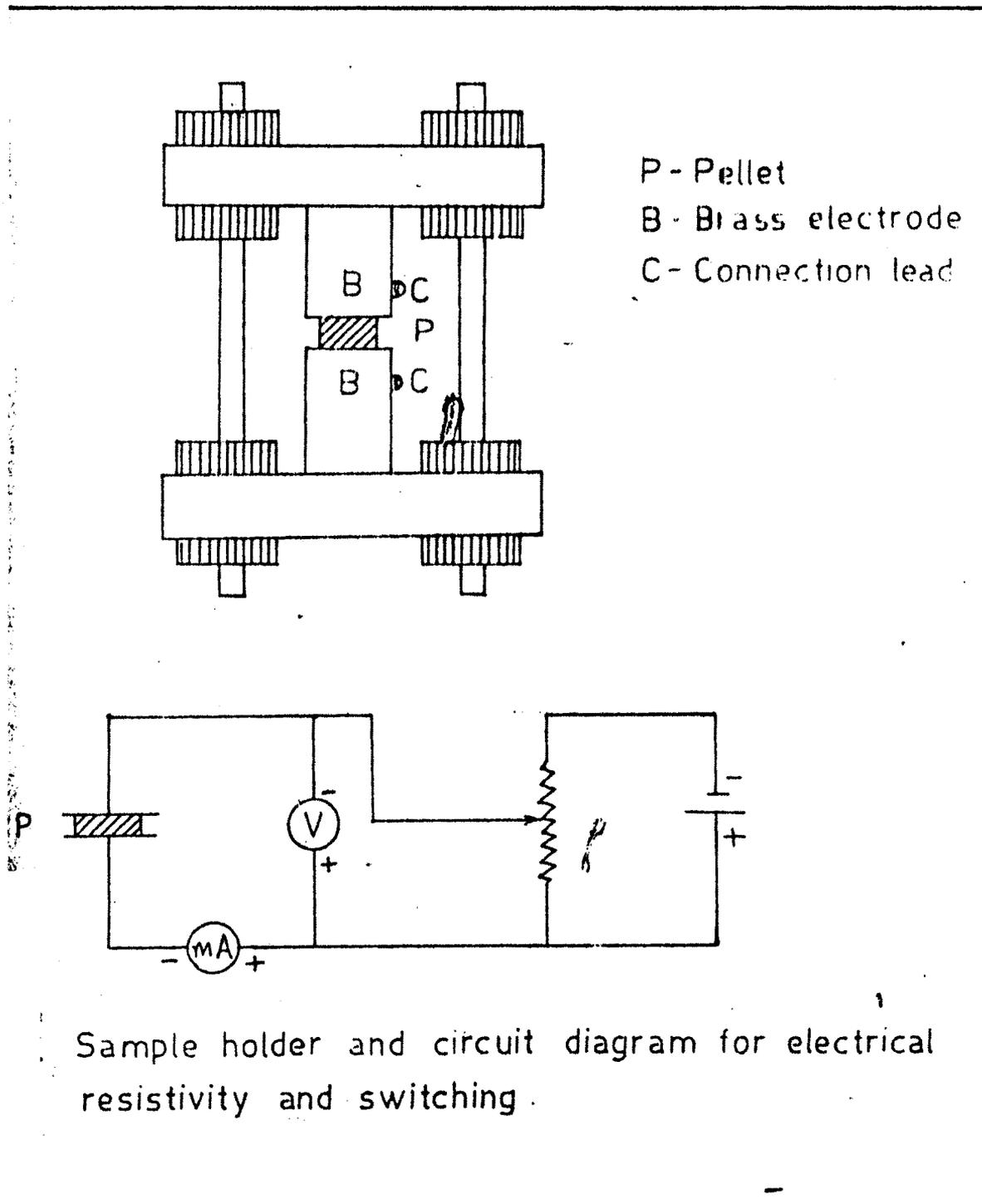


Fig.3-1



(Plate A)

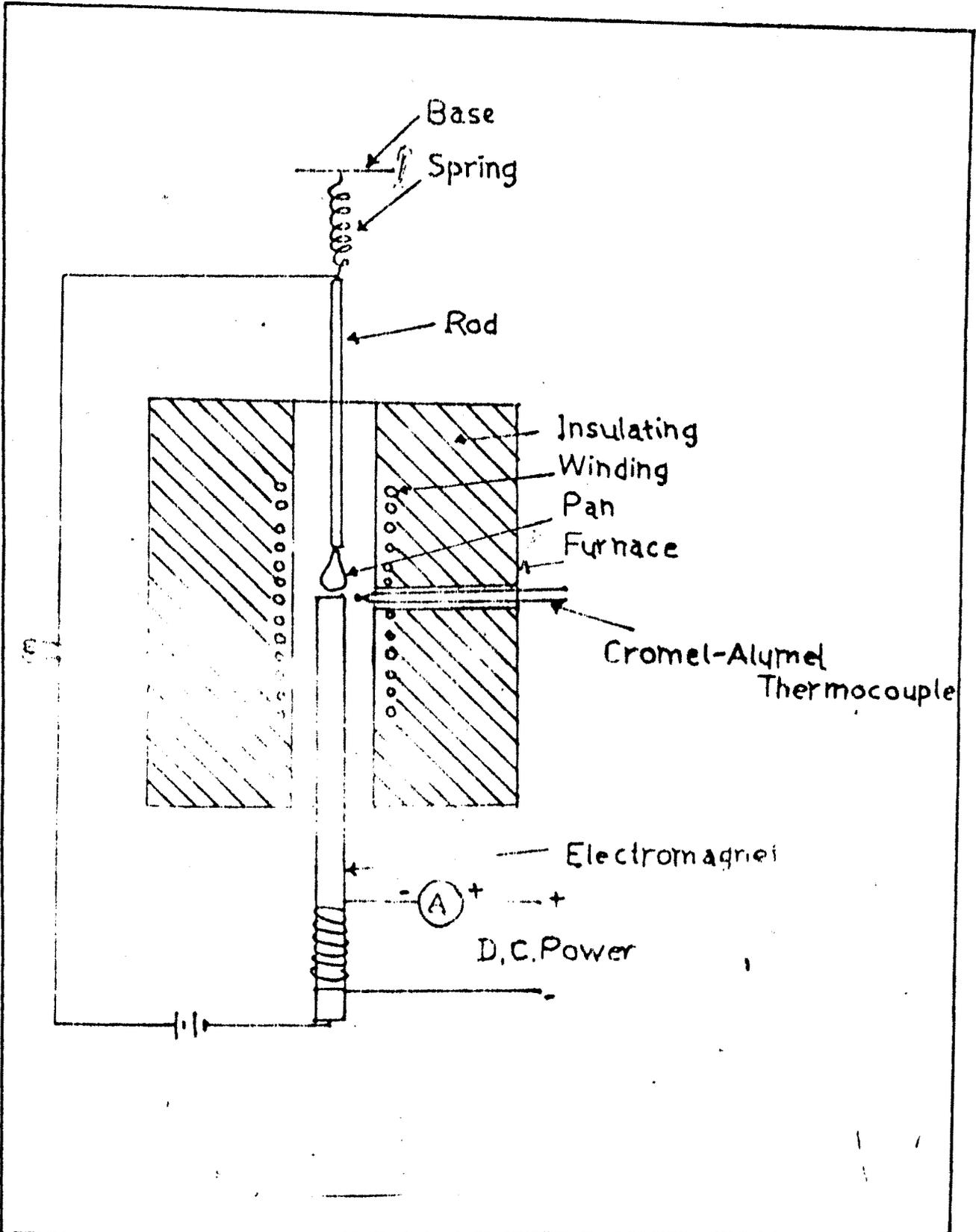
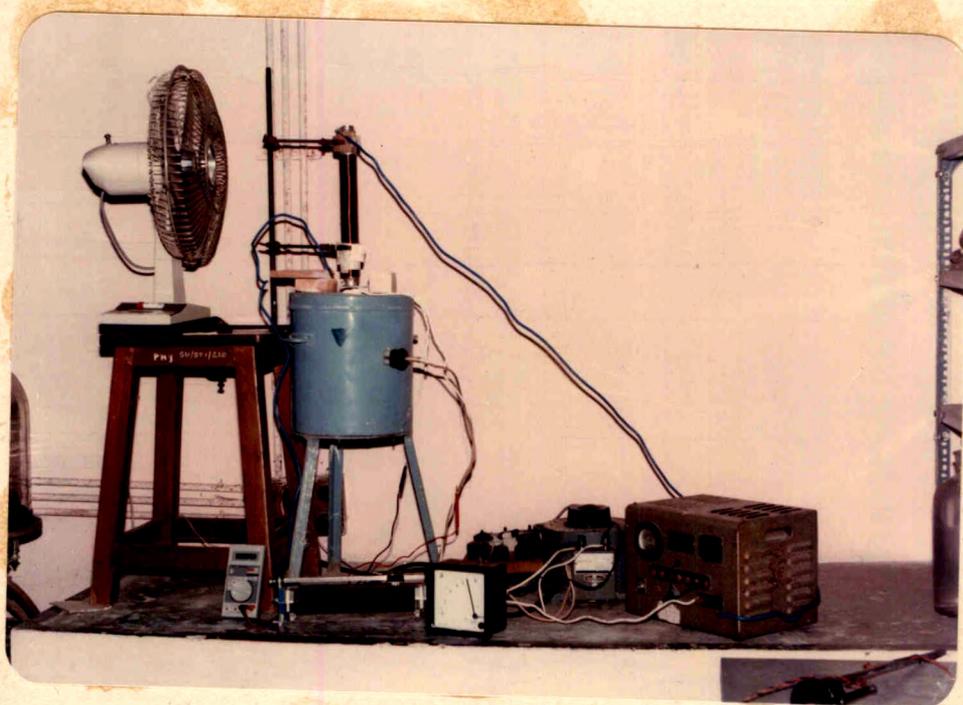


Fig. 3-2



(Plate B)

spring was located outside the furnace,⁽³¹⁾ A mild steel rod with diameter slightly less than the diameter of furnace was introduced in the furnace from the lower end which form the core of an electromagnet. The distance between upper end of the rod and lower end of the pan was adjusted less than half centimeter.

The mild steel rod was magnetised by passing 3-4 amps. Current through the coil of 2000 turns of copper wire, wound on its lower end. A copper pointer was fixed to the spring to complete electrical circuit when the sample touches magnetic pole. The test sample remained in contact with the magnet till curie temperature was attained. As soon as curie temperature was crossed, there was break in the continuity of the circuit and the temperature was measured immediately with the help of digital milivoltmeter, which was kept near and connected to thermocouple. The error in the measurement of curie temperature was kept minimum.

3.6 RESULT AND DISCUSSION :

In Fig. (3.3 to 3.10) etc. variation of $\log \xi$ Vs $-\frac{10^3}{T}$ for the slow cooled and quenched sample of $\text{Cu}_x\text{Co}_{1-x}\text{Fe}_2\text{O}_4$ is shown. It is seen that there are two breaks giving rise to three regions. Komar et al⁽²⁾ have observed breaks or discontinuities in $\log \xi$ Vs $1/T$ plots for many ferrites. This discontinuity occurs near curie temperature and

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In Fig. (3.3 to 3.10) etc. variation of $\log \epsilon$ Vs $\frac{10^3}{T}$ for the slow cooled and quenched sample of $\text{Cu}_x\text{Co}_{1-x}\text{Fe}_2\text{O}_4$ is shown. It is seen that there are two breaks giving rise to three regions. Komar et al⁽²⁾ have observed breaks or discontinuities in $\log \epsilon$ Vs $1/T$ plots for many ferrites. This discontinuity occurs near curie temperature and

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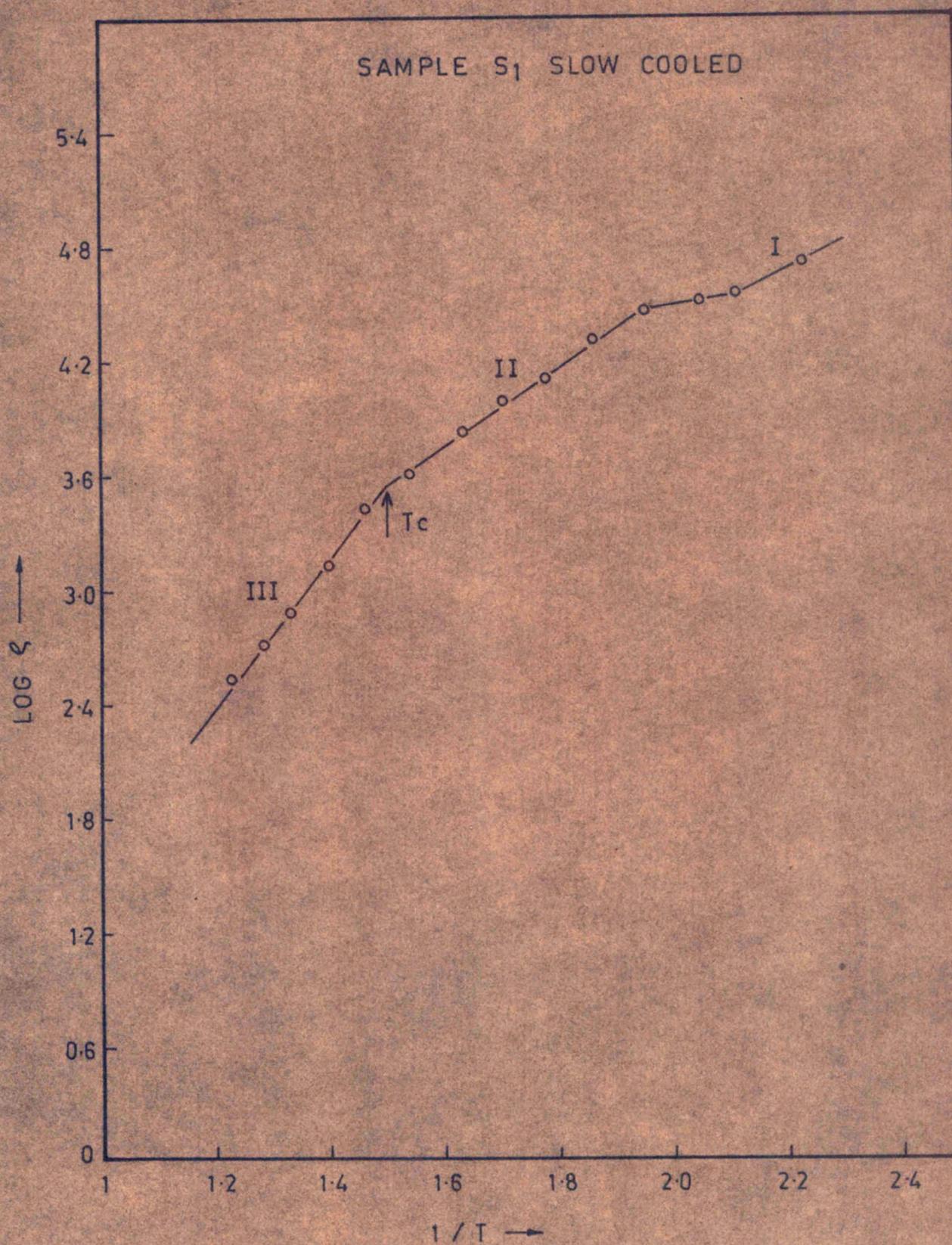


Fig. 3.3

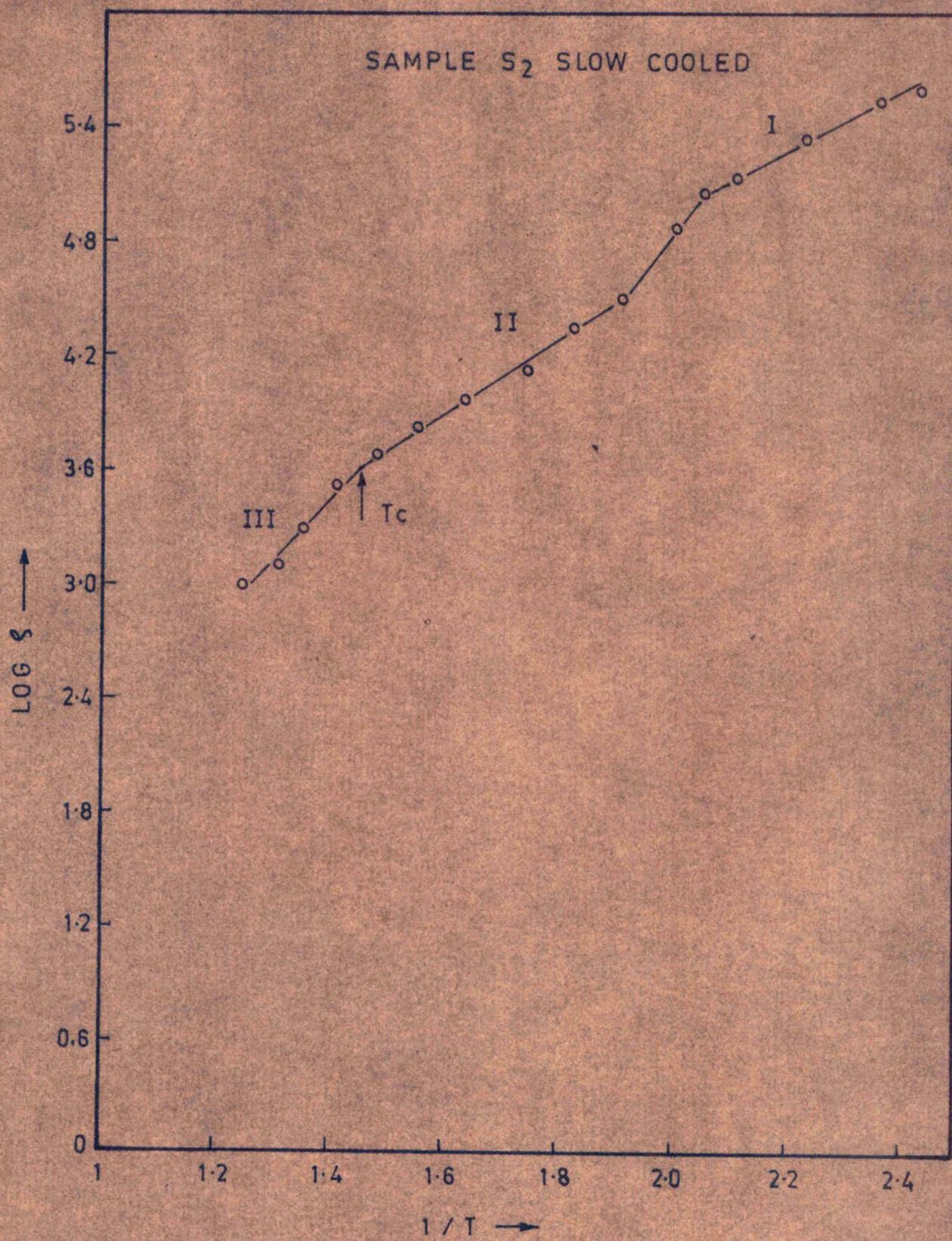


Fig. 3.4

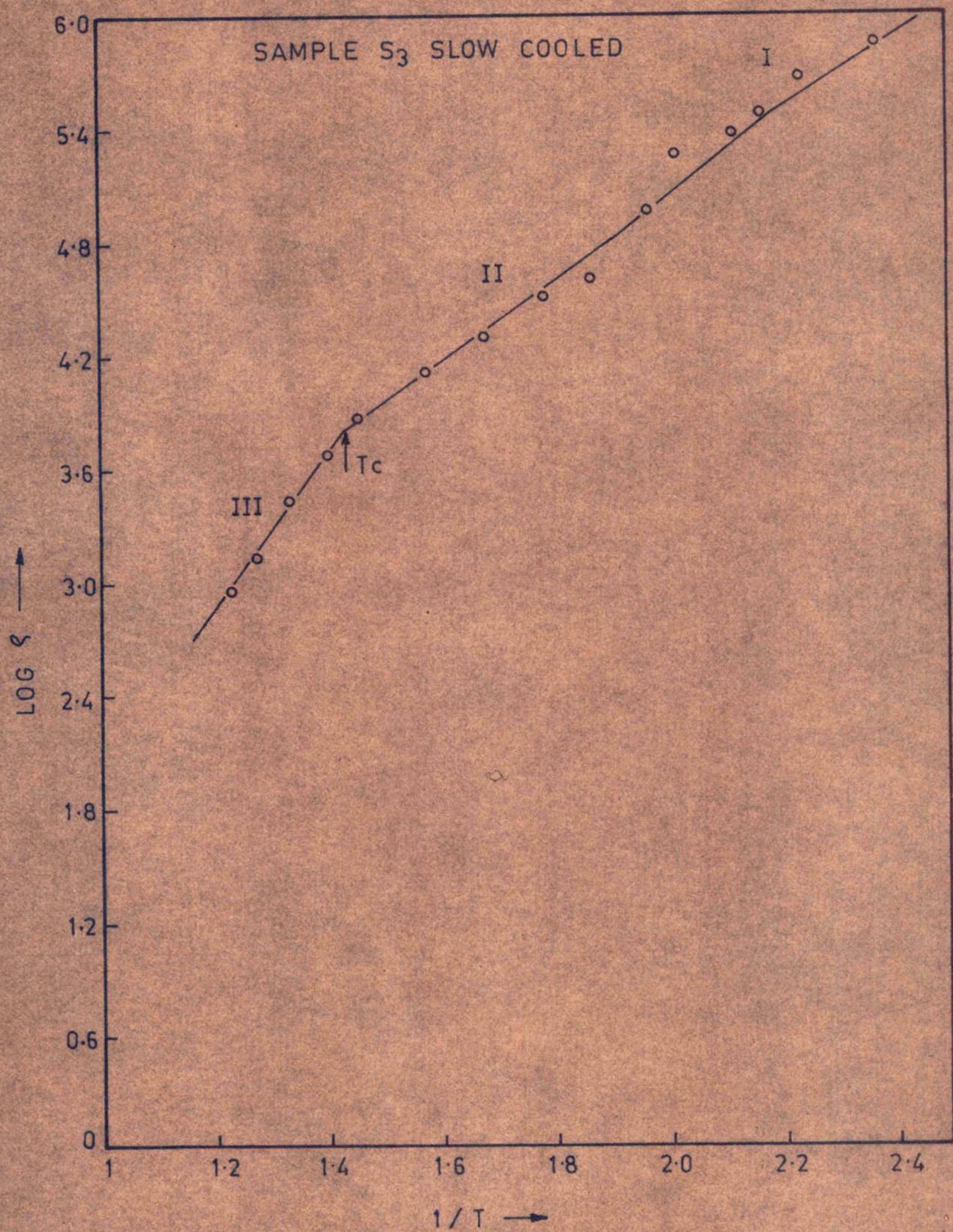


Fig. 3.5

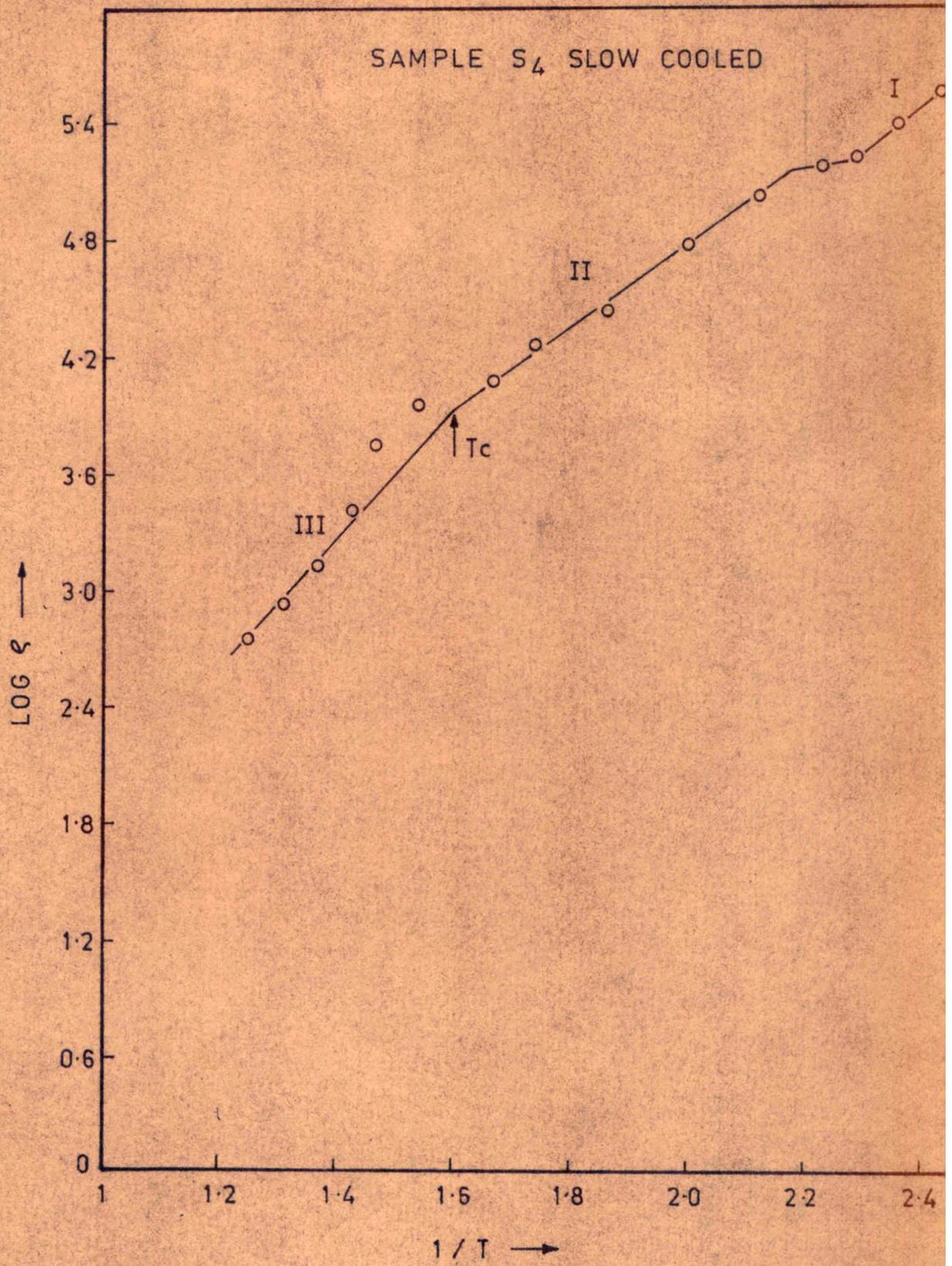


Fig. 3.6

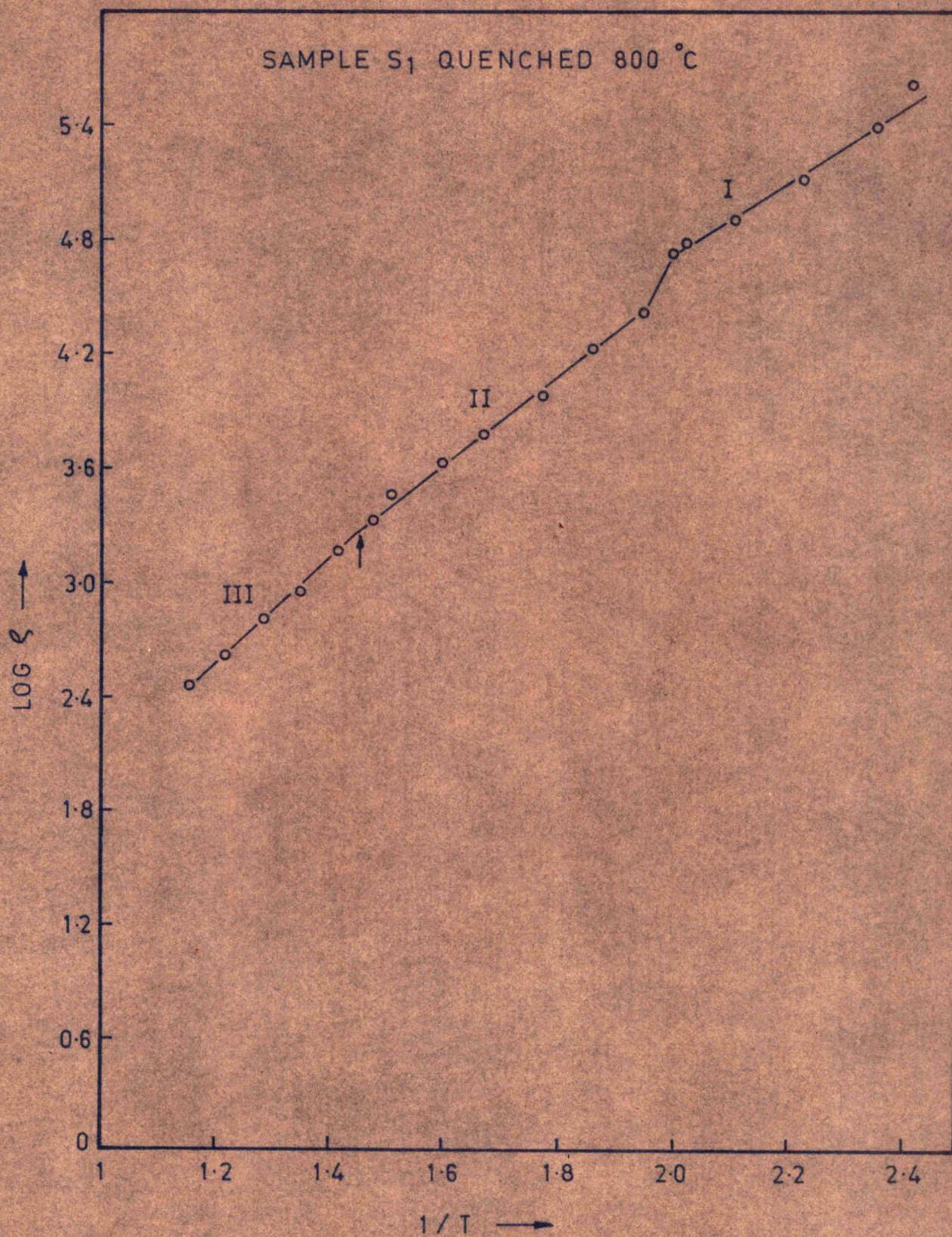


Fig. 3-7

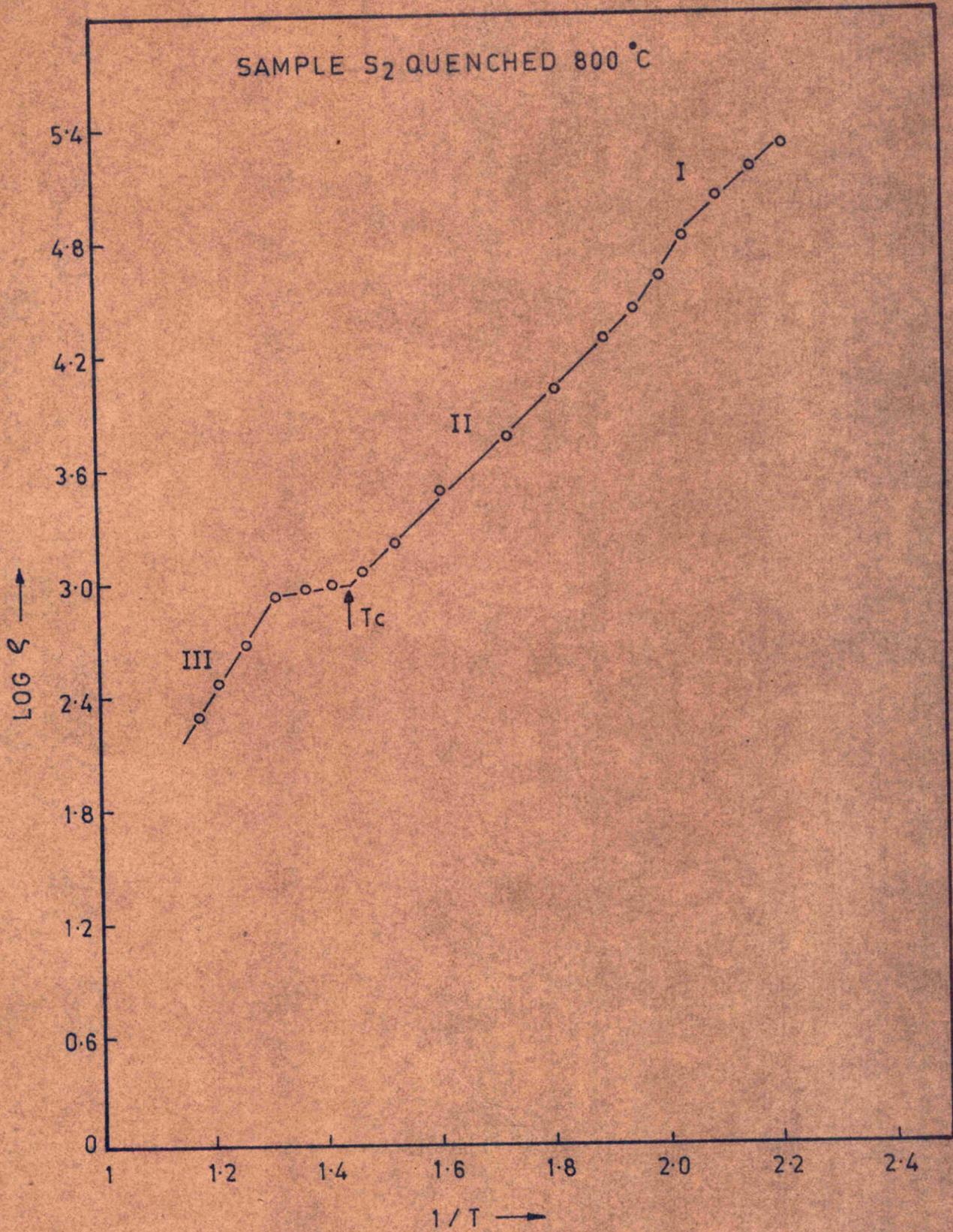


Fig. 3·8

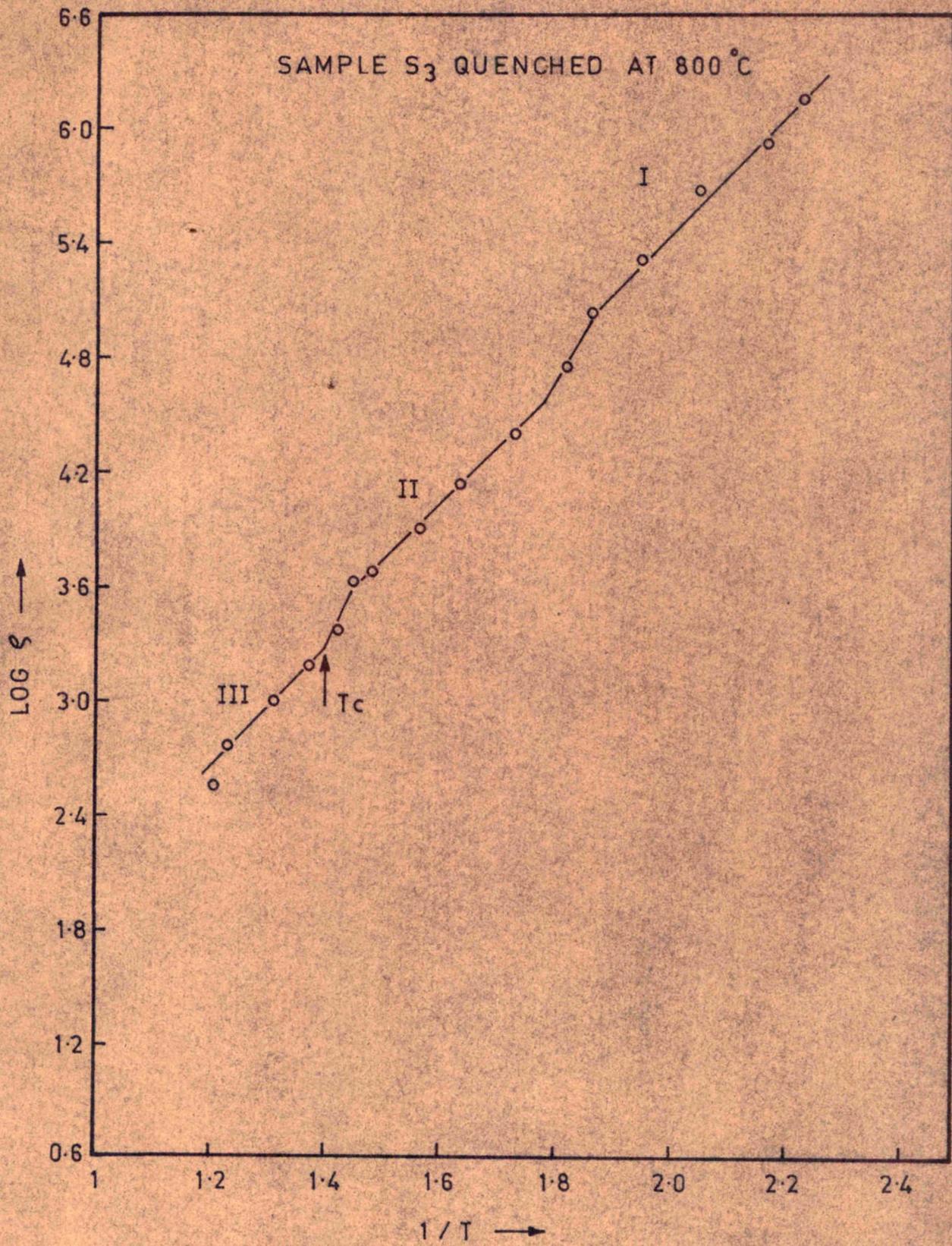


Fig. 3-9

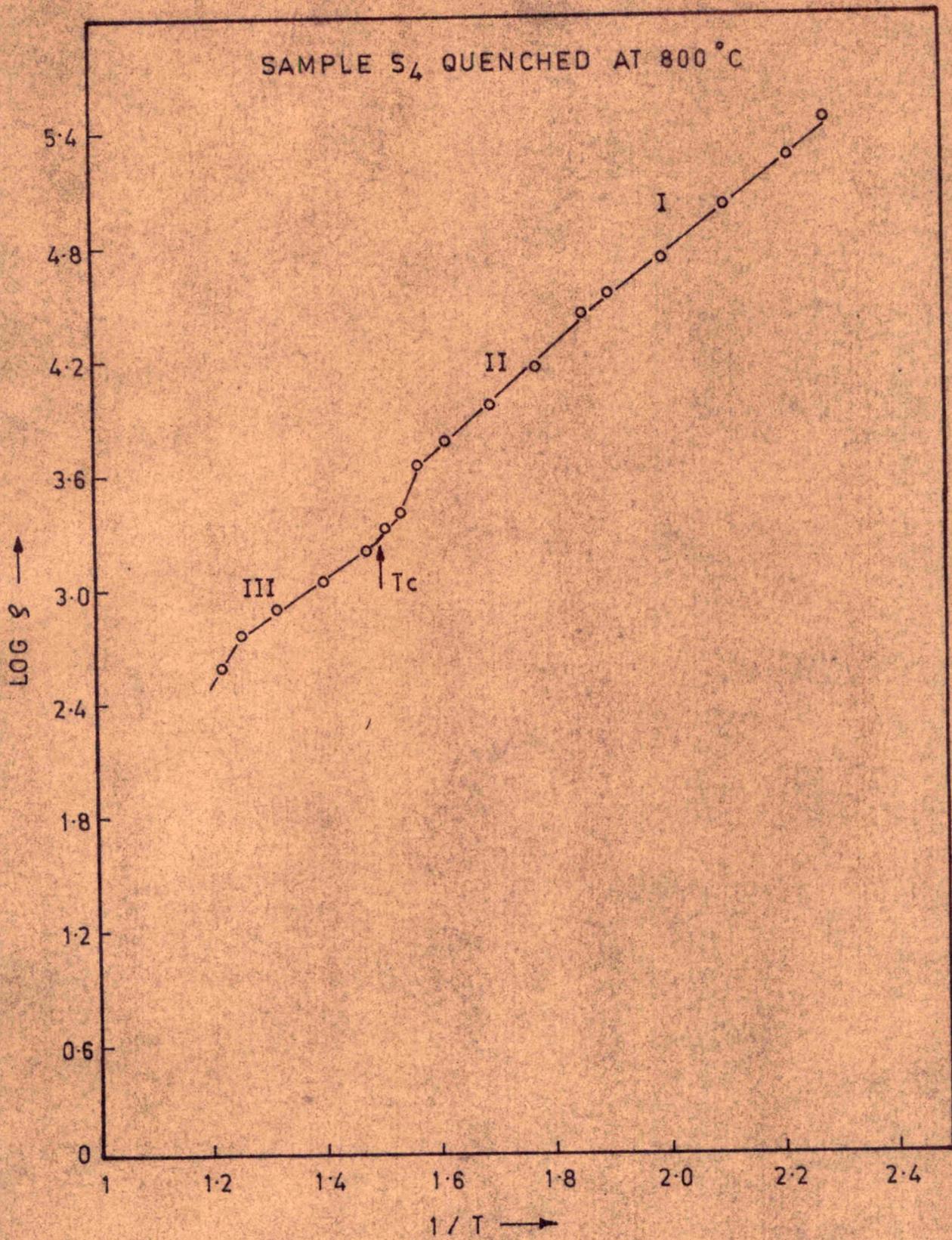


Fig. 3-10

segments ^{show} different conduction mechanisms Verwey et al⁽¹⁾ have observed this type of discontinuities in Mn Ni ferrites and that these discontinuities shifted towards higher temperatures where Mn content increases. They observed high activation energy of materials to be due to high resistivity at room temperature. Romejin⁽¹⁶⁾ has observed such discontinuities in Mn_3O_4 and Fe_3O_4 at low temperature, He observed thermal hysteresis in the neighbourhood of transition of Mn_3O_4 .

The activation energy for different conduction process ΔE have been calculated using the relation.

$$\rho = \rho_0 \exp (\Delta E / K_T) \dots\dots (3.8)$$

The detectable changes in the slope indicate the change in activation energy ΔE . The temperatures indicated arrow, Where the change occurs nearly coincide with the Curie temperature.

The first break is observed at low temperature which separates region I and region II. GHANI⁽¹⁷⁾ et al, have observed such breaks in Cu Ni ferrite and Clinton⁽¹⁸⁾ for Ni ferrite.

It is assumed that Cu-ferrite samples are partly compensated. This means that besides the donor centers in the

lattice of the ferrite there are acceptor centers. The formation of both types of centers arises from the loss of oxygen during the sintering process. For charge compensation a part of Fe^{3+} ions transforms to Fe^{2+} ions. The Fe^{2+} ions acts as donor centers. Additionally the loss of oxygen may cause a part of cations to occupy interstitial sites. These interstitial cations act as acceptor centers and P-carrier may be identified as holes on oxygen ions $\text{O}^{(19)}$

Copper ferrite shows a remarkable variation in its magnetic properties depending critically on the thermal history ⁽²⁰⁾. This is attributed to the distribution of Cu^{+2} and Fe^{+3} among the two non-equivalent sites, tetrahedral (A) and octahedra (B) provided by the spinel structure. It is well known that slowly cooled copper ferrite has tetragonal deformed spinel structure at room temperature, Snock ⁽²¹⁾ and Bertaut ⁽²²⁾ reported that CuFe_2O_4 is cubic above 760°C , but Ohnishi and Miyahara ⁽²³⁾ found however, the temperature of the transition from tetragonal to cubic structure to be 360°C . Rosenberg ⁽²⁴⁾ et al found that the transition of the tetragonal to the cubic phase for the slowly cooled composition is 175°C . Also Awad and Ahmad ⁽²⁵⁾ studied this transition and their results indicated a transition occurring at about 257°C . This transition phenomena affect the magnetic thermal and electrical properties of CuFe_2O_4 .

This change in the conduction can be attributed to tetragonal-to-cubic phase change that occurs in CuFe_2O_4 .

In table No.(land 2) data on activation energy, and curie temperature, are given for the slow-cooled as well as quenched samples.

In fig (3.11, 3.12) compositional variation of activation energy ΔE is shown for slow cooled as well as the quenched sample. The activation energy is minimum corresponding to 40% copper. However this change is more appreciable in the quenched samples.

In fig.(3.13) the compositional variation of curie temperature is shown. It is seen that there is no appreciable change in T_c on quenching, however as the content of Cu is increased the T_c values show decreasing trend.

The conduction mechanism in ferrites is due to exchange of electron from Fe^{+3} to Fe^{+2} in the same (octahedral lattices) lattice. This requires little energy to move and electron under the influence of electric field these extra electrons can be considered to constitute the conduction current by hopping from one site to the other . Recently Klinger has reviewed the conduction mechanism in ferrites. He stated that hopping of polarons is the main conduction mechanism in ferrites. He has suggested two phase mechanism for hopping of polarons.

T A B L E No. 1

THE DATA OF ELECTRICAL RESISTIVITY OF SLOW COOLED SAMPLES

F e r r i t e s	: A ctivation energy (E)	: Curie temp.
: Region I	: Region II	: Region III
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Cu _{0.2} CO _{0.8} Fe ₂ O ₄	0.327 0.425 0.765	441 455
Cu _{0.4} CO _{0.6} Fe ₂ O ₄	0.297 0.357 0.595	426 447
Cu _{0.6} CO _{0.4} Fe ₂ O ₄	0.418 0.424 0.714	416 440
Cu _{0.8} CO _{0.2} Fe ₂ O ₄	0.396 0.440 0.700	393 432

I A B L E N o . 2

THE DATA OF ELECTRICAL RESISTIVITY OF QUENCHED SAMPLES

Ferrites	Activation energy (E)	Region I	Region II	Region III	Curie temp. from graph (Tc)	Curie temp. from experiment (Tc)dc
$Cu_{0.2}CO_{0.8}Fe_2O_4$	0.357	1.488	1.891	451	445	
$Cu_{0.4}CO_{0.6}Fe_2O_4$	0.336	0.549	0.833	446	426	
$Cu_{0.6}CO_{0.4}Fe_2O_4$	0.654	0.744	0.850	441	423	
$Cu_{0.8}CO_{0.2}Fe_2O_4$	0.773	0.892	1.488	416	420	

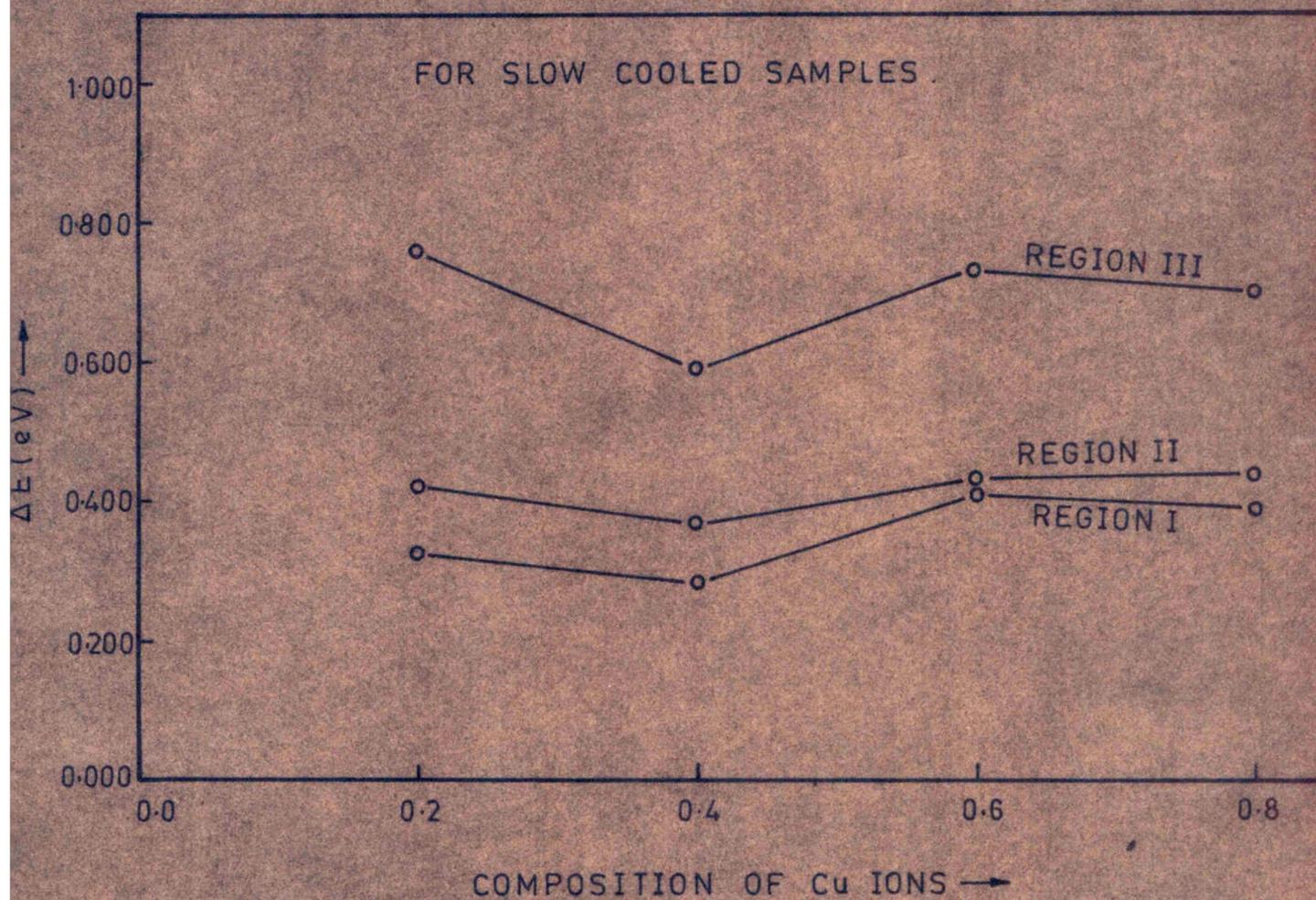


Fig. 3-11



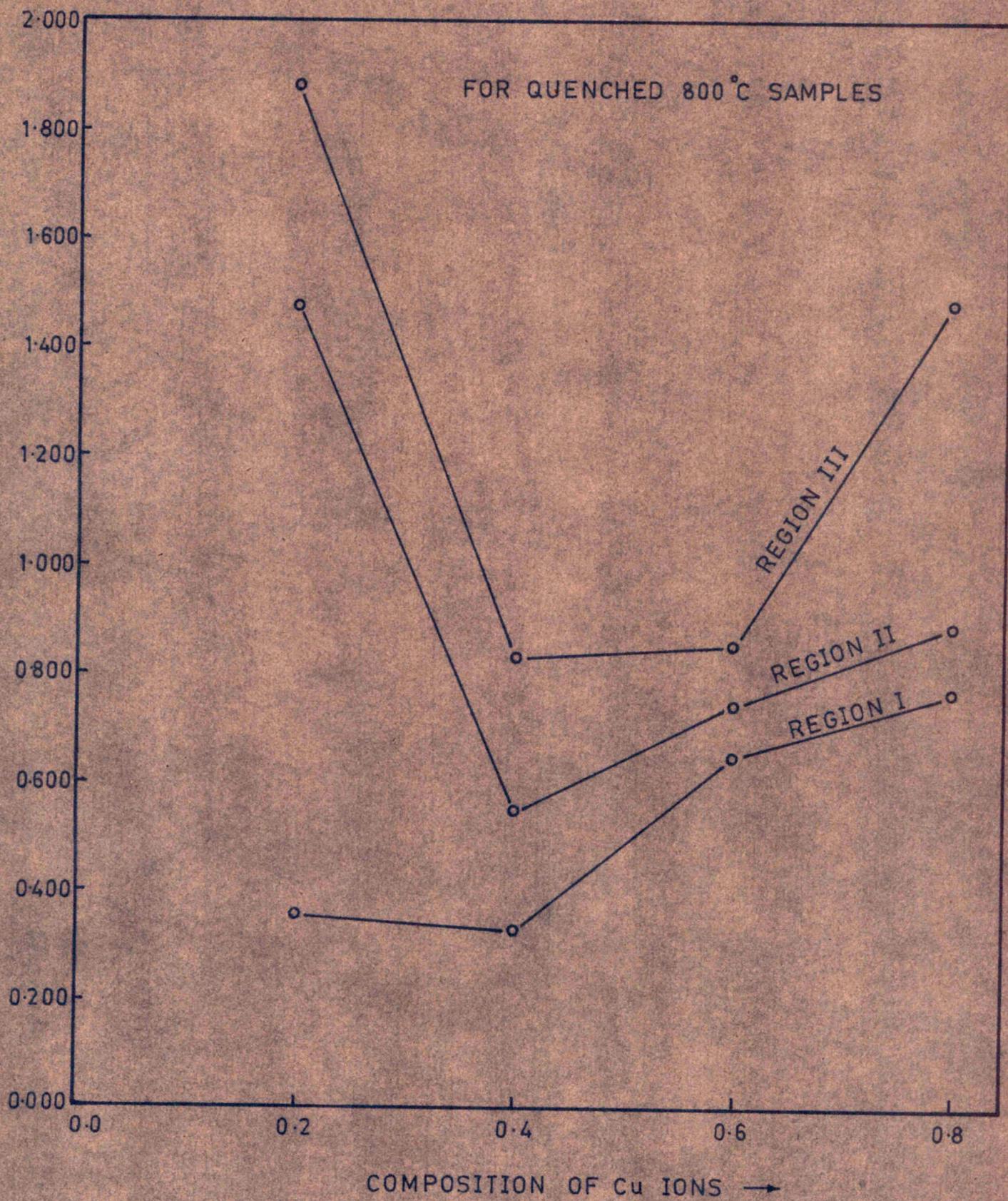
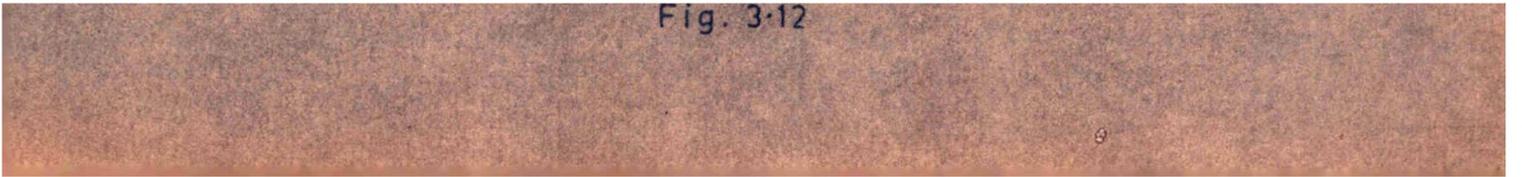




Fig. 3-12





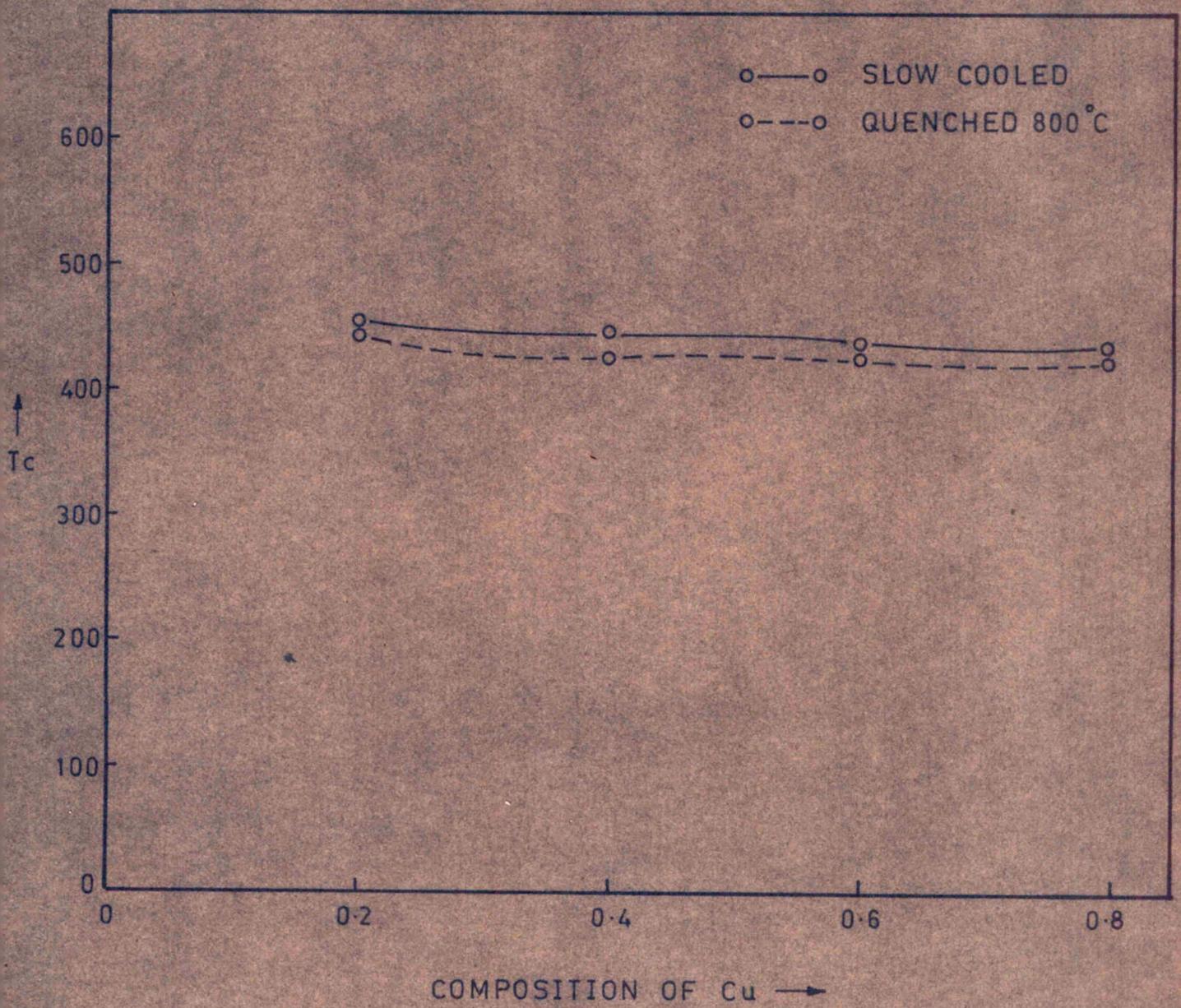


Fig. 3-13







It is reported that for the values of ΔE in the range of 0.2 eV lead to usual hopping of electron of the form $Fe^{+2} \rightleftharpoons Fe^{+3}$

Our values of E are far in excess of this value suggesting that the conductivity in our ferrite both at low and high temperature is governed by hopping process of Polaron.

Another co-relation may also play a role in determining the mechanism of charge transport. The electrostatic interaction between a conduction electron or hole and nearby ions may result in a displacement of the latter and hence in polarisation of the surrounding region. So the carrier becomes situated at the center of the polarisation potential well.

If this well is deep enough a carrier may be trapped at a lattice site and its translation to a neighbouring site may be determined by thermal activation. This has been described by hopping mechanism. That electron takes part in a ~~diffusion~~^{migration} process by jumps from one lattice site to another. The thermal activation process is encountered in ionic diffusion and ionic conductivity.

The following expression for mobility can be written.

$$\mu = \frac{ed^2}{kT} \exp \left\{ -\frac{q}{kT} \right\} \dots \dots \dots (3.9)$$

Where d = distance between nearest neighbours
 r = Frequency of Vibration
 q = activation energy for hopping process.

This has been used by many authors in discussion of conductivity with the development of Polaron theory it has become evident that equation (3.9) represents a special case of much more complicated relationship between mobility ^{μ} and a parameter of ionic lattice.

For relastic model for the calculation of mobility a knowledge of approximate spatial extent of the potential well

is required. If the potential well extends over many lattice units in the crystal, the excess charge may be considered to interact with a dielectric continuum and this model has been employed by Frohlich to Formulation interaction Hamiltonian for large Polarons. He gave the Parameter known as electron-Phonon coupling constant for the discussion of Polarons, α . For small Polarons the well is confined to a volume comparable to the ionic volume. It is self evident for small Polaron the interaction between the individual near neighbour ions is important. The actual Polaron size can be inferred from consideration of the free energy. Small Polaron formulation is favoured in solids which combine a large coupling constant with a narrow conduction band.

At sufficiently low temperature a small Polaron should behave as particle moving in a very narrow band. At high temperatures small Polaron motion may result from the absorption of one or more phonons and this process is essentially the hopping mechanism. If the time of tunnelling of the excess charge from one ion to the next is less, than the time T between successive hopping transitions, small polaron conduction will be dominant transport mechanism. There is strong experimental evidence for the existence of small polarons and for the hopping process.

From the values of ΔE in the region II it can be concluded that the conduction mechanism is due to hopping of

polaron because ΔE value are in excess of 0.2 eV.

The break which separates region II and III nearly coincides with the ferrimagnetic Curie temperature for the ferrites. This break also indicates that the conduction mechanism changes because of the transition from ferrimagnetic to paramagnetic transition.

On addition of copper Curie temperatures are lowered this is expected because a more magnetic cobalt ion is replaced by less magnetic copper ion the reduction in the Curie temperature indicates reduction in the A-B interaction. This fact is also corroborated by the observation of reduction in the magnetisation due to addition of copper in the mixed system.

The trend exhibited by the quenched sample is also similar in the regions I, II, III as far as the conductivity variations with temperature are concerned. However there is a considerable increase in the values of ΔE in the quenched samples indicating that more impedance to the hopping of the polarons is offered by the quenched samples. This can be correlated with the cation distribution obtained on the basis of Gilleo's formula.

It is seen that there is more migration of copper from B-site to A-site on quenching. This means the

contribution to the conductivity from $\text{Cu}^{+2} \rightleftharpoons \text{Cu}^{+1}$ is reduced while the contribution to the resistivity from $\text{Fe}^{+2} \rightleftharpoons \text{Fe}^{+3}$ is increased the net result is increase in the resistivity on quenching. Thus it is the reduction in the content of Cu^{+2} ions on B-site that leads to increase in ΔE due to increase in the impedance offered to polaron hopping.

On quenching the curie temperature in the mixed $\text{CuCoFe}_2\text{O}_4$ system have shown increase. This is in one to one correspondence with the increase in the magnetisation observed on quenching. Thus on quenching the A-B interaction are strengthened which can be related to more migration of Cu ions to A-site leading to increase of magnetisation of B-site.

In figure (3.14, 3.15) compositional variation of resistivity at various temperatures for the slow cooled and quenched (800°C) sample is shown. It is seen that on addition of Cu upto 60% the resistivity increases while further addition of copper decreases the resistivity similar trend is exhibited by the quenched samples except that the resistivity has increased on quenching. These behaviour may be due to cation migration.

The conductivity of ferrites presents many complexities. The interpretation of D.C. resistivity in polycrystalline material becomes rather involved, due to the presence of grain boundaries, giving rise to additional

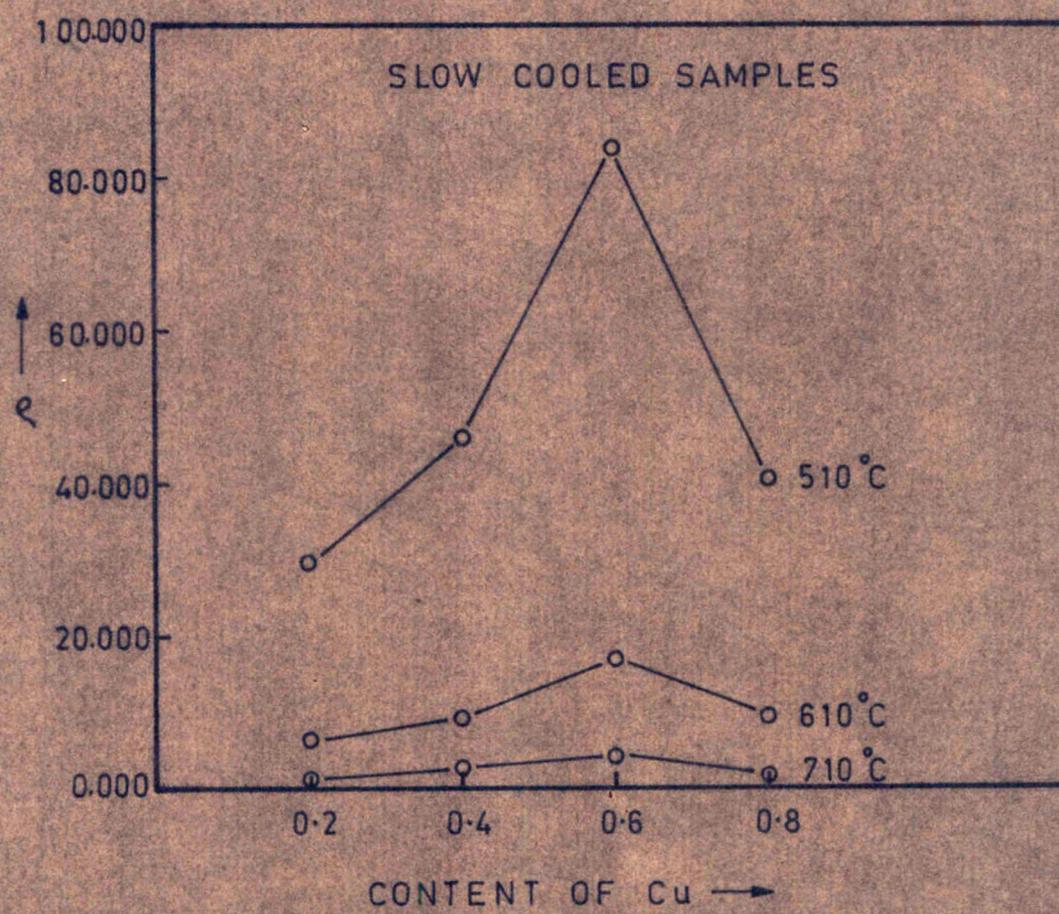


Fig. 3-14

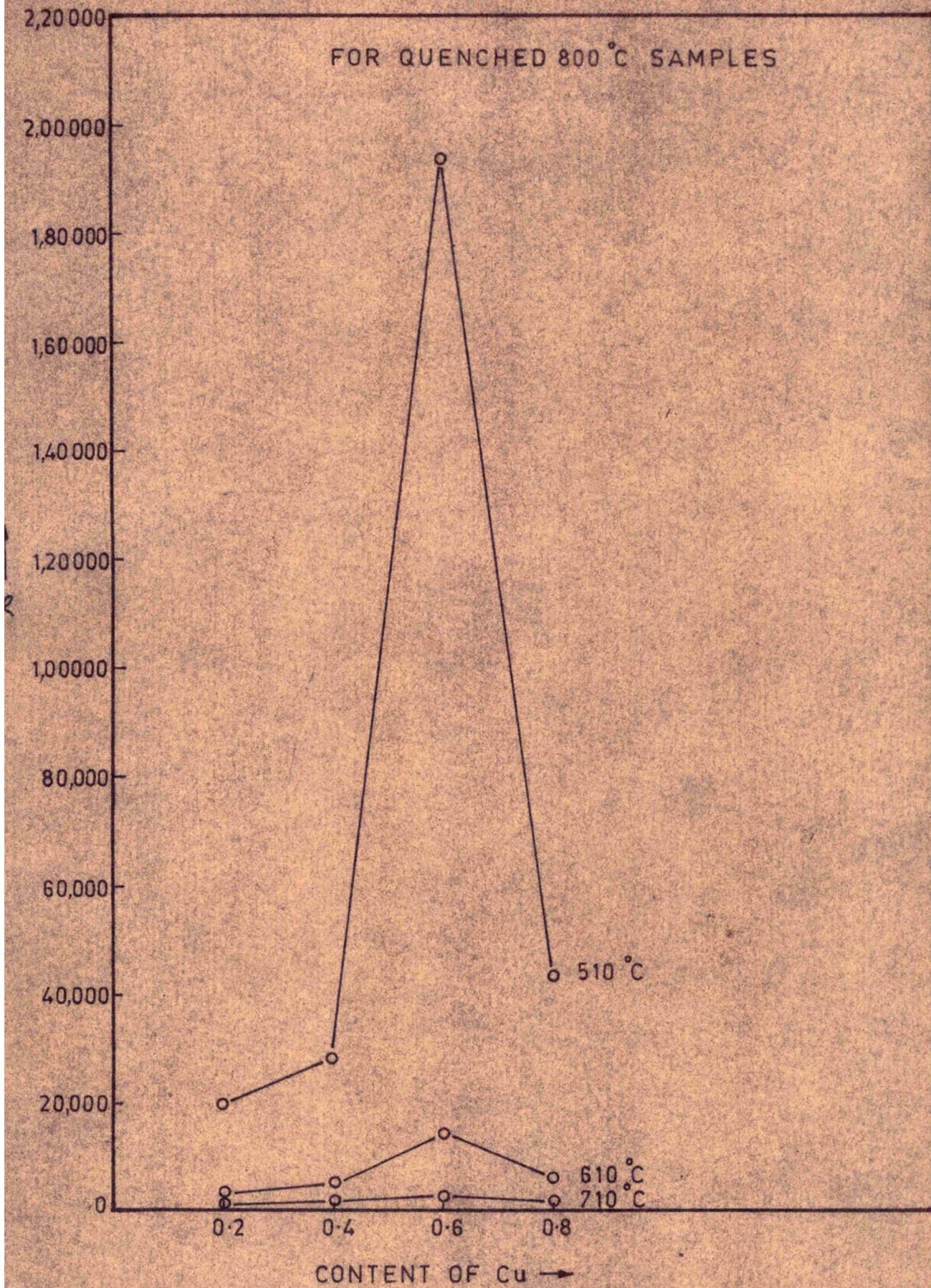


Fig. 3-15

scattering. The cation or anion vacancies may be responsible for acceptor and 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 anion or cation vacancies may also contribute a component of ionic conductivity to the D.C. conductivity of the sample.⁽²⁷⁾ Various conduction mechanisms based on different models for the donor levels have been given by different workers.⁽²⁷⁻³⁰⁾

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