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Magnetic after-effect in transformer steel and other magnetic alloys.

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MAGNETIC AFTER-EFFECT IN TRANSFORMER STEEL
AND OTHER MAGNETIC ALLOYS

by

JAMALUDDIN AHMED

A Dissertation
Submitted to the Faculty of Graduate Studies through the Department of Electrical Engineering in partial fulfilment of the Requirements for the Degree of Doctor of Philosophy

UNIVERSITY OF WINDSOR
1974
ABSTRACT

The phenomenon of magnetic after-effect has been studied by measuring the time decay of initial permeability in various temperature regions in Fe-Cu, Fe-Al, Fe-Si and Fe-Ni alloy. The time decay of initial susceptibility was investigated by measuring the time decay of secondary induction of a toroid. Results show that the after-effect is predominant in the temperature region between $-50^\circ$C and $+70^\circ$C and between $350^\circ$C and $525^\circ$C where the initial permeability exhibits minima when measured as a function of temperature. The disaccommodation in the low temperature region disappears after totally decarburizing the specimen and reappears with the introduction of carbon which shows that the magnetic after-effect in the low temperature region in these alloys is due to the interstitial atomic configuration. The after-effect in the high temperature region is found to be associated with the substitutional atomic orientation and in this temperature region removal of carbon has no effect. The after-effect has been found to be independent of frequency in both the low and high temperature regions. The various results described above are found to be similar in all the above alloys.
Experiments were performed with Fe-Al alloys containing various amounts of Aluminum in the high temperature region. The after-effect was found to be proportional to the square of Al-content which is in conformity with the theoretical predictions [1]. However, the phenomenon in Fe-Ni-alloy specimens is found to be due to vacancy diffusion.

In the low temperature region the percent drop in permeability at a particular temperature has been found to be directly proportional to the carbon content in solution. The effect of the interstitial carbon impurity on the phenomenon has been further studied by measuring the loss factor tan δ of specimens with different carbon contents at various temperatures and the peak value of loss factor has been found to be influenced by the carbon content in solution. The experimental results of loss factor vs temperature have been compared with the computed values (using an IBM 360 computer) from internal friction theory using the theoretical model of Koiwa [2].

The effect of plastic deformation on the after-effect has been investigated in both the low and high temperature regions. In the low temperature region the after-effect is found to be suppressed by the introduction of dislocations but the mechanism of diffusion remains unaltered.
and the activation energy remains unchanged. The total decay in initial permeability is found to be proportional to the stabilized value of initial permeability for various deformed samples containing a particular amount of carbon. The proportionality constants of these lines are found to vary with the carbon content in solution thus showing that this proportionality constant is a measure of the stabilization field. The loss factor $\tan \delta$ has been found to decrease with increasing deformation. The after-effect in the high temperature region has been found to be suppressed by deformation and the activation energy of the process decreases gradually with increasing deformation as the mechanism becomes very complicated. Magnetic after-effect has been found to be totally suppressed by the presence of minute traces of copper-precipitates in Fe-Cu alloy. The results are explained qualitatively in the absence of a suitable quantitative model.
ACKNOWLEDGMENTS

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CHAPTER I

INTRODUCTION

The magnetic after-effect is defined as the time dependent change in magnetization accompanying a change in the magnetic field. The phenomenon of magnetic after-effect has been known for a long time. It belongs to a general category of processes termed as relaxation process. Snoek (1941) gave an explanation of the process on an atomic basis. He showed that carbon and nitrogen in solid solution in the octahedral interstitial sites were responsible for the relaxation process. The same relaxation process can give rise to the various following phenomena.

Disaccommodation or Time Decay of Permeability:

The initial permeability $\mu'_1$ of a material is the slope of the linear and reversible part of the virgin magnetization curve of an ideally demagnetized specimen and is generally defined by the expression

$$\mu'_1 := \lim_{\Delta H \to 0} \frac{\Delta B}{\Delta H}$$

where $\Delta B$ is the change in magnetic induction corresponding to a small field change $\Delta H$. The change in magnetization is a measure of the mobility of the domain walls which in
turn may be affected by the metallographic state of the material. Thus initial permeability is greatly influenced by the microscopic structure of the specimen. Immediately after the demagnetization of a magnetic material the distribution of interstitials in the different octahedral sites can be considered to be homogeneous and the permeability is high. The redistribution of interstitials which then sets in will stabilize the new configuration of the domain walls thus reducing the mobility of the wall. This shows that the permeability decreases with time. This time decrease of permeability is called disaccommodation. Since the domain wall has less mobility in a stabilized condition the magnetization curves measured directly after demagnetization and a long time after demagnetization differ considerably. In the relaxed state a larger field is required to obtain a certain magnetization than the field needed immediately after demagnetization. The difference between the fields required for magnetization immediately after the specimen is demagnetized and long after it is demagnetized is called the Stabilization Field. Measurement of the stabilization field is another method of studying the disaccommodation.
Time dependence of Magnetic Viscosity: Loss Measurement:

If a material is subjected to a small alternating magnetic field the after-effect of the magnetization can cause a lag of the magnetization with respect to the magnetic field. This phase lag manifests itself as an energy loss termed as loss factor \( \tan \delta \) and the lag angle \( \delta \) is called the loss angle. This loss angle \( \delta \) is related to the relaxation time \( \tau \) of the process by the following expression.

\[
\tan \delta = A \frac{\omega \tau}{1 + (\omega \tau)^2} \quad (1.1)
\]

where \( \omega \) is the angular frequency and \( A \) is a constant related to the peak value of the loss factor.

Internal Friction or Elastic After-effect

In the unstressed condition of a material equal number of interstitial atoms will be found in all the available sites of a b.c.c. crystal lattice, while under the influence of an applied force a redistribution of the interstitial atoms takes place in the preferred sites with axes parallel to the direction of stress. If an oscillatory stress is applied, there will be phase difference between the stress and the strain thus resulting in a damping
effect. When the frequency of cyclic stress is very high compared to the jump frequency of the interstitials, the interstitial atoms cannot follow the stress change, the resulting loss is zero. Similarly when the frequency of stress is very low compared to the jump frequency, the strain can instantaneously follow the stress and there will be no phase difference between stress and strain. But when the frequency of stress is comparable to the jump frequency the strain will follow the stress with a definite lagging phase angle and the phase angle will be maximum at some intermediate frequency. Since it is difficult to vary the frequency over a wide range, the internal friction is generally studied by keeping the frequency constant and varying the temperature. As the jump frequency varies with temperature maximum damping is observed at some optimum temperature.

So far we have introduced the relaxation phenomenon caused by the preferred orientation of interstitial atoms. The same phenomenon can be caused by diffusion of vacancies as suggested by Dietze [3] and Balthesen [4] or by a preferential orientation of pairs of constituent solute atoms in substitutional ferromagnetic alloys as has been foreseen by Neel [1,5] and Taniguchi [6,7]. However, since
the substitutional elements are of much bigger size they can diffuse only at much higher temperature while the phenomenon with interstitial elements can take place even around room temperature. In general relaxation processes are measured by static or dynamic methods. In the static method of measurement a force is applied and the response is measured as a function of time. Dynamic method consists of cyclic application of a force and measurement of either the lag between the force and the response (or its equivalent, the energy loss per cycle) or the time decay of the amplitude of the response. The same relaxation phenomenon can be studied by both static and dynamic methods and both characterize the relaxation process by a relaxation time \( t \). Let us take a relaxing property \( P \) which has an initial value of \( P_0 \) and after complete relaxation it assumes a value of \( P_\infty \) and let \( P_t \) be the value at any time \( t \). Considering that the rate of change of the relaxing property at any time is proportional to the difference between the value of the property at that time and the equilibrium value of the property. This gives

\[
\frac{d}{dt} (P_t - P_\infty) = -\frac{1}{\tau} (P_t - P_\infty) \quad \ldots \quad (1.2)
\]
On integration

\[ P_t - P_\infty = Ke^{\frac{-t}{r}} \]

where \( K \) is a constant.

At \( t = 0 \)

\[ P_t = P_0 \]

\[ K = P_0 - P_\infty \]

\[ P_t - P_\infty = (P_0 - P_\infty)e^{\frac{-t}{r}} \ldots \text{(1.3)} \]

The relaxation time \( \tau \) is the time required for the relaxation process to undergo 62.8 percent of the total change.

In the present investigation the relaxation phenomena due to both interstitial and substitutional atomic diffusion were studied in different iron based alloys such as Fe-Cu, Fe-Al and Fe-Si. The samples were treated in the furnace and the time decrease of initial permeability was studied by energizing the primary of a toroid by a small a.c. signal and measuring the secondary induction. The toroid was placed in a furnace for measurement in the high temperature region. The loss factor \( \tan \delta \) was measured using a bridge method.

The effect of plastic deformation on the relaxation process was investigated in both the low and high
temperature regions by measuring disaccommodation and loss factor. Copper was precipitated in Fe-Cu alloy and its effect on disaccommodation investigated. The experimental values of loss factor in Fe-Si alloy was compared with the computed values obtained by using Koiwa's theoretical model.

In Chapter II the theories of interstitial and substitutional diffusion controlled after-effects are discussed. In Chapter III the experimental procedures are described and in the subsequent chapters the results are critically analysed and compared with the theoretically predicted values. Some of the results are discussed qualitatively in the absence of any adequate model. However it is expected that the findings of this investigation contribute towards further understanding of the relaxation process.
CHAPTER II

THEORETICAL CONSIDERATIONS

The interstitial diffusion controlled magnetic after-effect was first investigated by Ewing [8] in 1885. He observed that when a magnetic field is applied to a magnetic material, an appreciable time is required for the magnetization to obtain its final value. He measured the increase in magnetization as a function of time with a magnetometer in an iron wire specimen in the linear part of the magnetization curve. Subsequently, up till 1910 many such magnetic viscosity or after-effect (Nachwirkung) were observed using magnetometric methods and analogous elastic lag was observed during mechanical deformation [9]. Richter [10,11] (1937-38) and Wittke [12] (1938) confirmed these results by observing a well defined lag in specimens of carbonyl iron after removal of an applied magnetic field. Richter's magnetic measurements were made at low induction by ballistic and magnetometric methods at a field level of up to 0.1 oersted. He found the phenomenon to be highly temperature sensitive. His important contribution was the mathematical formulation of the after-effect based on a distribution of relaxation times which agreed with the experimental
results. Snbek [13] in 1938 first attempted to explain the phenomenon on an atomic basis and proposed a model for the after-effect based on diffusion of carbon to interstitial sites which are elongated by the magnetostrictive effect of the magnetization vector. In addition, Snoek showed that a type of internal friction (Elastic after-effect) and the magnetic after-effect were both caused by interstitial diffusion in iron.

Snoek's Model

According to this model, the basic cause for interstitial diffusion controlled magnetic after-effect is the preference of interstitial for certain interstitial sites depending on the direction of the magnetization vector. Iron has a body centred cubic structure and the interstitial sites in iron are midway between the corner atoms along the b.c.c. lattice edges. All of these interstitial sites \( \left( \frac{1}{2} \overline{0} \overline{0}; \frac{1}{2} \overline{0}; 0 \overline{0} \overline{2} \right) \) are energetically equivalent in the absence of any external stress or magnetization vector and are therefore the stable positions for the interstitials. In iron the easy direction of magnetization are the \( <100> \) directions. The \( \left( \frac{1}{2} \overline{0} 0 \right) \) interstitial site is considered to be parallel to the \( [100] \) direction and the \( (0 \frac{1}{2} 0) \) and
Interstitial sites in the body centered structure.

therefore either parallel or perpendicular to the direction of magnetization. Snoek [13] proposed a theory for the existence of preferred interstitial sites with respect to the magnetization. He reasoned that if the magnetization vector points in a direction of easy magnetization i.e. \( \langle 100 \rangle \) direction, the lattice is elongated in this direction by the magnetostriction constant \( \lambda_{100} \) due to magnetostrictive effect while in the two cubic directions perpendicular to the magnetization the lattice is shortened by \( \frac{1}{2} \lambda_{100} \). As a result of this lattice deformation the energy equivalence of the interstitials occupying the three types of sites will be destroyed. The energy of
the interstitial atoms in octahedral sites lying parallel to the direction of magnetization will be lower than those perpendicular to the magnetization vector. Due to this difference in energy of the atoms in different interstitial sites an anisotropic distribution of interstitial atoms will take place. Due to the accommodation of the interstitial atoms to the magnetostrictive strains the free energy becomes lower and as a result the magnetization will be stabilized. Compared with the state of isotropic distribution of interstitial atoms an extra energy is needed in order to turn the magnetization vector over a certain angle from the direction in which it is stabilized. This extra energy may be termed as stabilization energy and the corresponding field may be termed as stabilization field.

Tomono [14] in 1952 measured the magnetic after-effect with a heavily damped ballistic galvanometer in Armco iron after removing a magnetic field of 15 m.Oe. Tomono found that the after-effect induction decreased exponentially with time and is represented by a single relaxation time in contrast to Richter's observation at higher field level which indicated a distribution of relaxation times. Neel [15, 16] in 1952 criticized
Snoek's model and pointed out that the magnetostrictive coupling is too weak to explain the magnetic after-effect. His point of view was further supported by the experimental finding of Bosman [17] et al. in which the magnetostriction constant $\lambda_{100}$ for a Fe-6% Si alloy was found to pass a zero value while the after-effect remained constant. Bosman [18] et al also investigated the influence of pressure on the relaxation process and found that the application of a hydrostatic pressure of 3,000 kgm/cm$^2$ does not lead to change exceeding 1%. Neel therefore introduced an additional interaction between the interstitial atom and the magnetization vector. De Vries, Van Geest, Gerdsdorf and Ratheneau [19] later in 1959 experimentally confirmed Neel's conclusion by measuring the time change of strain of an iron sample during the magnetic after-effect and found that the length of the sample decreased in the direction of magnetization during the after-effect and therefore concluded that the interstitial atoms preferred sites which are perpendicular to the direction of magnetization vector. As a result, the diffusion of interstitials to energetically preferred sites with respect to the magnetization vector acts as a stabilizing
force for that particular direction of magnetization and consequently additional energy is required to change the direction of magnetization. The additional force required to overcome the stabilization effect of the interstitials, can be called the Stabilization Force. Neel calculated \[16\] the stabilization force for a 180° and 90° wall which lies on \{100\} planes. In the following figures, the stabilization energy \(E_S\) and stabilization force \(F_S\) are drawn schematically for a 90° and 180° wall according to Neel's calculation.

(a) Stabilization energy vs displacement.

(b) Stabilization force vs displacement.
The figure shows an important difference between the $90^\circ$ and $180^\circ$ wall. As the $90^\circ$ wall moves sufficiently far away from its initial potential well, wall movement results in a $90^\circ$ change in magnetization in the volume through which it passes and therefore energy increases linearly with distance and consequently the stabilization force is constant. On the contrary, as the $180^\circ$ wall moves far away from its initial stabilized potential well, wall movement reverses the magnetization in the volume swept except for two small regions—the initial place of the wall and the place where the wall is actually situated—and consequently does not alter the preferred interstitial sites. As a result the stabilization force on the $180^\circ$ wall first increases as it moves from its initial position, then decreases to zero. The instantaneous application of a magnetic field moves a domain wall until the force exerted on the wall by the applied magnetic field is balanced by the resisting force of the interstitial stabilization and other matrix disturbances such as inclusions, dislocations, and microstress gradients. However, after the instantaneous wall movement, diffusion of interstitials to energetically preferred sites begins. In the case of $90^\circ$ wall movement, the diffusion process
decreases the interstitial stabilization force. As a consequence, the applied magnetic field force becomes greater than the combined opposing forces and the wall moves again. This process continues until in the final stabilized condition of the wall, the field force equals the combined resisting forces. Thus the magnetic after-effect consists of the gradual movement of the wall caused by the imbalance of forces which are created by the diffusion of interstitials to preferred sites in the $90^\circ$ domain wall. The movement of the $180^\circ$ wall can cause after-effect in the same way as the $90^\circ$ wall only if the wall does not move so far away from its initial position that the stabilization force is zero. This requires the application of a small magnetic field for only then the $180^\circ$ wall movement will be small enough from its initial potential well and the interstitial diffusion will create a reduction in the stabilization force and allow subsequent wall movement.

Brissonneau [20,21] investigated the time decrease of initial permeability after demagnetization of iron. He measured the stabilization force proposed in Neel's theory in terms of a stabilization field as a function of carbon
concentration. Rathenau [22,23,24] in 1958 investigated the same decrease of permeability caused by interstitial diffusion of nitrogen in iron as a function of time. Tomono [14] investigated the magnetic after-effect using dynamic experimental methods and found that the process can be represented by a single time constant \( \tau \) at a given temperature according to the relation

\[
\tau = \tau_0 \exp \left( \frac{Q}{RT} \right)
\]

where \( Q \) is the activation energy of the process and \( R \) is the gas constant.

Bosman and Schenkel measured the loss factor \( \tan \delta \) (\( \delta \) is the lag angle between the magnetization \( B \) and magnetic field \( H \)) using a bridge method at various temperatures and calculated the relaxation time using the relationship

\[
\tan \delta = (\tan \delta)_{\text{max}} \frac{\omega \tau}{1 + (\omega \tau)^2}
\]

where \( \omega \) is the angular frequency.

The experimental values were compared with the calculated values obtained from

\[
\tau = \tau_0 \exp \left( \frac{Q}{RT} \right)
\]
where $\tau_0$ was arbitrarily selected and $Q$ was taken as the activation energy for diffusion of carbon in iron. Good agreement was found between theoretically and experimentally determined values of $\tau$.

The influence of plastic deformation on the interstitial diffusion controlled magnetic after-effect has been first investigated by Schulze [25] (1938). He made dynamic measurements of loss angle in carbonyl iron as reported by Richard and Lynch [26] (1953). Schulze found that the interstitial diffusion controlled after-effect was not present in as rolled material with 90% reduction. The material was then recrystallized and subjected to small amounts of deformation. He observed that after-effect decreased with cold work and disappeared totally at 2.3% deformation. The magnetic after-effect in cold-rolled iron was also studied by Tomono [14] (1952) by semi-static and dynamic measurements. He annealed cold rolled Amco iron at various temperatures before measuring the after-effect. He found that the static after-effect in the weak field is not observable in contradiction to Schulze's measurement but the after-effect is observable in the range of irreversible magnetization. The after-effect in the
range of initial permeability became observable in a recrystallised state in agreement with Schulze's result. Brissonneau [20] in 1957 made measurement of time decay in permeability of an iron sample with 39 p.p.m. carbon after subjecting the sample 2 to 5% plastic strain. He found that the maximum stabilization field of the deformed samples was reduced by 10% from that of the annealed samples. He attributed this decrease to an increase in resistance to domain wall movement by various matrix disturbances such as vacancies, dislocations etc. introduced during the deformation process and a decrease in the carbon concentration available for participation in the after-effect phenomenon as the dislocations tie up some of the available interstitial carbon.

The relaxation process has also been investigated by loss measurement: both magnetic and elastic. Since iron has a body centred cubic structure, the octahedral sites $(\frac{1}{2}, 0, 0); (0, \frac{1}{2}, 0); (0, 0, \frac{1}{2})$ etc. are the stable interstitial sites and energetically equivalent. If the material is subjected to a small alternating magnetic field the after-effect of the magnetization can cause a lag of the magnetization with respect to the magnetic field thus producing
an energy loss. To measure this energy loss the specimen
is placed in a coil and the effective resistance and self
inductance of the coil are measured using a precision
impedance bridge. The loss factor $\tan \delta$ is then given by

$$\tan \delta = \frac{\Delta R}{\omega L}$$

where $\Delta R$ = Equivalent resistance of after-effect loss.

$\omega$ = Angular frequency.

$L$ = Self inductance of the coil.

Analogously in the case of Elastic after-effect, under the
influence of an applied force a redistribution of the
interstitial atoms takes place so as to occupy the
ergetically most stable positions. Under the influence
of a cyclic stress, there will be a phase difference
between the stress and the strain depending on the temp-
erature thus resulting an energy loss. If the temperature
is low, the jump frequency of the interstitials is low
and the strain cannot follow the stress, so energy loss
is zero. Again if the temperature is high, the jump
frequency of the interstitials is high and the strain can
instantaneously follow the stress. There is no phase
difference and the energy loss is again zero. But when
the temperature is such that the jump frequency is com-
parable to the stress frequency, the strain can follow the stress with a definite lagging phase angle and the loss will be maximum at an optimum temperature. This energy loss is measured with the help of a torsion pendulum [27]. The specimen is loaded elastically and then unloaded; the elastic strain does not relax instantaneously to zero but relaxes to zero in a time dependent manner. The most fundamental measure of this internal friction is the fractional energy loss per cycle, called the specific damping capacity

\[
\frac{\Delta W}{W} = \frac{\text{Energy loss per cycle}}{\text{Total vibrational energy}}
\]

A widely used method to measure internal friction is to determine the rapidity with which the oscillations of the system die out. If \( A_n \) is the amplitude of the \( n \)th oscillation and \( A_{n+1} \) is the amplitude of the \( (n+1) \)th oscillation then the internal friction is measured by the logarithmic decrement

\[
Q^{-1} = \frac{1}{\pi} \ln \frac{A_n}{A_{n+1}}
\]

The relaxation is characterized by a relaxation time \( \tau \) which is related to the time for interstitial atom to move.
from its original position to one of lower energy position
and is related to the internal friction by the relation

\[ Q^{-1} = (Q^{-1})_{\text{max}} \frac{\tau \omega}{1 + (\omega \tau)^2} \]

where \( \omega \) is the angular frequency and \( \tau \) is dependent on temperature by

\[ \tau = \tau_0 \exp \left( \frac{E}{RT} \right) \]

where \( E \) = Activation of the jump process,

\( R \) = Gas constant,

\( T \) = Absolute temperature.

The relaxation phenomenon due to the presence of carbon
and nitrogen in alpha iron has been investigated by a
number of workers [28,29] by measuring internal friction.
The results obtained by experimental methods show agreement
with the computed values. The experimental curves were
slightly wider than the theoretical curves (assuming a
single relaxation time) obtained from the relation

\[ Q^{-1} = Q^{-1}_{\text{max}} \frac{\omega \tau}{1 + (\omega \tau)^2} \]

However the phenomenon becomes quite complicated in
ternary substitutional alloys due to lattice distortion
caused by the substitutional atom. It has been shown by Jack [30,31] that the presence of interstitial nitrogen in the b.c.c. iron lattice causes a distortion. Let us consider the octahedron of iron atoms as shown in the figure. Nitrogen occupies the interstice at the centre of this octahedron. This results in a lattice distortion by expansion along the tetrad axis and contraction along the diad axis. Then silicon atom sitting substitutionally in the iron lattice contracts the lattice. Thus the expansion required by the interstitial nitrogen atom is partially compensated by the contraction required by the silicon atom, thus reducing the energy of the system. As a result the nitrogen atom will prefer to sit in
octahedra of which the tetrad axes contain silicon atom. Under an alternating stress the nitrogen atom will jump into equivalent sites around the Si atom. The activation energy corresponding to the change of these sites will be less than for Fe-sites due to energy gained by accommodating Si atom. In the presence of the substitutional ternary element the Debye peak is shifted and/or changed in shape and in some case the phenomenon may be reduced in magnitude or be even totally suppressed in the presence of a particular substitutional element. In this case the solute is said to act as scavenger.

Limited work has been done on internal friction measurement in ternary alloys. Snoek [29] studied iron alloys containing small percentage of silicon, cobalt and chromium. He found that the internal friction was lower than for the equivalent binary alloys. The effect of ternary addition on internal friction was first studied by Dijkstra and Sladek [32] and they investigated the effect of manganese addition on the internal friction peak of Fe-Mn-N alloys. The presence of 0.5% Mn broadened the nitrogen peak and shifted it to a slightly higher temperature. They attempted to synthesize the experimentally observed internal friction curve by the super
position of two ideal Debye peaks. Later on several
workers [33-36] investigated the Fe-Mn-N system and
attempted to analyse the result by breaking into component
Debye peaks caused by jumps of the type
(a) from Fe-Fe site to Fe-Fe site
(b) from Fe-Mn site to Fe-Fe site
(c) from Mn-Mn site to Fe-Fe site
(d) from Fe-Fe site to Fe-Mn site.

The Fe-Mn-N system was further investigated by Couper
and Kennedy [37] who used alloys with different Mn and N
content. For a N concentration of 0.25\% the damping
spectrum could be synthesized from three ideal Debye
peaks at 7\°C, 23\°C and 34.5\°C. They concluded that the
peak at 34.5\°C could be attributed to nitrogen atoms
jumping from Fe-Mn to Fe-Fe sites. The second peak at
23\°C is the normal peak for jumping Fe-Fe to Fe-Fe site.
The third peak at 7\°C was accounted for being a jump from
Fe-Mn site to Fe-Fe site and jump of N atom associated
with pairs of Mn atoms. Fe-Mn-N system was further
investigated by Enrietto [38] who showed from his results
that the influence of the substitutional Mn atom extended
over 3 to 4 atomic distances. Ratchie and Rawlings [39]
studied the effect of Mn, Cr and Ni in Fe-N ternary alloys. They synthesized the damping spectrum observed by superposition of a large number of ideal Debye peaks. They attributed these individual peaks to different types of interstitial atomic jumps. Dijkstra and Sladek [32] have widely studied the effect of Cr, Mo and V on internal friction in Fe-N ternary system and synthesized the spectrum by superimposing individual peaks. An interesting result was reported by G. Szabo-Miszenti [40] while investigating the internal friction in Fe-Ti-N system after nitriding with ammonia at 590°C. Two peaks were observed at low Ti concentration. An increase in the concentration of Ti eliminated all the interstitial atom from solution by forming the compound TiN thus suppressing the relaxation process.

On the contrary very limited amount of work has been done in the study of relaxation process by magnetic loss measurement. Bosman [41] has investigated the relaxation process in Fe-C and Fe-Si-C system by loss measurement. The values for the pure iron are the same as the computed values from the ideal Debye equation. But the curve for Fe-3.2% Si alloy has become broader
and more asymmetric. The asymmetry has been attributed to the presence of a second peak at 145°C due to carbon atoms occupying octahedral sites with one iron atom and one silicon atom. He has calculated the difference in activation energy for these two different types of jump to be 3000 cal/mole.

Until recently all the experimental results of relaxation phenomena in ternary b.c.c. alloys have been synthesized by the superposition of a suitable number of Debye peaks for different component relaxation processes. The height and position of the peaks were derived from the process of fitting the individual curves to the observed damping spectrum. This form of analysis is an oversimplification and a more rigorous analysis of the effect of ternary substitutional alloy on the interstitial distribution has recently been published by Koiwa [2].

Koiwa divided the lattice surrounding a substitutional atom into a number of spheres or shells centred on the substitutional atom. The surface of each shell contains interstitials equidistant from the substitutional atom. It is assumed that the motion of interstitial atoms
outside the sphere is not influenced by the substitutional atom and also it is assumed that the behaviour of the interstitials in each unit sphere is identical. Thus the first shell is of radius \( R \) (\( R \) is half lattice constant of a b.c.c. cell) and has four interstitial sites. Fig. (2-1) shows two unit cubes of the b.c.c. lattice in which a substitutional atom is contained. The curve shows the energy of interstitials belonging to different shells. The distance, number of sites and neighbouring sites of the other various shells up to the 13th are shown in Table (2-1). This division of interstitial sites into shells was further subdivided in the 3rd and 7th to 10th shells because of different surroundings of the interstitial sites in each particular shell even though the distance of each shell from the S atom is constant.

Koiwa then assumed that the presence of the S atom would influence the energies of the interstitial sites only up to the third shell and beyond this the behaviour of the i atoms would not be influenced by the presence of S atom. The jump frequencies of i atoms from the pth to the qth shell, \( K_{pq} \), are given by

\[
K_{pq} = \nu_{pq} \exp \left( -\frac{E_{pq}}{RT} \right) \quad \ldots \quad (2.1)
\]
Figure (2-1). The arrangement of atoms and interstices around a substitutional atom (the filled circle). The curve shows the energy of interstitial atoms. Numbers at the bottom indicate the shell numbers of respective interstices. After Koiwa (2)

<table>
<thead>
<tr>
<th>Shells</th>
<th>1st</th>
<th>2nd</th>
<th>3rd†</th>
<th>4th</th>
<th>5th</th>
<th>6th</th>
<th>7th†</th>
</tr>
</thead>
<tbody>
<tr>
<td>Distance†</td>
<td>$R$</td>
<td>$\sqrt{2}R$</td>
<td>$\sqrt{5}R$</td>
<td>$\sqrt{6}R$</td>
<td>$3R$</td>
<td>$3R$</td>
<td>$\sqrt{10}R$</td>
</tr>
<tr>
<td>Number of sites</td>
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<td>12</td>
<td>24</td>
<td>24</td>
<td>6</td>
<td>24</td>
<td>24</td>
</tr>
<tr>
<td>Neighbouring sites</td>
<td>2nd $\times$ 4</td>
<td>1st $\times$ 2</td>
<td>2nd $\times$ 1</td>
<td>3rd $\times$ 2</td>
<td>7th $\times$ 4</td>
<td>4th $\times$ 2</td>
<td>3rd $\times$ 1</td>
</tr>
<tr>
<td></td>
<td>3rd $\times$ 2</td>
<td>4th $\times$ 2</td>
<td>6th $\times$ 2</td>
<td>7th $\times$ 1</td>
<td>9th $\times$ 2</td>
<td>5th $\times$ 1</td>
<td>8th $\times$ 1</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>10th $\times$ 1</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Shells</th>
<th>8th†</th>
<th>9th†</th>
<th>10th†</th>
<th>11th</th>
<th>12th</th>
<th>13th</th>
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<tbody>
<tr>
<td>Distance†</td>
<td>$\sqrt{13}R$</td>
<td>$\sqrt{14}R$</td>
<td>$\sqrt{17}R$</td>
<td>$\sqrt{17}R$</td>
<td>$\sqrt{18}R$</td>
<td>$\sqrt{18}R$</td>
</tr>
<tr>
<td>Number of sites</td>
<td>24</td>
<td>48</td>
<td>24</td>
<td>12</td>
<td>24</td>
<td></td>
</tr>
<tr>
<td>Neighbouring sites</td>
<td>7th $\times$ 1</td>
<td>6th $\times$ 1</td>
<td>7th $\times$ 1</td>
<td>9th $\times$ 2</td>
<td>8th $\times$ 2</td>
<td>10th $\times$ 2</td>
</tr>
<tr>
<td></td>
<td>9th $\times$ 2</td>
<td>8th $\times$ 1</td>
<td>13th $\times$ 2</td>
<td>6th $\times$ 2</td>
<td>6th $\times$ 2</td>
<td>6th $\times$ 2</td>
</tr>
<tr>
<td></td>
<td>12th $\times$ 1</td>
<td>11th $\times$ 1</td>
<td>0th $\times$ 1</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>14th $\times$ 1</td>
<td></td>
<td></td>
<td></td>
<td></td>
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</tr>
</tbody>
</table>

† $R$ is a half of the lattice constant of the h.c.c. cell.

Table (2-1). Classification of interstices around a substitutional atom. After Koiwa (2).
for pq = 12, 21, 23, 32, and 30

\[ K_{30} \] refers to jump out of the third shell to shells outside the first three and the other jump frequencies are given by

\[ k = v \exp\left(-\frac{E}{RT}\right) \]

where \( v \) = frequency factor \( E \) = Activation energy

\( R = \) Gas constant \( T = \) Absolute temperature

For simplicity, consider the case in which the external stress is applied in such a direction that the energies of x and y sites are equally affected while the energy of z site is differently affected. The probability of occupation of an interstitial site (p.o.i.) in the x-orientation in the jth shell is denoted by \( n_{jx} \). Similarly \( n_{jz} \) for z sites. For the 3rd and 7th - 10th shells the additional variables are \( m_{jx} \) and \( m_{jz} \) for interstitial sites that were distinguished from others on ground of different surrounding.

Now rate equations for jumps from and to each type of site can be written in terms of the number of type of nearest neighbour sites surrounding each shell. This yields 31 rate equations in terms of \( n_{jx} \) and \( m_{jz} \). In these equations \( K \)'s are jump frequencies i.e. \( K_{2x \times 1} \) is a jump frequency from a 2X site to neighbouring 1z or 1x site. When an
external stress $\sigma = \sigma_0 f(t)$ is applied then $K_{1,2}$ and $K_{1x2}$ are different as because of the change of energy in the X-site due to application of $\sigma$. The interaction energy $E_x$ of the tetragonal dipole in x-orientation is given by

$$E_x = v_0 \sigma (\lambda_1 \alpha_x + (\lambda_2 (\alpha_y + \alpha_z)) \ldots \quad (2.2)$$

where $v_0$ is the atomic volume, $\lambda_1$ and $\lambda_2$ are the principal value of the $\lambda$ tensor, $\alpha_x, \alpha_y, \alpha_z$ are the direction cosine of the stress. Using eq. (2.1) and (2.2)

jump freq. $K_{1x2} = \nu_{12} \exp \left( -\frac{E_x + E_{12}}{RT} \right)$

$$\approx \nu_{12} \left( 1 - \frac{E_x}{RT} \right) \ldots \quad (2.3)$$

$$= \nu_{12} \left( 1 - U_x \right)$$

where $U_x = \frac{E_x}{RT} = \hat{U}_x f(t)$

In the absence of an applied stress the p.o.i. are the same for the x, y, z sites. The p.o.i. for the jth site $n_j$ can be shown from the rate equation as

$$n_j = a_j^p$$
where \( P \) is the p.o.i. on the sites not affected by the S atom \( \{ j \geq 4 \} \) and

\[
a_1 = \frac{K_{21}K_{32}K}{K_{12}K_{23}K_{30}}, \quad a_2 = \frac{K_{32}K}{K_{23}K_{30}}, \quad \ldots (2.4)
\]

\[
a_3 = \frac{K}{K_{30}} \quad \text{and} \quad a_j = 1 \text{ for } j \geq 4.
\]

The factors \( a_j \) are called enrichment factors, expressing increased occupancy of the sites near the S atom. Since the enrichment factors are temperature dependent for conservation of i-atoms within the crystal \( P \) is also temperature dependent and given by

\[
P = \frac{c_0}{3} F(T)
\]

\[
F(T) = \frac{1}{1 + 2c_m(a_1 + 2a_2 + 4a_3 - 7)} \ldots (2.5)
\]

where \( c_0 \) is concentration of i atoms in mole fraction

\( c_m \) is concentration of S atom in atomic fraction

Koiwa showed that if the stress is pure shear the probability of occupation of a particular shell by interstitials is constant and the effect of stress is to only cause redistribution between \( x \) and \( z \) sites in that shell.
The conservation law greatly simplifies the rate equations and reduces the total number of equations to 18 as shown in Table (2-2). The coefficients \( d_k \) are given by

\[
\begin{align*}
    d_1 &= \frac{2}{s} \\
    d_2 &= 4 + d_1 \\
    d_3 &= \frac{\tau_2}{\tau_2} s \\
    d_4 &= \frac{\tau_2}{\tau_3} \\
    d_5 &= 4 + d_3 + d_4 \\
    d_6 &= \frac{2\tau_2}{\tau_4} \\
    d_7 &= \frac{\tau_3}{\tau_5} \\
    d_8 &= 4 + d_6 \\
    d_9 &= \frac{d_5}{2} \\
    d_{10} &= d_7 \\
    d_{11} &= 6d_{10}
\end{align*}
\]

and

\[ s = \frac{\lambda_1^{(1)} - \lambda_2^{(1)}}{\lambda_1^{(0)} - \lambda_2^{(0)}} \]

called the relative anisotropy factor. The quantities \( \tau_k \) and \( \tau_k' \) above are defined as

\[
\begin{align*}
    \frac{1}{\tau_1} &= 6K_{12} \\
    \frac{1}{\tau_2'} &= 6K_{21} \\
    \frac{1}{\tau_3'} &= 6K_{23} \\
    \frac{1}{\tau_4} &= 6K_{32} \\
    \frac{1}{\tau_5'} &= 6K_{30} \\
    \frac{1}{\tau_2} &= \frac{1}{2} \left( \frac{1}{\tau_2'} + \frac{1}{\tau_3'} \right) \frac{1}{\tau_3'} = \frac{1}{4} \left( \frac{1}{\tau_4'} + \frac{3}{\tau_5'} \right)
\end{align*}
\]

The \( \tau \)'s can be expressed as
\[
N_1 = \frac{1}{6r_1} [-4N_1 - d_1N_3 + d_2f(t)],
\]

\[
N_2 = \frac{1}{6r_2} [-4N_2 - d_2N_1 - d_3N_3 + d_4f(t)],
\]

\[
N_3 = \frac{1}{6r_3} [-4N_3 - d_3N_2 + d_4(2N_4 - N_7 - M_7) + d_5f(t)],
\]

\[
M_3 = \frac{1}{6r_3} [-4M_3 + d_5N_2 + d_7(2M_7 - N_4) + d_8f(t)],
\]

\[
N_4 = \frac{1}{6r_4} [-4N_4 - M_3 - N_8 + 6f(t)],
\]

\[
N_5 = \frac{1}{6r_5} [-4N_5 - 2M_7 + 6f(t)],
\]

\[
N_6 = \frac{1}{6r_6} [-4N_6 - N_4 - N_9 + 6f(t)],
\]

\[
N_7 = \frac{1}{6r_7} [-4N_7 - N_3 - M_3 - N_5 + N_6 - N_{10} - M_{10} + 6f(t)],
\]

\[
M_7 = \frac{1}{6r_7} [-4M_7 + M_3 - 2N_5 - M_8 - M_8 + M_{10} + 6f(t)].
\]

\[
N_8 = \frac{1}{6r_8} [-4N_8 - N_7 - M_7 + 2N_9 - 2N_{12} + 6f(t)].
\]

\[
M_8 = \frac{1}{6r_8} [-4M_8 + 4N_7 - 2N_9 - 2M_9 + N_{12} + 6f(t)].
\]

\[
N_9 = \frac{1}{6r_9} [-4N_9 - 2N_4 + N_8 + N_{11} - 2N_6 + 6f(t)].
\]

\[
M_9 = \frac{1}{6r_9} [-4M_9 + N_6 - N_8 - M_8 - 2N_{11} + N_0 + 6f(t)].
\]

\[
N_{10} = \frac{1}{6r_{10}} [-4N_{10} - N_7 - M_7 + 2N_{13} - 2N_0 + 6f(t)],
\]

\[
M_{10} = \frac{1}{6r_{10}} [-4M_{10} + M_7 - 4N_{13} + N_0 + 6f(t)].
\]

\[
N_{11} = \frac{1}{6r_{11}} [-4N_{11} - M_8 - N_0 + 6f(t)],
\]

\[
N_{12} = \frac{1}{6r_{12}} [-4N_{12} - N_8 - N_0 + 6f(t)],
\]

\[
N_{13} = \frac{1}{6r_{13}} [-4N_{13} - M_{10} - N_0 + 6f(t)].
\]

Table (2-2). After Koiwa (2).
\[ \tau = \tau_0 \exp \left( \frac{E}{RT} \right), \quad \tau_1 = \tau_{10} \exp \left( \frac{E_{12}}{RT} \right) \]

\[ \tau_2 = \tau_{20} \exp \left( \frac{E_{21}}{RT} \right), \quad \tau_3 = \tau_{30} \exp \left( \frac{E_{23}}{RT} \right) \ldots \quad (2.8) \]

\[ \tau_4 = \tau_{40} \exp \left( \frac{E_{32}}{RT} \right), \quad \tau_5 = \tau_{50} \exp \left( \frac{E_{30}}{RT} \right) \]

For convenience the variable \( n_{jx}, n_{jz} \) etc. are redefined as normalized probability of occupation \( N \) such that

\[ N_{jx} = \frac{n_{jx} - \bar{n}_j}{n_{jx} - \bar{n}_j} \]

\[ \ldots \quad (2.9) \]

Under a cyclic stress \( \sigma = \sigma_0 \exp (i\omega t) \), the steady state solution has a form

\[ N_j = \tilde{N}_j \exp (i\omega t) \]

where \( \tilde{N}_j \) is a complex number.

\[ \tilde{N}_j = N_{jR} - iN_{jI} \]

The forcing function \( \exp (i\omega t) \)

and replacing \( \frac{d}{dt} \) by \( i\omega \)
\[ [i\omega \tau_1 + 2/3] \tilde{N}_1 + \frac{d_1}{6} \tilde{N}_2 = \frac{d_2}{6} \]

\[ [i\omega \tau_2 + 2/3] \tilde{N}_2 + \frac{d_3}{6} \tilde{N}_1 + \frac{d_4}{6} \tilde{N}_3 = \frac{d_5}{6} \]  \hspace{1cm} (2.10)

\[ [i\omega \tau_3 + 2/3] \tilde{N}_3 + \frac{d_6}{6} \left( 2\tilde{N}_2 - \frac{d_7}{6} \tilde{N}_4 - \tilde{N}_7 - \tilde{M}_7 \right) = \frac{d_8}{6} \]

\[ \cdots \]

\[ \cdots \]

Where \( \tau \)'s are related to \( k \)'s for different type of jump and the coefficient \( d \)'s are functions of various values for the different jump processes as described before.

Solving these simultaneous equations it is possible to find the real and imaginary components of the normalized probability of occupation of interstitials.

Now the internal friction is defined as the ratio of imaginary part of an elastic strain to the total elastic strain of crystal and is given by

\[ \Delta E_{an} = \frac{3}{2} \alpha z^2 \left( \lambda_1^{(0)} - \lambda_2^{(0)} \right) \frac{\nu \sigma_0^0}{RT} \left[ a_1 f_1 S^2 N_{1z} \right. \]

\[ \left. + \sum_{j=2}^{14} a_j f_j N_{jz} \right] \]  \hspace{1cm} (2.11)

where \( a_j \) = Enrichment factor for each shell
\( f_j \) are coefficients consisting of one third of the number of sites in each shell multiplied by concentration \( C_m \).

\[ h_{1(0)} \] = Principle value of tensor in direction of nearest neighbour metal atoms.

\[ h_{2(0)} \] represents the other two principle values of \( h \) tensor.

The internal friction \( Q^{-1} \) is obtained by dividing the imaginary part of \( \Delta \epsilon_{an}^{\nu} \) with the elastic strain \( (\sigma_0/M_0) \),

\[ Q^{-1} = \frac{M_0^\nu}{2R} (h_1^{0} - h_2^{0})^2 a_z^2 C_0 \frac{1}{T} Q(T) \ldots (2.12) \]

where \( Q(T) = F(T) N(T) \)

and \( N(T) = a_1 f_1 s^2 N_{1zI} + \sum_{j=2}^{1} a_j f_j N_{jzI} \)

where \( M_0^\nu \) is the unrelaxed modulus in the direction of applied stress.

Thus a theory has been developed concerning the relaxation of interstitial atoms in b.c.c. iron lattice in the presence of a substitutional foreign atom. The damping spectrum can be calculated if numerical values
of the relative anisotropy factor $S$ and activation energies and frequency factors for jumps of interstitial atoms are given.

So far we have discussed the observations of various interstitial diffusion controlled phenomenon and the theory dealing with interstitial diffusion controlled relaxation processes. In principle, the same phenomenon can be caused by an anisotropic distribution of constituent atoms in substitutional ferromagnetic alloys. The possibility of observing a magnetic viscosity in ferromagnetic alloys due to the preferential orientation of pairs of solute atoms has been foreseen by Neel [5] and Taniguchi [6]. Their qualitative consideration on this subject allow one to explain some results on anomalies of permeability observed by Ferro and Montalenti [42], Kulwein [43] and Fahlenbrach [44]. The time decrease of initial permeability in the temperature region $350^\circ C$ to $550^\circ C$ has been observed by different authors [45,46,47]. Fahlenbrach investigated the disaccommodation of permeability in various iron alloys. He found that the after-effect is prominent in substitutional iron alloys like Fe-Si and Fe-Al alloys. He investigated the time decay of initial
permeability at various temperatures and evaluated the activation energy of the process to be 65 Kcal/mole.

Brissonneau and Moser [45] first used direct measurement of induced anisotropy in single crystal picture frames to measure the stabilization field as a function of magnetization. In 1959, Dietze [3] came out with a theory that holds that the diffusion of vacancies into or out of the wall is the cause of disaccommodation. However, most authors assume the mechanism of pair ordering to be at the base of the observed effects. The theory of the magnetic viscosity due to solute atom pairs is presented by Biorci et al [46] and is very similar to that of the viscosity due to interstitial atoms.

Neel [16] considers a small volume dv of the material, where the magnetization vector has rested in a given direction \((\alpha, \beta, \gamma)\) for a long time. If the magnetization vector in that small volume is suddenly turned to a new direction \((\alpha', \beta', \gamma')\), this rotation requires an energy per unit volume

\[
E_d = W_0 \left( \alpha'^2 \alpha^2 + \beta'^2 \beta^2 + \gamma'^2 \gamma^2 \right)
\]  \hspace{1cm} (2.13)

the direction cosines being referred to the crystallographic axes. From this property of a small volume dv
Neel [16] deduced the existence of a viscosity field in a multidomain structure under proper condition. Taking the anisotropy energy as that given by Neel, the anisotropy energy induced by heat treating the alloy in a magnetic field, under the assumption that the interaction energy between a pair of solute atoms and the magnetization vector $J_s$ is of the type $W \cos^2 \phi$ ($\phi$ being the angle between $J_s$ and the axis of the pair) is given by

$$E_d = W_c \left[ \frac{1}{2} + S_{22} + (S_{44} - S_{22})(\alpha'^2 + \beta'^2 + \gamma'^2) + \frac{1}{4}S_{22}(\alpha \beta' + \beta \gamma' + \gamma \alpha') \right] \quad (2.14)$$

where $\alpha, \beta, \gamma, \alpha', \beta', \gamma'$ have the same meaning as in eq. (2.13) and the $S$'s are constants for each crystallographic structure. Then this energy is referred to the small volume $dv$ ultimately showing that this anisotropy energy can give rise to a magnetic viscosity. According to Neel the drop in permeability is caused by additional stabilization of the demagnetized state causing anisotropy in the atomic configuration which leads to an increase in the local minima of the potential energy in
places where Bloch walls are found. This will tend to inhibit the motion of the Bloch walls thus decreasing the permeability.

Our investigations are aimed at determining the influence of carbon content, plastic deformation and minute traces of non-magnetic inclusions on the magnetic after-effect of Fe and Fe-3.25% Si alloy samples. For this, three series of measurements are made. (i) Specimens with various carbon contents are studied and the same specimens are investigated after being decarburized in a wet hydrogen atmosphere. (ii) The influence of plastic deformation is investigated on specimens containing various carbon contents. (iii) Specimens with minute traces of non-ferromagnetic materials with heterogeneous solution are studied and the influence of precipitation in various stages on the magnetic after-effect of the initial permeability is investigated. The various above studies are conducted by making two parallel measurements. (i) Time decay of initial permeability, and (ii) relaxation loss factor tan δ.

Further investigations are aimed at determining the influence of plastic deformation on the magnetic after-
effect in Fe-3.25% Si alloy in the high temperature region. Magnetic after-effect in Fe-Al alloys with different Al content is studied to determine if the after-effect in the high temperature region is due to the pair ordering of the solute atoms. The primary aim of the theoretical investigation is to calculate the relaxation loss curve using the internal friction model of Koiwa and compare it with the experimentally obtained results.
CHAPTER III

EXPERIMENTAL PROCEDURES

SAMPLE PREPARATION

The Fe-Si samples used mostly in the present investigation was grain oriented silicon steel sheet of thickness 0.30 mm made by Westinghouse Company. Specimens of 25 mm width were prepared with their length parallel to the rolling direction. The surface of the samples was cleaned first with Ethyl alcohol and then with a dilute hydrochloric acid solution. In order to eliminate the effect of work hardening all the samples were stress annealed in pure dry hydrogen atmosphere at 1000°C. After annealing at this temperature for 10 hours the furnace was turned off and the sample allowed to furnace cool. The chemical composition of the specimen was:

Si-3.25%  Cr-0.05%  Ni-0.02%
Mn-0.02%  Cu-0.02%  Ti-0.001%
C-0.008%  other impurities 0.03% and the remainder iron

The Fe-Al alloy samples containing various amounts of Al used in our experiment were also in the sheet form and were treated in a similar way as the Fe-Si alloy. The
Fe-Cu alloy was heat treated in pure dry hydrogen atmosphere at 875°C for 10 hours and then quenched to keep all the copper in solution.

The interstitial content of the samples was controlled by thermal treatment in a suitable atmosphere inside the furnace. Heat treatment was performed in a pure dry hydrogen atmosphere; the hydrogen was passed through a heated palladium cell. Interstitial carbon was removed by holding a sample at 1000°C and passing the hydrogen through distilled water. Conversely diffusion of various amounts of carbon into the specimen was achieved by holding the sample at 1000°C in hydrogen passed through n-heptane, a saturated hydrocarbon.

**SAMPLE DEFORMATION AND STRAIN MEASUREMENT**

The samples of this investigation were deformed by an Instron tensile machine. The samples used for deformation were 12 inches long and had no reduced cross-section. The samples were always strained at room temperature at a strain rate of 0.2% per minute. The values of the net plastic strain was evaluated from the stress-strain chart.
MAGNETIC MEASUREMENTS

3 (a) Measurement of Disaccommodation

The time decay of the initial permeability of deformed and undeformed samples was measured by a toroidal method. Strips of 25 mm width were wound into cores of 35 mm i.d. and 45 mm o.d. Primary and secondary windings with glass insulation were placed on the cores and a magnetic field of 5 \text{m.Oe.} was applied by passing a small signal through the primary coil and the time decay of initial permeability was measured at various temperatures by measuring the secondary induction. Measurements at high temperature were made in a resistance furnace. The measurements at low temperatures were performed by placing the toroid in suitable liquid baths with different melting and boiling points. The specimen temperature was measured with a thermocouple inbedded in each core. Before each reading the specimen was demagnetized by passing an alternating current (60Hz) of gradually diminishing amplitude through the primary winding. An automatic set-up was made with relay circuitry as shown in Fig. (3 a-l) so that immediately after demagnetization is over, the primary is energized with a small signal of the desired frequency and the secondary induction is recorded with time. In the beginning of a measurement
Figure 3 a-1 Automatic set up to measure time decay of initial permeability.
cycle, the primary of the coil is connected to the 60 Hz
demagnetizing unit and the secondary is disconnected
from the scope. Microswitch M is coupled to the
demagnetizing unit in such a way that immediately after
demagnetizing is over the microswitch is activated. Relay
A is energized and it works as a locking device, irrespective
of the position of the microswitch. Relay B is
energized which in turn energizes relay C and D. Relay
C disconnects the demagnetizing unit from the primary of
the coil and relay D energizes relay E and F. Relay
E transfers the primary of the coil from 60 Hz source to
25 Hz source. Relay F connects the secondary side of
the coil to the scope. After one set of reading has been
taken the 12-V source is switched off and then on and the
circuit is again ready for another set of measurement.
3(b) Measurement of Loss Factor \( \tan \delta \)

The loss factor \( \tan \delta \) was determined by using the bridge circuit used originally by Wilde [49] and then later by Bosman [41]. The principle of the measuring circuit can be seen in Figure (3b-1(a)). A double wound coil of 250 turns each is placed on a nonmagnetic former. The core material in the form of a thin sheet of 1" X 9" size is placed inside the former. By using a double winding the inductance is separated from the resistance.

The losses in the core are

(a) Eddy current loss
(b) Hysteresis loss
(c) Relaxation loss

The eddy current loss is small on account of low frequency and small thickness of the sheet. The hysteresis loss is small because the measuring field is very small (0.5m.0e.). The relaxation loss is small because the mean time of stay \( \tau \gg \frac{1}{f} \) where \( f \) is the frequency of the alternating field. Figure 3b-1(b) shows the equivalent circuit of the measuring bridge. \( r_L \) is the ohmic resistance of the coil.

To determine the influence of losses we introduce a complex permeability \( \mu = \mu' - i\mu'' \) corresponding to a
Figure (3 b-1) (a) The bridge circuit used for measuring loss factor tan δ.

(b) Equivalent circuit of the above bridge in Fig. (a).
complex impedance $Z = iwL_s + R_s$

where $L_s$ is the self inductance of the coil and $R_s$ is the equivalent resistance of the after-effect loss.

For balance

$$(r_s + r_L)R = (R_s + iwL_s)(R_c - i\frac{1}{wC}) \quad \ldots \quad (3b-1)$$

Comparing the real and imaginary parts

$$(r_s + r_L)R = R_s R_c + \frac{L_s}{C}$$

and $wL_s R_c - \frac{R_s}{wC} = 0 \quad \ldots \quad (3b-2)$

$\therefore R_c = \frac{1}{wC} \cdot \frac{R_s}{wL_s}$

and $R = \frac{L_s}{(\nu_s + \nu_L)C} \left( 1 + \left( \frac{R_s}{wL_s} \right)^2 \right)$

To see which part of permeability contributes to the after-effect loss, induced voltage $\nu = \frac{d\Phi}{dt}$

$$= A\mu \frac{N}{\ell} \frac{dI}{dt}$$

$$= K\mu \frac{dI}{dt}$$

where $\mu = \frac{AN}{\ell}$.

where $N = \text{Number of turns}$. 
\( A = \text{Cross-sectional area of specimen.} \)

\( l = \text{length of specimen.} \)

\[
\begin{align*}
\text{So } v &= K (\mu' - i\mu'') \frac{d}{dt} (I_0 e^{i\omega t}) \\
&= K (\mu' - i\mu'') i\omega I_0 e^{i\omega t} \\
&= iK\omega\mu' I_0 e^{i\omega t} + K\omega\mu'' I_0 e^{i\omega t} \\
&= (iK\omega\mu' + K\omega\mu'') I_0 e^{i\omega t} \\
&= \frac{v}{i} = K\omega\mu'' + iK\omega\mu' \\
&= R_s + i\omega Ls \\
\end{align*}
\]

From the balance equation

\[
R_c = \frac{1}{wC} \frac{R_s}{wLs} \\
= \frac{1}{wC} \left( \frac{\mu''}{\mu'} \right) \\
\]

Loss factor \( \tan \delta = \frac{\mu''}{\mu'} \) \\
\( = \omega C R_c \)

In our measurement circuit \( C = 10^{-7} \text{ F} \).

For a particular frequency the bridge was balanced at different temperatures by varying \( R \) and \( R_c \) and the
value of loss factor $\tan \delta$ was computed using the relation

$$\tan \delta = \frac{wCR}{C}$$

The loss factor $\tan \delta$ was measured at different temperatures by placing the double wound sample in a constant temperature liquid bath.

**Temperature Control**

A constant temperature liquid bath was constructed out of a large size dewar flask. A heating element was placed in the liquid bath and the temperature was maintained uniform by a stirrer. A thermometer was used to measure the temperature. All components (heating element, thermometer, stirrer) were mounted through a nonconducting cork fitted on the top of the flask. The stirrer was made of glass rod to avoid any heat loss. The stirrer had two propellers to ensure that there was no dead volume of liquid.

**Alternative method**

The results of loss measurement were checked by using an alternative method. For the measurement of effective resistance and self inductance the Owen bridge shown in Figure (3b-2) was used in conjunction with the relay.
Figure (3 b-2). Series Owen bridge used for measuring loss factor, $\tan \delta$. 

SERIES OWEN BRIDGE
circuitry as shown in Fig. (3b-3) to automatically switch on the measurement field at the end of demagnetization. In the beginning of a measurement cycle the microswitch M is open and relay B, C and D are deenergized and the relay E is energized thus disconnecting the measurement coil from the unknown terminal of the bridge. The demagnetizing coil is connected to the 60 Hz demagnetizing unit. At the end of demagnetization the microswitch M is activated automatically. Relay A is energized and it locks automatically irrespective of the position of the microswitch. Relay B is energized and the 25 Hz signal is connected to the generator terminal of the bridge. Relay C is energized and the demagnetizing coil is disconnected from the demagnetizing unit. Relay D is energized which in turn deenergizes relay E and as a result the measuring coil is connected to the unknown terminal of the bridge. After the bridge is balanced the circuit is initialized by switching off and then on the 12V supply and the circuit is ready for the next measurement.

The values of resistance and self inductance of a coil was measured at various temperatures once with the
Figure (3 b-3) Automatic set up to measure $\tan \delta$ with series Owen bridge.
sample inside the coil and then without the sample. The loss angle due to eddy current is given by

\[
\tan \delta = \frac{\Delta R}{wL}
\]

where \( \rho, \mu_0 \) and \( d \) are resistivity, initial permeability and thickness of specimen respectively. As at room temperature the time of relaxation of after-effect is much larger than \( \frac{1}{f} \), the observed loss angle is independent of after-effect. The resistance of the coil was measured at room temperature at different frequencies. The value of \( \frac{W_T}{f} \) was plotted against \( f \), where \( W_T \) is the total loss and the hysteresis and eddy losses at room temperature were separated. At higher temperatures the time of relaxation of after-effect becomes smaller. This makes direct determination difficult. Hence the eddy loss at higher temperatures is estimated by correcting that of room temperature by using the known temperature variations of \( \mu_0 \) and \( \rho \). Thus taking care of the ohmic losses for coil, hysteresis and eddy current the effective value of loss due to after effect and the self inductance are evaluated and the loss factor determined from the relation

\[
\tan \delta = \frac{\Delta R}{wL}
\]

where \( \Delta R = \text{after-effect loss} \)

\( L = \text{self inductance of the coil} \)
3 (c) **Measurement of Initial Susceptibility**

In this investigation initial susceptibility was measured at various temperatures ranging from \(-196^\circ C\) to \(650^\circ C\). Since initial susceptibility is the slope of the linear and reversible part of the virgin magnetization curve of an ideally demagnetized sample, a small magnetic field in the primary of a transformer type device was suddenly reversed and the corresponding change in flux across a search coil was measured with a highly sensitive ballistic galvanometer.

For measurement at room temperature, a suitable yoke was used as part of the magnetic circuit. On a non-magnetic former, 9 cm long, a primary winding of 20 turns was used to produce the field and a secondary winding of 4500 turns was used as the search coil. The specimen of the size 9 cm X 1.5 cm X 0.3 mm was put inside the former and two semi-circular mumetal yokes of 2.25 sq. cm cross-section and very high permeability (\(\mu_r = 20,000\)) were clamped to the two sides so as to make a complete magnetic circuit. A highly sensitive galvanometer was used with a damping resistance of 40 ohms. The initial permeability at temperatures other than room temperature was measured.
by a toroidal method. The specimen strips 25 mm width
were wound into toroids of 35 mm i.d. and 45 mm o.d.
Primary and secondary windings with glass insulation
were placed on cores. For measurement at high temp-
eratures the toroid was placed in a resistance furnace
and for measurement at low temperatures the toroidal
sample was placed in a suitable liquid bath. A small
field was suddenly reversed in the primary winding and
the corresponding flux change across the secondary winding
was measured with a ballistic galvanometer. Before
each reading the specimen was demagnetized by passing
an alternating current (60Hz) of the gradually diminishing
amplitude through the secondary. The specimen temperature
was measured by a thermocouple imbedded in the specimen.
The ballistic galvanometer was calibrated using the
solenoid method.
CHAPTER IV

EXPERIMENTAL RESULTS AND DISCUSSION

Figure (4.1) shows the temperature dependence of the initial susceptibility of undeformed and deformed samples of Fe-3.25% Si alloy with various degrees of deformation [50]. The ratio of the initial susceptibility \( X_1(T) \) at temperature \( T \) to its value at \(-196^\circ C\) has been plotted as a function of temperature \( T \). The \( X_1(T) \) vs \( T \) curve of the undeformed specimen passes through a minimum around room temperature and a second minimum around \( 450^\circ C \). The maximum occurs at \( 350^\circ C \). This behaviour is in contradiction to the theoretical prediction.

The theoretical relation expressed by Kersten [51] is based on the bending of Bloch walls around dislocations under the influence of an external magnetic field and leads to a temperature dependence of \( X_1(T) \) as

\[
X_1(T) = \text{Const.} \frac{M_s}{K_1^{1/2}} \quad \ldots \quad (4.1)
\]

where \( M_s \) = Saturation magnetization at temperature \( T \)

\( K_1 \) = Crystal anisotropy constant

According to Trauble's model [52],

\[
X_1(T) = \frac{M_s}{GAdK_1^{1/4}} \quad \ldots \quad (4.2)
\]

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Figure 4-1: Ratio of the Initial Susceptibility $X_i(T)$ at Various Temperatures to its Value at $-196\,^\circ C$, $X_i(-196\,^\circ C)$ as a Function of Temperature of Undeformed and Deformed Fe-3.25% Si Alloy Specimens.
According to both the models the initial permeability of a ferro-magnetic material is expected to increase due to the fast drop in the crystal anisotropy $k_1$ as compared to drop in saturation magnetization $M_s$ with rising temperature up to slightly below the Curie temperature. The $X_1$ then drops steeply at the Curie temperature. However from the experimental results it is observed that the value of $X_1$ for the undeformed sample is abnormally low in the temperature region between $-50^\circ C$ and $+70^\circ C$ and between $350^\circ C$ and $600^\circ C$. The minima and maxima are gradually flattened with increasing deformation until the specimen with 7% deformation is almost flat. The peak temperature is shifted from $350^\circ C$ to lower temperature as deformation increases. Such type of anomalous behaviour in temperature dependence of initial susceptibility was previously observed by Snoek in carbonyl iron between $-50^\circ C$ and $+5^\circ C$ and by Fahlenbrach, Sommerkorn and others in Fe-Si, Fe-Cr and Fe-Ni alloys. The reason for this anomalous behaviour has been attributed to the diffusion after-effect or the so-called disaccommodation of initial permeability. In order to have further insight into the phenomena the time decay of initial susceptibility has been investigated in both the low and high temperature regions.
(a) **After-Effect in the Low Temperature Region.**

The results of the measurement of the time decay of the initial permeability of undeformed Fe-3.25% Si Alloy specimens is shown in fig. (4 a-1). The ratio of the initial permeability $\mu_i$ to its value at $-50^\circ$C has been plotted against temperature at various times after demagnetization. At a temperature higher than $70^\circ$C, the process is too fast to be observed. The sample was then completely decarburized and after-effect was found to be totally absent as seen in the same figure. This shows that the relaxation process in this low temperature region is mainly due to the interstitial element carbon. As a further evidence the activation energy of the process has been evaluated and the results are shown in fig. (4 a-2). The time required for fractional change in initial permeability at different temperatures has been plotted against $\frac{1}{T}$. For a thermally activated process it gives straight line and the slope of the line corresponds to the activation energy of the process. In this case the activation energy of the process has been found to be about 20 Kcal/mole which is the activation energy for diffusion of carbon in alpha iron [37].
Fe-3.25% Si Alloy

The graph shows the ratio of the initial permeability $\mu_i$ of Fe-3.25% Si Alloy at various temperatures compared to the permeability at 50°C. The temperature range is from -40°C to 0°C.

The lines represent different time intervals: 0, 5, 15, 60, and 300 seconds. The permeability ratio decreases as the temperature increases, indicating a decrease in permeability with rising temperature.

Decarburized samples are indicated as 'DECARBURIZED' on the graph.
Figure (1: a-2): Temperature Dependence of the Time Required for Fractional Change in Initial Permeability of Undeformed Fe-3.25% Si Alloy (Low Temperature Region)
To generalize the validity of this observation, further investigation has been carried out with other iron alloys. The results of the measurement of time decay of initial permeability at different temperatures are presented in fig. (4 a-3) for Fe-5% Al alloy. The ratio of the initial permeability $\mu_i(T)$ at a temperature $T$ to its value at $-50^\circ C$ has been plotted against the temperature $T$ of measurement. In all those observations the permeability decays to very low values. Decarburization of the specimen has lead to total disappearance of the phenomenon. The activation energy of the disaccommodation of initial permeability $\mu_i$ for the different iron alloys with copper and aluminium have been evaluated in a similar way and found to be the same as that of Fe-3.25% Si alloy. Thus it is evident that the anomaly observed in the temperature dependence of initial susceptibility in the low temperature region is caused by the presence of carbon. As has been mentioned in the introduction section, the permeability of the specimen immediately after demagnetization is high and the interstitial carbon atoms are uniformly distributed in all the octahedral positions. Under the influence of a small magnetic field the domain wall moves about its mean position and
Figure (4 a-3): Ratio of the Initial Permeability $\mu_i(T)$ of Fe-5% Al alloy at Various Temperatures to its Value at -50°C as a Function of Temperature $T$. 
interact with the interstitial impurities. The interstitial impurities, in order to minimize the energy, diffuse into the wall and pins it down. Thus the stabilization of the demagnetized state causes anisotropy in the interstitial atomic configuration which leads to an increase in the local minima of the potential energy in places where Bloch walls are found. This will tend to inhibit the motion of the Bloch walls thus causing a drop in permeability.

Fig. (4 a-1) shows the percent decay in initial permeability as a function of carbon content in the sample at a constant temperature (20°C); the after-effect is found to be proportional to the carbon content.

Some interesting results were observed when an experiment was conducted to investigate the influence of nonmagnetic inclusion on the magnetic after-effect in iron. The Fe-2% Cu specimen was homogenized by annealing at 875°C in pure dry hydrogen atmosphere. After 8 hours of annealing at this temperature, the specimen was quenched to room temperature and the results of time decay of initial permeability $\mu_1$ are shown in Fig. (4 a-5). Then the specimen was heat treated at 710°C to
Figure (a-4): Percent Reduction in $\mu_i$ as a Function of Carbon Content in Fe-3.25% Si Alloy Specimen.
Figure 1 (a-5): Ratio of the Initial Permeability $\mu_i(T)$ of Homogenized Fe-2% Cu Alloy at Various Temperatures to its Value at -50°C as a Function of Temperature T, representing the material's behavior after demagnetization.
introduce minute traces of the copper precipitates. The combined results of measurement are presented in Fig. (4 a-6). The ratio of the relaxed values of initial permeability \( \mu_i(T) \) at any temperature \( T \) to its value at \(-50^\circ C\) has been plotted against the temperature \( T \) at various stages of precipitation. It is observed that as long as the additional element remained in solution the disaccommodation of initial permeability is controlled by carbon content only. However, the after-effect is suppressed heavily as soon as minute traces of this element are precipitated. This amount of precipitated copper is sufficiently small and the precipitated particles are of such small size that the coercive field of the specimens remained unaltered. The disaccommodation reappeared after the precipitated copper was dissolved in iron by suitable heat treatment. A similar scavenging effect of Ti in the internal friction measurement of Fe-Ti-N system has been reported by G. Szabo-Miszenti [40]. An increase in the concentration of Ti eliminated all the interstitial atom from solution by forming the compound TiN thus suppressing the relaxation effect. In our case it is quite possible that the interstitial carbon
atoms are arrested by the formation or copper-carbide or that some kind of clustering takes place, causing disappearance of the disaccommodation of permeability. We have observed similar results in our loss factor measurement, where the point has been further discussed.

To investigate the effect of frequency on the relaxation phenomenon, experiments on the undeformed alloy were performed at various frequencies. The results are presented in Fig. (4 a-7 and 4 a-8). In these diagrams the relaxation time $\tau$ has been plotted against $\frac{1}{T}$ for various frequencies. Both in the low and high temperature regions the relaxation time $\tau$ at a fixed temperature is found to be independent of frequency up to 5000 Hz. From the physical point of view such result can be expected since if the frequency of the measured alternating field is so large that the diffusion phenomena cannot follow its time change, it cannot influence the shape of the potential minimum inside which reversible displacements of the wall take place.
Figure (a-7): Relaxation time $\tau$ as a function of $1000/T$ for different frequencies. Temperature region for different frequencies.
Figure (\(\tau\) vs. \(1000/T^\circ K\)): Relaxation Time \(\tau\) as a Function of \(1000/T^\circ K\) (High Temperature Region) for Different Frequencies.
(b) **Effect of Deformation**

In order to investigate the effect of deformation on the relaxation process, well annealed samples of Fe-3.25% Si alloy were deformed by tension with an Instron machine and the time decrease of initial permeability measured at various temperatures. Fig. 4(b-1) shows the plot of $(\mu_t - \mu_\infty)/(\mu_0 - \mu_\infty)$ as a function of time for a deformed and an undeformed sample at a constant temperature (0°C). The constancy of the fractional drop in permeability for both the deformed and the undeformed samples suggests that the mechanism of the process remains unchanged by deformation.

Our result is in contradiction to Tomono's observation. Tomono found no diffusion controlled after-effect in cold worked armco iron and even after annealing at the recrystallization temperature. Tomono's samples were heavily cold worked as a result of which all the carbon atoms might be tied up in the dislocations. Especially if the carbon content is low then his sample might not recover enough to permit measurement with the sensitivity of his instrument. However, our result is in agreement with the results of Schulze. He found after-effect up
Figure (4 b-1): Fractional Change in Initial Permeability of Va Deformed Fe-3.25% Si Alloy Specimens as a Function of Time After Demagnetization. (Carbon Content
to 2.3% deformation but he could not observe after-effect in 90% cold-worked carbonyl iron. Actually this depends to a great extent on the carbon content. After-effect will be observed as long as there is free carbon in solution to stabilize the Bloch walls. The mechanism of the diffusion process is not affected by the deformation perhaps due to the fact that the carbon atoms, on account of their small size, have a mobility in the relatively loose body centred structure of iron that permits them to diffuse easily from one interstitial position to another, even in a deformed specimen. However, the total drop in permeability will depend on how much carbon is available in the solution. Since some of the carbon atoms are trapped in the dislocations (due to their favourable energy position) introduced during the deformation process, the total amount of carbon available to participate in the phenomenon is decreased. This explains our result showing a drop in total decay in permeability with increasing deformation.

To have further insight into the phenomenon, stress annealed samples of Fe-3.25% Si alloy were deformed up to various extent and the total decay of initial permeability
measured at °C. The results are presented in Fig. (1 b-2).
The drop in initial permeability has been plotted as a function of relaxed value of initial permeability at a filed level of \( \frac{1}{2} \) mee. The total decay in initial permeability is found to be directly proportional to the initial permeability. The significance of this result becomes clear by treating the process analytically.

![Diagram of After-Effect Model]

The after-effect phenomenon can be explained by the above model. In the unrelaxed state flux density \( B_i \) is produced by a field level \( H_o \). After the walls are fully stabilized the flux density drops to \( B_\infty \). Now an extra field \( H_s \) called stabilization field is required to increase the flux density to \( B_i \).

So \( B_i \text{ (at } H_o) = B_\infty \text{ (at } H_o + H_s) \) \ldots \ (11 b-1)
or \( B_i \text{ (at } H_o) = \mu_\infty \text{ } (H_o + H_s) \)
or \( B_i \text{ (at } H_o) = \mu_\infty \text{ } H_o + \mu_\infty \text{ } H_s \)
or \( B_i \text{ (at } H_o) = B_\infty \text{ (at } H_o) + \mu_\infty \text{ } H_s \)
or \((\text{Bi} \text{ (at } H_0) - B_\infty \text{ (at } H_0) = H_s \mu_\infty)\)

or \(\mu_i H_0 - \mu_\infty H_0 = H_s \mu_\infty\)

\[\Delta \mu = \mu_i - \mu_\infty = \frac{H_s}{H_0} \mu_\infty \quad \ldots \quad (4 \ b-2)\]

Thus the total decrease in permeability is directly proportional to the initial permeability, and the proportionality constant is \(\frac{H_s}{H_0}\). Ho being held constant throughout this experiment, the slope is proportional to the stabilization field \(H_s\).

Since the fractional decrease of permeability increases with increasing carbon content (Fig. (4a-c)) using eqn. (4b-2) it is seen that the value of \(H_s\) will also increase. Thus, there appears to be a relationship between the carbon content and proportionality constant \(H_s\). To verify this, various groups of specimens, each group containing a particular carbon content, were deformed for each separate group and the total decay in initial permeability were measured. The results of the measurement are shown in Fig. (4 b-3). Each of the straight line represents the results of a group of specimens with a fixed carbon content. The proportionality constant of each line is plotted against the carbon content and shown in Fig. (4 b-4).
Figure (4 b-3): Total change in permeability versus the relaxed value of initial permeability of various deformed Fe-3.25% Si alloy samples. Each group of samples contains 3
It shows that the proportionality constant $H_s$ is directly proportional to the carbon content. It is further noticed that from the above relation

$$\frac{\mu_i - \mu_\infty}{\mu_\infty} = \frac{H_s}{H_0}$$

Since for specimens with a particular carbon content $\frac{\Delta \mu}{\mu_\infty}$ is a constant (Klet's)

$$H_s = K H_0 \quad \ldots \quad (4 \ b-3)$$

So the value of the stabilization field depends on the field of measurement which means that the slope of the line of $H_s$ vs. carbon content will depend on the field of measurement. From the recent experimental investigation by Qureshi and Chaudhary [53] the initial permeability of a deformed sample is related to the strain $\varepsilon$ by the relation

$$\frac{1}{\mu_\infty} = \text{Const.} \times \varepsilon^{1/4} \quad \ldots \quad (4 \ b-4)$$

and from our experimental observation we found

$$\Delta \mu = \text{const.} \times \mu_\infty$$
Using these relations one expects

\[ \frac{1}{\Delta \mu} = \text{const.} \times \varepsilon^{1/4} \quad \text{(\ref{eq:b-5})} \]

where \( \Delta \mu \) is the total decrease in permeability at a constant temperature and \( \varepsilon \) is the percent deformation.

The values of \( 1/\Delta \mu \) are plotted against \( \varepsilon^{1/4} \) as shown in Fig. (\ref{fig:b-5}). The resulting straight line further supports the validity of our experimentally observed relationship.

\[ \Delta \mu = \text{const.} \times \mu_\infty \]
Figure (4 b-5): $\frac{1000}{\Delta \mu}$ Plotted as a Function of $\epsilon^{1/4}$, is Percent Strain of Fe-3.25% Si Alloy.
(c) **Magnetic Loss Measurement**

As has been mentioned before in the introduction section, the elastic after-effect (Internal Friction) and the magnetic after-effect (Loss factor) are both caused by the preferential distribution of the interstitial atoms and the two effects are analogous to each other. In this section an attempt has been made to calculate the loss factor curve using Koiwa's Internal Friction model and then compare the results with the experimentally obtained loss factor curve. From our previous investigations (Fig. (4 a-2) of the time decay of the initial permeability of Fe-3.25% Si alloy specimens containing 20 ppm interstitial carbon, the calculated value of the Activation Energy was about 20 Kcal./mole and preexponential factor \( T_0 \) was of the order to \( 10^{-15} \) sec. These values were used in the computation to calculate the loss factor curve using various values of the relative anisotropy factor \( S \). The loss factor \( \tan \delta \) was measured using a bridge method and the combined results are presented in Fig. (4 c-1). In this figure the relative loss factor \( \tan \delta / (\tan \delta)_{\text{max}} \) has been plotted as a function of temperature. The solid line shows the experimentally
Figure (4 c-1): Ratio of Loss Factor $\tan \delta$ to its Maximum Value Plotted as a Function of Temperature for Fe-3.25% Si Alloy. For Theoretical Analysis:

$E = 20$ kcal, $E_{12} = 21.5$ kcal/mole, $E_{21} = E_{23} = E_{32} = E_{30} =
\tau_0 = 5 \times 10^{-15}$ Sec, ($\phi$) $S = 1.0$ (o) $S = 1.2$ ($\Delta$) $S = 1.5$ ($\Omega$).
obtained values and the dotted lines show the computed values using Koiwa model for different values of S. The ideal Debye peak as shown in the figure has been calculated using the ideal Debye eq.

\[
\frac{\tan \delta}{(\tan \delta)_{\text{max}}} = \frac{\omega \tau}{1 + (\omega \tau)^2}
\]

where \( \tau = \tau_0 \exp \left( \frac{Q}{RT} \right) \), \( \omega = 2\pi f \).

The values used in this computation are

\[ f = 25 \text{ c/s} \quad Q = 20 \text{ kcal/mole} \]

and \( \tau_0 = 5.0 \times 10^{-15} \text{ sec} \).

As has been mentioned before, the presence of the substitutional foreign atom stresses the lattice thus making the phenomenon complicated. For the case of pure Fe-C alloy the loss factor curve can be quite satisfactorily represented by an ideal Debye equation but the presence of the substitutional atom introduces asymmetry and the peak becomes broad. The computed curve becomes closer to the experimental curve as the value of S is increased. Firstly the curve improves due to assumption of a type I
case where the value of $E_{12}$ is different from that of $E$.  
As the value of the anisotropy factor $S$ is increased, it gives rise to a small secondary peak and this broadens the spectrum. For a value of $S = 1$, the loss factor curve is quite improved. However, attempts have been made to further improve the computed curve using different activation energies for the different jump processes. The combined results of computational and experimental findings are presented in Fig. (c-2). As discussed before, the experimental values are quite different from the values given by ideal Debye peak. However, for a type 5 case where the substitutional atom has influence up to the 5th influence sphere, the computed curve closely follows the experimental curve up to about $140^\circ C$. This type of analysis is unique in its nature, however it needs further modification to predict the loss factor more accurately.

The effect of deformation on the loss factor in Fe-Si-C alloy has been investigated. The results of measurement are presented in Fig. (c-3). The deformed samples show the peak value at the same temperature as the undeformed one but the magnitude of the peak value decreases with increasing deformation. This can be
Figure (4 c-2): Ratio of Loss Factor $\tan \delta$ to its Maximum Value Plotted as a Function of Temperature for Fe-3.25% Si Alloy, For theoretical Analysis $S = 1.0$

(x) $E = 20$ kcal/mole, $E_{12} = 21.5$, $E_{21} = E_{23} = E_{30} = E_{31} = E = (0)E = 20$ kcal

---

Equations:
- $E_{12} = 21.5$
- $E_{21} = E_{23} = E_{30} = E_{31} = E = 20$ kcal

---
Figure (4 c-3): Loss Factor $\tan \delta$ as a Function of Temperature for Various Deformed Fe-3.25% Si Alloy Specimens.
explained by the fact that the carbon atoms are tied up by the dislocation and as a result less amount of carbon is available for participation in the phenomenon. As the specimen is more deformed more and more, carbon goes out of solution and thus the relaxation process is more suppressed with increasing deformation.

Now that we have investigated the interstitial diffusion controlled relaxation phenomenon in the various deformed samples by measuring both (1) disaccommodation of initial permeability, and (2) loss factor. We will see if both measured quantities are similarly influenced by deformation. Fig. (4 c-4) shows a plot of total drop in initial permeability as a function of peak values of loss factor tan δ, and it shows a linear relationship thus showing that both the parameters measured are affected identically by the plastic deformation.

In the previous result and discussion part on our disaccommodation measurement we discussed about the possible scavenging effect of minute traces of copper in Fe-cu-C ternary solution. Further work on this point has been performed by measuring the loss factor tan δ of 2% Cu-Fe solution at various stages of precipitation.
Figure (a-c). Total Change in Permeability as a Function of the Peak Value of Loss Factor \( \tan \delta \) for Various Deformed Fe-3.25\% Si Alloy Specimens.
The combined results of measurement are presented in Fig. (4 c-5). The peak temperature is found to be at about 67°C which is the same as that for pure Fe-C system. As long as the copper is in solution in iron the loss factor has been found to be unaffected and determined by carbon content only. After annealing for ten minutes at 710°C, minute traces of the additional element is precipitated and as a result the peak value of loss factor has been found to be heavily suppressed. Similar results were observed during disaccommodation measurement. This is probably because of the formation of iron carbide precipitates or some kind of clustering takes place, thus removing the carbon from the matrix. The after-effect was found to reappear after homogenizing the specimen at optimum temperature.

Further experiments have been performed with the Fe-Cu alloy to see the effect of frequency on the loss factor. The combined results of the measurement are presented in Fig. (4 c-6). In this figure the loss factor $\tan \delta$ has been plotted against temperature for different frequencies. The peak temperature is found to be shifted at higher frequencies. This can be explained by some...
Figure (4 c-5): Loss Factor $\tan \delta$ as a Function of Temperature of Fe-2\% Cu Alloy Specimen at Various Stages of Precipitation
Homogenized Fe-2% Cu Alloy

Figure (4 c-6): Loss Factor tan δ of Homogenized Fe-2% Cu Alloy Specimen as a Function of Temperature at Different
analytical treatment. The relaxation time of this thermally activated process is given by

\[ \tau = \tau_0 \exp \left( \frac{Q}{RT} \right) \]

where \( Q \) is the activation energy of the process.

Loss factor is given by

\[ \frac{\tan \delta}{(\tan \delta)_\text{max}} = \frac{2 \omega \tau}{1 + (\omega \tau)^2} \]  \hspace{1cm} (4 c-1)

let \( \omega \tau = e^x \)

\[ \frac{\tan \delta}{(\tan \delta)_\text{max}} = \frac{2e^x}{1 + e^{2x}} = \text{Sech } x \]

and

\[ e^x = \omega \tau_0 e^{\frac{Q}{RT}} \]

for peak temp \( X = 0 \)

\[ \tau = \frac{Q}{R} \frac{1}{\cosh(\omega \tau_0)} \] \hspace{1cm} (4 c-2)

From this expression it is clear that with the increase in frequency the peak temperature is higher as we have found in our experiment. Using the values of \( Q \) and \( \tau_0 \) for diffusion of carbon in iron, the peak temperatures at various frequencies were calculated and found to be in close agreement with our experimental observations.
(d) **After-Effect in the High Temperature Region**

As we have seen in the result and discussion chapter that according to theoretical models of Kersten and Trauble the initial susceptibility $X_1$ of a ferromagnetic material would be expected to increase with temperature up to slightly below the Curie temperature, due to the faster drop in anisotropy energy $k_1$ as compared to the drop in saturation magnetisation $M_s$ with rising temperature. However, from the experimental results (Fig. 4-1) it is observed that the initial susceptibility drops in the temperature range $-50^\circ$C to $+70^\circ$C and $350^\circ$C to $550^\circ$C. The anomaly in the low temperature region has been investigated and the results discussed in the previous section. In this section we will investigate the anomalous behaviour in the high temperature zone. The anomaly in the high temperature zone is assumed to be due to the diffusion after-effect or the so-called disaccommodation of permeability. Such anomalous behaviour of permeability was first studied by Fahlenbrach [44] and later by Fahlenbrach and Sommerkorn [46]. Boesono [54] et al studied the after-effect phenomenon by internal friction measurement. The time decrease of initial
permeability has been investigated by Balthesen. The combined result of these investigators after measurement of the time decay of various different parameters gives the activation energy of the process to be $65 \pm 5$ Kcal/mole.

The time decay of initial permeability has been measured using the toroidal method and the results presented in Fig. (4 d-1). In this figure the initial susceptibility $X_1(T)$ has been plotted as a function of temperature $T$ at various time after demagnetization. The after-effect starts at $350^\circ C$ and the value of $X_1$ decays to one twentieth its initial value in five minutes after demagnetization. As the temperature is increased the process becomes faster, and at $525^\circ C$ the phenomena is too fast to be observed. The activation energy of the process has been evaluated by plotting the time required for fractional drop in initial permeability at various temperatures against $\frac{1}{T}$. (Fig. 4 d-2). Since this is a thermally activated process, the slope of the straight line gives the activation energy of the process. The activation energy for the undeformed sample has been found to be $65 \pm 5$ Kcal/mole.
Figure (4 d-1): Initial Permeability $\mu_i$ as a Function of Temperature at various Time After Demagnetization for an Undeformed Fe-3.25% Si Alloy Specimen.
Figure (4 d-2): Temperature Dependence of the Time Required for Fractional Change in Initial Permeability of Undeformed Fe-3.25% Si Alloy (High Temperature Region).
To ascertain if interstitial carbon has any influence on the relaxation process at such high temperatures, the sample was decarburized and found that it did not show any after-effect in the low temperature region. Then the time decay of initial permeability was measured in the high temperature region and it was found that the after-effect was not affected by the removal of carbon thus showing that interstitial carbon has no influence on the process in the high temperature region.

To investigate the effect of deformation on the relaxation phenomenon in this high temperature range, well annealed samples of Fe-3.25% Si alloy have been deformed up to various extent and the disaccommodation of initial permeability measured. The results are shown in figure (4 d-3) to (4 d-7). These figures show the time decay of initial permeability $X_i(T)$ at various constant temperatures. In the deformed samples the after-effect appears at a temperature below 350°C and the effect is suppressed with increase in deformation and the resulting dislocation density. In the 7% deformed sample there is only 20% change in permeability. The activation energy of the process in the various deformed samples have been
Figure 4d-3: Initial permeability as a function of temperature at various time after demagnetization for a 1% deformed Fe-3.25% Si alloy specimen.
Figure(4 d-4): Initial Permeability as a Function of Temperature at Various Time After Demagnetization for a 2% Deformed Fe-3.25% Si Alloy.
Figure (4 d-5): Initial permeability as a function of temperature at various time after demagnetization for a 3% deformed Fe-3.25% Si alloy specimen.
Figure (4d-6): Initial permeability as a function of temperature at various time after demagnetization for 5% and 7% deformed Fe-3.25% Si alloy specimens.

Figure (4d-7):
evaluated from these figures by plotting the temperature
dependence of the relaxation time for various amounts of
fractional change in the initial permeability of the
deformed samples as shown in figure (4 d-8) and (4 d-9).
In these figures \( \ln t \) are plotted against \( \frac{1}{T} \) for con-
stant values of \( \frac{\mu_i - \mu_\infty}{\mu_0 - \mu_\infty} \). The slope of this
Arrhenius plot is a measure of the activation energy of
the process. The complete results of the activation
energy is shown in Fig. (4 d-10). The activation energy
is plotted against \( 1/\varepsilon \) where \( \varepsilon \) is the percent deformation.
It is observed that there is a considerable drop in
activation energy with increase in deformation which
suggests that the diffusion mechanism gradually changes
due to the presence of the stress field in deformed samples.
In the absence of any quantitative model these observed
results can be only qualitatively explained. The initial
permeability \( \mu_i \) which represents the average distance
moved by the domain walls under the influence of an
applied magnetic field decreases as a function of time at
a constant temperature probably because directional ordering
is occurring, for the ordering will tend to inhibit the
motion of the domain walls. In a deformed specimen the
Figure (4 d-8): Temperature dependence of the time required for fractional change in initial permeability of 1% deformed Fe-3.25% Si Alloy.
Figure (4 d-9): Temperature Dependence of the Time Required for Fractional Change in Initial Permeability of 2% Deformed Fe-3.25% Si Alloy.
Figure (4 d-10): Activation Energy $Q$ as a Function of $1/\%\varepsilon$. $\varepsilon$ is Deformation of Fe-3.25\% Si Alloy Samples.
ordering process is suppressed due to the interaction between the diffusing atom and various matrix disturbances such as vacancies, dislocations etc. introduced during the deformation process.

In order to gather further experimental evidence on similar effect of deformation on disaccommodation of initial permeability in substitutional alloys, the relaxation phenomenon has been investigated in Fe-Al alloy specimens with various Al contents. The choice of the specimens was suggested by the consideration that in order to observe relaxation effects caused by rotation of solute atom pairs, substitutional disordered alloys are suitable. These conditions are fulfilled by Fe-Si alloys for low silicon content. Below 6% wt Si, the number of Si pairs increase as the square of the Si content. The Fe-Al alloys are somewhat similar to Fe-Si alloys but have less tendency to order. From the fundamental work of Bradley and Jay [55] and from a paper by Taylor and Jones [56] it is known that the solid solution of Al in Fe can be considered random for Al content up to 18 atomic percent.

The temperature dependence of initial permeability
of Fe-Al alloys with 2, 3, 4 and 5% of Al has been investigated between \(-196^\circ C\) and \(550^\circ C\) and the results are presented in Fig. (4 d-11). The experimental result shows anomaly in the temperature region between \(-50^\circ C\) and \(+70^\circ C\) and between \(350^\circ C\) and \(550^\circ C\). It is observed that in the low temperature region the drop in permeability increases with high Al content. This is due to the fact that the presence of Al increases the solubility of carbon in Fe. The disaccommodation of initial permeability in Fe-5% Al has been discussed previously in this chapter.

On the high temperature side the maximum permeability for Fe-2% Al alloy is attained at \(300^\circ C\) and this peak temperature increases slightly with increase in Al content. For the 5% Al-Fe alloy, the peak temperature is \(350^\circ C\).

Similarly the minimum permeability for 2% Al-Fe alloy is at \(425^\circ C\) and it increases slowly with increase in Al content. The dip in the curve in this high temperature range is found to increase with higher aluminium content.

The time decrease of initial permeability for the alloy has been investigated in the temperature range between \(350^\circ C\) and \(525^\circ C\) and the results are presented in Fig. (4d-12) to (4 d-15) for the different Al contents. The
Figure (4 d-11): Ratio of the Initial Permeability $\mu_i(T)$ at Various Temperatures to its Value at $-196^\circ$C as a Function of Temperature $T$ for Various Fe-Al Alloys.
Figure (4 d-12): Ratio of the Initial Permeability $\mu_i(T)$ at Various Temperatures to its Value at $23^\circ C$ as a Function of Temperature $T$ at Various Time After Demagnetization for a Fe-2% Al Alloy.
Figure (4 d-13): Ratio of the initial permeability $\mu_i(T)$ at various temperatures to its value at $23^\circ$C as a function of temperature $T$ at various time after demagnetization for Fe-3.25% Al alloy.
Figure (4 d-1%): Ratio of the initial permeability $\mu_i(T)$ at various temperatures to its value at 23°C as a function of temperature $T$ at various time after demagnetization for Fe-4% Al alloy.
Figure (4 d-15): Ratio of the Initial Permeability $\mu_i(T)$ at Various Temperatures to its Value at 23°C as a Function of Temperature $T$ at Various Time After Demagnetization for a Fe-5% Al Alloy.
activation energy of the system has been determined from the Arrhenius plot as shown in Fig. (4 d-16) to (4 d-19). From the combined results the activation energy has been found to be 50 kcal/mole. Our result is comparable to the value of 56 kcal/mole for Fe-17% Al alloy samples as found by Maringer [57] from magnetic after-effect measurement and by Fischbach [58] from Zener relaxation measurement. Then the Fe-5% Al alloy was deformed to various extent and the temperature dependence of initial susceptibility $X_i$ measured, Fig. (4 d-20) shows the ratio of initial susceptibility $X_i(T)$ at a temperature $T$ to its value at 23°C as a function of temperature $T$. The result is similar to that of Fe-Si alloy. After-effect commences at a lower temperature for the deformed samples. Fig. (4 d-21) shows the time change of initial permeability in the 1% deformed samples. The effect is suppressed by deformation. The activation energy of the system decreases with deformation as shown in Fig. (4 d-22). On the whole the effect of deformation on the relaxation phenomenon in Fe-Al alloy is similar to that of Fe-Si alloy.

According to theoretical prediction [1] it the
Figure (4d-10): Temperature Dependence of the Time Required for Fractional Change in Initial Permeability of Fe-2% Al alloy.
Figure 4: Temperature dependence of the time required for fractional change in initial permeability of Fe-3% Al alloy.
Figure (4 d-18): Temperature dependence of the time required for fractional change in initial permeability of Fe-4% Al alloy.
Fe - 5% Al Alloy

Figure (4d-19): Temperature Dependence of the Time Required for Fractional Change in Initial Permeability of Fe-5% Al Alloy.
Figure (4d-20): Ratio of the Initial Susceptibility $X_i(T)$ at Various Temperatures to its Value at 23°C As a Function of Temperature of Undeformed and Deformed Fe-5% Al alloy Specimens.
Figure (4 d-21): Ratio of the Initial Permeability \( \mu_i(T) \) at Various Temperatures to its Value at 23°C as a Function of Temperature \( T \) at Various Time After Demagnetization for a 1% Deformed Fe-5% Al Alloy.
Fe - 5% Al Alloy
\( \epsilon = 1\% \)

Figure (4 d-22): Temperature Dependence of the Time Required for Fractional Change in Initial Permeability of 1% Deformed Fe-5% Al Alloy.
disaccommodation of initial permeability is due to the pair ordering of substitutional atoms the after-effect is expected to vary as the square of substitutional atomic concentration. To verify this the total change of initial permeability after complete relaxation for alloys with various Al contents has been plotted against the Al percent square as shown in Fig. (4 d-23). As the relation is linear it is evident that disaccommodation in the high temperature region is caused by pair ordering of the substitutional atoms.

While investigating the presence of any relaxation phenomenon in Fe-50% Ni alloy specimens some interesting results were observed. For this investigation the samples were stress-annealed to 1000°C and then quenched to room temperature. The temperature dependence of initial permeability was investigated, and the result is presented in Fig. (4 d-24). In this figure, the ratio of the initial permeability $\mu_i$ at a temperature $T$ to its value at 23°C has been plotted against the temperature $T$. There is distinct after-effect above 200°C. Then the sample was stress annealed and furnace cooled and the initial permeability measured at different temperature.
Figure (4 d-23): Stabilization Field Plotted as a Function of Aluminum Content Square.
Figure 1 (a.2.1): Ratio of the Initial Permeability \( \mu_i(T) \) at various temperatures to its value at 23°C as a function of temperature for Fe-50% Ni Alloy. Specimens quenched from different temperatures.
This time there was no after-effect observed. One possible explanation is that vacancies are generated during quenching the specimen and the time decrease of initial permeability is caused by the vacancy diffusion and once the specimen goes through a heat treatment, all the vacancies are annealed out as vacancy movement begins at above 200°C. To have further insight, an attempt has been made to correlate the experimental results with the available analytical expressions in literature. According to Kittel [59] the number of vacancies formed by quenching is related to the quenching temperature by the relation

\[ n = N e^{-\frac{E}{KT}} \]

\[ \ln \frac{n}{N} = -\frac{E}{KT} \]

where

n = number of vacancy
N = total number of atoms
E = activation energy
K = Boltzmann constant

If the observed phenomenon is caused by vacancy diffusion then one may expect
\[ \ln \Delta \mu = - \frac{E}{kT} \quad \ldots \quad (4 \text{ d}-2) \]

To verify this relationship specimens were quenched from different high temperatures and time decrease of initial permeability measured at different temperatures. The maximum change in permeability was observed at about 300°C. Fig. (4 d-25) shows \( \ln \Delta \mu \) plotted as a function of \( \frac{1000}{T^0 K} \) and it shows a linear relationship thus suggesting that the observed phenomenon is due possibly to the vacancy diffusion.
Figure (4d-25): Total Change in Permeability Plotted as a Function of $1000/T^\circ K$ for Various Fe-50% Ni Alloy Specimens Quenched from Different Temperatures.
CHAPTER V

CONCLUSIONS

The temperature dependence of the initial susceptibility of substitutional Fe-3.25\% Si alloy disagrees with all theoretical models. The anomalous behaviour in the temperature range between -50\°C and +70\°C and between 350\°C and 550\°C cannot be explained with the present theories of initial permeability, and is attributed to the time decay or disaccommodation of permeability. In the present work the influence of carbon content, plastic deformation and minute traces of non magnetic inclusions on the magnetic after-effect of Fe and Fe-3.25\% Si alloy has been investigated by measurement of time decay of permeability and the loss factor \( \tan \delta \) in the low temperature region. The experimental results of loss factor vs temperature have been compared with the computed values from internal friction theory using the theoretical model of Koiva. The effect of deformation on the magnetic after-effect in Fe-Si and Fe-Al alloys has been investigated in the high temperature region. The results show that:

1. The after-effect phenomenon is predominant in the temperature region between -50\°C and +70\°C and between
$350^\circ \text{C}$ and $525^\circ \text{C}$ where the initial permeability exhibits minima when measured as a function of temperature.

2. In the low temperature region the carbon content greatly influences the time dependence of the initial permeability and the total after-effect is found to be proportional to the carbon content in solution.

3. The time decay of initial permeability has been found to be independent of frequency in both the low and high temperature regions over the frequency range 500 to 5000 Hz.

4. The effect of deformation on the initial permeability is that the time decay in the upper temperature region in Fe-3.25% Si specimens commences at temperature lower than $350^\circ \text{C}$ and the after-effect is suppressed with increasing deformation.

5. Experiments with Fe-Al alloys produced results similar to that of Fe-Si alloys. The after-effect stabilisation field was found to be proportional to the square of the Al content thus showing that the after-effect in high temperature region is associated with substitutional atomic orientation (pair ordering).

6. The after-effect in the Fe-Ni alloy specimens is probably due to vacancy diffusion.
7. The effect of deformation on the initial permeability is that the total decay in the low temperature region in Fe-3.25\% Si specimens is proportional to the stabilized value of initial permeability and the constant of proportionality is a measure of the carbon content. This shows agreement with the relations obtained by analytical treatment.

8. As a result of deformation, the total decay of permeability in the low temperature range in Fe-Si specimen is suppressed but the mechanism of diffusion remains unchanged.

9. The loss factor \( \tan \delta \) in Fe-Si samples has been found to decrease with increasing deformation.

10. Both the time decay of initial permeability and the loss factor \( \tan \delta \) in Fe-C alloy have been found to be suppressed by the presence of minute traces of a non-magnetic inclusion (cu).

11. The loss spectrum of the ternary Fe-Si-C alloy specimen becomes asymmetric due to the presence of the substitutional atom. The theoretical model of Koiwa can be used with considerable success to calculate the loss spectrum. However, the theory needs further improvement to calculate the loss spectrum more accurately.
The present investigations are an attempt to study the relaxation phenomenon in different temperature regions under different metallographic conditions of the material—namely in the presence of dislocations, nonmagnetic inclusions and foreign solute atoms. These results should help in better understanding of the relaxation phenomenon.

An immediate extension of the work could be to study the magnetic after-effect by direct observation of the domain wall motions under the influence of an alternating magnetic field of various frequencies. Modern optical techniques and high speed photography of domain walls in motion will give further clues towards better understanding of the relaxation process. The scavenging effect of copper to obtain interstitial free iron may be better understood by further studies using optical and electron microscopy. It is quite possible that interstitial free magnetic materials may be obtained by introducing minute traces of some other element with a minimum deterioration of technically important magnetic properties.
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