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TECHNICAL REPORT NO. 2

KILOCYCLE DAMPING IN PURIFIED IRON
BETWEEN 77°K AND 300°K

By

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BY

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Submitted in partial fulfillment of the requirements
for the degree of Doctor of Philosophy
in the Faculty of Pure Science
Columbia University
1963

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ABSTRACT

↓ Studies of damping in electron beam zone purified iron indicate that there is no low temperature dislocation relaxation peak such as is found in most other materials.

It was found that a modulus defect could be induced by high amplitude oscillations. This is shown to be due to dislocations which have been displaced from equilibrium positions. The activation energy for dislocation motion is estimated to be no higher than .07 e.v. and possibly as low as .01 e.v.

In view of this, it is predicted that the Peierls stress mechanism for the Bordoni peak put forward by Seeger and others, would require the peak in iron to appear below 70°K at about 30 kcps. Consideration of the damping structure in this region shows that the behavior is principally magnetic in character and leads to a possible explanation for the absence of the Bordoni peak.

→ The amplitude dependent damping was studied as a function of temperature, applied magnetic field, and mechanical state. The Granato-Lücke theory of dislocation damping was found not to apply under the conditions met in this study. ↖

Auxiliary studies of damping in nickel show that there is a damping peak in the expected temperature range which appears to be a Bordoni peak. The peak shows annealing characteristics similar to those of copper but at higher temperatures.

I. INTRODUCTION

Since the introduction of dislocation theory, enormous strides have been made towards understanding the nature of the fundamental processes governing the macroscopic mechanical behavior of solids. It has been applied with some success to such phenomena as creep, work hardening, the yield point,¹ and so on. Unfortunately perhaps, the versatility of the theory is such that a phenomenon may be explained satisfactorily by several dislocation models. It is at this point that theorizing must give way to experimentation so that with sufficient facts on hand one may determine the model which most nearly describes the event.

A number of the phenomena of interest depend upon the ability of dislocations to move through the material. Because this is a process that is not clearly understood in the case of iron, a program of study of the motion of dislocations in iron was undertaken.

Because of its particular advantages, internal friction was chosen as the means by which to investigate this problem. It has been amply demonstrated that such methods are capable of providing valuable information concerning the nature and interactions of lattice imperfections. One of the chief virtues of this method is that it is able to focus attention upon what is essentially an atomic level. As such it has contributed materially to

understanding the macroscopic behavior of solids in terms of the underlying atomistic processes.

Internal friction generally refers to energy lost in a solid. Specifically, solids are capable of dissipating energy when they have been set into oscillatory motion even though the material may be so well insulated from its surroundings that losses to its environment are negligible.

Phenomenologically, internal friction arises from the fact that real materials exhibit imperfect elastic behavior. A truly elastic material will not dissipate energy during oscillations since the stress and strain are always in phase. Internal friction may be manifested through static anelastic phenomena such as creep, or stress relaxation, and dynamically through changes in the modulus and energy absorption peaks^{2,3,4}; or by means of a static hysteresis mechanism such as described by Nowick.⁵

A convenient measure of the internal friction, and the one used throughout this paper, is the logarithmic decrement which is defined as follows

$$\Delta = \ln (A_n/A_{n+1}) = dW/2W \quad (1.1)$$

where A_n and A_{n+1} are successive amplitudes of a body oscillating in free decay, dW is the energy dissipated per cycle, and W is the energy stored in the body.

The decrement is further related to the angle ϕ by which the strain lags behind the stress, and the width

of the resonance response curve of an oscillating body by the following ^{2,3,4}

$$\Delta = \pi \phi = \pi (\delta w) / w_r \quad (1.2)$$

where δw is the width of the response curve at one half of the maximum power point, and w_r is the resonant frequency. The above relations are correct when the damping is low and must be modified otherwise.⁶

The primary objective of the present investigation was a search for the Bordoni peak^{2,7,8,9} since this phenomenon is generally considered to be associated only with the motion of dislocation segments through the lattice. The study of amplitude dependent internal friction has also been shown to be useful in studying dislocation motion and this too was observed. Most important in this context is the theoretical study of Granato and Lüke¹⁰ where it was found that dislocation damping should be described by the formula

$$\Delta = \Delta_0 + (B/\epsilon^{1/2}) \exp(-A/\epsilon) \quad (1.3)$$

where A and B are constants and ϵ is the strain amplitude. This expression was derived under the assumption that a dislocation segment is strongly pinned at dislocation nodes, and less strongly by impurities. At sufficiently high strain amplitudes the dislocation line will break away from the impurity pinning points, but not from the nodal pinning points, contributing a significant increase in damping. The constants A and B of equation 1.3 are related to the number of pinning points per unit length of dislocation and

to the network loop lengths respectively. Consequently, whenever the theory is applicable, information may be obtained relating to important dislocation parameters. In general, damping described by equation 1.3 is known as Granato-Lüdtke damping.

The moduli of elasticity also are important means of observing changes in the state of a specimen. A deviation from the true modulus is referred to as a modulus defect and is an extremely sensitive indicator of dislocation behavior, for example, compared to damping, because of the high precision with which it may be determined. As such, this too was observed. In addition, the magnetic field dependence was studied to be sure that this variable was properly controlled.

To accomplish all this, an apparatus was designed and constructed which could be utilized over a range of temperatures from ambient down to liquid helium. In order to bring the effects which were of interest to higher temperatures, it was decided to operate in the kilocycle region.

Since it was intended to vary the temperature over a considerable range, it was desirable to eliminate the use of bonding cements which are troublesome under these conditions. Hence, it was decided to use an electromagnetic drive which, in the case of iron, requires nothing to be affixed to the specimen. An added virtue is the fact that

the electromagnetic drive is capable of achieving higher strain amplitudes than others such as, for example, an electrostatic drive. The apparatus is described in greater detail below.

In order to establish the best conditions for a study of dislocation motion, considerable effort was directed towards ensuring that the dislocations would indeed be free to move. In practice this meant first, using material of the highest purity available, and second, arranging the experiments so that there would be present the greatest opportunity for unimpeded dislocation motion. This was accomplished by treating the specimens to various aging treatments designed to precipitate and stabilize as much interstitial impurity as possible before cold work. On the other hand, deformation was also performed in certain instances at temperatures below which interstitials were capable of migrating to, and pinning, dislocations.

Though internal friction studies have been fairly extensive in fcc metals, studies of dislocation motion in bcc materials, especially iron, have been somewhat of a rarity. Among the papers dealing primarily with dislocation phenomena in iron there are the studies of transients induced by cold work by Köster¹¹, which is probably related to dislocation rearrangement and defect pinning.²

Recently there have been two papers concerned with the amplitude dependence of the damping, one in deformed

pure iron,¹² and the other in deformed iron containing carbon.¹³

There are other phenomena which may be related to dislocations or dislocation-defect interactions. First, there are the trio of small peaks known as the Hasiguti peaks^{14,15,16,17} which appear between -40°C and -110°C at 1050 cps. These peaks are interpreted as being due to vacancy and interstitial interactions with dislocations in a manner that is at present not understood. There is also the "cold work" peak which appears in cold worked iron containing nitrogen.^{2,18} This phenomenon is also not clearly understood, but it is tentatively interpreted as being associated with the motion of impurity atoms and dislocations, or of impurity atoms in the stress field of dislocations. The latter has been observed at about 1 cps.

A study has recently been made on iron whiskers by Conte et al.¹⁹ Whiskers allow studies to be made under very special circumstances, such as very high purity and a nearly perfect structure. The chief significance of this particular paper is the demonstration of a large modulus defect at 20°K , the importance of which is discussed below.

There are two cases where mention is made of a search for a Bordoni type peak in iron. In their review of dislocation damping, Niblett and Wilks⁸ merely mention that no such peak was found without presenting any details. In addition, Bruner²⁰ reported similar results, however, in this case only two specimens were examined under identical

conditions and without close regard for the effects of interstitial pinning. Hence, it was felt that the absence of a peak in iron was not clearly established and that justification existed to pursue this matter further.

Though Bruner found no Bordoni type peak in iron, he did come upon a significant feature at relatively low temperatures. The damping was observed to rise rapidly and then become constant as the temperature was decreased to that of helium. In addition, Heller²¹ while studying the damping behavior of hydrogen and deuterium in iron found additional damping structure below 10°K at 1 cps which he identified with the increase observed by Bruner. This increase in damping at very low temperatures will be referred to as the "Bruner-Heller rise" throughout the paper.

At the time of Bruner's paper, no Bordoni type peaks had been observed in bcc metals. This fact prompted Bruner²⁰ to propose a mechanism which predicted that bcc metals could not produce this phenomenon. Since then Chambers²² has demonstrated the existence of deformation induced relaxation peaks in bcc refractory metals. These peaks share many, though not all, of the characteristics of the fcc peaks. The differences notwithstanding, it was concluded for a time that the peaks observed in these materials probably possessed an origin similar to that of the Bordoni peaks in copper. However, in a very recent paper Chambers²³ has attempted to demonstrate that these

deformation-induced peaks are of a fundamentally different nature in the fcc and bcc metals. In particular, he has concluded that Seeger's Peierls stress mechanism²⁴ is consistent with the results for the bcc metals only. His conclusions are reinforced by the recent work of Bruner and Mecs²⁵, who demonstrated that copper, freshly deformed at low temperatures, exhibits a large modulus defect at 4.2°K.

Thus it was expected that a deformation-induced peak should appear in highly purified iron if conditions for the peak apply equally to all bcc metals.

II. EXPERIMENTAL PROCEDURE

A. Apparatus and Techniques of Measurement

1. Excitation. The specimen in the form of a cylindrical rod is driven in longitudinal oscillation by a telephone type electromagnetic exciting device first used by Wegel and Walther,²⁶ and also by Pittenger²⁷ and Chambers.²⁸

A schematic of the excitation and detection circuits, and the specimen position is shown in Figure 1. (The dotted lines represent alternate signal routes.) The output of a Hewlett-Packard Model 200T Test Oscillator may be directed either to a General Radio 3 Watt amplifier or to a McIntosh 75 Watt amplifier and thence to an electromagnet to excite the specimen. A Hewlett-Packard Model 522B electronic counter measures the frequency at the oscillator output since the signal generated by the specimen vibrations is generally too low to trigger the counter. However, the frequency of the generated signal could be compared to that of the driver when displayed on a Tektronix 545A oscilloscope.

The amplitude of the driver signal is measured across the coil by a Hewlett-Packard Model 400D voltmeter. The signal produced by the oscillating specimen (see below) is amplified by a Dynakit PAM-1 preamplifier and then displayed on a Hewlett-Packard Model 400H voltmeter and/or the oscilloscope mentioned above. Provisions were made to

allow a signal to be sent directly through the specimen for purposes of calibration (see below).

The magnet cores are constructed of 5 mil hipernik laminations which were heat treated after fabrication to minimize hysteresis losses. The magnet arms contain 40 laminations and are approximately $5/16$ " square. The laminations are insulated from each other by means of a silicone varnish (Dow Corning 980) which was properly thinned and baked on. The gap between arms is $1/16$ ", while the gap between the magnet and specimen varied between one to ten mils.

Two windings are positioned on the vertical arms of the magnet, one for the A.C. drive and the other for a D.C. bias. The A.C. windings were constructed so that reasonable impedance matching is achieved between the driving amplifiers and the windings. The D.C. field makes it possible to increase the driving force many times over that produced by an unaided A.C. field²⁹.

The magnet and windings were secured to a copper shield which in turn was secured to a geared drive that may be operated manually from outside the evacuated specimen chamber. This drive permits the magnet to be positioned at the optimum gap which depends upon the temperature and applied magnetic fields.

For non-ferromagnetic specimens, a small magnetic pole piece was affixed to one end of the specimen,

which was carefully machined flat, drilled, and tapped to a depth sufficient to allow a few threads. Then a steel screw was tightly screwed in and machined flat to a thickness of less than 1/16" with the sides flush to the specimen. The results were very satisfactory.

2. Gripping device. The specimens are gripped in the center at a nodal point. The actual grips are a pair of straight brass pins which are mounted in oversized holes in brass sections by means of liberal amounts of soft solder. Such a method was found to decouple adequately the specimens from the support rods. With more rigid couplings, the support rods may be caused to resonate when one of their natural frequencies is close to the natural frequency of the specimen. The resulting coupled oscillations give rise to spurious results.

One of the brass grips mentioned above is threaded and tightens the specimen against the other grip. The second grip is spring loaded and together they afford a fairly secure support. However, the grips cannot be tightened too much before the pins bend, thus preventing more than a minimal amount of local deformation from being produced in the specimen.

The specimen and grips are mounted on a pair of support rods in such a manner that the specimen may be positioned about two mutually perpendicular axes in the horizontal plane.

3. Detection. A simple capacitor type microphone serves as the method of detection. The end of the specimen opposite to the magnet forms one plate of a parallel plate capacitor. (See Fig. 1.) When a D.C. voltage is applied across the capacitor, small changes in the gap (due to specimen oscillations) will produce an A.C. voltage of the same frequency as that of the vibrating specimen, as shown below:

The voltage across a capacitor is related to the capacitance as follows

$$V = Q_0/C_0 \quad (2.1)$$

Now, considering Figure 2 in an open circuit condition, the capacitor C_0 will be charged by V_{DC} (Fig. 1) with an amount of charge Q_0 . If the rod vibrates, causing the static gap width d_0 , to change, C_0 will also be caused to change. No charge is flowing at this point, but a voltage δV is generated

$$\begin{aligned} \delta V &= (-Q_0/C_0^2) \delta C_0 \\ &= (-V_{DC}/C_0) \delta C_0 \end{aligned} \quad (2.2)$$

The capacitance of a parallel plate condenser is given as:

$$C_0 = K'A/d_0 \quad (2.3)$$

where K' is a constant, A is the area of the plate, and d_0 is the distance between them (the static gap width above). Therefore, a variation in the gap will produce a corresponding change in the capacitance

$$\delta C_0 = -C_0 \cdot \delta d_0/d_0 \quad (2.4)$$

Substituting in equation (2.2) we arrive at

$$\delta V = V_{DC} \cdot \delta d_o / d_o \quad (2.5)$$

Thus it is shown that the voltage generated by the vibrating specimen varies directly as the change in gap width, which in turn is directly related to the change in dimension of the specimen.

Again consider Figure 2. Z_1 represents all impedances faced by the generated voltage, including the grid leak resistor of the amplifier. When the circuit is completed, the current is given by

$$I = \delta V / (Z_1 + X_c) \quad (2.6)$$

where X_c is the impedance of the pickup capacitor. V_1 , the voltage to be amplified, is simply

$$V_1 = IZ_1. \quad (2.7)$$

Substituting equation 2.6, we have

$$V_1 = IZ = V_{DC} (\delta d_o / d_o) Z_1 / (X_c + Z_1) \quad (2.8)$$

which may be reduced to

$$V_1 \cong V_{DC} (\delta d_o / d_o) Z_1 / X_c \quad (2.9)$$

since $X_c \gg Z_1$. Furthermore, since the capacitive reactance of the pickup capacitor is given by

$$X_c = 1 / 2\pi f C_o = d_o / 2\pi f K' A \quad (2.10)$$

equation 2.9 becomes

$$V_1 \cong V_{DC} w K' A Z_1 (\delta d_o / d_o) \quad (2.11)$$

The RMS voltage read on the voltmeter will be

$$V_{o1} \cong (B V_{DC} w K' A Z_1 / \sqrt{2}) \delta d_o / d_o^2 \quad (2.12)$$

where B is the effective amplification of the signal.

This expression may be rewritten in terms of the strain amplitude if the following substitutions are made.

$$\epsilon_o = 2 \cdot \delta d_o / L, \text{ and } \gamma = BZ_1 K' A w$$

(L is the length of the specimen.) The strain amplitude is

$$\epsilon_o \approx 2\sqrt{2} d_o^2 V_{o1} / (\gamma V_{DC} L) \quad (2.13)$$

The only unknown in this expression is the factor γ which is a constant of the system for any specific setup and may be readily determined. As indicated above and in Figure 2, the detection circuit is the equivalent of a voltage generator in series with a capacitor. It is therefore possible to substitute a known voltage for the voltage developed by the oscillating specimen. In practice, this is accomplished by switching an oscillator-power amplifier combination in series with the pickup and cutting out the driver coils.

Under these conditions, and following the same procedure as before, this second output voltage is given by

$$V_{o2} = B V_{ac} Z_1 / (Z_1 + X_c). \quad (2.14)$$

V_{ac} is the impressed voltage, and the other terms have the same meaning as before.

Making the appropriate substitutions and rearranging, we arrive at

$$V_{o2} = \gamma V_{ac} / d_o \quad (2.15)$$

If there are any stray capacitive couplings to the pickup, equation 2.15 must be modified to read

$$V_{o2} = [(\gamma/d_o) + \beta] V_{ac} \quad (2.16)$$

β is also a constant.

It remains then to measure V_{O2} versus V_{ac} at several known static gap widths in order to determine the constants δ and β . In order to do this, the second plate of the pickup capacitor was mounted on a micrometer which permits the distance between the plate and the specimen to be measured to within $\pm .0002$ ".

Once the constants of the system have been determined for a given specimen and amplification, the static gap may be determined at any time by again placing a known voltage across the pickup capacitor. This permits the determination of ϵ_0 under almost any conditions. For example, it may be done at temperatures other than ambient when thermal expansions have altered the gap. The output voltage (V_{O1}) may therefore be considered a direct measure of the strain amplitude.

Equations 2.13 and 2.16 were verified by direct measurements of the output voltages as a function of each of the parameters, i.e. d_0 , V_{ac} , and V_{DC} .

4. Measurement of decrement. The width of the resonance response curve is directly related to the decrement.³ Use was made of this fact to determine the decrement when the specimen damping was independent of the strain amplitude.

It was found that damping measurements are independent of the signal-to-noise ratio so long as the ratio was 4:1 or greater. Under conditions that did not provide maximum sensitivity, i.e. with narrow diameter specimens,

or large static gap, it was necessary to operate at higher strain amplitudes in order to meet this criterion.

When the damping depends on strain amplitude, use is made of the fact that in a specimen vibrating at resonance, the amplitude of oscillation is proportional to the driving force and inversely proportional to the damping.³ Or in terms of the damping

$$\Delta = C \cdot F / \epsilon_0 \quad (2.17)$$

where F is the driving force, and C is a constant.

It has already been shown that the strain amplitude is directly proportional to the output voltage (equation 2.13). A simple and rapid means of damping measurement would be available if the driving force were directly proportional to the output voltage of the power amplifier. Accordingly, a sensing coil of ten turns of No. 34 teflon coated copper wire was placed about the magnet gap. The induced voltage was measured as a function of the power amplifier output voltage and it was found that the induced voltage which is proportional to the change in flux, and hence to the driving force, is linearly related to the driving voltage over the entire range of operation. This is true both with and without a D.C. bias.

It is necessary therefore, only to measure V_D (driving voltage), V_{O1} , and the decrement at a low strain amplitude (where the damping is independent of amplitude) in order to determine C . Following that, the strain amplitude dependence of the decrement may be determined simply and rapidly by measuring V_D and V_{O1} .

5. Cryostat. The driving coils, magnet, specimen and grips, and pickup are suspended from a pair of support rods which are affixed to a stainless steel plate. This plate forms the cover for a stainless steel vacuum chamber into which everything is placed. A mechanical forepump is sufficient to maintain a vacuum of about 5 to 10 microns.

This vacuum chamber is inserted into a standard type double dewar cryostat. The outer jacket is an evacuated, double walled stainless steel dewar. The inner member is a double walled pyrex dewar with silvered inner surfaces. A notable feature is the final length of liquid gas transfer tube which is enclosed within the double walls of the glass dewar.

Provisions were made for the introduction of dried helium or nitrogen gas into the vacuum chamber to promote heat exchange either in warming up or cooling down. Either of the gases may be used to pump liquid coolant into the dewar system.

Temperature measurements were effected by a pair of copper-constantan thermocouples. These were affixed to the steel specimen support approximately 1 cm. on either side of the specimen. Tests showed that the specimen's temperature did not differ significantly from that usually measured when the warmup rates were as low as those encountered in these experiments.

Warmup was usually effected by allowing the entire system to warm naturally after the liquid nitrogen boiled off.

The maximum warmup rate was about one degree per minute becoming slower as room temperature was approached under ordinary conditions.

6. External magnetic fields. A coil to provide a magnetic field parallel to the specimen axis was wound directly onto the outer surface of the vacuum chamber. As a consequence it is immersed in the liquid coolant which has the advantage of reducing the power necessary to produce the fields by a factor of ten or more. A field of 125 oersteds could be achieved.

7. Test of apparatus. In order to be assured that the apparatus was capable of measuring a Bordoni peak, the phenomenon was looked for in copper and aluminum. In both cases the peaks were readily observed and found to agree quite well with the literature, thus it was concluded the apparatus was in satisfactory operating condition and could be used for its intended purpose.

B. Specimen Material, Preparation and Treatments

The specimens used in this investigation were primarily purified iron, but other materials, ferromagnetic and otherwise, were used as the occasion demanded.

The ferrous specimens were of various purities, the lowest being shop grade drill rod which is an oil hardening steel containing 0.90 per cent carbon. Next in order of increasing purity comes a vacuum remelted Westinghouse iron; then a series of electron beam zone

melted material of one, two, and four molten passes utilizing Ferrovac E as a starting material. Also used was a sample of the high grade electrolytic iron, vacuum remelted and zone refined by the Battelle Memorial Institute the actual position of which in the scale of purity cannot be precisely determined since analyses of all the materials are lacking.

Analyses where known are presented in Table I. Relative purities in other cases can be inferred from the relative performance of the specimens under certain conditions, as will be indicated below.

Nickel specimens came in two groups of purity. The lowest purity was shop grade material. High purity specimens were kindly provided by the International Nickel Company in the form of nickel reference electrodes of 99.99+ per cent nickel.

The non-magnetic specimens consisted of 99.999 per cent copper from the American Smelting and Refining Company, and brass.

Specimens were in the form of cylindrical polycrystalline rods of about 3 to 4 inches in length, and between .150 and .187 inches in diameter. When possible deformation was performed by means of swaging, or a combination of compression followed by swaging. However, due to the limitations in the number of swaging dies available, it was found that the only feasible mode of deformation which

left a useable specimen was deformation in torsion. This was achieved by positioning the specimen in a lathe after carefully ensuring alignment from the head through the tail stock. The specimen was then twisted through the desired angle. When the specimen has been deformed in this manner the deformation is reported as per cent strain at the surface.

All heat treatments were performed in vacuum. The specimen was sealed with titanium getter in a carefully cleaned quartz tube using a mechanical forepump to evacuate the tube. The vacuum was improved by firing the getter to a red heat for a few minutes in order to remove oxygen and nitrogen. Frequently the getter was permitted to remain during the entire heat treatment. In these cases the specimen and getter were separated by a narrow constriction in the quartz tube.

In certain instances low temperature deformation was performed. The methods are described more fully in the appropriate sections under experimental results.

All iron specimens (except drill rod) used in this investigation received one of two alternative aging treatments designed to promote the production of unpinned dislocations. The two are distinguished from each other by whether a 300°C heat treatment occurs after recrystallization and prior to deformation, or whether a deformed specimen is aged for a short time at 300°C prior to additional deformation. In each case it is hoped that the interstitial

impurities will become trapped in the sub-structure remaining after the heat treatment and remain there during subsequent deformation. This hope is based upon an observation by Petarra,¹⁸ who found that not all the nitrogen which contributes to the cold work peak appears in the Snoek peak immediately after deformation. It is explained on the basis of the interstitials not leaving the substructure until the specimen is heated above the Snoek peak temperature. In addition, Fast and Verrijp¹² demonstrated that when a specimen containing .003 per cent C received the first type of aging treatment, the aging characteristics after deformation were consistent with those of a specimen containing about .0005 per cent C. Thus it appears such aging treatments are indeed capable of providing the advantage we seek.

III. EXPERIMENTAL RESULTS

The results of this investigation fall into four broad categories: (1) the search for a low temperature dislocation relaxation peak at low strain amplitudes which is primarily a study of damping as a function of temperature; (2) time dependent effects following cold work or high amplitude oscillations; (3) amplitude dependent damping; and (4) effects of applied magnetic fields upon the damping at constant strain amplitude. Such a subdivision of results serves to separate purely relaxation effects from hysteresis effects, and both, which are essentially independent of time, from those effects which depend strongly upon time. Lastly of course, are the phenomena which are purely magnetic in origin.

Some effects are unavoidably bound together so that they cannot be neatly categorized. Thus, the time-dependent recovery of the modulus which is induced by high amplitude oscillations is separated from the amplitude-dependent damping studies because the phenomenon was measured at low amplitudes, and is simpler and more readily understood than the complex amplitude dependence. The time-dependent recovery of the amplitude-dependent damping is also included here because it bears directly upon the understanding of the modulus defect.

In many cases the effect of a variable is compared for the purified iron in three states: (1) fully recrystallized; (2) lightly cold-worked (<5 per cent); (3) heavily

cold-worked (>5 per cent). Comparisons are also made to copper and brass.

A. Damping Versus Temperature

This section presents the results concerning the temperature dependence of the damping at low strain amplitudes (10^{-7}) for iron and other materials. Table II summarizes these results.

With respect to iron, the first point of note is the lack of any sort of deformation-induced damping peak which might be likened to the Bordoni peaks in face-centered cubic metals^{7,8,9} or the peaks found by Chambers²² in the body-centered cubic refractory metals. Instead, the typical curve of damping versus temperature for cold-worked purified iron (Fig. 3) decreases gently from liquid nitrogen temperature to room temperature as reported by Bruner. This is unaltered by applied magnetic fields or degree of cold work other than to change the level of damping, except for a Hasiguti peak.^{14,15,16,17} Large impurity concentrations do alter this behavior for in the case of drill rod the damping is quite low and nearly horizontal until near room temperature where it begins to increase. Variation of mechanical treatments and heat treatments to include recrystallization above and below the Curie temperature, aging, or varying the time of aging at room temperature after room temperature deformation, etc., produced no damping peaks either.

In two cases, the iron was deformed below room temperature and the damping measured from very low temperatures to room temperature without any intervening time above -30°C .

In the first case, a specimen was fabricated of Battelle iron and tested by the courtesy of Dr. L. J. Bruner and Mr. B. Mecs of the IBM Watson Laboratories in their equipment.²⁵ The flexural mode used has a fundamental frequency around 500 cps. The specimen was deformed at 105°K in the internal friction apparatus. However, only 1/2 per cent plastic strain was achieved before the specimen fractured at one of the grips. Fortunately, the internal friction portion of the specimen was left intact. The temperature was lowered to 4.2°K before measurements were commenced. The results of this test are shown in Figure 4. While reliable data could not be obtained below 20°K because of the effects of the boiling liquid helium, the Bruner-Heller rise is clearly visible. This rise may be characterized by the temperature at which the curve departs from the horizontal. For this experiment the characteristic temperature was about 28°K .

A careful scrutiny of the resonant frequency data suggests that there may be a relaxation of the modulus in the neighborhood of 45°K , and also at about 160°K . The first is within about 20° of the characteristic temperature defined above, while that of the higher temperature may correspond to a very small Hasiguti peak.

In the second case, specimen 3 was heat treated by the second method described above to produce sub-grain trapping. It was then immersed in liquid nitrogen until the specimen reached that temperature. Liquid nitrogen was continuously poured over this specimen as it was deformed in a prealigned lathe. Plastic deformation was approximately 3 per cent. After deformation the specimen was resubmerged in liquid nitrogen and then quickly inserted into the internal friction apparatus. The chamber was evacuated and inserted into the dewar which contained liquid nitrogen. The entire process took about five minutes. The damping versus temperature again showed the familiar gentle decrease with increasing temperature. Other aspects of the behavior of this specimen will be discussed in following sections.

In Table IV are presented some figures on the value of the damping in iron as a function of mechanical treatment. It is seen that cold work always decreases the damping at liquid nitrogen temperatures.

Investigations were then directed toward the role played by the ferromagnetic nature of the lattice and its possible effect on the 'Bordoni' peak. To this end nickel was chosen since it is face centered cubic, in common with copper, yet also ferromagnetic. A summary of the results for nickel appears in Table III.

The as-received nickel specimens represented a fairly heavily cold worked material which had been aged

for several years at room temperature. In this state a large well developed peak appeared in both specimens, one at -147°C (126°K) and the other a few degrees higher. When one specimen was annealed at 600°C for thirty minutes (presumably recrystallized), (a) no peak was observed, and (b) the damping level was three times the previous peak height. On the other hand, when the second specimen was subjected to an additional 5 per cent torsional deformation, the peak height increased.

The first specimen was then swaged to a 4-5 per cent reduction in area. This reintroduced the peak at a slightly lower temperature and with a reduced peak height. The material was then aged for fifteen minutes at successively higher temperatures and it was found that the peak height first decreased. In addition, after annealing at 250°C a second small, broad peak appears at about -40°C (233°K).

Annealing at higher temperatures causes both peaks to grow. After the 450°C anneal, the major peak is sharply reduced and the minor peak seems to disappear. Annealing at still higher temperatures causes the background to increase a great deal, but leaving a trace of the major peak.

These results are very similar to those of Bruner and Mecs²⁵ in which copper was deformed at liquid helium temperature and aged at successively higher temperatures up to room temperature.

One of the nickel specimens was reduced to a wire and installed in a torsion pendulum in an attempt to determine an activation energy for the peak. The data are somewhat erratic since the apparatus was being pressed to operate at temperatures below that for which it was originally designed. However, it is evident that one peak is present at -75°C (198°K) and there is the appearance that the damping is rising in the neighborhood of -160°C (113°K) presumably to another peak. Since this wire was not aged above room temperature, this peak cannot correspond to the peak at 233°K found at about 32 kcps. This 1 cps peak must therefore correspond to a peak that lies somewhere above room temperature at 32 kcps.

In as much as iron was of primary interest, studies of nickel were not pursued any further.

B. Time Dependent Effects

1. Induced by cold work. Among the phenomena observed were that of the time-dependent change in the modulus and damping after deformation, i.e. the K \ddot{u} ster effect. The results are shown in Figures 5 to 7.

In the first two figures, the change in resonant frequency and damping are compared for two levels of cold work at relatively low strain amplitudes ($<10^{-6}$). The damping is seen to recover at a more rapid rate and to a greater extent in the specimen with the higher degree of cold work. Similarly the changes in resonant frequency

are greater for the specimen with greater cold work. Note that the recovery continues for periods of the order of hours.

In Figure 5 there are some very obvious anomalies in the recovery curve for the resonant frequency of the specimen cold worked 6 per cent. These were induced by high amplitude oscillation and will be discussed in greater detail below.

Another point of interest is the fact that recovery affects high amplitude damping more than low amplitude damping which is particularly noticeable in lightly cold-worked specimens. (See Figs. 7, 9, 10.) It is apparent that more than one process contributes to the amplitude dependent damping.

Observations were made of the Koster effect in Battelle iron after very heavy deformation. The results were very complicated, but it is noted here that the damping went variously up and down with time with a range of 0.8×10^{-3} .

For comparison, the recovery of pure nickel swaged to about a 4-5 per cent reduction of area, was observed. The recovery of the damping was comparable to that of the iron specimen deformed only 1 per cent. The change in resonant frequency however, was much less than either the 1 per cent or 6 per cent deformed iron. Note also the anomalous behavior in the resonant frequency recovery curve which did not appear to recover quite as smoothly as for the iron (Fig. 8).

2. Induced by high amplitude oscillations. The anomalies observed in Figure 5, i.e. the increased modulus defects which appear periodically, were found each time at the termination of a measurement of the amplitude dependence when the specimen was driven once more at a relatively low strain amplitude. In each instance, the disturbance seems to disappear in several minutes time allowing the recovery to proceed more or less smoothly.

The effect is manifested in two ways: the first is a change of frequency which is introduced at a relatively high strain amplitude and disappears instantaneously as the amplitude is decreased to some relatively low value, e.g. 10^{-7} . The second type of frequency change remains even after the low amplitude is reached and disappears only with the passage of time. In as much as this phenomenon was observed primarily as a change in the resonant frequency, the total change in frequency will be referred to as the Δf effect, with Δf_H and Δf_L respectively applied to the two distinct types described above. The changes are related by the expression $\Delta f = \Delta f_H + \Delta f_L$.

(a) Generation of the Δf effect. The Δf effect was found to be fairly sensitive to a number of variables which are described in this section.

The difference between the two types of Δf is vividly demonstrated in the case of 99.999 per cent copper. In this specimen it is possible to induce a total Δf measured in the hundreds of cycles most of which disappears

directly as the strain amplitude is decreased leaving a Δf_L of approximately 30 cycles which then decays over a period of time. This difference is not nearly so marked in the case of iron or brass where Δf_H is only a few cycles at best. The threshold strain amplitudes for these processes are presented in Table V where it is seen that the differences between copper, brass, and iron for both thresholds are quite large. In addition, it is observed that the threshold is a function of temperature in iron (Table Vb).

The magnitude of the effect is a function of the amplitude and time of high amplitude oscillation, the magnetic field, and the temperature. Further, in each case the results are modified by the state of cold work of the iron. Figure 11 shows that, as a function of strain amplitude for a constant time of application, the Δf_L increases as the square of the strain amplitude for the recrystallized material. The lightly cold-worked material however, exhibits an initially linear increase with respect to strain amplitude which changes over a narrow interval to a greater slope and appears to continue linearly.

With respect to time at a constant amplitude of oscillation, Δf_L increases along a curve which is concave downwards (Fig. 12). The exception here is the recrystallized specimen which shows an arrest between 50 and 70 seconds (Fig. 13). The experiment was repeated to verify the presence of the arrested portion and both curves are

shown. The curve for a heavily cold-worked specimen, which is not shown, has the same form as exhibited for the lightly worked specimen in Figure 12.

All of the above experiments were carried out at room temperature with no externally applied magnetic fields. Figure 14 shows the results at room temperature with a field for recrystallized and heavily cold-worked specimens. Though the strain amplitudes are unequal, both show a decrease in Δf_L with respect to an increasing field. In fact, the effect in the recrystallized material is reduced to zero at a high field at the particular amplitude of observation. In all cases, the Δf_L produced after removal of the field is the same as at the beginning of the experiment.

The recrystallized and heavily cold-worked specimens show a markedly different response to lowered temperatures (Fig. 15). In both cases, the Δf_L is depressed by the application of a 125 oersted field. Though there is considerable scatter in the results for the recrystallized material, it is abundantly clear that the effect is increased by at least a factor of three at 77°K in the absence of a field. On the other hand, the cold-worked material exhibits only a slight increase at liquid nitrogen temperatures. Note also that the amount of the depression by magnetic field is nearly independent of temperature.

The specimen that was cold worked below 0°C and immediately taken down to liquid nitrogen temperature gave

Δf_L values exactly the same as those given by the specimen cold-worked and aged at room temperature.

The effect is also easily introduced in drill rod and shows the same qualitative dependences as the purer iron for all variables except temperature and magnetic field, for which no observations were made.

b. Recovery of the Δf effect. Studies of the time-dependent recovery of the Δf effect were made from liquid nitrogen temperatures to room temperature. Both the electron beam zone melted iron and drill rod were used in the absence of applied magnetic fields. In both cases, the recovery of the Δf_L was found to be fitted satisfactorily by the following equation:

$$\Delta f_L = \Delta f_0 \exp \left[-(t/\tau)^n \right] \quad (3.1)$$

The exponent n in equation 3.1 was observed not to be constant for a given experiment over the entire temperature range. Rather, it was found that two distinct values could be assigned in two different temperature ranges. From liquid nitrogen temperatures to about -10°C the data fit the expression above with n equal to 1. At 0°C the recovery curve first shows quite clearly a change over to a value of n equal to $2/3$. Fig. 16 shows typical behavior. Note in Fig. 16B that the initial portion of the curve satisfied equation 3.1 with n equal to 1.

If the event leading to the recovery of Δf_L is an activated process for which τ obeys the expression

$$\tau = \tau_0 \exp (Q/RT), \quad (3.2)$$

the activation energy may be estimated from a plot of $\ln \tau$ versus T^{-1} . Data were gathered for both drill rod and the purest iron in the absence of a magnetic field and for temperatures below 0°C (i.e. where $n = 1$). Unfortunately, the results of such an analysis (Fig. 17) showed considerable scatter. For the purified recrystallized material the apparent activation energy is $.010 \pm .003$ ev. For the drill rod it is $.002 \pm .002$ ev. For the purified, deformed iron the scattered points determine only an upper limit of $.017$ ev and a lower limit of zero.

As indicated before, the recovery curves above 0°C are characterized by a change in the value of n from 1 to $2/3$. It was found that in the absence of a magnetic field, the portion of the recovery curve that obeyed the $n = 1$ law at room temperature increased with the time of high amplitude oscillation. As the time of oscillation increased from thirty seconds to two minutes the $n = 1$ portion increased from about .4 to 1.4 in units of $\ln \Delta f_L$. The strain amplitude was approximately constant for these tests.

The application of an external magnetic field also produced significant effects in the time dependent recovery of Δf_L at room temperature. The addition of a field during the entire period of high amplitude oscillation and recovery greatly reduces, if not entirely eliminates, the portion of the curve obeying $n = 1$. Secondly, the rate of

recovery is increased by the field as evidenced by a 25% increase in $-d(\ln \Delta f) / d(t^{2/3})$.

In a critical experiment a specimen was permitted to oscillate at high amplitudes in the absence of a magnetic field and allowed to recover initially without a magnetic field (Fig.18). When a 125 oersted field was applied for a period of time and then removed, it was observed that there was no noticeable effect upon the recovery of Δf_L at room temperature. The section of the curve where the field was applied is seen to fit smoothly with the remainder. In as much as iron exhibits a " ΔE effect"³⁰ i.e. the modulus, and hence the resonant frequency, increase with magnetization, the Δf_L determined while the field was on represented the difference with respect to the modulus measured after the decay was over at the particular value of magnetization.

On the other hand, if the process is repeated at liquid nitrogen temperature, the application of the field produces a definite change. In Fig. 18B it is seen that a discontinuous, though small decrease in the defect (i.e. an increase in the modulus) coincides with the application of the field. When the field is removed, the curve reverts to the path it would have taken if no field were applied at all.

Changes in damping were also observed to coincide with the recovery of Δf_L . At every temperature the damping was found to have decreased in purified iron when the specimen was returned to low amplitude oscillations.

With time the damping increased. This was determined by the voltage ratios and reinforced by spot checks of the width of the resonance peak.

This contrasts sharply with the behavior of 99.999% copper where the damping appeared to increase after high amplitude oscillation at room temperature and below. At liquid nitrogen temperature, and up to about 140°K, the data on this point are irregular showing both an increase and a decrease.

Figure 19 illustrates the changes in the resonant frequency and the damping as a function of time. As may be seen, the two quantities change in the same way. In order to be ensured that the change in damping represents a real change in the behavior of the specimen and not an instrumental disturbance, measurements were also made of the apparent static gap. Since any of several constants, such as the amplifier gain, might have varied, these measurements actually test the constancy of the whole detection system. The apparent gap width shows an apparently random scatter of 3/4% on either side of its mean value, while the damping changes monotonically by 9% in the same time interval. It does not seem possible for the variation of the damping to be due to changes in the gap.

3. Hysteresis. Niblett and Wilks⁸ have described the results of other investigators where significant hysteresis was developed in certain strain amplitude measurements. Such phenomena were absent in the purified iron,

unless the specimen was held for a considerable length of time at high strain amplitudes. Even then the differences were small.

C. Damping Versus Strain Amplitude at Various Temperatures

The amplitude dependence of the damping is possibly the most complicated of the effects observed in iron. There are at least two important sources of damping, i.e. dislocations and magnetic effects, and it is not a simple task to separate the two at all times. Furthermore, there does not appear to be any damping which might be definitely classified as Granato-Lücke.¹⁰ No generalized description can be given of the shapes of the curves for that depends intimately upon the temperature, externally applied fields and the degree of cold work. A detailed case-by-case description of the various aspects is therefore necessary.

1. Without magnetic field. Figure 20 (A to G) presents a series of amplitude-dependent curves for the electron beam zone refined material (specimen 9) which are typical for the temperature ranges noted. The material is recrystallized and aged. The measurements were carried out in the absence of an applied magnetic field.

Several features are clearly visible in the data. At low temperatures, the damping first falls and then rises slightly as the amplitude is increased. At higher temperatures the damping increases monotonically with amplitude at a rate which is fairly rapid for iron. At room temperature

the increase tapers off at high amplitudes and then begins a new region of increase at very high amplitudes which is visible only near room temperature. The variety of complex shapes observed at low temperatures may be described as a mixture of the fall-off at low amplitudes and the rise at high amplitudes, each of which is affected differently by temperature. The fall-off may still be present at 168°K, but only to such a small extent that the combination results in a nearly level curve. The damping which is linear with amplitude at 188°K may be a distinctly different component from the one which rises so slowly at lower temperature.

The major effect of an 8% deformation at room temperature is to depress the damping at every temperature (measured with zero field). The shapes of the curves are, in general, not altered, nor are the progression of shapes altered by the cold work with the exception of the following details. The difference from the maximum to the minimum at liquid nitrogen temperature is decreased, and the transition to the shape corresponding to Figure 20(D) is lowered 10 to 15 degrees. Furthermore, while the same complex inflected shape is retained at room temperature, the damping at the maximum strain amplitude represents a greater net change in the decrement.

2. With magnetic field. An externally applied magnetic field brings about marked changes in the damping

behavior. Figures 21, 22, 23 illustrate the results at room temperature in a recrystallized specimen (No. 9), a lightly cold-worked specimen (No. 3), and a heavily cold-worked specimen (No. 3) respectively.

The first important effect of the field is the depression of the overall level of damping as the field strength is increased. The second most noticeable effect is the depression of the amplitude dependence. The damping curve changes progressively from the complex inflected shape at zero field to an approximately horizontal plateau at 125 oersteds. At very low amplitudes a fairly rapid rise in damping remains at the maximum applied field.

The most immediate effect of light cold work is to depress the overall level of damping. The shapes of the curves and the effect of a magnetic field are about the same as before. The major difference here is the apparent retention of amplitude dependence to higher fields. Note particularly the differences in the curve for 25 oersteds in Figure 21 and that for $37 \frac{1}{2}$ oersteds in Figure 22.

When the cold work is increased to about 8-10%, additional changes are noted. At zero field the damping curve has nearly lost the inflection point. The damping at lower strain amplitudes does not decrease uniformly as the field is increased, but goes through a maximum. Furthermore, the rise at lowest strain amplitudes observed earlier is almost eliminated.

The behavior of the damping at liquid nitrogen temperature already has been shown to differ greatly from that at room temperature. The application of an external magnetic field similarly produces changes at these lower temperatures, though of a different sort. It is observed that the field reduces the overall damping as before (Figs. 24 and 25). Most important is the reduction in damping in the low amplitude region such that the falloff with increasing strain amplitude found earlier is almost eliminated in the recrystallized specimen. It is entirely removed in the cold-worked specimen when a field of 125 oersteds is reached; in fact, the damping increases slightly before leveling off. Furthermore, there is no evidence of an increase in damping with increasing strain amplitudes (Note: the data for $H = 0$ actually extended to $\epsilon_0 = 7 \times 10^{-5}$ where the curve appears to be leveling off in Fig. 25.)

Measurements, the same as above, were made in specimen 3 which had been deformed below room temperature in torsion to a maximum plastic strain of about 3%. Qualitatively, the results are very similar to those of the recrystallized material. The fact that some cold work had actually been achieved is evidenced by a decrease in damping for the same field strength shown in Figure 24. Unlike the more heavily cold worked material (Fig. 25) the fall-off in damping is not entirely eliminated.

For the sake of comparison, the amplitude dependent damping at liquid nitrogen temperatures was observed for

drill rod and for pure copper. In neither case was there observed a decrease in damping at low strain amplitudes. In fact the drill rod showed considerably more amplitude dependence of positive slope than did the purer iron at the low temperatures.

Almost all of the data on amplitude dependence presented so far represent what might be considered a rather gross view of the damping behavior. Starting at strain amplitudes of about 10^{-7} and working upwards does not permit one to observe some interesting phenomena that lie below 10^{-7} down to perhaps 10^{-9} .

First there is a view of the damping in the 10^{-7} - 10^{-6} region on a somewhat different scale than had been used earlier (Fig. 26). This has the advantage of permitting us to examine somewhat more closely the differences in damping between specimens of different purity but comparable cold work at room temperature.

It appears that in the absence of a field, the higher the purity the higher is the level of the damping. This order is however, modified by the degree of cold work (note that specimen 9 which was deformed about 8% lies below that of specimen 2 which received about 4% cold work). Of more significance perhaps, is the slope of the curves. This appears to be largely unaffected by the cold work, but proceeds in a regular progression from the least pure to the most pure. Drill rod gives a horizontal curve, while specimen 2 (electron beam, 1 molten pass) starts out

almost horizontal and begins an upward turn at about 1.5×10^{-6} . Specimen 9 (electron beam, 4 molten passes) gives an almost linear plot with the greatest slope and with essentially no amplitude independent regions. It is apparent that a magnetic field and impurity content play similar roles in defining these curves. For comparison, the behavior of 99.999% copper is included.

In one series of experiments very high sensitivity was achieved and it was possible to reach down to 10^{-9} strain amplitude. The results of these measurements (Fig. 27) suggest that in the vicinity of liquid nitrogen temperature there is a peak in damping versus strain amplitude, of which only the fall-off is ordinarily observed. As the temperature is increased the peak diminishes and is eliminated by 200°K . At room temperature the rapid rise in damping from below 10^{-8} still remains.

Light deformation lowers the damping at the peak and also decreases the fall-off.

D. Damping Versus Magnetic Field

One other relationship worth considering is that of the damping versus the applied magnetic field at constant strain amplitude. Here too, the degree of cold work is an important factor. In a recrystallized specimen (Fig. 28) the damping generally decreases with the field and passes through an inflection point at about 60 oersteds. It appears to be approaching a constant value at 125 oersteds.

The damping of a 'heavily' cold worked specimen on the other hand, goes through a maximum at about 60 oersted and then decreases with increasing field (Fig. 28). In addition, hysteresis is clearly evident as the curve taken upon decreasing the field lies above the curve for increasing field.

Lightly cold-working purified iron usually leads to unpredictable results. The damping has been observed to go through both a relative minimum and maximum, as well as to behave similarly to the annealed material.

Another interesting relationship may be found in cross-cuts of the amplitude dependent studies mentioned earlier. For example, Figure 25 provides the data to produce Figure 29. Here are plotted the damping versus applied field at two temperatures as a function of relatively high and low amplitudes. Note the curious inversion of shapes: at liquid nitrogen temperatures it is the high strain amplitude data that go through a maximum while at room temperature it is that due to the low amplitude data. There is a similar inversion for the other shape.

IV. DISCUSSION

Perhaps the most significant aspect of the damping in iron is the lack of a deformation-induced low temperature relaxation peak such as may be found in many other materials. Though similar results had been reported for iron previously^{8,20} the material had not been subject to as detailed a scrutiny as in the present survey. The important question of why there is no such peak in iron is thus raised.

When one considers further the results of this investigation, it becomes clear that there are additional questions of major interest that should be answered. These are: (1) Is the Bruner-Heller rise actually a disguised deformation peak? If not, what is it? (2) What is the Δf effect? (3) Of what significance are the data concerning the amplitude dependence, and the Köster effect? The primary purpose of this section is to attempt to answer these questions.

A. Bruner-Heller Rise

A survey of damping studies in iron at very low temperatures indicates that there is very little which might be attributed to dislocation behavior. We have mentioned the damping structure observed by Heller,²¹ and the damping rise observed by Bruner²⁰ which is unusual in that the damping does not decrease to form a bona fide peak

by 4.2°K . The above results are similar to those found in this investigation (Fig. 4), it being impossible in this case to determine whether a peak is actually formed. In the absence of a clear peak, it is necessary to find another characteristic temperature which may be used instead of the peak temperature. The choice made was the shoulder temperature, defined as the temperature where the rise levels off to a plateau as one goes down in temperature. A deviation of about 5×10^{-4} from the plateau was considered significant. The magnitude and characteristic temperature of the rise recorded by the various observers are summarized in Table VI.

In all probability these "peaks" are manifestations of the same phenomenon in spite of the disparity of shapes. Bruner³¹ suggests that the difference in shapes may be due to the substantially different frequencies employed. In addition the data is rather sparse in this region in Heller's plot³⁰ so that the true shape of the peak is uncertain.

The question, as noted above, becomes one of whether or not this damping rise is a manifestation of a Bordoni type peak. We believe it is not for the following reasons.

In all respects, except for the position on the temperature scale, the behavior is incorrect for a deformation induced relaxation peak such as the Bordoni type

peaks. First, the increase in damping is present in recrystallized material²⁰ whereas the peaks found in other materials are very small or non-existent in recrystallized specimens. Secondly, Bruner²⁰ showed that the height of the damping is decreased by room temperature deformation and that in addition there is a considerable downward shift in the shoulder temperature.

Observations in the present investigation also bear upon the matter. The damping was observed to rise at low temperatures, and while the actual plateau was not achieved at kilocycle frequencies, a comparison of the present data with Bruner's leaves no doubt that the present rise is identical with his and that almost all the rise was observed, so that the major part of the damping at 77°K is to be associated with the rise. Accordingly, the substantial amplitude and magnetic field dependences observed at 77°K are also to be associated with the rise. The first point to be made is that deformation always lowered the damping at 77°K. The data further show a sharp decrease in damping with increasing strain amplitude and with an applied magnetic field. Cold work also reduces this damping, though for the degrees of deformation employed here, strain amplitude and magnetic field prove to be more potent. Finally, the suggestion that the damping actually goes through a peak at 77°K and above, as a function of strain amplitude (Fig. 27), is sufficient to discard the idea that this low

temperature damping phenomenon is a relaxation caused by a Peierls stress barrier. If this were the case, the damping would show a rapid increase with strain amplitude, and not a decrease, when a stress had been reached that could propel the dislocations over the Peierls barrier.

All the behavior noted is entirely consistent with damping of a ferromagnetic nature. Heller also concluded that the peak is related to magneto-mechanical damping. When the frequencies of measurement of the various "peaks" mentioned above are plotted against the reciprocal of the shoulder temperature in degrees absolute, it is seen that the points fall on straight lines which extrapolate to a point obtained from the measurements of Kreielsheimer^{32,33,34} (Fig. 30) as explained below.

Note that in Figure 30 the point from Heller's data²¹ is displaced to the right of the line connecting the other points. It is apparent from Bruner's work however, that cold work displaces the data point to the right of the point for recrystallized material. Heller's point represents a specimen which is both cold-worked and charged with hydrogen (which is known to introduce deformation²¹). The percentage displacement is the same in both cases (though the point plotted here represents a high estimate of the peak temperature in Heller's data) as is the direction of displacement. Thus one may conclude that the points are in reasonable agreement with each other and are associated with the same phenomenon.

Kreielshelmer's³² measurements were concerned with the permeability of iron as a function of frequency. He found that there are irreversible and reversible components of the permeability. The contribution of the irreversible component is sharply diminished in the region of 10^6 cycles per second. This may be interpreted as reaching a frequency that is too high to be followed by the irreversible domain wall motion.

The elements necessary for a relaxation peak are thus available: when the applied frequency is low, the domain walls can follow easily and the damping is quite low for a given strain amplitude. When the applied frequency is too high to permit the domain walls to follow irreversibly, then the damping will also be low. Between these two regions lies the point at which the applied frequency will equal the characteristic frequency of the irreversible domain wall motion and thus where the damping will be a maximum. Furthermore, the curve in Figure 30 is indicative of a relaxation phenomenon from which the activation energy for the process is estimated to be 460 ± 230 cal/mole ($.02 \pm .01$ e.v.). It is noteworthy that the damping data alone, considered without Kreielshelmer's point for the moment, imply that the pre-exponential factor in $f = f_0 e^{-Q/RT}$ is several orders of magnitude smaller than is found for dislocation or atomic processes.

In summary, it is concluded that the low temperature damping rise observed by Bruner, Heller and the present investigator is a ferromagnetic phenomenon and may be associated with the irreversible movements of domain walls, with an activation energy of about .02 e.v.

B. Δf Effect

As we turn to consider the Δf effect it must be emphasized that, in as much as we are observing a decrease in the modulus, there must be an increment of strain above the purely elastic strain produced by the movement of a particular entity. In iron the modulus, or resonant frequency, may be decreased (1) by the movement of domains, or (2) by the movement and, possibly, the multiplication of dislocations. In addition there is the third possibility of a temperature rise due to energy dissipation which can lower the resonant frequency. These possibilities shall be considered in turn in an effort to determine what causes the effect.

Considering the last possibility, it is possible to estimate the maximum frequency change caused by a rise in temperature due to the dissipation of heat assuming that all the energy dissipated during a given period of oscillations is converted into heat and uniformly distributed throughout the specimen with no energy lost to the surroundings during this time. Then, taking the logarithmic

decrement to be equal to the energy dissipated per cycle divided by twice the stored energy, the total energy loss may be solved for and hence Δf_L may be found. This equals

$$\Delta f_L = K(T) \cdot \Delta \cdot \epsilon_0^2 \cdot f_r \cdot t \quad (4.1)$$

where $K(T)$ is a temperature-dependent factor incorporating the specific heat, the rate of change of the resonant frequency with temperature, Young's modulus, etc.

Choosing values of the variables such as were usually encountered during the investigation, e.g. $\epsilon_0 = 4 \times 10^{-5}$, $\Delta = 2.5 \times 10^{-3}$ for heavily cold-worked iron (Fig. 10) and $f_r = 32,000$ cps, one calculates a maximum $\Delta f = 1.5$ cps at room temperature. Compare this to that found in Figure 11 for identical conditions, i.e. a Δf_L of 11 cps, almost a factor of 10 greater. At liquid nitrogen temperature, the decrease in the heat capacity is nearly canceled by the decrease in the rate of change of resonant frequency with temperature, and the calculated Δf_L is also much smaller than the measured value.

A number of additional, more qualitative arguments reinforce the conclusion that thermal effects are not responsible. From equation 4.1, it is expected that Δf_L will be proportional to time and to the square of the strain amplitude, at least initially. If account is taken of the fact that the decrement is a function of amplitude also, the actual dependence on strain amplitude will be greater than the second power after the initial region.

The recrystallized and cold-worked specimens both show ϵ_0^2 dependence, but the lightly cold-worked specimen shows a break in the curve and an initial linear dependence on ϵ_0 which is inconsistent with the above suppositions (Fig. 11).

As far as time dependence is concerned, there is insufficient data to determine whether there is an initial linear dependence (Figs. 12, 13). The data of Figure 12 fit a $t^{1/2}$ or $t^{2/3}$ dependence better than t . Furthermore, the arrest in Δf_L versus t exhibited in Figure 13 is clearly inconsistent with the expected behavior of thermal effects.

Thus it may be concluded that under the worst conditions met in this investigation, heat generated by an oscillating specimen will contribute only a small fraction of the observed Δf_L .

In iron the possibility exists that the movements of domains contribute to Δf_L . The modulus is known to be decreased by the movement of domain walls at very low strain amplitudes, a phenomenon known as the ΔE effect.³⁰ There is sufficient evidence to indicate that domain walls are free to move under most of the conditions encountered with the purest iron: the change in decrement with H , the ΔE effect, and the linear increase in damping with strain amplitude at zero field all indicate the presence of freely moving domain walls. Though all these phenomena are observable at the very lowest strain amplitudes (for example, the linear relationship between damping and strain amplitude

is found to commence below 5×10^{-7}), no Δf_L is found until a strain amplitude of about 1×10^{-5} is achieved in the case of heavily cold-worked iron, or about 5×10^{-6} in the case of recrystallized material.

Furthermore, in studying Δf_L as a function of applied magnetic field in the recrystallized material it was found that a field of about 125 oersteds reduced Δf_L to zero at room temperature (Fig. 14). Concurrent measurements on the damping as a function of strain amplitude at the same field strength show that the magnetic damping, while greatly reduced, has not been entirely eliminated (Fig. 26, specimen 9). Therefore, up to the maximum ϵ_0 achieved in this experiment, domain walls were still available for movement, yet no Δf_L was observed.

Last, and perhaps most significant, is the fact that the sudden application of a 125 oersted field during the decay of Δf_L for a relatively short period of time did not in any way alter the decay at room temperature, and produced only a very small shift in the effect at liquid nitrogen temperatures. From this we may draw two conclusions. First, the fact that the structure undergoing a change, which in turn produces a change in the resonant frequency, is essentially independent of an applied magnetic field indicates that domain walls are not the active agents in the Δf effect. Second, the fact that there is a small effect at 77°K when the field is applied suggests that the

recovery mechanism at low temperatures may be somewhat different from that at room temperature.

Thus, because domain walls are found to be moving freely without developing a Δf_L , and because the recovery of Δf_L is largely unaffected by externally applied magnetic fields, one may conclude that the Δf_L produced by high amplitude oscillations is not caused by the movement of domain walls. Therefore, the only reasonable alternative, indeed the only alternative, left to explain the Δf effect is dislocations.

Support for the dislocation hypothesis for the Δf effect is given by the fact that the threshold varies with the material as shown in Table V. In particular, the values for the initiation of the Δf_H and Δf_L described above agree quite well with certain measurements made by means of an etch pit technique in copper of similar purity.³⁵ In this work, single crystals were stressed by applying a pure bending moment. The resolved shear stresses necessary to move grown-in dislocations and cause multiplication were determined to be 4 gr/mm² and 18 gr/mm². The stress necessary to move a fresh dislocation was inferred to be lower than 4 gr/mm². The shear strains corresponding to these stresses are about 1.3×10^{-6} & 6×10^{-6} respectively. It seems rather straight forward to identify the Δf effects with the breakaway and multiplication of dislocations in the case of copper. The values of 3×10^{-6} and 9×10^{-6}

for the threshold tensile strains for the Δf effects are equivalent to maximum shear strains of 1.5×10^{-6} and 4.5×10^{-6} respectively. Since the copper specimen used in the present investigation was lightly cold-worked, the numerical agreement may be fortuitous, especially for the stress necessary to move the dislocation. However, the correlation is still considered to be valid.

The work mentioned above concerning the effect of an externally applied field on Δf clearly implies that there is a coupling of magnetic properties with dislocation motion. Such behavior is supported by recent direct observation in nickel.³⁶ Experiments on nickel foil, using an x-ray diffraction extinction contrast technique, showed a definite change in the dislocation structure when magnetic flux was imposed upon the specimen.

Note that while the transient internal friction due to plastic deformation and the Δf effect may have much in common, the times associated with recovery are vastly different (compare Figs. 5 and 18). The recovery after deformation has been interpreted as the transfer of the dislocations from an irregular array to more stable positions involving climb.² This is illustrated by the differences in the rates of recovery between iron and nickel (Figs. 5 and 8) presumably because in the bcc metals climb is much easier. Thus, because of the differences in magnitude of the changes and the times involved to complete

the change, it appears that recovery of the Δf effect involves much smaller scale adjustment, presumably rearrangement and retrapping over only a few lattice distances.

If one considers the recovery of Δf_L , the first noteworthy point concerning this process is the fact that the decay law expressed by equation 3.1 is satisfied by two values of the exponent in different temperature regions: $n = 2/3$ above 0°C , and $n = 1$ below that point. Dealing as we are with dislocations, the $t^{2/3}$ dependence of the recovery is best explained in terms of the stress-assisted diffusion of point defects to dislocations according to the analysis of Cottrell and Bilby³⁷ and Harper.³⁸ The fact that the $t^{2/3}$ dependence disappears in a temperature range where interstitial diffusion is expected to become unimportant marks the principle recovery mechanism at higher temperatures as one of interstitial pinning of dislocations. This observation reinforces the previous conclusion that there are different agents in the recovery process in different temperature ranges.

It is possible at this point to introduce a model of the Δf effect. At high amplitudes of oscillation, dislocations are freed from certain low energy positions and move freely, thus lowering the modulus. When the amplitude is reduced the motion of these dislocations is reduced, restoring most of the modulus defect. Some of the dislocations may find a barrier impeding their return to

their original positions and thus continue to vibrate freely leaving part of the induced modulus defect. With time the dislocations surmount the barriers and return to their original low energy positions. Above 0°C the freely moving dislocation is also pinned by point defects which can move to the dislocation.

The damping decreases with time after high amplitude oscillations in copper indicating that the dislocation motion giving rise to the Δf was associated with a hysteresis damping mechanism. In iron, the damping rises indicating that the dislocations undergo large motion with small damping in the freed condition, but give rise to greater damping, though smaller strains, in their preferred positions.

The relative ease with which the Δf is introduced in the recrystallized material when compared to the "heavily" cold-worked material is consistent with many observations that a high degree of cold-work depresses the effects due to dislocation motion which were enhanced by small deformation. This may be related to the observations of the dislocation distribution in deformed iron^{39,40} which have shown that the heavily deformed structure consists of cells, the interior of which are relatively free of dislocations. Most of the dislocations are in the boundaries of the cells. Such a configuration restricts dislocation motion.

The second point of interest, if not actually of major interest, concerning the recovery of the Δf_L is the fact that its dependence upon temperature is quite small

(Fig. 17). This means that those dislocations which are free to move do so with very little lattice resistance which in turn is barely altered by a change in temperature. Our interpretation envisions dislocations tied up at secondary pinning points when displaced from equilibrium or otherwise experiencing a barrier to return to their original positions. Therefore, the activation energy determined from the recovery is actually that necessary to free the dislocations from these secondary traps or overcome a barrier.

It must be pointed out that the present investigation distinguishes between two types of resistance stress associated with dislocations. First is a "static resistance stress", which is represented by the threshold values for the Δf effect, necessary to force a dislocation to move. Once the dislocations are moving freely, they are confronted by a "dynamic resistance stress", which is much less than the static stress. This is evidenced by the fact that once a Δf_L has been induced, it may be observed at very low strain amplitudes, i.e. 10^{-7} . The data on the recovery of Δf apply to the dynamic situation. Since the dislocations move easily in the recovery (shown by the fact that the damping is low) the time necessary to surmount the dynamic resistance stress is much less than the time to free the dislocation from the barriers preventing immediate return to the rest position. If we equate the dynamic resistance stress with the Peierls stress, we may inquire as to the limits placed on a possible relaxation process such as is observed by Chambers and

Schultz.²² No sign of a peak has appeared at 77°K, and thus the τ of such a process is much less than the characteristic time of the vibrations: $\tau \ll 1/\omega = 5 \times 10^{-6}$. If we assume $\tau = \tau_0 \exp(Q/RT)$, with $\tau_0 \approx 10^{-11}$ as found by Chambers and Schultz,²² the activation energy Q is no larger than .07 e.v. (1660 cal/mole).

Such a low energy barrier to dislocation motion contradicts the viewpoint that the large change in yield point in iron and other bcc metals is due to a temperature dependent Peierls-Nabarro force, first put forth by Heslop and Petch.⁴¹ The various ideas concerning the temperature dependence of the yield point are summarized in a recent review by Conrad⁴² and therefore need not be considered here.

There are recent papers which generally support the present findings. The study of iron whiskers by Conte et al¹⁹ showed that the modulus of rigidity measured at one cycle per second was considerably lower than the theoretical modulus near 20°K. Irradiation with fast neutrons increased the modulus nearly to the theoretical value. Clearly this phenomenon represents the pinning by the neutron bombardment of dislocations which had hitherto been mobile under the low stresses realized in the torsion pendulum. If we use this to estimate the limit on the activation energy for a relaxation process in iron as before, we find that the value is probably less than .04 e.v. which is lower than before.

Lawley et al⁴³ studying electron beam zone refined molybdenum, found the temperature dependence of the yield

stress was influenced by the purity and concluded that the Peierls stress is much less temperature dependent than previously supposed.

In a paper which bears more directly upon the present work, Brown and Ekvall⁴⁴ studied the temperature dependence of the yield point in iron by means of a relatively high sensitivity extensometer. They demonstrated that the stress required to move dislocations was independent of temperature down to 90°K but depended upon impurity content. They concluded that the large temperature dependence ordinarily observed with macroscopic measurements of the yield point was associated with the rate of work hardening in the region just prior to the macroscopic yield point.

Though the important conclusions of this paper, e.g. that the Peierls stress is low at 77°K, and that there is little evidence for temperature dependence for the Peierls stress, agree in substance with our present findings, we wish to point out that our findings are based upon more stringent limits than theirs: the present plastic strain sensitivity is more than three orders of magnitude greater (7×10^{-10} versus 4×10^{-6}), and it is possible to assign an upper limit to the Peierls stress at 77°K, depending on whether it is identified with the static or dynamic resistance stress. If the static resistance stress is "the Peierls stress," then the upper limit is about 450 psi, compared to Brown and Ekvall's 2700 psi. If the dynamic resistance stress is chosen, then the upper limit is 3 psi.

In addition, it is possible to assign to Brown and Ekvall's static tensile tests an effective frequency so that the two means of testing may be more equably compared. This is accomplished by equating the number of Peierls barriers that must have been crossed per unit time during the determination of the elastic limit, at the limit of sensitivity in plastic strain, to the frequency of oscillation in a dynamic test assuming uniformity of strain. They could detect a plastic strain of 4×10^{-6} which appeared at an elastic strain of about 3×10^{-4} . Since the overall strain rate was 0.1 min^{-1} , the plastic strain rate was 10^{-5} sec^{-1} . Using the relation $\dot{\epsilon} = \rho b v$ for the plastic strain rate, where b is the Burgers vector and v the average velocity of the dislocations, and assuming that the active dislocation density, ρ , was 10^8 cm/cm^3 , the effective frequency is taken as v/b . This is estimated to be about 200 cps for the static test compared to about 30 kcps for the present investigation. It appears that we have been able, in effect, to make the observations at much lower temperatures due to the shift in temperature scale occasioned by the substantial difference in frequencies.

It would have been desirable to make a similar estimate of effective frequency for the data of Heslop and Petch,⁴¹ but because in tensile testing of iron Lüders bands are formed, the simple analysis above is not valid. It is possible that under such conditions the effective frequency

is quite high. In that case, the results of Brown and Ekvall do not apply to the observations on the lower yield stress.

In a number of instances the application of a magnetic field has been observed to affect the production and recovery of Δf . To summarize briefly, a magnetic field depresses the production of Δf at constant strain amplitude by an amount which is nearly independent of temperature. At room temperature, where the recovery is characterized by both $n = 1$ and $n = 2/3$, a magnetic field reduces or eliminates the $n = 1$ portion, and also decreases the time of recovery. Figure 18 shows that an applied field produces a definite effect upon the recovery of Δf_L at liquid nitrogen temperatures but not at room temperatures. These results indicate that dislocations interact with magnetic fields, and perhaps moving domain walls, though in a manner which is at present undetermined.

The dependence of the production of Δf_L upon temperature is not clearly understood. The large Δf_L generated in a recrystallized specimen at 77°K compared to room temperature (Fig. 15) implies that a difference exists possibly in the mechanism of dislocation displacement or movement. In a recent paper, Mura and Brittain⁴⁵ reported that the presence of a yield point at 303°K did not necessarily imply a yield point at 77°K which may be another manifestation of the same phenomenon. As shown in

Table V, the strain at 77°K at which a Δf_L is first detected in a lightly cold worked specimen is about three times that at room temperature. This increase might be expected on the basis of dislocation breakaway from pinning points.

C. Amplitude Dependence of Decrement

Some aspects of the amplitude dependence have already been touched upon, e.g. the decrease in damping with increasing amplitude at liquid nitrogen temperature and its relationship to domain motion. The complexity of the phenomenon is amply illustrated by Figures 20 to 28. The problem of understanding the behavior is made more complex by the presence of the magnetic damping which is either enhanced or diminished depending upon the degree of deformation and the strain amplitude.

Figure 20 indicates that there are at least two, and possibly more mechanisms operating. The damping at the lower amplitudes has been demonstrated to be primarily associated with magnetic phenomena at liquid nitrogen temperature. As the temperature increases from liquid nitrogen temperature, the low amplitude magnetic phenomenon becomes less prominent. At about 170°K the onset of entirely different behavior is observed. We note that it is in this range that the Hasiguti peaks^{14,15,16,17} are observed. When room temperature is reached, the damping is frequently observed to evidence yet another damping mechanism at highest strain amplitudes.

At room temperature and at low strain amplitudes the damping is also primarily associated with ferromagnetic phenomena as evidenced by the effect of applied fields, cold work, and impurities all of which tend to lower the damping (Fig. 26). From the data it is apparent that the damping is a linear function of the strain amplitude. This is undoubtedly an example of the magnetomechanical damping described by Bozorth.³⁰ Though this has been observed in nickel and certain alloys containing iron, as far as we can determine this is the first observation in highly purified iron.

Observations of the Küster effect also help to distinguish between the two types of damping mechanisms. In every case examined, the effects of recovery affect the damping at higher strain amplitudes to a greater extent. This is illustrated best in the recovery of a lightly cold worked specimen (Fig. 9). This recovery may be more closely linked with dislocation rearrangement, and also possibly pinning, rather than with domain movement.

It must be pointed out that results published in two recent papers show significantly different amplitude dependence for iron.^{12,13} Both these papers cite a linear amplitude dependence at room temperature in the presence of a large magnetic field, with a slope of damping versus amplitude about 10 times higher than that found in the present work. The reasons for such disparity are not

readily apparent. Several possibilities are immediately suggested by the obvious differences in frequency, 1 cps. versus 30 kcps, the methods of inducing oscillations, the modes of oscillations (torsional versus longitudinal), and possibly differences in purity.

D. Effect of Magnetic Field at Constant Strain Amplitude

The shapes of the curves of damping versus applied magnetic field (Figs. 29 and 30) are of interest and may be discussed on the basis of recent studies of the megacycle attenuation in iron.⁴⁶ As such the results serve chiefly to demonstrate the mobility of domain walls. To briefly summarize the results of the above investigation, three differently shaped curves were found for a 1010 steel specimen depending upon the magnetic and stress states; (1) annealed-demagnetized: sigmoidally decreasing; (2) cold worked-demagnetized, and annealed-remanent: passes through a maximum; (3) cold-worked-remanent: passes through both a minimum and a maximum.

Two simple assumptions are sufficient to explain the curves. They are that the domain wall area varies, and that the internal stresses induced by cold work immobilize the walls at low fields. Considering those cases which apply to the present case such as (1) above, the walls are able to move easily but the area decreases steadily with increasing field and so the damping decreases. The specimens in Figure 28 were not treated to produce a

demagnetized state, however in a specimen of such purity there is not likely to be a very great difference between an annealed-demagnetized and an annealed-remanent condition.

The cold worked specimen in Figure 28 also resembles the demagnetized specimen above. In this case, the applied magnetic field acts to overcome the internal stresses which inhibit the wall motion initially, and though the wall area may be initially decreasing, the damping increases. As the field strength increases further, the wall area is decreased to such an extent that the damping once again decreases.

Figure 30 indicates that the damping at liquid nitrogen temperatures decreases monotonically with increasing field strength. Not much may be inferred from the apparent peak at higher amplitudes because of the fact that one cannot be sure that a magnetic hysteresis effect is not operating.

E. Damping Peak in Nickel

Evidence is presented which indicates that nickel exhibits a damping peak very similar to the Bordoni peak in copper. In particular, the aging characteristics resemble those for copper²⁵ with the following exception. In copper, the increase during annealing occurs at about 230°K whereas for nickel this occurs between 520°K and 620°K. In both cases these events appear to be explained by the annealing first of interstitials, which lower the peak height by

shortening loop lengths; then the interstitials are annihilated by migrating vacancies which causes the peak to grow.

The temperatures for these occurrences in nickel agree fairly well with those determined by Clarebrough et al⁴⁷ who made measurements of the stored energy released and changes in electrical resistivity accompanying annealing after deformation. Two stages of annealing prior to recrystallization were found to be centered at 393°K and 533°K and could be explained best by the annealing of interstitials and vacancies respectively.

Comparing the results of iron and nickel in this respect, one may infer that ferromagnetism affects the deformation peak in bcc metals but not in fcc. Therefore, the results suggest that the two mechanisms are not the same, thus reinforcing the Chambers-Bruner-Mecs conclusion.

F. Absence of Deformation Peak in Iron

As one speculates upon the reasons for the absence of a deformation induced low temperature relaxation peak, three possibilities come immediately to mind: (1) crystal structure, i.e. bcc as opposed to fcc; (2) purity; and (3) the ferromagnetic nature of iron.

The first possibility is immediately dismissed on the basis of Chambers' observations.²²

The effect of impurities has been to depress or even to eliminate the low temperature deformation

peak.^{7,8,48,49} Most of the models which have been advanced to explain these low temperature peaks are based upon the motion of dislocation segments which are confronted by an energy barrier characteristic of the given metal. Generally speaking, the impurities act to pin dislocations and leave the energy barriers unaffected. Consequently, if the situation is such that the impurity level is sufficiently low, leaving freely moving dislocations, one might expect to observe the peak. However, in the present case no such peaks were observed, yet it is believed that there were segments of freely moving dislocations present which could have contributed to a Bordoni type peak. The reasons for this belief are concerned with the Δf effect.

A specimen deformed well below 0°C exhibited Δf_H 's at liquid nitrogen temperatures which were the same as those found in a specimen deformed at room temperatures. We have demonstrated that the Δf effect is in all probability due to dislocation motion. Thus, if a specimen is deformed at a temperature where interstitial motion is negligible, which provides the greatest opportunity for the production of free lengths of dislocations, and it is found to produce a dislocation effect equal in magnitude to that for the case where no such precautions were taken, one may conclude that the available impurities did not interfere with the particular dislocation motion involved. This is not to imply that the specimen receiving four molten passes in an

electron beam zone refining apparatus is necessarily pure enough to allow the damping effect to take place unimpeded. It is also possible that the aging treatment completed the purification process by precipitating sufficient impurities from the matrix so that the fresh dislocations were presented with a highly purified environment.

The problem of ferromagnetism is not so easily dismissed. On the one hand, the presence of a peak in nickel and its absence in iron indicate a difference in mechanism such that the peak is affected in iron. On the other hand, if one accepts the premise of different mechanisms in the two crystal structures, one cannot argue that the presence of a peak in ferromagnetic nickel implies a peak in ferromagnetic iron. In any case, the possibility exists that ferromagnetism is instrumental in preventing the formation of a Bordoni type peak.

The absence of a peak in iron might be explained by one of at least two guesses related to the nature of dislocation motion and the very low Peierls stress. As it happens, one is dependent upon the ferromagnetic properties of iron and the other not.

Results of various investigations have indicated that there is some coupling between magnetic properties and dislocations, as shown above. We are also struck by the fact that in the region where investigation suggests the Bordoni type peak will most likely be found, there is considerable activity of a magneto-mechanical nature. We

suggest that there may be a coupling between dislocation and domain wall motion so that the dislocation "sees" a substantially reduced Peierls stress. This might be brought about if the dislocation in iron possessed an intrinsic local magnetization such that moving domain walls are able to supply the energy necessary to propel a dislocation over a Peierls hill.

Since the motion of the domain walls and dislocations are coupled, the motion of the dislocation would also be frozen out at the very low temperatures which stop domain motion. Under these conditions, the dislocation peak, if it exists, would be coupled with that of the domain walls.

The second possibility for a Peierls stress which is consistent with our data is that the dislocation core has two atomic configurations (perhaps only at low temperatures): one in which the dislocation may be easily moved, and the other in which the line is immobile. A stress would then be required to transform the dislocation from the static to the dynamic configuration which might be the threshold stress observed for the Δf . The time dependent recovery would be the conversion from one configuration to the other at low stresses. The increase in damping during Δf recovery might reflect the ease of motion of the dynamic configuration, with small associated damping, while the static configuration might move with difficulty. Note that the recovery of Δf , which is observed at very low strain

amplitudes (e.g. 10^{-7}), suggests that the dislocations face a vanishingly small Peierls stress.

The two configurations supposed here are analogous to the two configurations which have come to be accepted for the interstitial in fcc metals: it propagates as a crowdion and rests as a split interstitial.⁵⁰

V. SUMMARY

1. No low temperature dislocation relaxation peaks are found in iron.
2. It is possible to produce a modulus defect by high amplitude oscillations. This is concluded to be principally a dislocation effect.
3. The fact that dislocation motion occurs at extremely small stresses even at 77°K, and that no dislocation relaxation peak is observed suggests that the Peierls energy barrier is very low. It is estimated to be no higher than .07 e.v. and possibly as low as .01 e.v.
4. Evidence of so small an activation energy for motion and hence a very low Peierls stress at low temperatures, is contrary to earlier ideas concerning increased yield points at low temperatures⁴¹. Support is drawn from several recent investigations^{19,43,44}, which conclude that dislocations move freely at low temperatures and that increased yield points at these temperatures are not due to an increased Peierls stress.
5. For an activation energy for dislocation motion of .07 e.v. one expects to find a Bordoni type peak at very low temperatures, i.e. below 70°K at 30 kcps based upon a Seeger type mechanism. In this region are found some interesting damping phenomena. However, because of the fact that this damping is reduced by magnetic fields, cold work, and goes through a peak with strain amplitude, it is concluded to be principally of magnetic origin. By correlation with measurements of permeability,

it is shown to be due most probably to domainwall motion.

6. Observed interactions between domains and dislocations suggest that domains may lower the activation energy for dislocation motion. Furthermore, if there is a dislocation peak it may be coupled to that of the domain walls because of the interaction.
7. An alternative explanation for the absence of a Bordoni type peak is proposed on the basis of a static and dynamic configuration for the dislocation core suggested by the difference in stress necessary to initiate dislocation motion and to allow return to low energy positions.
8. The presence of a damping peak in nickel and not in iron reinforces the conclusion that the mechanism for the Bordoni type peak is different in fcc and bcc metals.

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ACKNOWLEDGEMENTS

The author wishes to express his sincere appreciation to Professor D. N. Beshers for his support, encouragement, and guidance since the inception of the project.

Thanks are also due the American Iron and Steel Institute for donating the zone-refined iron produced at the Battelle Memorial Institute, and to Dr. Harvey Schadler of the General Electric Research Laboratory and Dr. George Murray of The Material Research Corporation for electron-beam zone purification of other specimen material.

The research was conducted under the sponsorship of The Office of Naval Research under contract Nonr 266(61).

TABLE I
Specimen Analysis

	Battelle Fe	Ferrovac E	Ferrovac E 2 passes	99.999% Cu	Ni
Carbon	10	30	20	-	-
Oxygen	17	55	8	-	-
Nitrogen	1	5	nd<0.1	-	-
Sulfur	12	-	-	< 1	-
Aluminum	15	-	nd<5	-	1-10
Boron	5	-	-	-	< 1
Chromium	15	100	nd<10	<.5*	nd
Cobalt	5	70	nd<10	-	1-10
Copper	7	60	1	-	1-10
Manganese	0.1	10	nd<1	-	nd
Nickel	20	150	nd<5	< 1*	-
Silicon	10	-	5	<.1*	1-10
Tin	5	-	nd<50	< 1*	1-10
Titanium	1	-	nd<10	-	< 1
Vanadium	1	40	nd<10	-	< 1
Molybdenum	5	100	nd<10	-	nd
Magnesium	-	-	1	-	1-10

- Note: 1. 'nd' indicates element was not detectable
 2. * signifies spectographic analysis
 3. Analysis is given in parts per million by weight

TABLE II
SUMMARY OF TEMPERATURE DEPENDENT DAMPING

SPECIMEN (FE) TREATMENT	SPEC. 15 DRILL ROD 0.90% C STEEL	SPEC. 1 VACUUM REMELTED	SPEC. 2 ELECTRON BEAM ZONE REFINED, 1 MOLTEN PASS	SPEC. 4 ELECTRON BEAM ZONE REFINED, 2 MOLTEN PASSES	SPEC. 3 ELECTRON BEAM ZONE REFINED, 4 MOLTEN PASSES	SPEC. 9 ELECTRON BEAM ZONE REFINED, 4 MOLTEN PASSES	SPEC. 6, 13(W) BATTELLE FE VACUUM REMELTED & ZONE REFINED
ANNEALED	--	No Peaks; Varied H	No Peaks; Little Rise in Damping to 77°K	No Peaks; Annealed Above & Below Curie Temp. Before C.W.	No Peaks	No Peaks	No Peaks; Damping High
LIGHTLY DEFORMED, < 1%	--	--	No Peaks	No Peaks	--	No Peaks	No Peaks; Spec. 13(W): Deformed 1/2% at 105°K, Rise in Damping Below 28°K
MODERATE DEFORM'N. 1 - 5%	No Peaks; Damping Very Low, Δ In- creases Near Room temp.	No Peaks; Varied H and Aging Time; Δ Greater at 77°K	Hasiguti Peak; ≈ 95°C, 23.1 KC; Re- mains After R.T. Aging; Varied H Does Not Change	Hasiguti Peak; ≈ 95°C, Re- mains After R.T. Aging; Varied H	No Peaks; Varied H; Agd'l. Small Amts. C.W. Produce No Peak	--	Very Small Peak ~-165°C to ~170°C Could Not Be Maximized
HEAVY DEFORM'N. > 5%	--	--			Hasiguti Peak; ~90°C; Varied H & Aging Times	Hasiguti Peak; ~95°C, Aging at 200°C Leaves Peak	No Peaks; Varied H

Cu 99.999% ASR C.W. ≈ 3% Peak -190°C, 19.8 KC	Al Shop Grade Lightly Deformed Peaks: -160°C, -140 at 17.4 KC	70 Cu -30 Ni C.W. Light to Very Heavy Indication of Low Broad Peak at About -170°C, 21 KC	Ni Shop Grade C.W. ≈ 3% No Peak, Damping Very Low	Ni 99.99+ % (INCO) Peak at About -140°C to -145°C. See Table III
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TABLE III

Summary of results concerning deformation induced peak in nickel

Treatment	$\Delta_{\max.} \times 10^3$	T_p °C	Est. Δ_{\log} at peak $\times 10^3$	f_r (cps) at T_p	Secondary Peak T_p °C	$\Delta_{\max.} \times 10^3$
10:As rec'd	4.1	-140	.9	35,000	-	-
600°C, 30 min	Damping very high, no peak apparent					
11:as rec'd	3.8	-147	.8	34,950	-	-
Add 5% c.w.	5.1	-142	1.5	34,852	-	-
10:5% R.A. after rex'l	1.65	-144	1.70	31,096	-	-
Age 15 min. 150°C	1.35	-145	0.90	31,960	-	-
250°C	1.20	-145	0.90	31,963	-40	.2
350°C	2.40	-148	1.60	31,909	-30	.3
450°C	0.3	-150	3.60	31,990	not visible	
600°C	0.4 ?	-155 ?	15	31,916	-	-

TABLE IV

Effect of cold work on low amplitude
damping at liquid nitrogen temperatures

Specimen	Treatment	Field	$\Delta \times 10^3$
1	Annealed	40 oe	1.90
1	5% R.A.	42	.60
3	Annealed	30	2.10
3	8% R.A.	30	1.90
3	8% R.A.	0	1.70
6	Annealed	0	3.25
6	5% Tors.	0	2.80
9	Annealed	0	3.00
9	8% R.A.	0	2.50
9	10% Tens.	0	1.70

TABLE V

(a) Threshold strain amplitudes for Δf at room temperatures

Material	First noticeable Δf_H at high ϵ_0	Onset of time dependent Δf_L
99.999% Cu	3×10^{-6}	9×10^{-6}
Fe (No. 9) ¹	$1 - 2 \times 10^{-6}$	2×10^{-5}
Brass	$6 - 9 \times 10^{-5}$	1.5×10^{-4}

(b) Threshold for Δf_L compared at liquid nitrogen and room temperature

	Room temperature	Liquid Nitrogen temperature
Fe (No. 3) ²	5×10^{-6}	1.5×10^{-5}

¹Spec. No. 9 lightly cold-worked²Spec. No. 3 heavily cold-worked

TABLE VI

Low temperature damping 'peaks' in iron

State	$\delta\Delta \times 10^3$	T ($^{\circ}$ K)	Frequency	Source
Annealed	1.98	60	26.41 kcps	Ref. 20
Deformed	1.51	40	26.35 kcps	Ref. 20
Light c.w.(?)	2.60	28	520.5 cps	Fig. 4
C.W. & H ₂ charged	0.85	7-10	1.67-1.11 (cps)	Ref. 21
C.W. & D ₂ charged	1.6	7-10	1.67-1.11 (cps)	Ref. 21
Deformed	.5	88	89.38 kcps	-

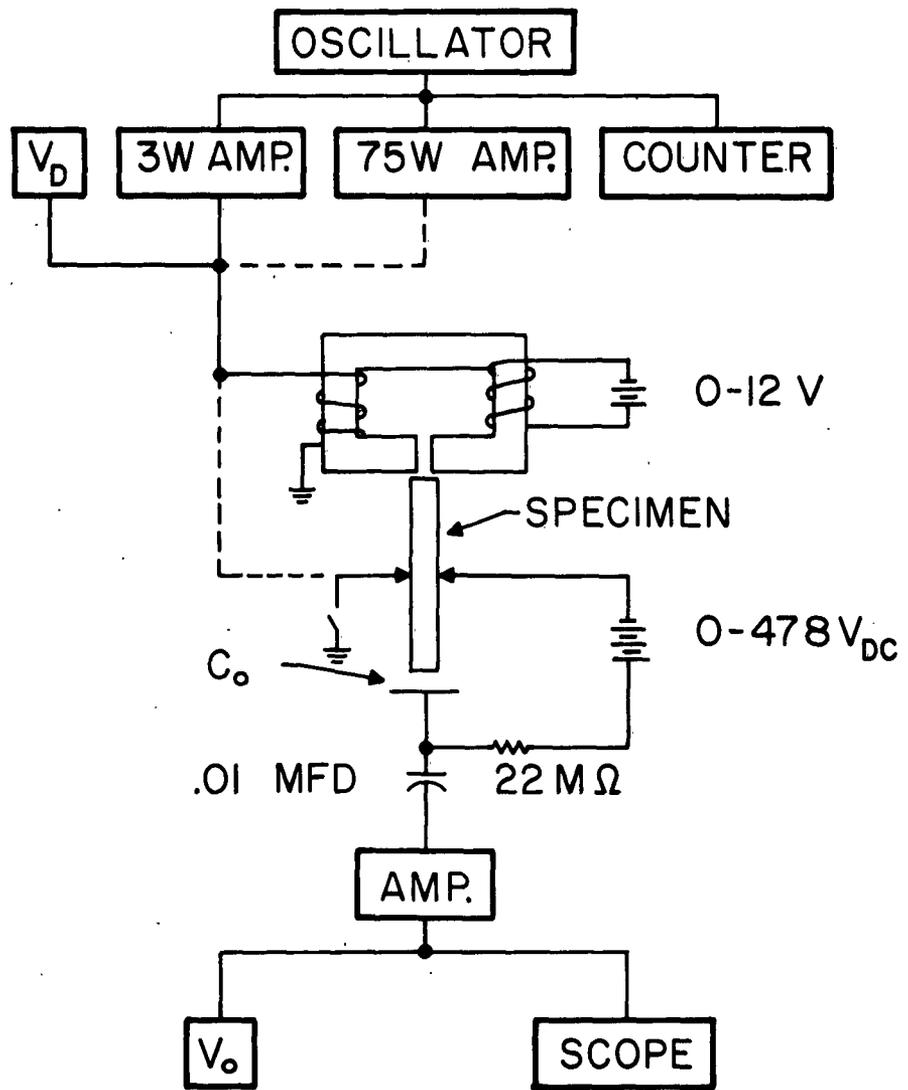


Fig. 1 SCHEMATIC LAYOUT OF INTERNAL FRICTION APPARATUS.

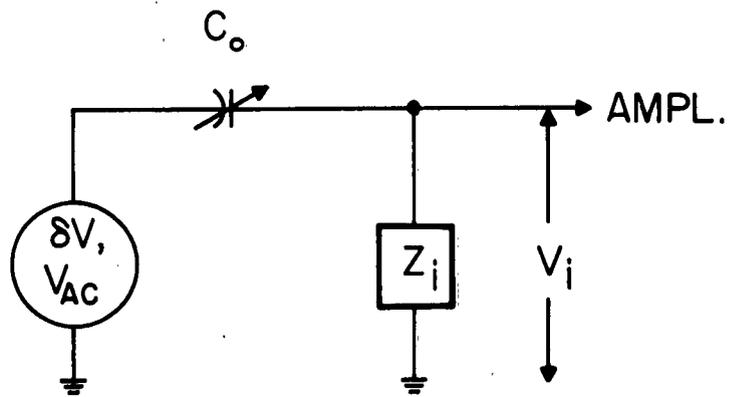


Fig. 2 EQUIVALENT DETECTION CIRCUIT

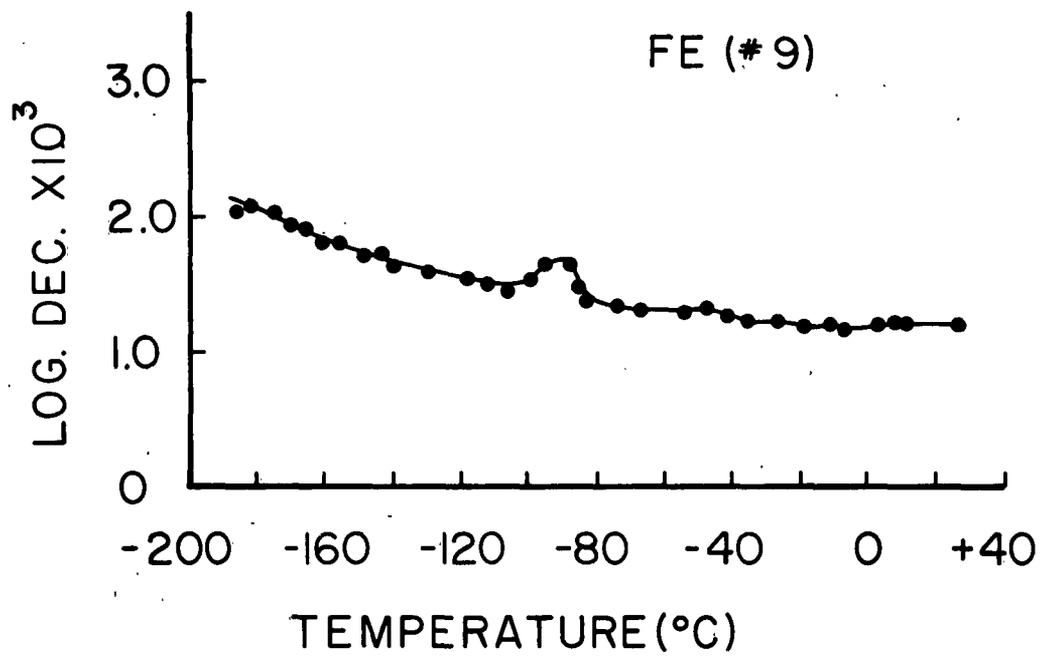


Fig. 3 TYPICAL DAMPING VS. TEMPERATURE CURVE
 8½% Reduction in area, aged at room
 temperature.

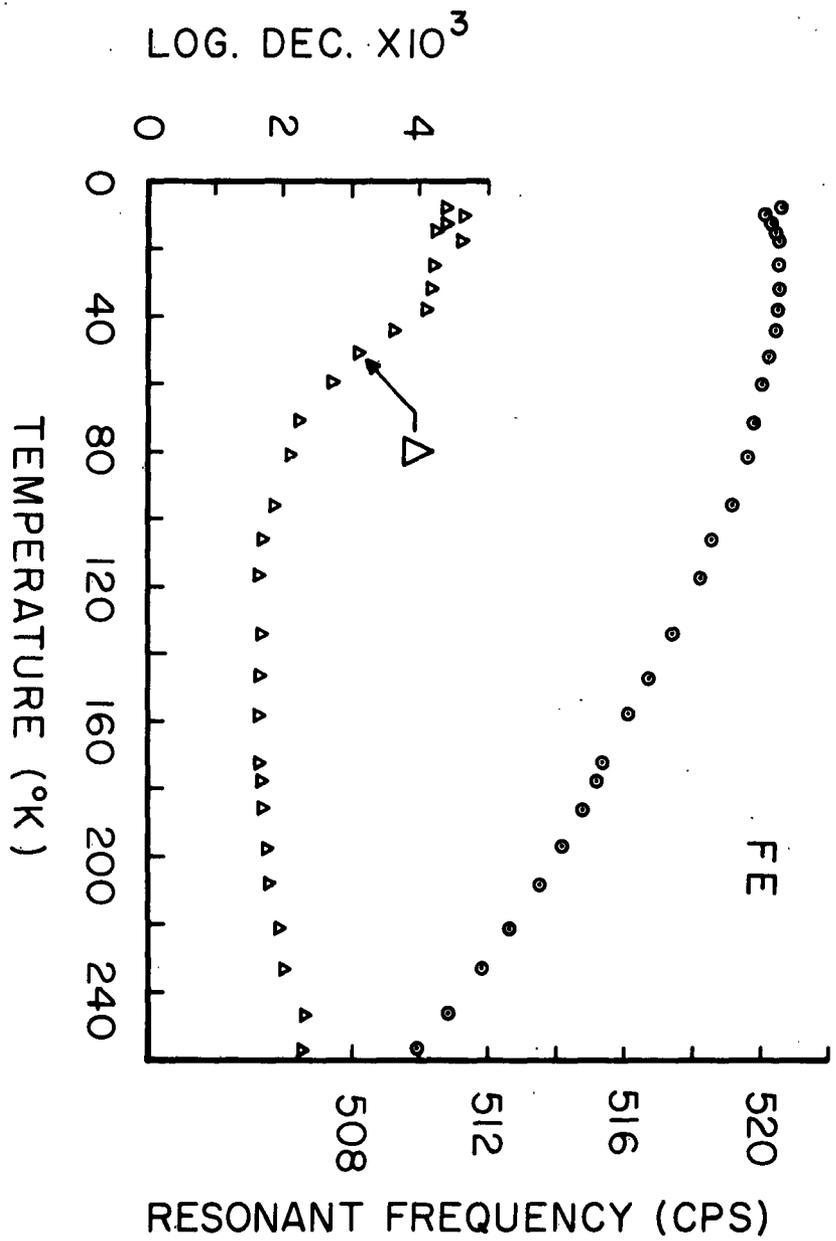


Fig. 4 DAMPING AND RESONANT FREQUENCY VS. TEMPERATURE
 Battelle iron, deformed 1/2% at 105°K.

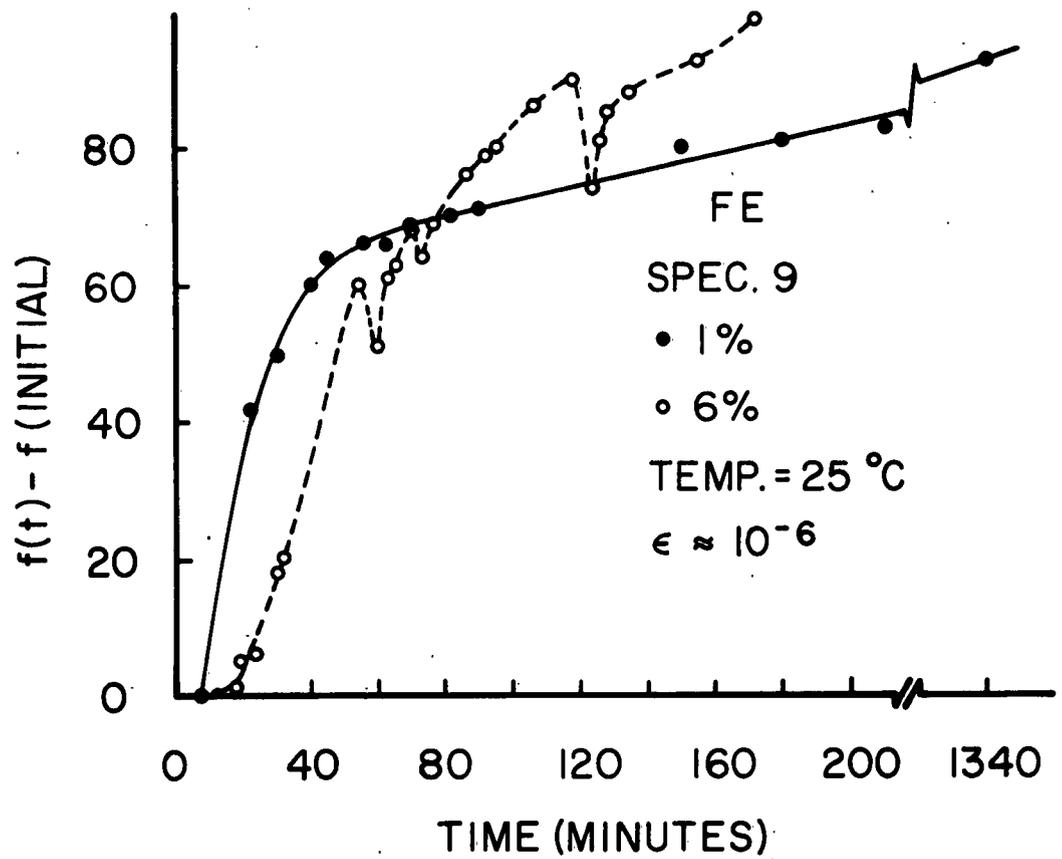


Fig. 5 CHANGE IN MODULUS VS. TIME AFTER DEFORMATION
Values in Graph refer to amount of deformation.

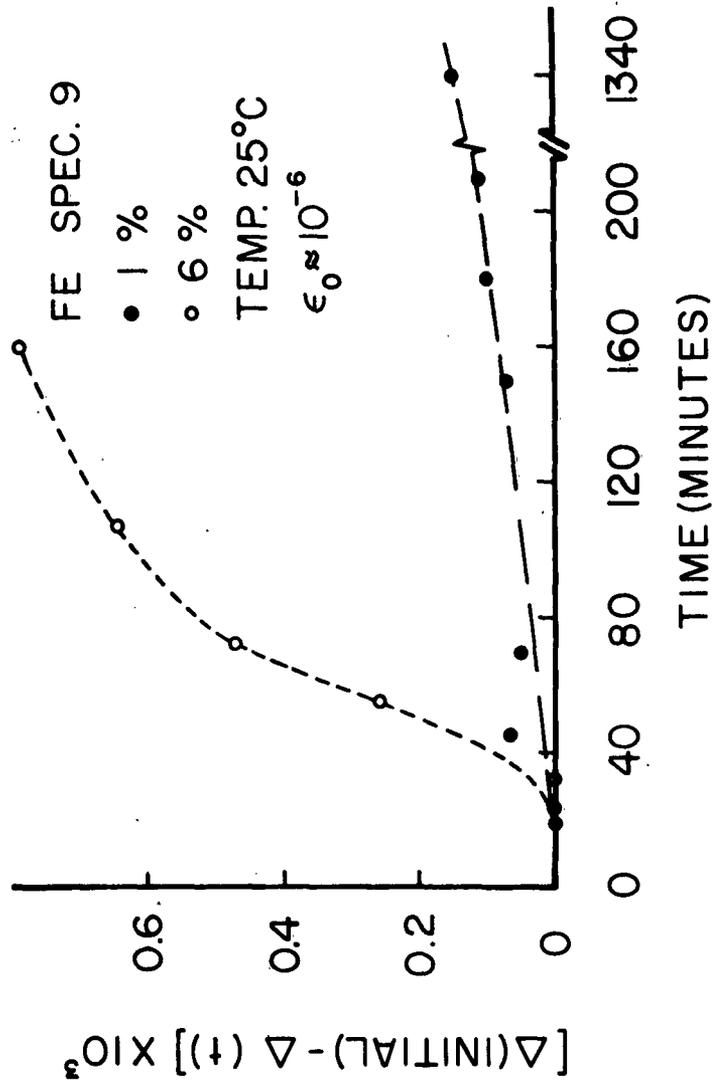


Fig. 6 CHANGE IN DAMPING VS. TIME AFTER DEFORMATION
 Values in graph refer to amount of deformation.

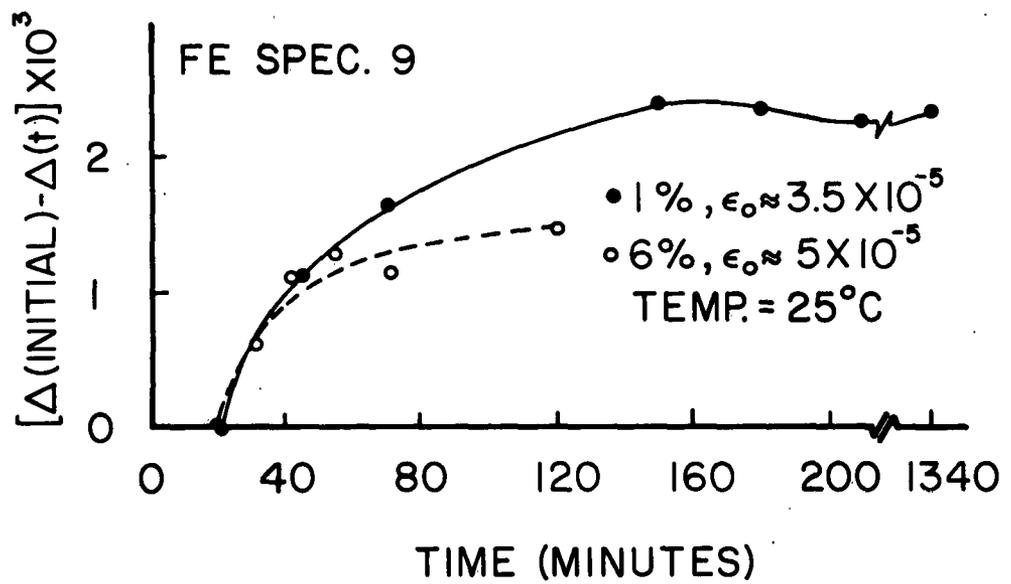


Fig. 7 CHANGE IN DAMPING VS. TIME AFTER DEFORMATION
 Values in graph refer to amount of deformation.

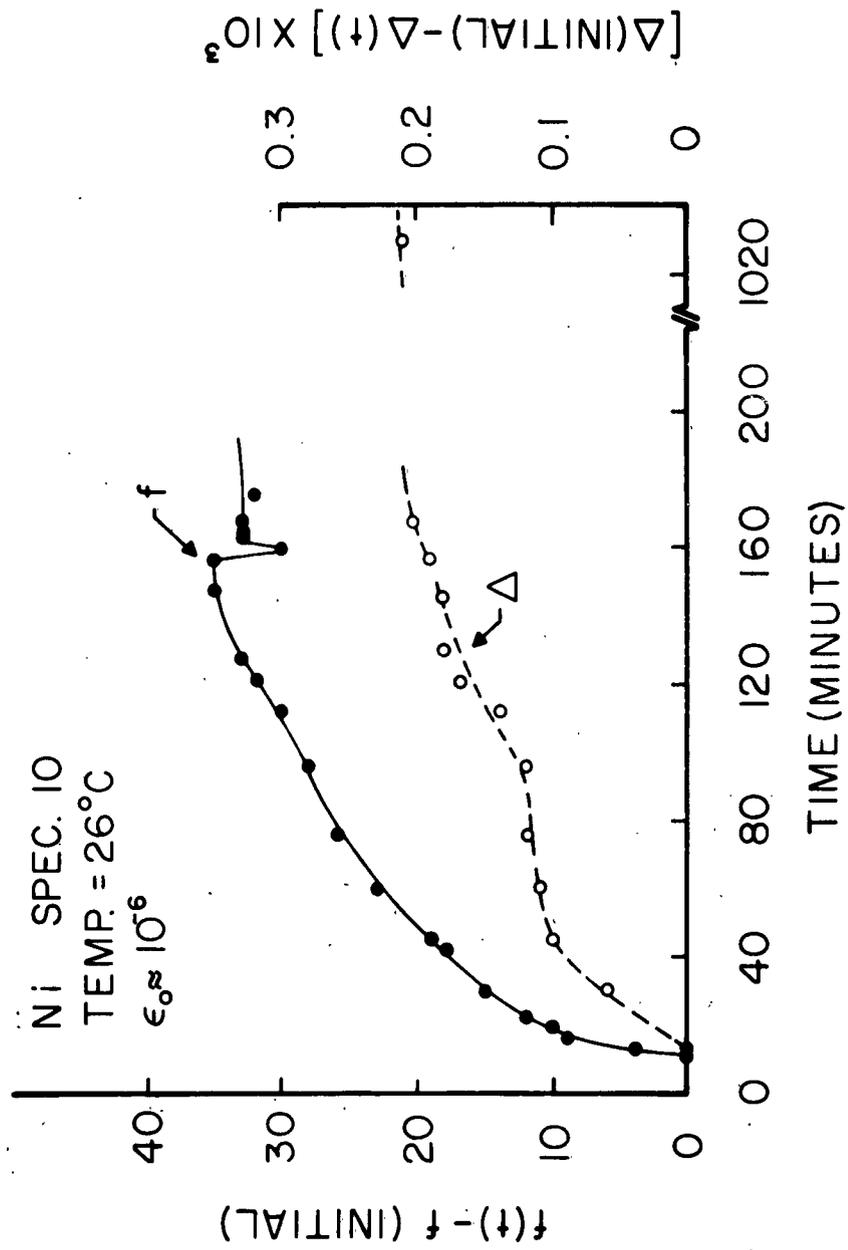


Fig. 8 CHANGE IN DAMPING VS. TIME AFTER DEFORMATION
 $4\frac{1}{2}\%$ reduction in area.

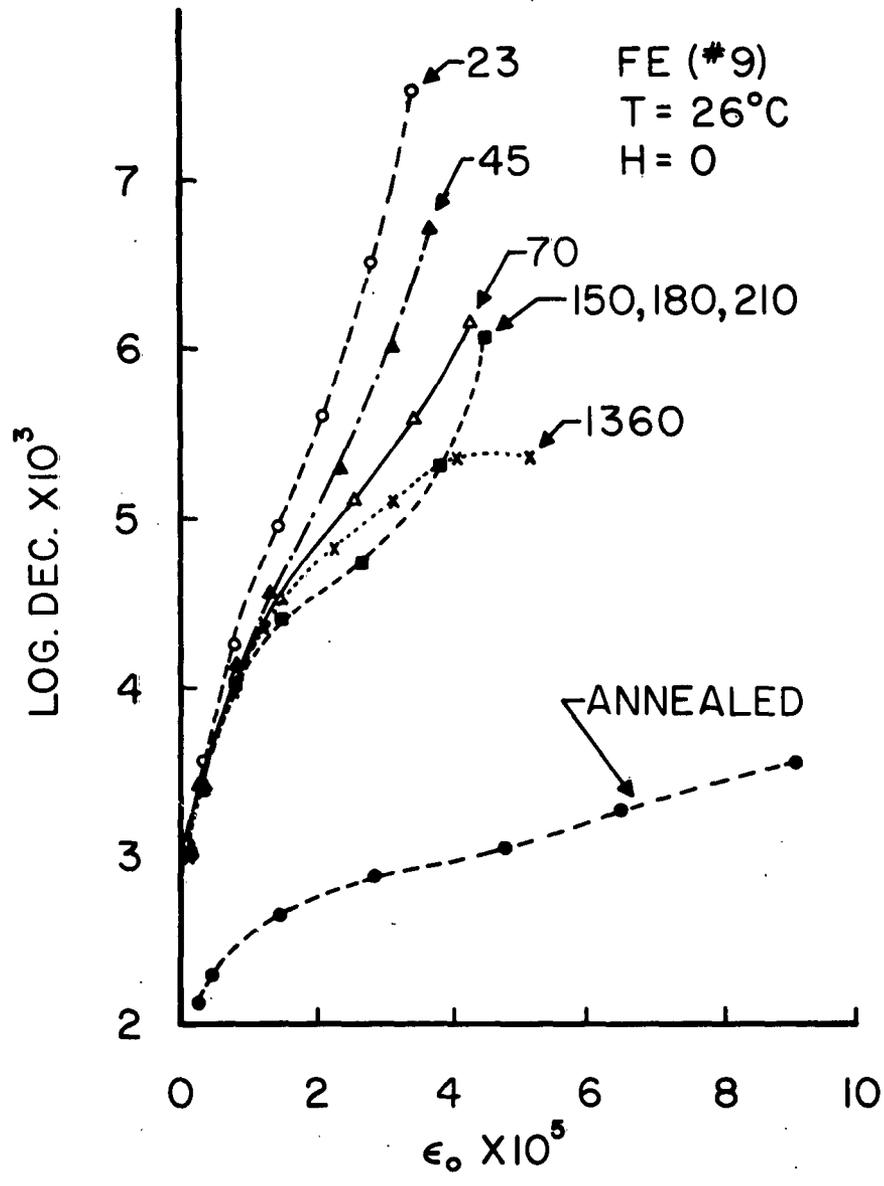


Fig. 9 DAMPING VS. STRAIN AMPLITUDE
Deformed 1% in compression. Values in graph refer to time in minutes after deformation.

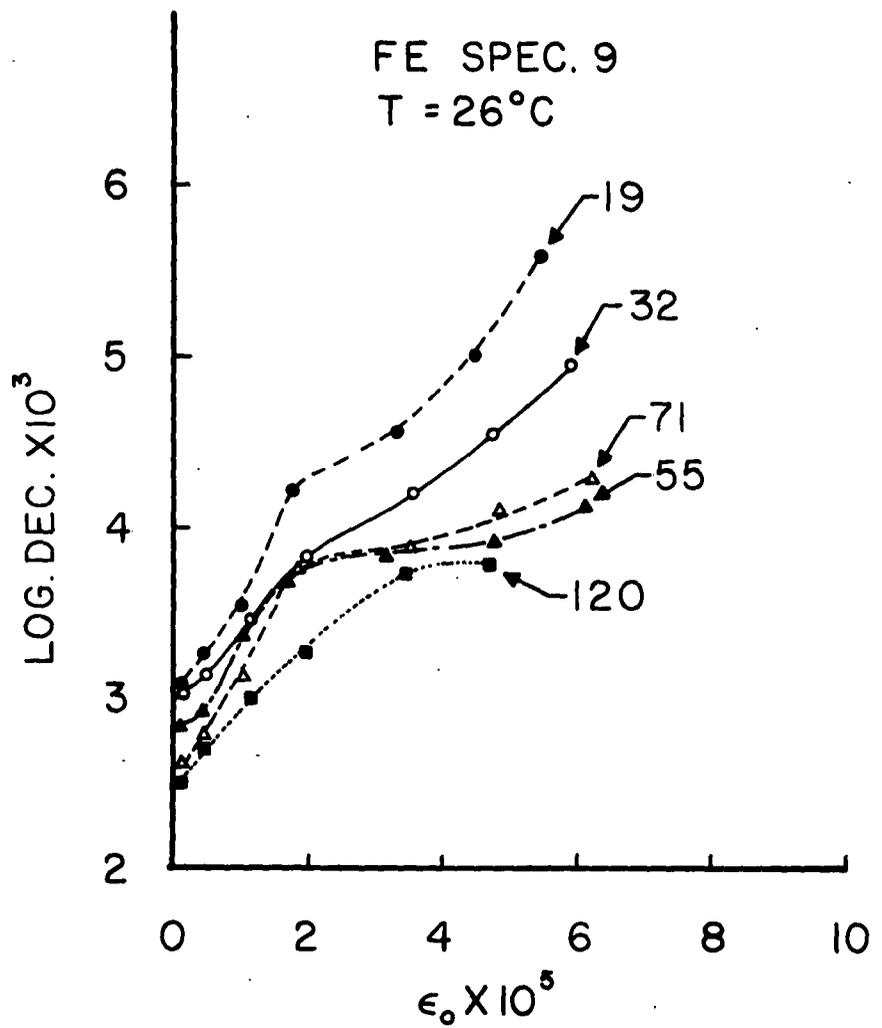


Fig. 10 DAMPING VS. STRAIN AMPLITUDE
Deformed 6% in compression. Values in graph refer to time in minutes after deformation.

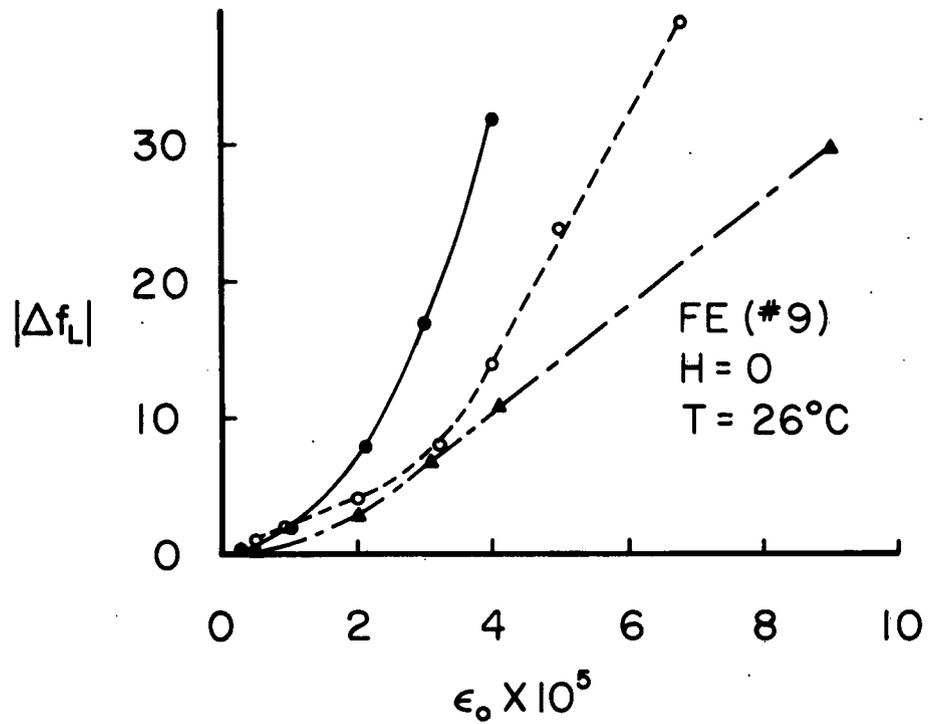


Fig. 11 MODULUS DEFECT AT LOW AMPLITUDE VS. STRAIN AMPLITUDE
One minute at high amplitudes (● Recrystallized;
○ lightly cold-worked; ▲ Heavily cold-worked).

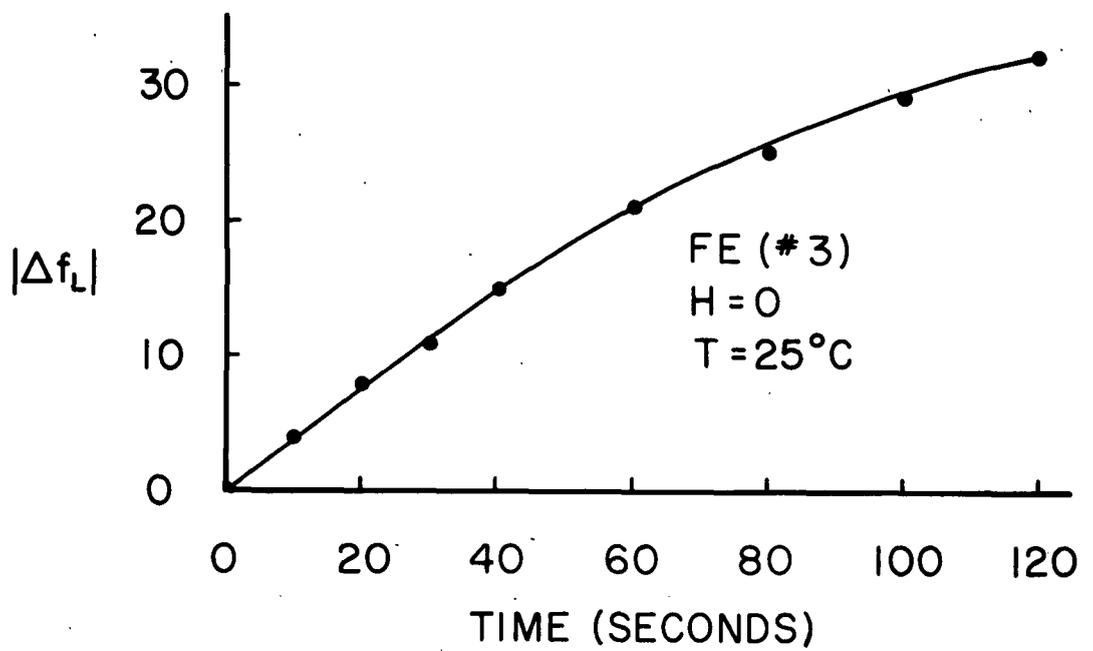


Fig. 12 MODULUS DEFECT AT LOW AMPLITUDE VS. TIME OF HIGH AMPLITUDE OSCILLATION
Lightly cold-worked specimen. High amplitude oscillations at $\epsilon_0 = 3 \times 10^{-5}$

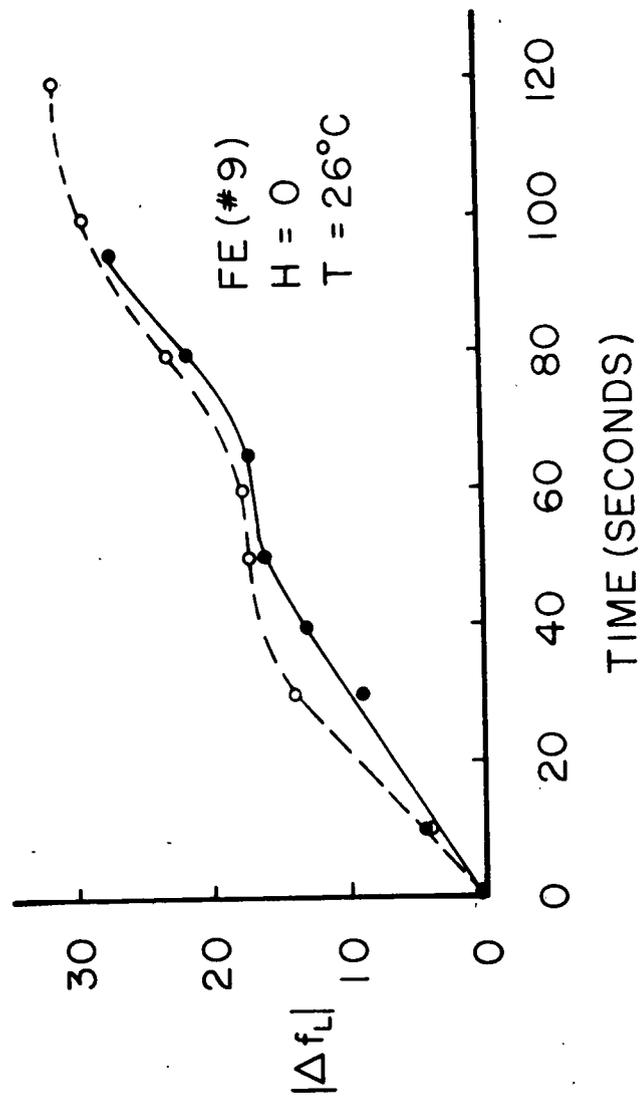


Fig. 13 MODULUS DEFECT AT LOW AMPLITUDE VS. TIME OF HIGH AMPLITUDE OSCILLATION
 Recrystallized specimen. High amplitude oscillations at $\epsilon_0 \approx 3 \times 10^{-5}$.

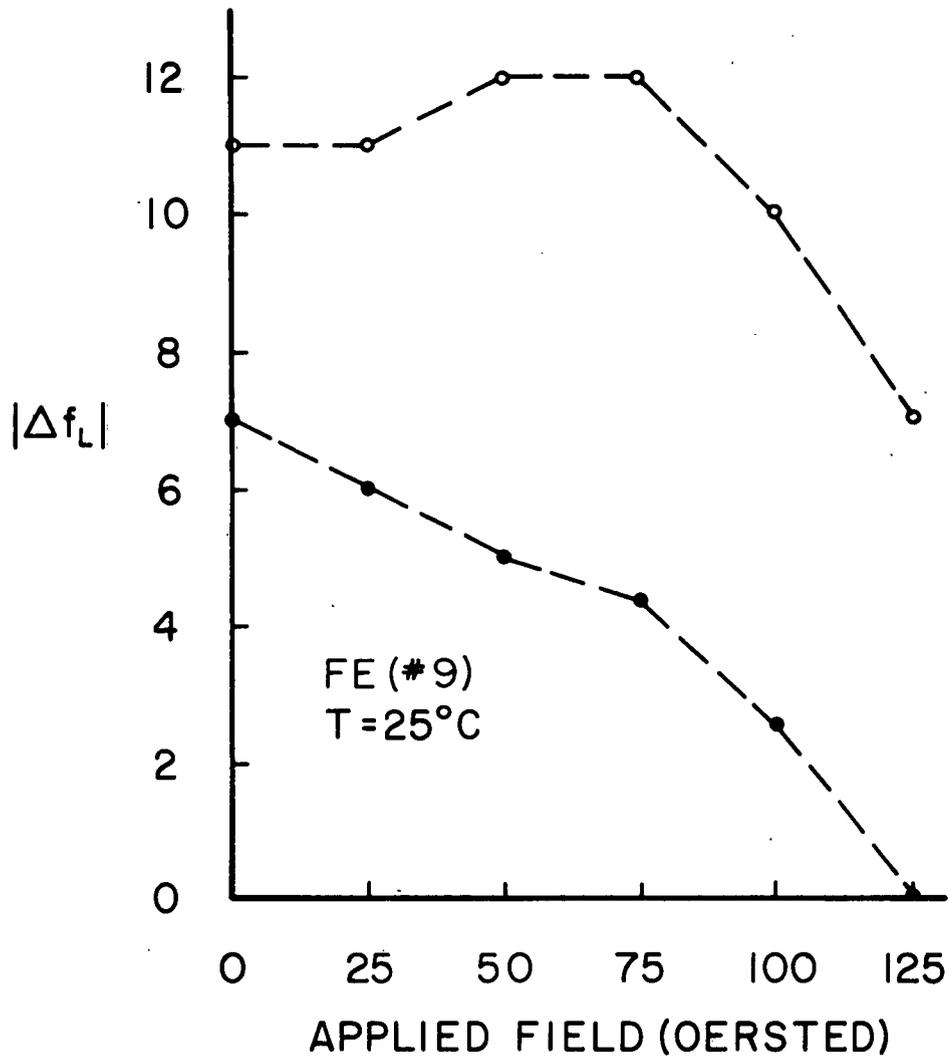


Fig. 14 MODULUS DEFECT AT LOW AMPLITUDES VS. APPLIED MAGNETIC FIELD
 One minute at high amplitudes: (○) $\epsilon_0 = 4 \times 10^{-5}$, heavily cold-worked; (●) $\epsilon_0 = 1.5 \times 10^{-5}$, recrystallized.

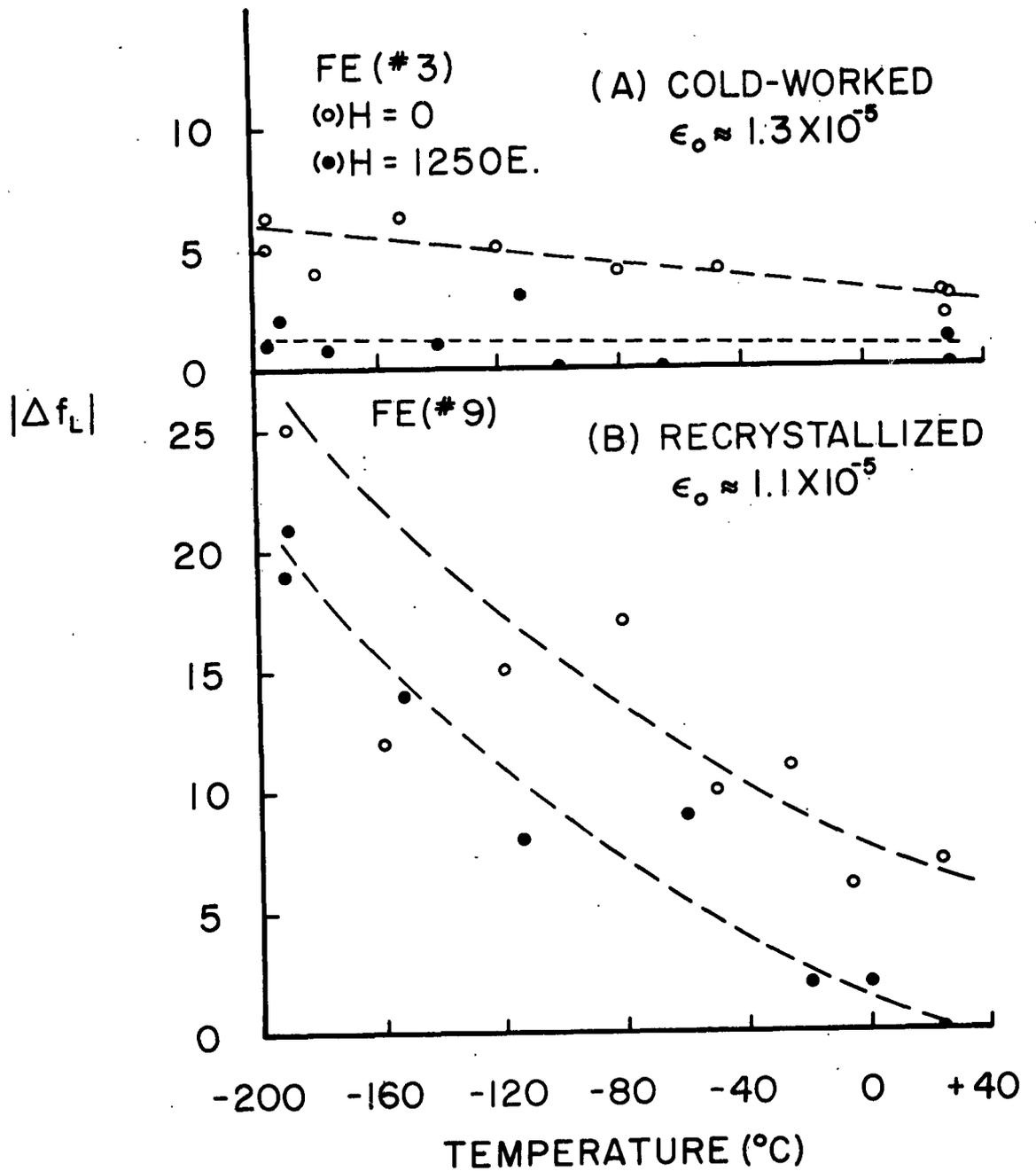


Fig. 15 MODULUS DEFECT AT LOW AMPLITUDES VS. TEMPERATURE
 One minute at high amplitudes noted in figure.

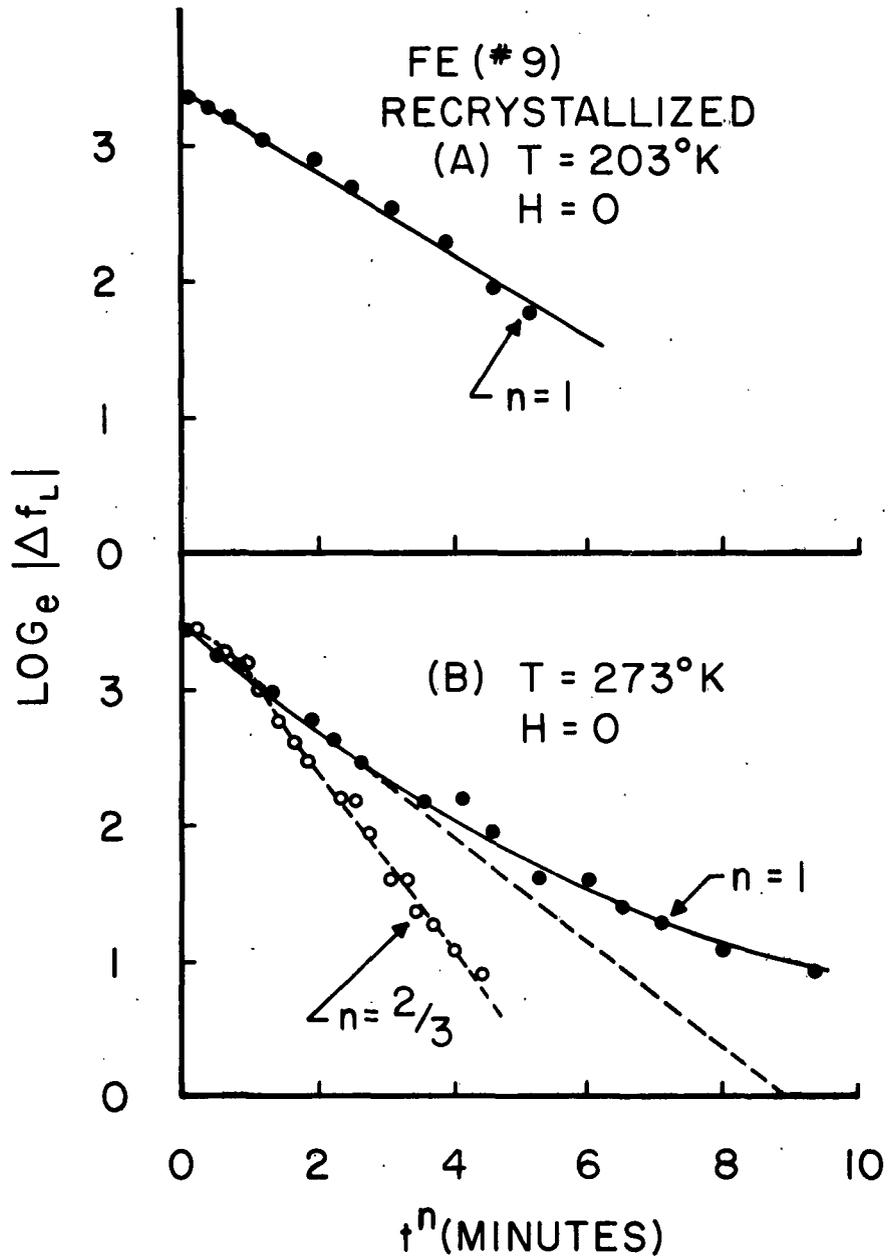


Fig. 16 LOG OF CHANGE IN FREQUENCY VS. TIME AFTER HIGH AMPLITUDE OSCILLATIONS

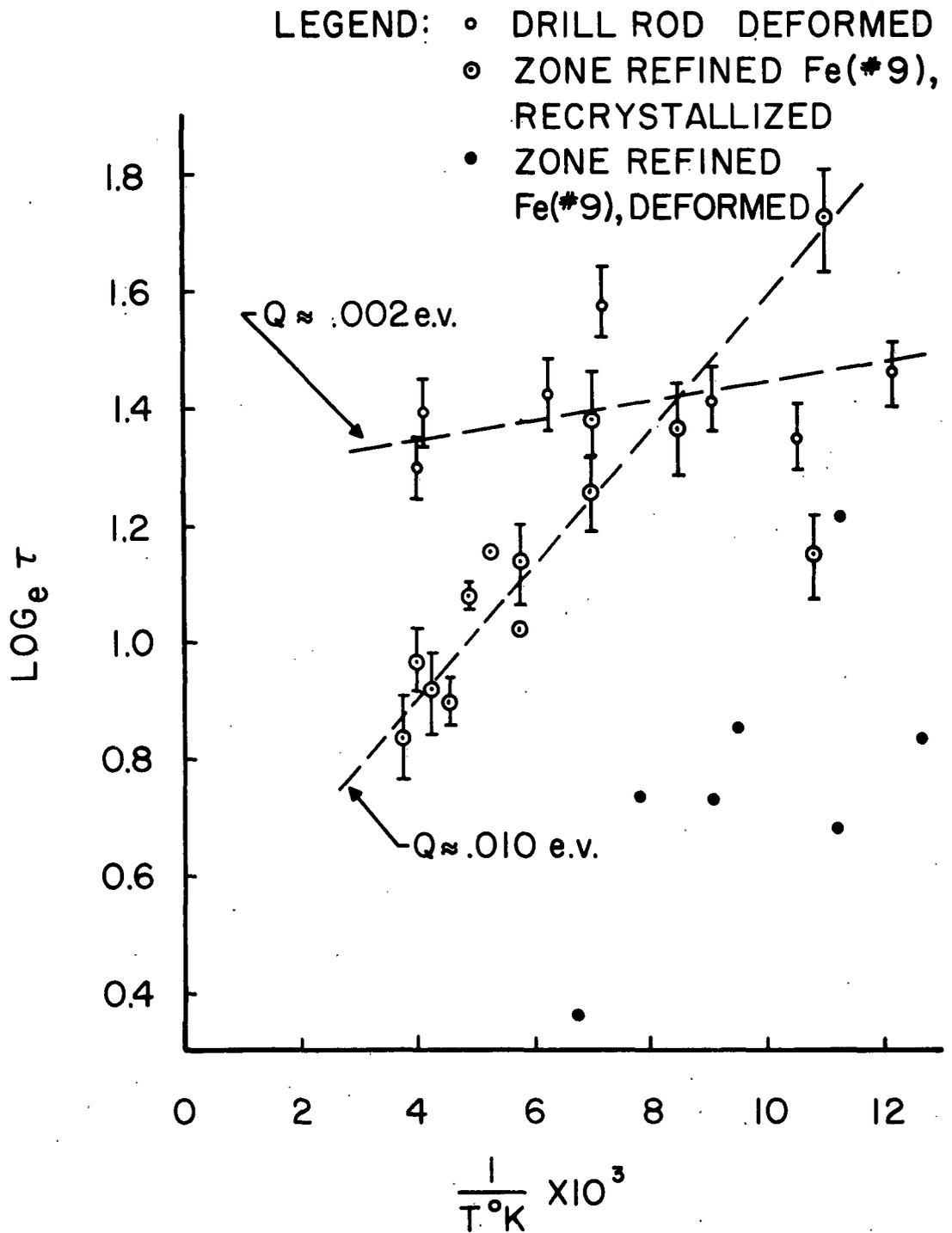


Fig. 17 LOG OF RELAXATION TIME VS. RECIPROCAL ABSOLUTE TEMPERATURE

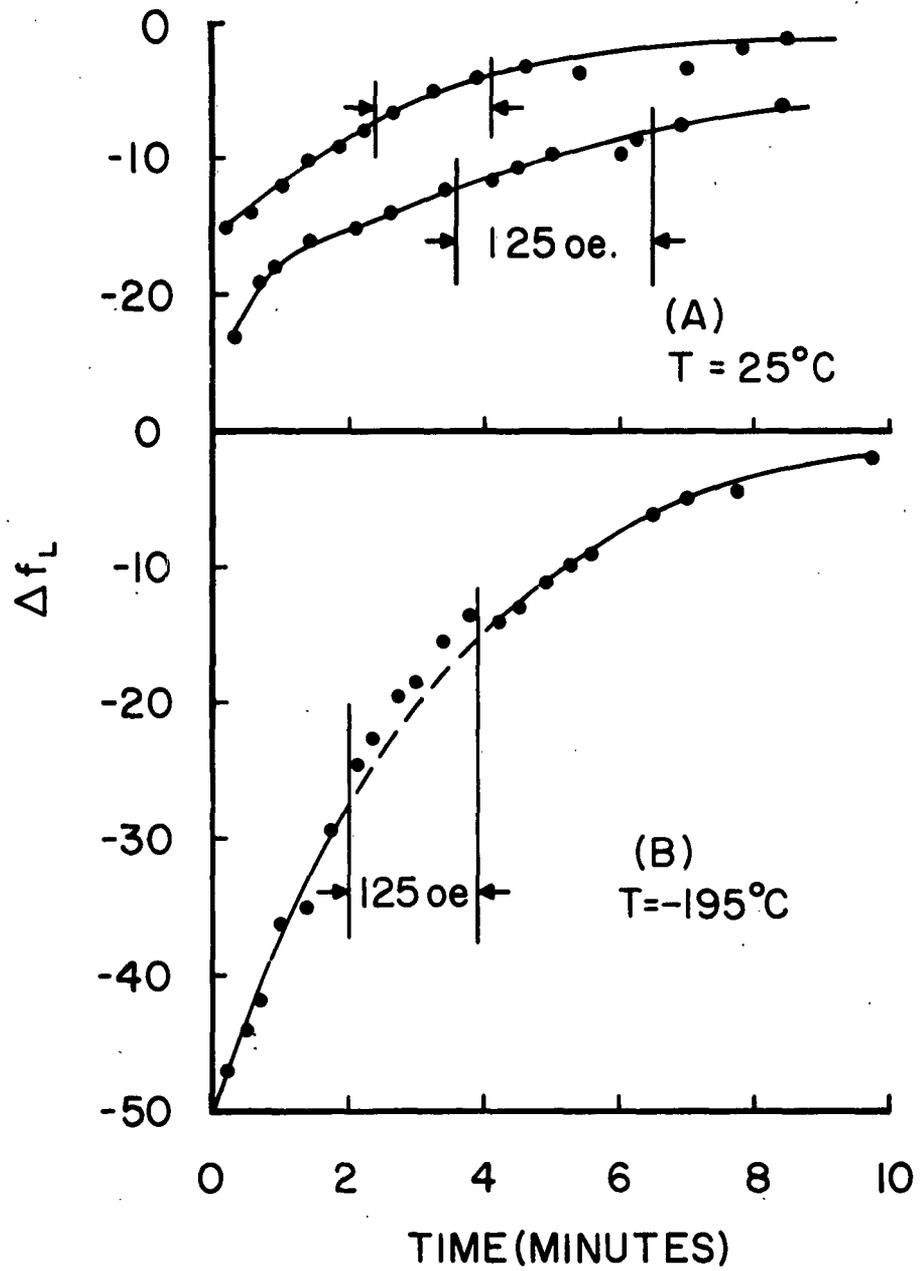


Fig. 18 MODULUS DEFECT VS. TIME AFTER HIGH AMPLITUDE OSCILLATIONS
Magnetic field applied only during time interval indicated by arrows.

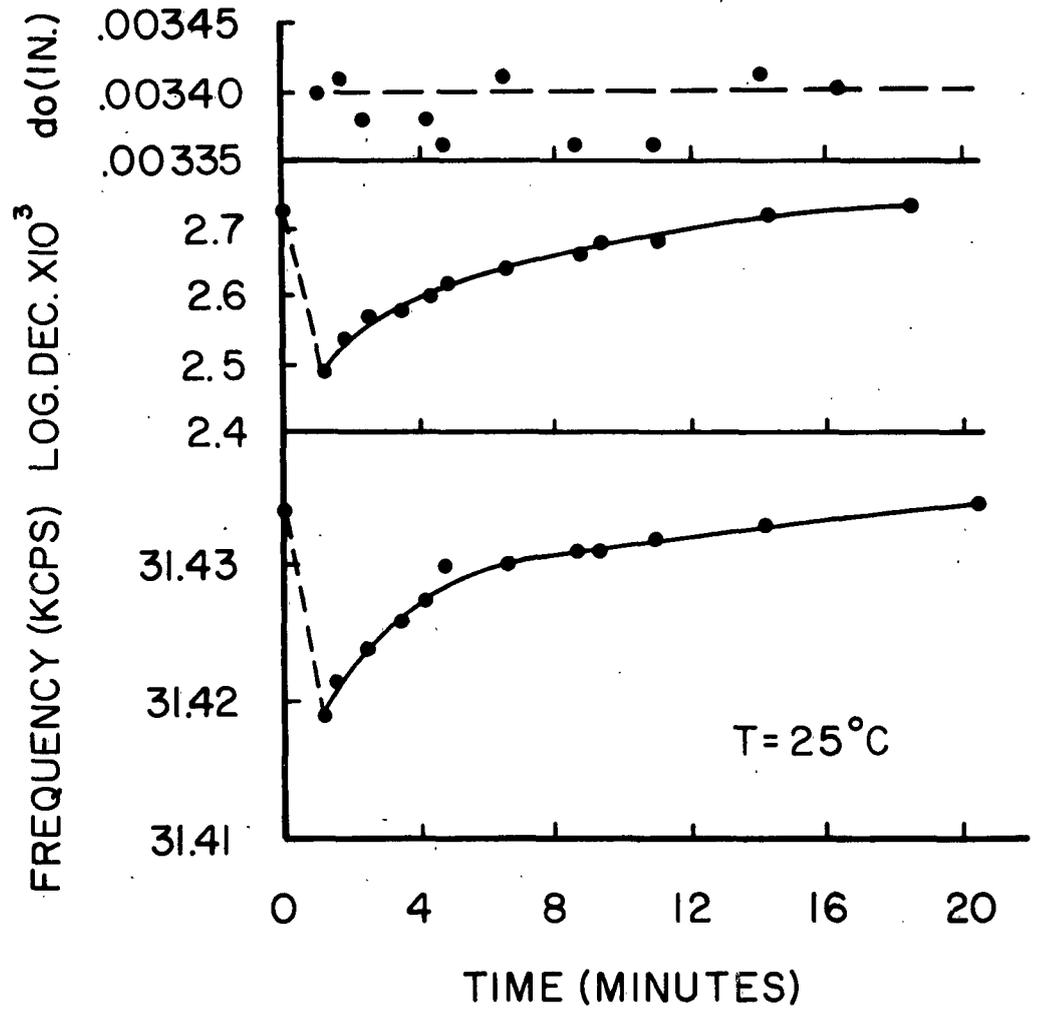


Fig. 19 FREQUENCY, DAMPING, AND GAP WIDTH VS. TIME AFTER HIGH AMPLITUDE OSCILLATIONS
 Dashed line represents time of high amplitude oscillation.

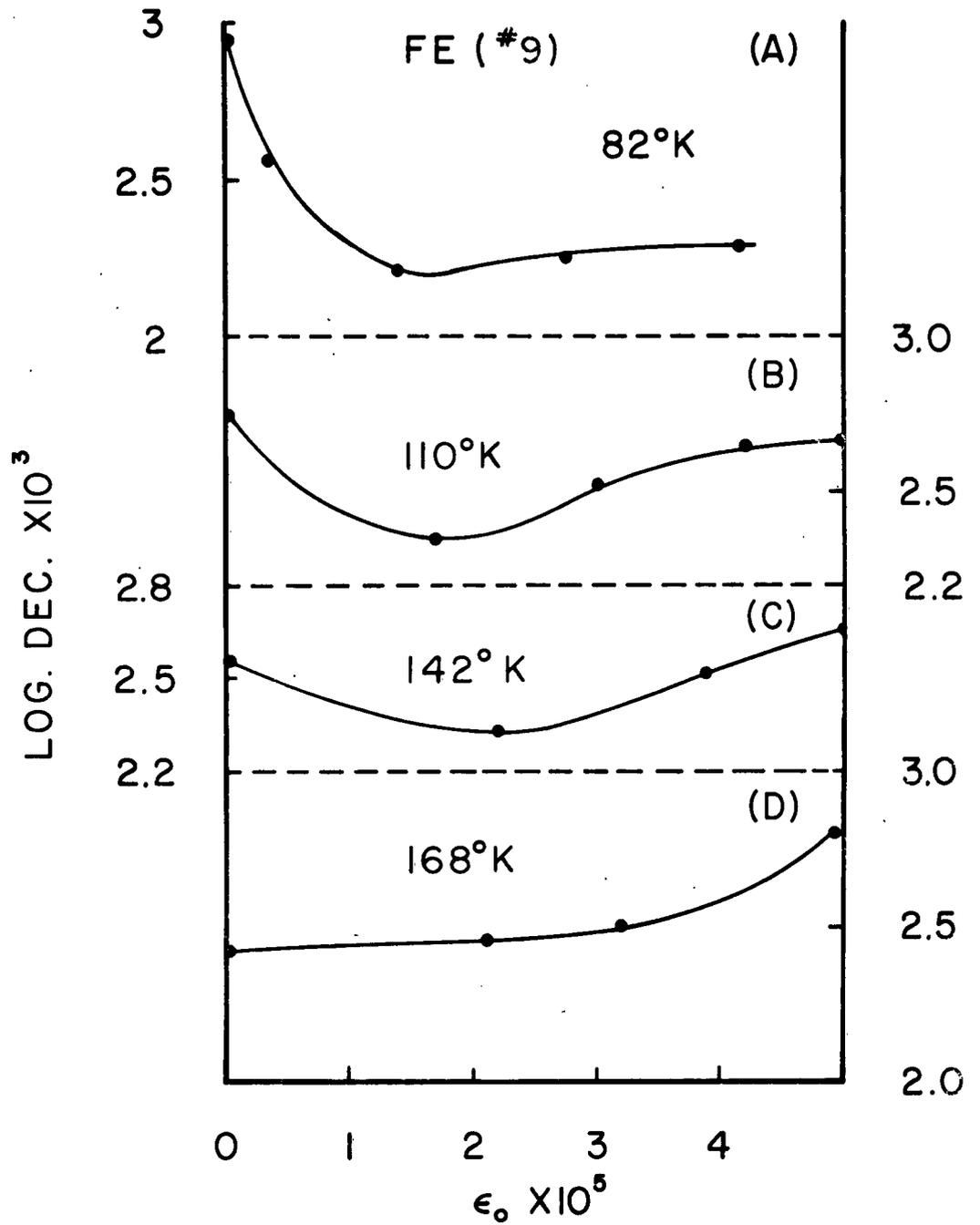


Fig. 20 DAMPING VS. STRAIN AMPLITUDE AT VARIOUS TEMPERATURES
 Specimen is recrystallized; $H = 0$.

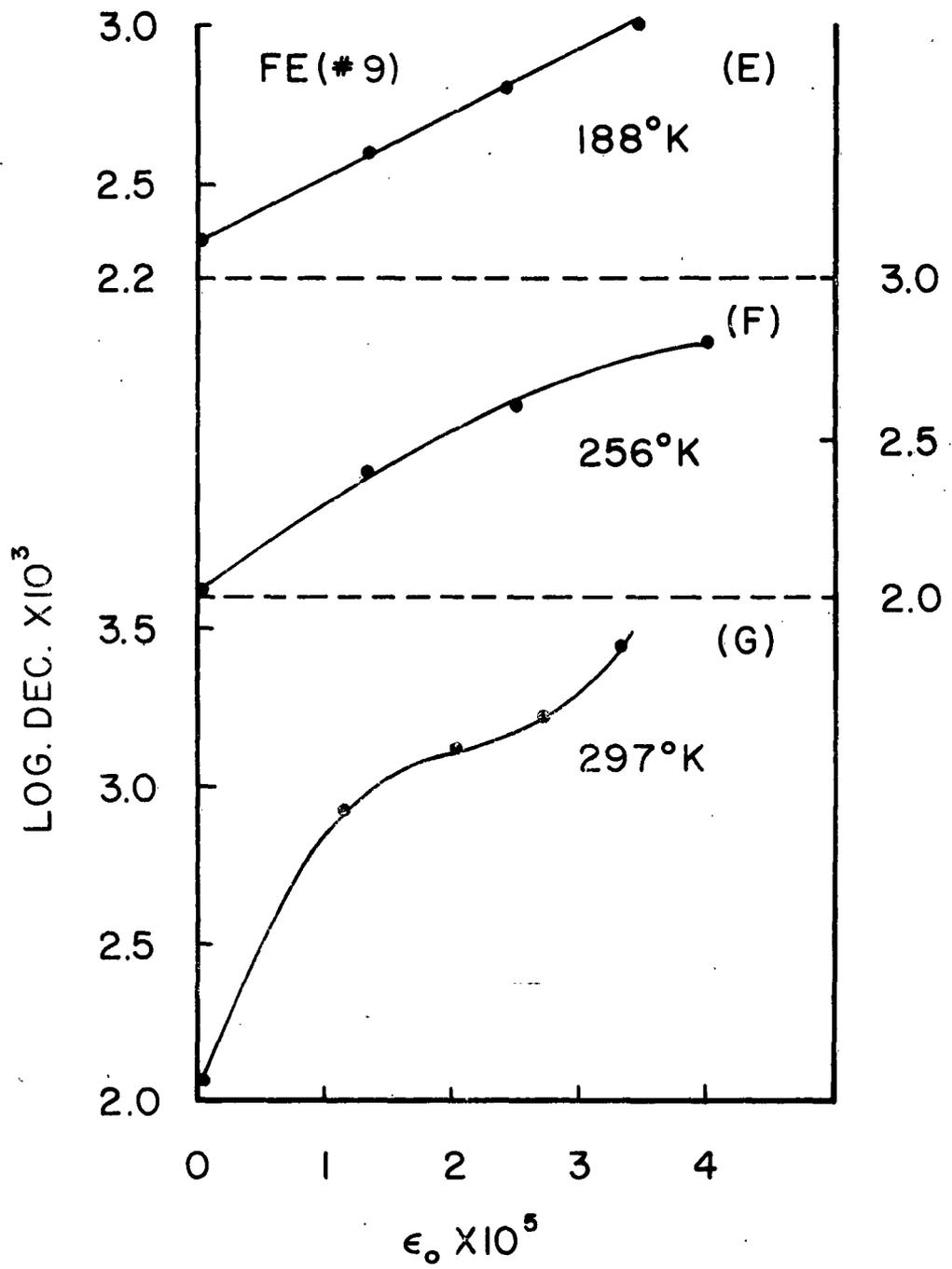


FIG. 20 DAMPING VS. STRAIN AMPLITUDE AT VARIOUS TEMPERATURES
 Specimen is recrystallized; $H = 0$.

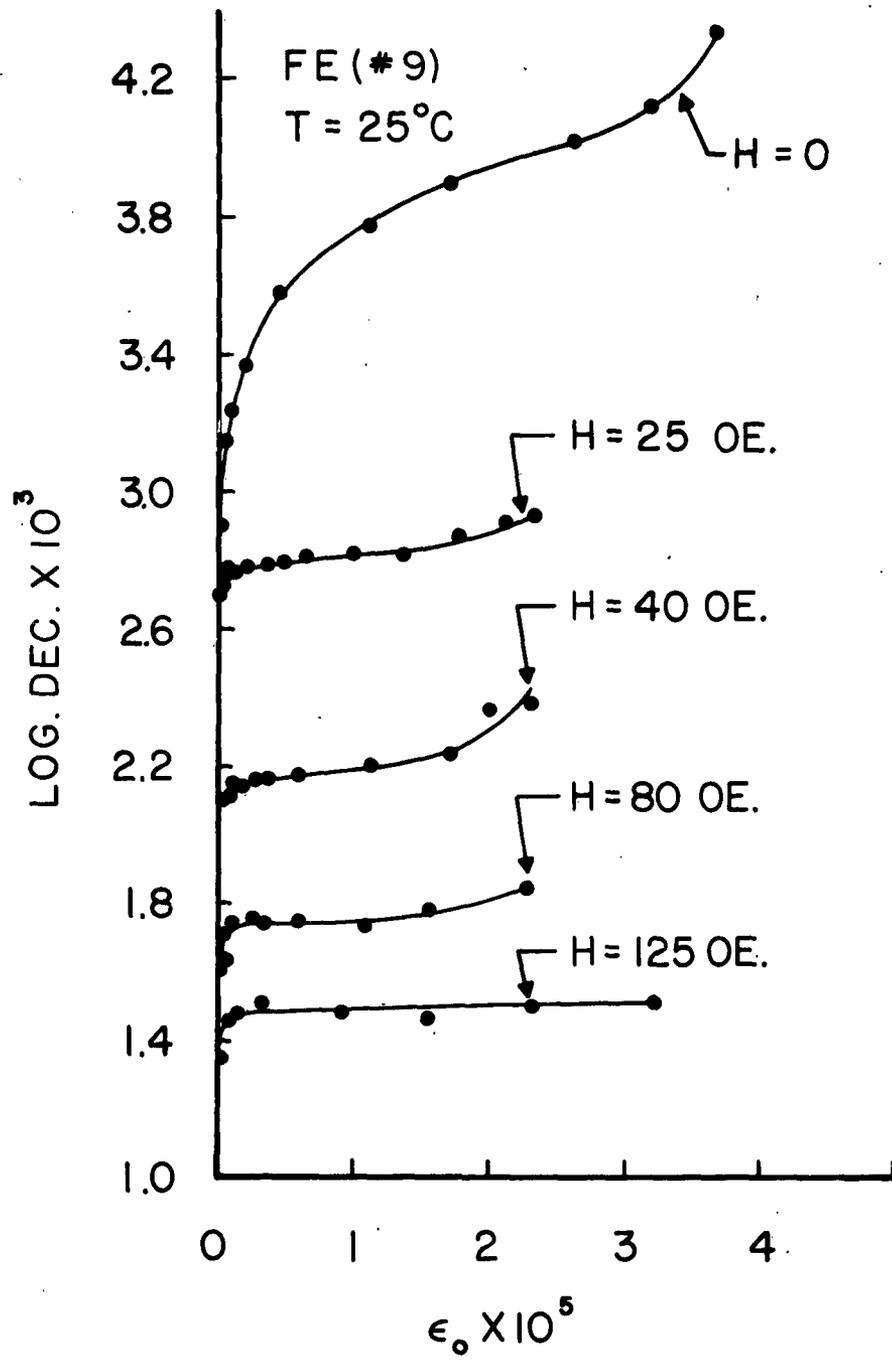


Fig. 21 DAMPING VS. STRAIN AMPLITUDE
Specimen is recrystallized.

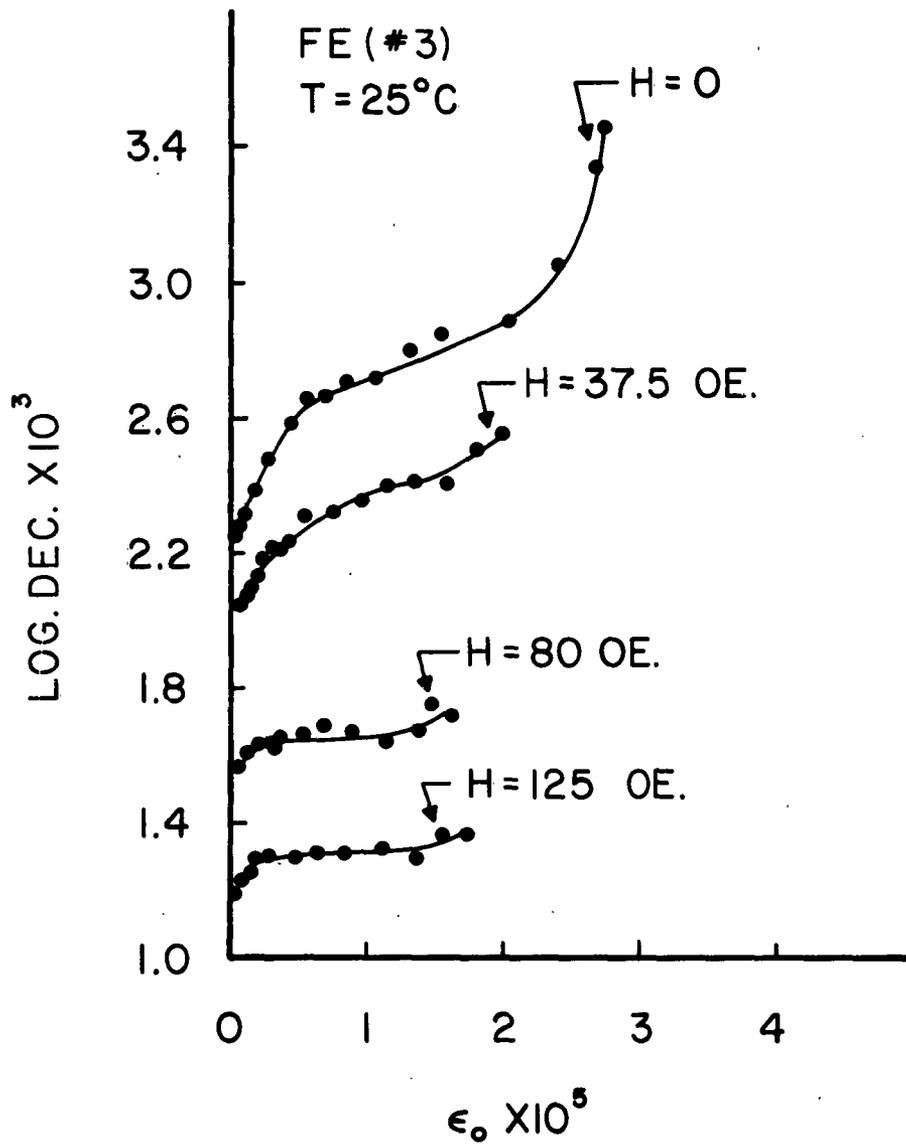


Fig. 22 DAMPING VS. STRAIN AMPLITUDE
Specimen is lightly cold-worked.

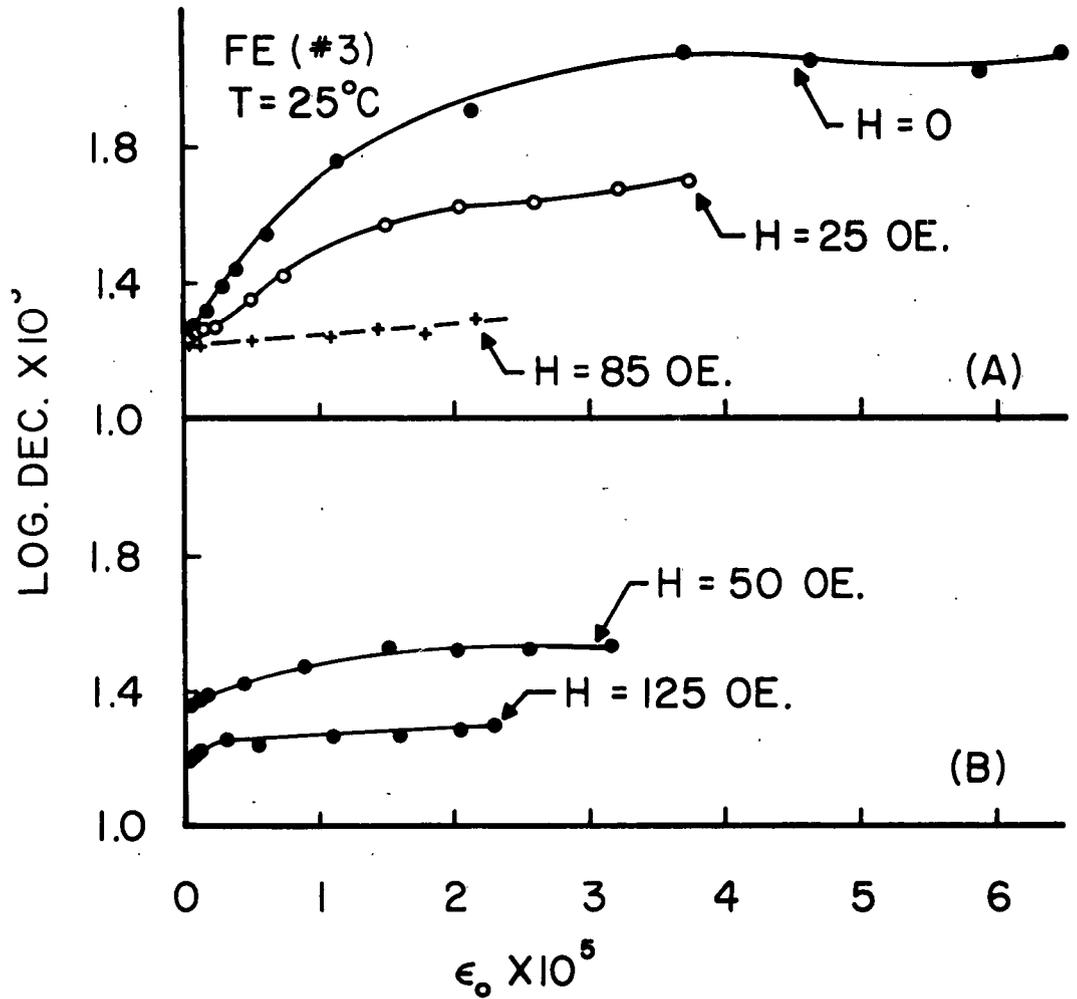


Fig. 23 DAMPING VS. STRAIN AMPLITUDE
Specimen is heavily cold-worked.

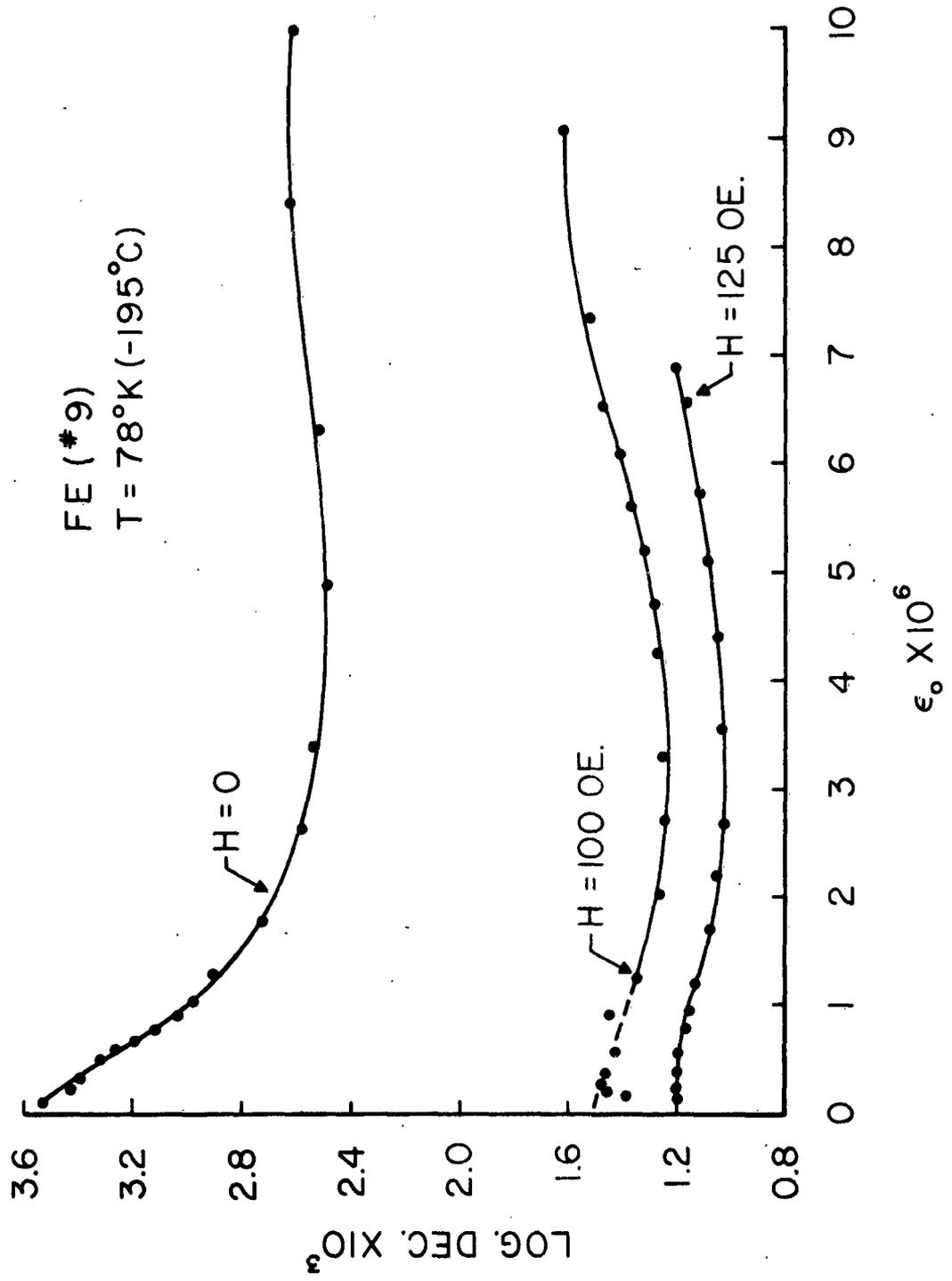


Fig. 24 DAMPING VS. STRAIN AMPLITUDE
 Specimen is recrystallized.

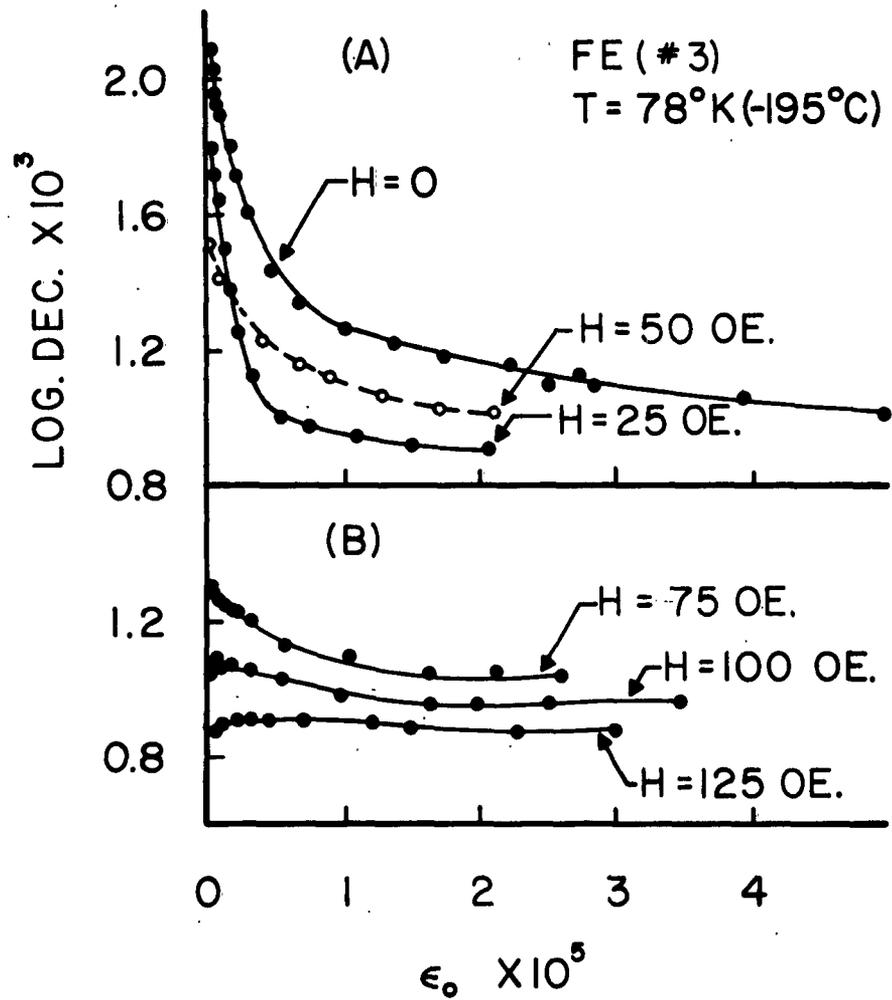


Fig. 25 DAMPING VS. STRAIN AMPLITUDE
Specimen is heavily cold-worked.

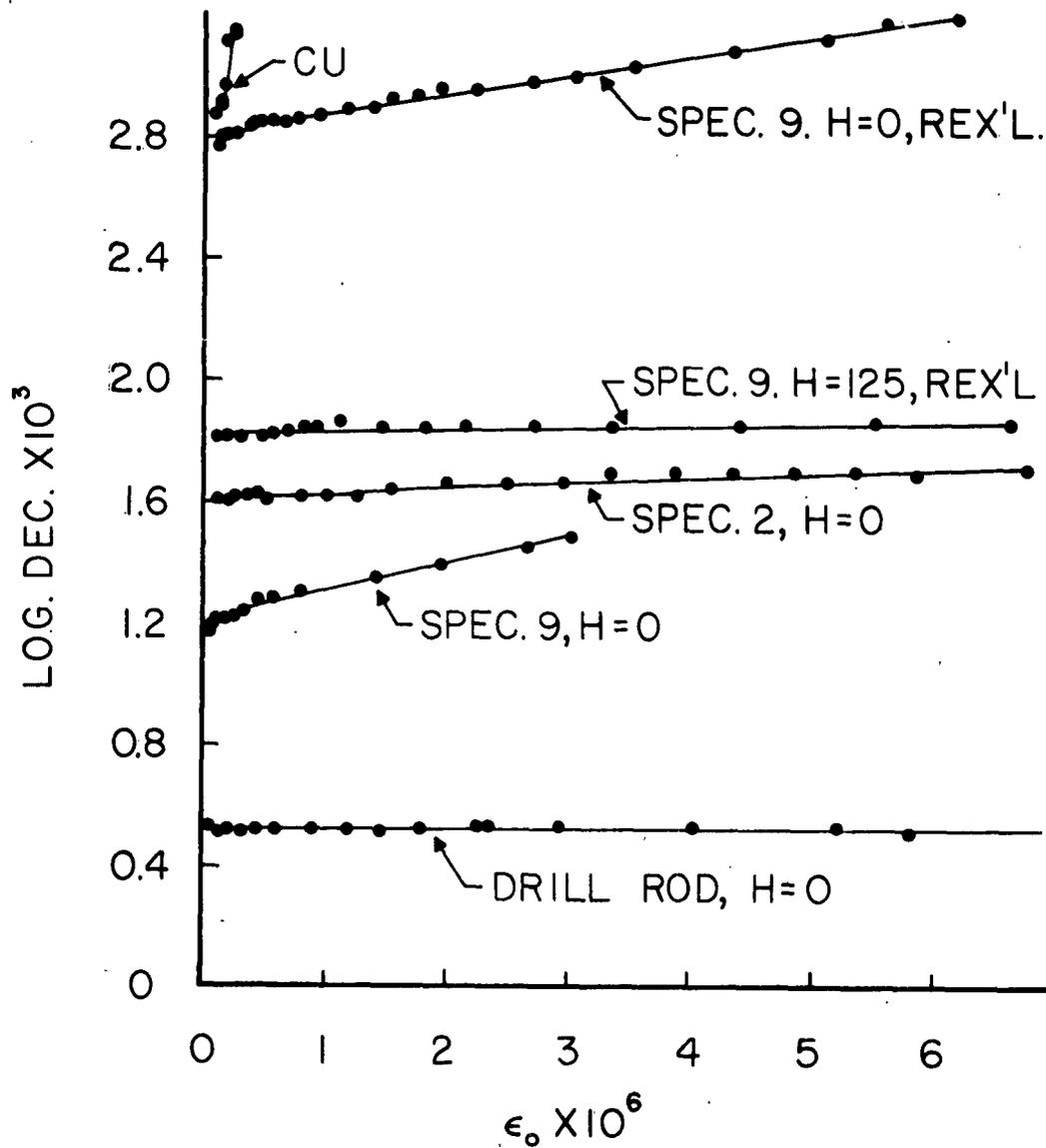


Fig. 26 DAMPING VS. STRAIN AMPLITUDE
 Specimens cold-worked except as noted.
 "REX'L" denotes recrystallized.

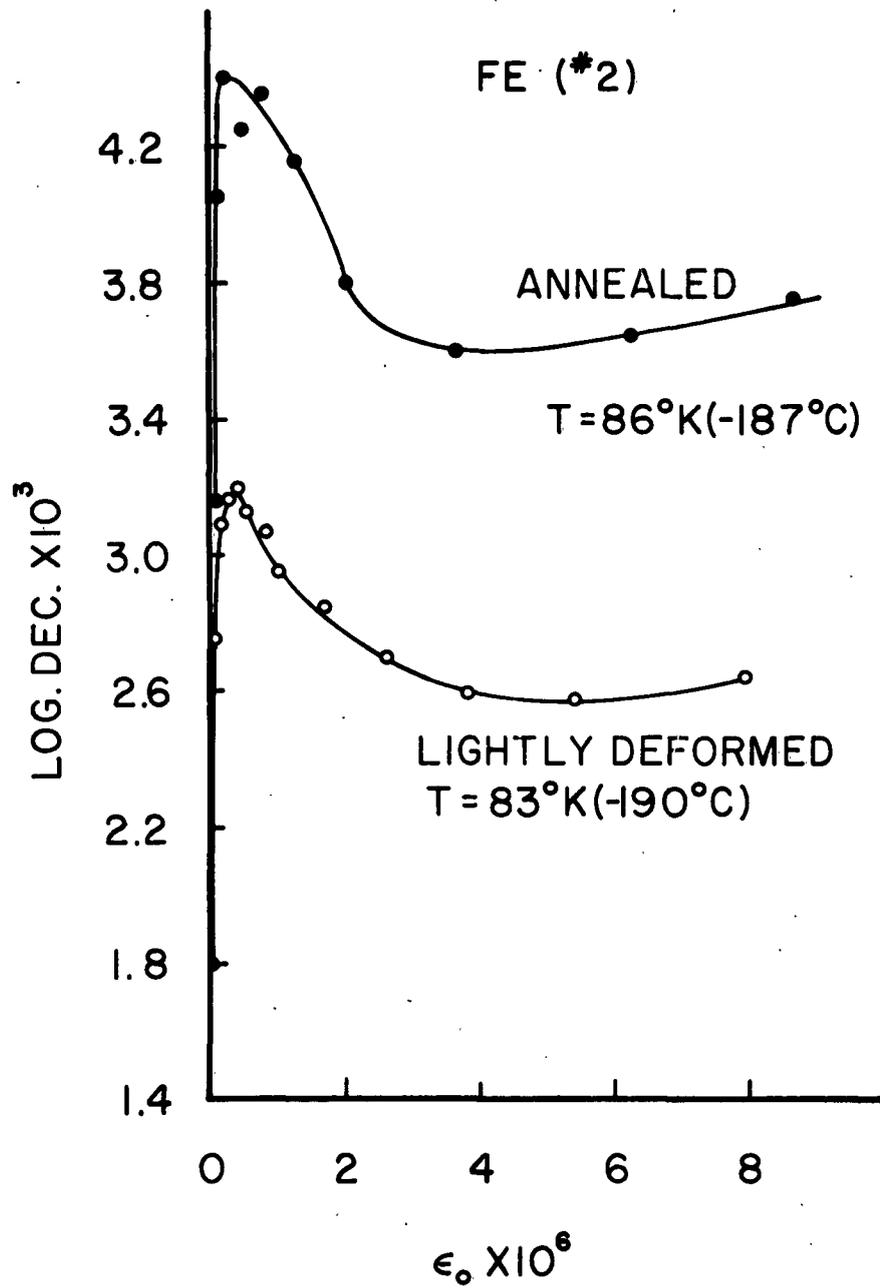


Fig. 27 DAMPING VS. STRAIN AMPLITUDE
H = 0.

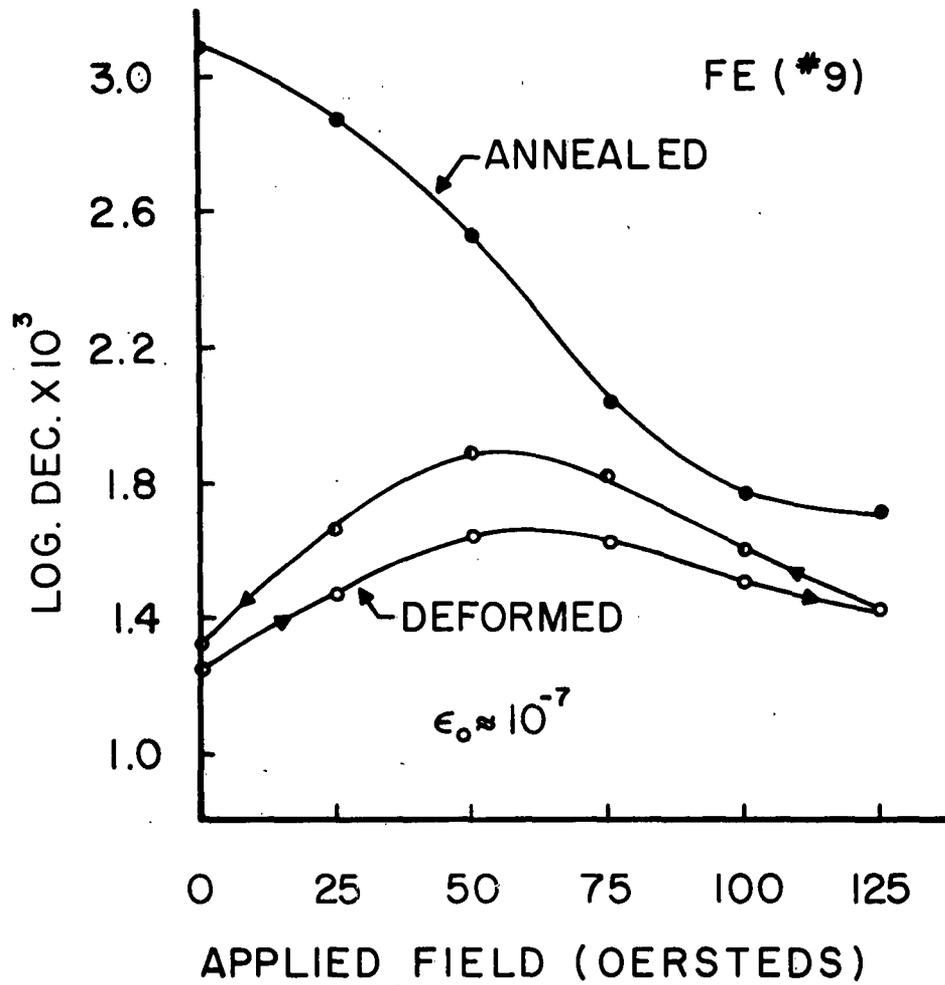


Fig. 28 DAMPING VS. APPLIED FIELD AT ROOM TEMPERATURE

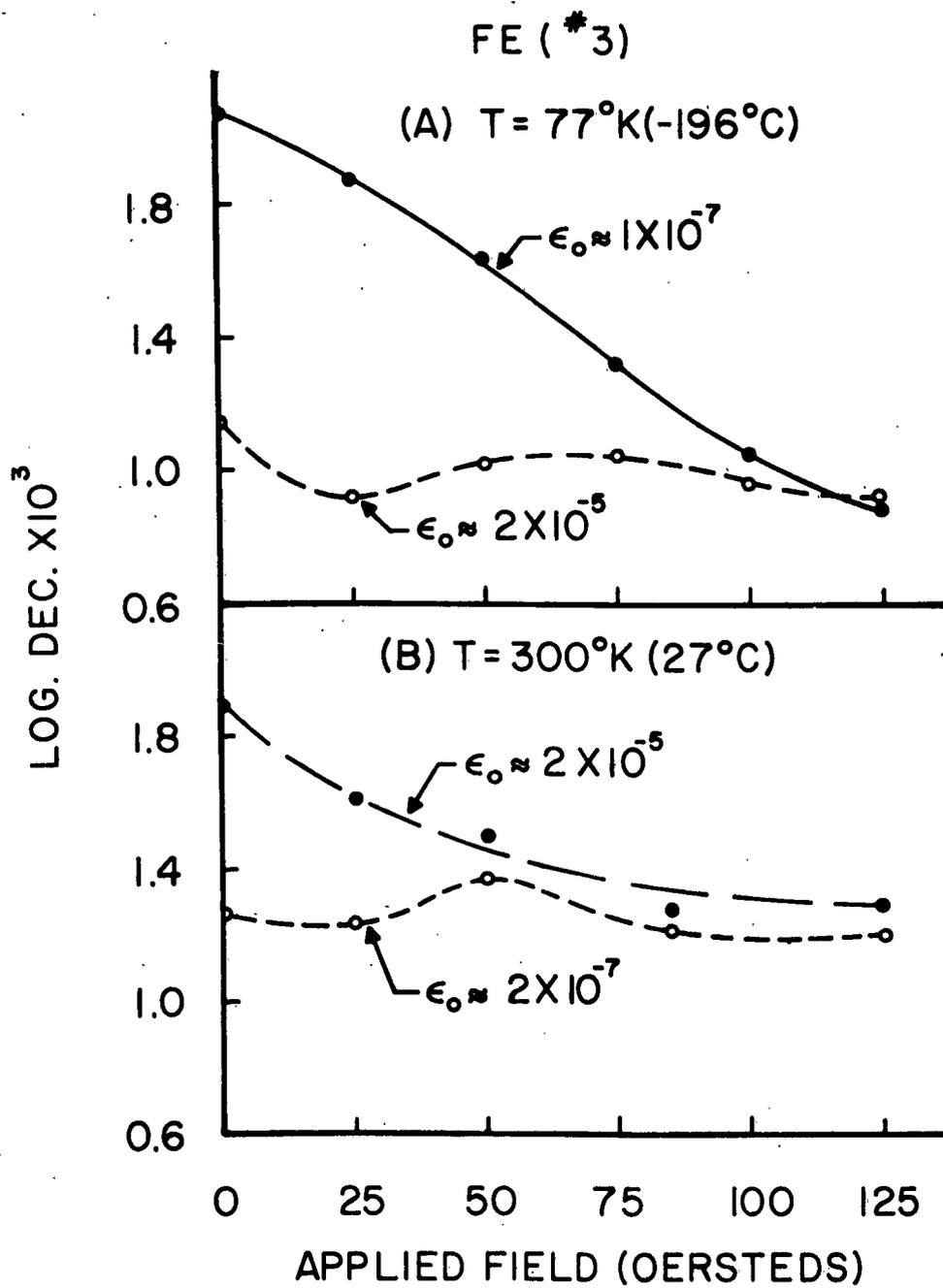


Fig. 29 DAMPING VS. APPLIED FIELD
Specimen is cold-worked

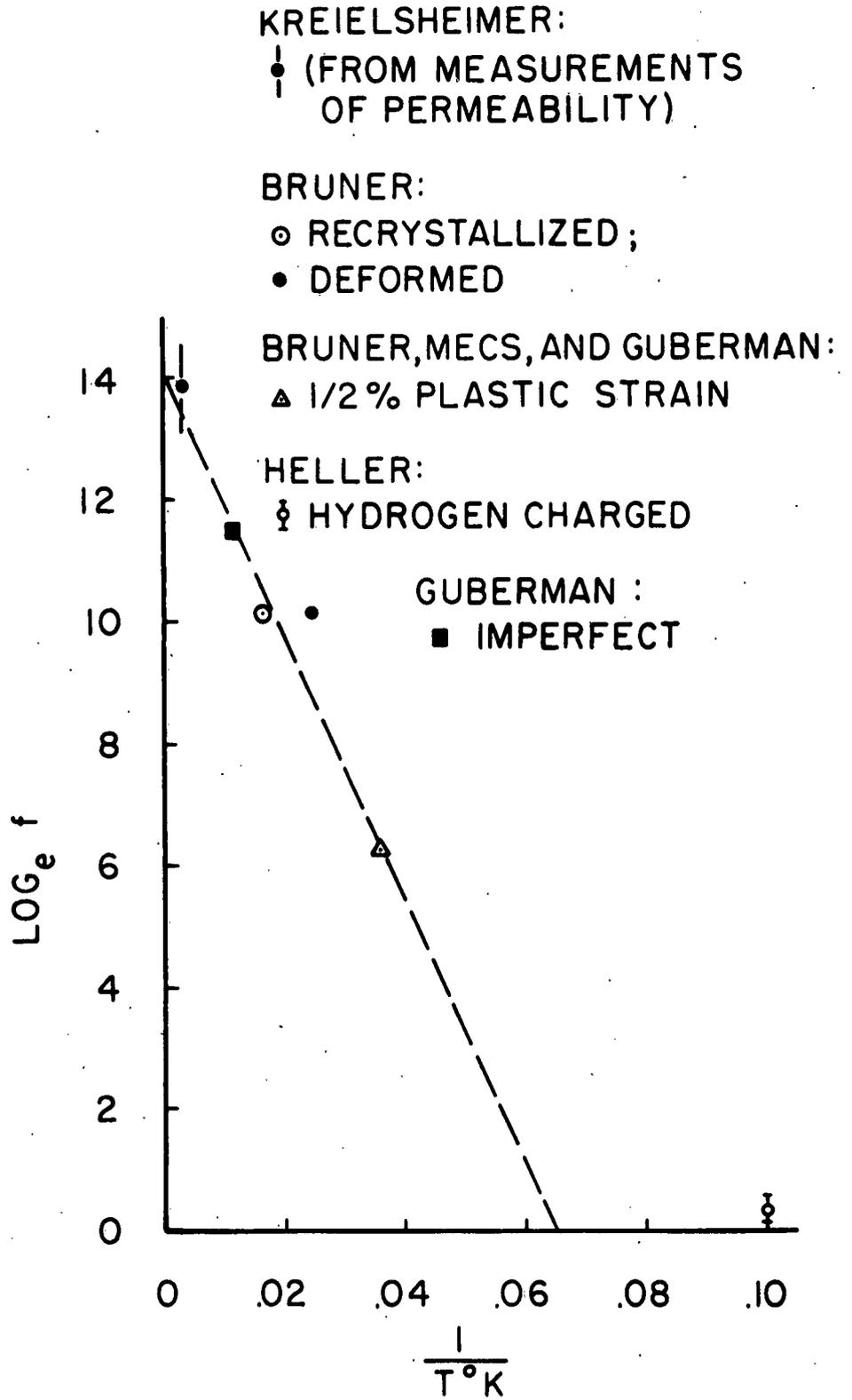


Fig. 30 LOG OF FREQUENCY OF MEASUREMENT VS. INVERSE SHOULDER TEMPERATURE.