RICE UNIVERSITY

DISLOCATION DAMPING IN MAGNESIUM MONOCRYSTALS

by

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ABSTRACT

By employing a micro-strain gage, hysteresis loops were observed as a function of stress amplitude in magnesium single crystals from 83°K to 323°K, in the low frequency range (0.1 cps.).

Broad relaxation peaks were found around 180°K. Activation energies associated with the peaks were about 0.33 ev., with a frequency factor of $10^8$ cps.

The stress amplitude dependence of the decrement was analyzed according to the Granato-Lucke and Teutonico-Granato-Lucke theories. The experimental results support the Teutonico-Granato-Lucke concept of pinned dislocations.

A qualitative model is suggested to explain the symmetrical nature of the hysteresis loops, and the origin of the relaxation peak.
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INTRODUCTION

When subjected to an external cyclic stress, metal crystals experience an energy loss, a phenomena known as internal friction. A measure of internal friction can be defined as the ratio of the energy lost per unit volume per cycle to the total vibrational energy density. Thus:

\[ \Delta_T = \frac{W_{irr}}{W_T} \] (1)

where \( \Delta_T \) is termed the "decrement" and Wirr and \( W_T \) are as defined above.

Experimentally the decrement is found to be a function of stress or strain amplitude, prior deformation or history, temperature, frequency of applied stress, impurities, and crystal orientation. Comprehensive reviews of these experimental findings are presented by Lücke and Granato(1) and Roberts(2).

First proposed by T.A. Read(3), it is now generally accepted that this energy loss is due to the motion of dislocations. Theories relating the motion of dislocations to the decrement have developed from two basic concepts presented by J.S. Koehler(4) and A.S. Nowick(5).

Nowick pictured the loss mechanism to be a hysteresis type, in which dislocations are moved from one potential
minimum to another. Using this concept, Weertman and Salkowitz(6) constructed specific hysteresis models enabling semi-quantitative calculations of the modulus and decrement changes to be made. The assumption is made that a dislocation length under an applied stress must move over distances greater than \( b/c_0^{1/3} \), where \( b \) is the Burgers vector and \( c_0 \) is the atomic concentration. Granato and Lucke(7) refute this theory on the basis that the dislocations cannot move such large distances.

Koehler assumed an edge type dislocation to be pinned by impurities by the Cottrell(8) mechanism. Using an analogy between the motion of the dislocation under an applied stress and that of a string under forced damped vibrations, he developed a quantitative theory relating the decrement and modulus changes with the stress amplitude and applied frequency. Granato and Lucke(7), using Koehler's model, found that dislocations oscillating under an applied stress lead to two types of losses, one frequency dependent, and the other frequency independent. The latter occurs at frequencies in the kilocycle range and below. The first loss is due to the fact that the measurement is a dynamic one. Since the motion of the dislocation is opposed by some damping mechanism, such as proposed by Eshelby(9) and Liebfried(10), it causes a phase lag for an oscillating stress, such that the strain lags the stress. This type loss will not be discussed further in this paper.
The second type loss, frequency independent, is based on the following model: There is a dislocation network present in single crystals such as proposed by Mott\(^{(11)}\). The average distance \(L_n\) between nodes in this network is assumed to remain constant. Impurity atoms pin the dislocation along the length \(L_n\) by the Cottrell mechanism, with an average distance between these pinning points being \(L_c\). Under the action of an applied stress the dislocation bows out between the pinning points, causing dislocation strain in addition to the elastic strain. When the stress is large enough to break the dislocations away from the impurity atoms an increase in the dislocation strain is observed, causing a deviation from the linear stress-strain curve. When the stress is removed the line tension in the bowed dislocation causes it to return to its initial position in a different manner than bowing out. Thus a closed hysteresis loop is predicted, providing the stress necessary to activate a Frank-Read source\(^{(12)}\) is not reached. By predicting a stress-dislocation strain law, both for loading and unloading portions of the cycle, they are able to predict the dependence of the decrement on the stress amplitude. Their derived equation for the decrement is

\[
\Delta_1 = \frac{A_1}{T_0} \exp\left[-\frac{B}{T_0}\right]
\]

where \(A_1\) and \(B\) are constants for any one temperature, and
contain properties such as dislocation density, Burgers vector, shear modulus, $L_c$, $L_n$ and the Cottrell binding force. $\Upsilon_o$ is the stress amplitude of the observed hysteresis loop. In this derivation thermal effects were ignored.

Teutonico-Granato-Lucke\(^{(13)}\) revised the Granato-Lücke theory by accounting for thermal fluctuations and thermal breakaway from the impurities. Their derived decrement has the following temperature and stress amplitude dependence:

$$\Delta_1 = \frac{A^2}{\Upsilon_o^{1/4}} \exp[-U_o/KT - \Gamma/\Upsilon_o^{1/2}] \quad (3)$$

This particular derivation was for discrete pinning along the dislocation line. Other specific models, such as continuous pinning, are presented.

Using the torsional pendulum method developed by Cooke and Brown\(^{(14)}\), strains are readily computed, but the stress is unknown and can at best only be estimated. Since all quantitative theories of internal friction are derived in terms of stress amplitude the correlation between theory and experiment is difficult to evaluate.

Lucke and Granato\(^{(1)}\) suggest that in the low frequency range damping can be determined by direct measurements of the stress-strain curves and the hysteresis loop, but that no systematic investigations of dislocation damping in this frequency range are known. Roberts\(^{(2,15)}\) describes a technique whereby hysteresis loops are directly observable, in-
volving measurements of strains of the order of $10^{-6}$. In these measurements the state of stress is accurately known and the decrement can easily be evaluated over a frequency range of 0.001 to 1 cycle per second.

Roberts' measurements on zinc single crystals were in good agreement with both the Granato-Lucke and the Teutonico-Granato-Lucke theories as related to the stress amplitude dependence of the energy lost per cycle.

This work is an attempt to experimentally check the validity of the Teutonico-Granato-Lucke theory with respect to the dependence of the decrement of the hysteresis loops on stress amplitude and temperature.
EXPERIMENTAL PROCEDURE

Specimen preparation -

Extruded magnesium bar stock, 1/2 inch in diameter with a purity of about 99.95%, was used as the raw material*. From this stock cylindrical single crystals were grown in a graphite container under an argon atmosphere using the Bridgman\(^{(16)}\) technique. The crystals were 6 to 7 inches long and 1/2 inch in diameter. The growth rate was 3 cm./hr. through a temperature gradient of 15°C/cm. A spectrographic analysis of several of the specimens after growth is given in Table I.

Chemical etching and X-Ray analysis were both used to determine if the grown specimens were single crystals. The most successful etchant was found to be 5% glacial acetic acid in distilled water, with an etching time of the order of two minutes. Three Back-Reflection Laue patterns, as described by Barrett\(^{(17)}\), were taken on each crystal, one on either end and one in the middle after the crystal was rotated through 180° with respect to the cylindrical axis. Exposure times of 30 minutes were found satisfactory, using a silver target, 40 kvp., and a pinhole diameter of 0.025". Table II shows important crystallographic angles with respect to the cylindrical axis of the specimen, as determined by

*The author is indebted to the Dow Chemical Co., Midland, Michigan, for supplying the raw material and performing the spectrographic analysis.
the Laue patterns. These angles are considered accurate to within $\pm 2^\circ$.

The specimens tested were cut in half using a jewelers saw and mounted in aluminum grips using Shell Epon adhesive VI. The grips were cylindrical, with tapped threads in one end and bored at the other end to an internal diameter approximately 0.003" larger than the diameter of the crystal being gripped. This assured alignment of the grips with the specimen axis. Approximately 3/8" of each end of the specimen was gripped, with the smallest distance between grips for any specimen being 2.75". After curing the adhesive for one hour at 110°C in an open air furnace, which resulted in a crystal anneal of the same duration, the crystals were chemically polished by gently rolling in a 10% solution of HNO$_3$ in distilled water, followed by a rinse in distilled water and a warm air blast drying.

The average diameter of each crystal was determined using a micrograph, with the largest deviation from the mean being 0.003".

The bond strength of the adhesive was found to be greater than 500 pounds at room temperature and about 250 pounds in liquid nitrogen. The load applied during testing never exceeded 45 pounds. Once the adhesive was cured, it was necessary to bake it for one hour at 300°C if the grips were to be removed without damaging the specimen.
Stress measurement -

All tests were performed in simple tension on an Instron testing machine. The load was determined by a type "C" Instron load cell, with a range of 0 to 50 pounds. The signal from the load cell, amplified to a millivolt output, was simultaneously applied to the Instron recorder and the Y axis of a Moseley X-Y recorder. Using calibration weights, the load signal was found linear over the 50 pound range, with an error less than 0.1%. The Instron recorder recorded load versus time, which was linear in all cases, except when approaching the yield point of the specimen, and upon initial loading. These load time curves supplied the stress rate and the period to form a hysteresis loop. The reciprocal of the period was used as the applied frequency.

The dead load on each specimen was the sum of the bottom grip, the lower universal joint, the retaining plug, and the lower half of the capacitance gage. The total weight of these items was 0.18 pounds. The smallest load at which hysteresis loops were analyzed was approximately 4 pounds.

Strain measurement -

Strains were measured using a capacitance gage first described by Roberts\(^{2,15}\). Figure 1 shows a photograph of the gage mounted on a specimen. The gage employs six set screws for mounting to the crystal.

The capacitance of a parallel plate capacitor is given
by

\[ C = 0.225KA/d \]

(4)

where

\[ C \text{ = capacitance in picofarads} \]

\[ K \text{ = dielectric constant of medium between plates} \]

\[ A \text{ = area of ungrounded plate in square inches} \]

\[ d \text{ = separation of the plates in inches} \]

Since \( K \) and \( A \) are constant at any one temperature, the change in capacitance corresponding to a change in displacement is

\[ -\Delta C = 0.225KA \frac{Ad}{d^1d^2} \]

(5)

If the change in displacement is small, then \( d_1d_2 \) can be replaced by \( d^2 \). Thus, the change in displacement can be given by

\[ \Delta d = -\frac{d^2}{0.225KA} \Delta C \]

(6)

Using a Fielden Proximity Meter*, which can measure a minimum \( \Delta C \) of 0.01 picofarads full scale, with a plate area of 1 square inch and an initial spacing between plates of 0.010", displacements of the order of \( 10^{-8} \) inches can be measured. However, thermal and mechanical stability limit the useful sensitivity to about \( 10^{-6} \) inches displacement. For this work \( d_1 \) ranged from 0.010" to 0.020". The maximum displacement measured for any one hysteresis loop was of the

order of $10^{-4}$ inches, hence the approximation that $d_1d_2 = d_1^2$ is justified. As shown in Figure 2, a schematic of the measuring circuit, the output of the Proximity Meter was applied to the X circuit of the X-Y recorder. Thus, load-displacement curves were obtained directly and permanently recorded. To assure that no stray capacitance signal was picked up, it was essential that all component parts of the apparatus were grounded, including the grips on the specimen, and that a shielded tri-axial capacitance cable was employed from the gage to the Meter.

In order to accurately determine the absolute value of observed strain it was necessary to know $d$ in equation (6) to at least 0.0002". This initial displacement was extremely difficult to determine at room temperature, and impossible to determine at any other temperature. To alleviate this difficulty the initial slope of the load-displacement curve was taken to be the elastic modulus, corrected for temperature and orientation factors, using elastic constants found in the literature (18).

A steel specimen was employed as a standard. The modulus of a steel bar was determined to be $30.2 \pm 0.5 \times 10^6$ psi, using a dial gage extensometer and a drop weight testing machine. A section of this bar, with the same dimensions as the crystals, was mounted in the same fashion. With the gage on the bar a linear load-unload curve was observed. (See Figure 4). Hence for this particular displacement between gage plates
the value of displacement along the X axis was known. With the Proximity Meter balanced at the last reading, the gage was transferred to a magnesium crystal. Using the movable plate the initial separation was varied until the Proximity Meter yielded the same balance as with the steel specimen. By applying a small load, a linear load-unload curve for the magnesium specimen was observed. Since the X axis was now calibrated for displacement and the Y axis for load, the modulus of the magnesium specimen could be determined. Values of observed moduli using this method are compared in Table II with those calculated from known orientations and elastic constants.

Errors in strain measurements by taking the initial slope of the hysteresis loop as being modulus are of the order of 10%. However, since the decrement involves the ratio of two areas (See Figure 5) the errors in strain cancel out. Results reported in this work are of decrement versus stress amplitude, temperature, and frequency, all of which are known to a good degree of accuracy.

One obvious question which arises is whether the deviation from a linear stress-strain curve is due to bending in the specimen. Three experimental observations assure that this is not the case. First, with a different gripping system, which did not assure alignment of the crystal, bending was observed. This bending was slight enough so that it could not be detected visually, but was detected by the gage.
A load-displacement curve for this particular arrangement was apt to take any shape, including negative strain for positive load. Second, with the present gripping system a linear stress-strain curve at high load-displacement sensitivity was observed. Third, a linear stress-strain curve was observed at low strain sensitivity up to the macro-yield point. In addition the linearity of the system was checked prior to each test by employing the standard steel specimen.

To test the effects of the capacitor plates not being parallel, a test was performed on the steel specimen with an angle between the plates of approximately 5°. No deviation from linear stress-strain curves was observed. In all subsequent tests the plates were made as parallel as could be determined visually.

Temperature control -

Figure 3 shows a schematic of the low temperature apparatus. The specimen and gage were surrounded by a brass bucket, made from a 3 1/2" O.D., 1/8" wall thickness, 15" length of yellow brass tubing, with a brass plate silver-soldered to the bottom. The upper portion of the tube was left open to the atmosphere to prevent constriction of the capacitance cable and the load sensing device. The tube served the purpose of isolating the capacitance gage from the cooling medium, isopentane. If the gage were submerged directly in the isopentane local variations in the dielectric
constant of the medium greatly affected the functioning of the gage. A stainless steel Dewar was used to contain the isopentane, and liquid nitrogen was introduced through cooling coils suspended in the Dewar. Nylon insulated Nichrome heating elements were wound around the cooling coils. An iron-constantan thermocouple suspended in the bath supplied a signal to a Wheelco controller, the output of which was applied to a saturable reactor which heated the nichrome elements. Thus, at any one temperature a balance between the rate of flow of liquid nitrogen and the power to the heating elements was obtained. In this fashion the bath was kept at a constant temperature until the supply of liquid nitrogen diminished (approximately 24 hours for a 110 liter container during one testing period). Three iron-constantan thermocouples resting against the specimen were used to determine specimen temperature. After reaching equilibrium the maximum gradient between the top and bottom thermocouples was 0.5°C. A gradient was expected since the brass bucket was open at the top. During the course of any one test no temperature variation was detectable with the three thermocouples. The soaking time required for the specimens to reach equilibrium temperature after the cooling medium had reached that temperature was of the order of one hour. The time required for testing at any one temperature was of the order of 10 minutes. The lowest temperature obtainable using the isopentane was -157°C. Liquid air was used as the
surrounding medium for tests at -189°C. Since the bucket was open at the top, when liquid nitrogen was used as the surrounding medium it condensed air from the atmosphere thus defeating the purpose of the bucket.

**Hysteresis loops**

Upon initial loading of each crystal the stress-strain curve was linear for small applied loads. Hysteresis loops were not observed until after the specimen was subjected to a slight amount of pre-strain. The specimens were pre-strained until the hysteresis loops were judged to be large enough to be easily measured. The amount of pre-strain for a crystal was of the order of 1%. After a pre-strain of the order of $10^{-4}$ the capacitance gage would become loose, evidenced by completely unstable deflections in the X-Y recorder. Tightening of the set screws would then set up a local stress field which resulted in an increase in the non-linearity of the stress-strain curve and a lowering of the yield point of the order of 10%. The crystal was then strained until the previous yield was reached and easy glide was again observed.

As soon as possible after straining, the tests on the crystals were performed. This time varied from 30 minutes to two hours. A check of the recovery of the decrement for crystal 61 as a function of time was performed at room temperature.
All tests were performed at a constant cross-head speed of 0.02" per minute, except for crystal 62, for which the cross-head speeds were 0.02, 0.05 and 0.10 inches per minute. The usable limits of the cross-head speed were found to be 0.20 and 0.005 inches per minute over the load range used. The upper limit was dependent on the ability of the X-Y recorder to follow, while the lower limit was a function of the drift-stability of the Proximity Meter. The drift stability was dependent on temperature variations. The maximum time to complete any hysteresis loop was less than 30 seconds.

Drift was measured as a function of time before each test, with observed values of $5 \times 10^{-6}$ inches per 5 minutes being representative. This drift was apt to be either in the positive or negative direction of strain, and constituted the largest error in the measurement of the decrement. If the drift were in the positive direction the loop tended to be larger than expected, and vice versa. Any hysteresis loops that showed overlapping, or were not completely closed, were not analyzed.

Temperature increments were of the order of 20°C, but varied among the crystals tested. Crystals 31, 42 and 61 were tested in decreasing temperature intervals after straining at room temperature. Crystal 62, the other half of 61, was strained to 5% in liquid nitrogen, brought to room temperature in order to attach the gage, then immediately lowered in temperature. The time involved in bringing the cry-
stal from $-200^\circ C$ to room temperature and then back to $0^\circ C$ was 23 minutes. Total time to reach $-100^\circ C$ was 36 minutes. Crystal 62 was then tested with increasing temperature up to $-10^\circ C$.

After testing over the desired temperature range, each crystal was etched to determine the extent of recrystallization. Crystals 31 and 42, each, showed two recrystallized areas, approximately 0.020" in diameter, at set screws indentations. Crystal 62 showed twin bands formed in the area of both grips, but at least 1/2" away from the gage section. No visible deformation was present in crystal 61.
ANALYSIS

Figure 5 shows a typical page of hysteresis loops recorded at constant temperature. The zero stress actually corresponds to 0.44 psi due to the weight of the grips and gage. A zero control on the recorder was used to reposition the recording pen to keep the loops from overlapping. Without this control each successive loop would be contained within the previous loop.

The first loop on the page is not completely closed, exhibiting plastic deformation of the specimen. This plastic deformation was termed prior strain, \( \gamma_p \).

The vertical scale measured load \( P \) directly, and the horizontal scale measured changes in displacement. By taking into account the cross-sectional area, \( A \), and the orientation of the specimen the vertical scale could be calibrated in resolved shear stress units:

\[
\tau_0 = \frac{P}{A} \cos \theta \cos \phi
\]  

(7)

\( \theta \) and \( \phi \) are given in Table II for each crystal, \( P \) is in pounds, and \( A \) in square inches.

From the known initial modulus, the gage length, and the orientation factor, the horizontal scale could be calibrated in terms of resolved shear strain.

The cross-sectional area and the gage length (1.00 inches) were assumed independent of temperature for strain calibrations.
The errors introduced by these assumptions are far less than the error associated with determining the initial slope of the hysteresis loop.

\( \tau_0 \) and \( \gamma_0 \) are the shear stress and shear strain amplitudes, respectively, associated with each hysteresis loop. \( \gamma_B \) is the deviation from the initial modulus, and is a direct measure of the dislocation strain. The stress rate, determined from the Instron recording of load versus time, gives a direct measure of the period necessary to form a hysteresis loop for a particular value of stress amplitude. The reciprocal of the period necessary to form the loop is the applied frequency, \( \omega \). Thus, if a loop had a value \( \tau_0 \) associated with it, the period to form the loop would be the stress amplitude divided by the stress rate. The reported values of stress rate were obtained from plots of stress amplitude versus period of formation of hysteresis loops. The stress rate remained constant over the temperature range covered for each particular crystal. Note that a different definition of frequency is employed here since the usual definition is the reciprocal of the time required for one cycle of positive and negative stress.

The area within a closed loop is the energy lost per cycle per unit volume, \( \text{Wirr} \). The total area under the loading portion of the loop is the total vibrational energy per unit volume, or the total work done on the crystal, \( W_T \). The ratio of these two areas is the decrement, \( \Delta_T \), which must al-
ways be less than unity. Since the calibrations for determining Wirr and \( W_T \) in work units is identical, the decrement, being the ratio of the two, is insensitive to errors in strain calibrations. Wirr and \( W_T \) were measured by planimetering. Figure 6 shows a representative plot of Wirr and \( W_T \) versus stress amplitude. Figure 7 is a plot of log Wirr and log \( W_T \) versus log \( T_0 \). This linear relationship was observed for all tests. The significance of this empirical observation will be discussed later. It is mentioned here, for the decrement as a function of temperature at a constant stress amplitude was determined from these curves. These plots had the effect of reducing the error in decrement determinations due to planimeter errors.
RESULTS

The hysteresis loops appeared symmetrical about the axis defined by the reduced modulus line (Figure 5). The initial portion of the loading side of the loop corresponds with the initial unloading portion at the peak. Prior strain and temperature had no obvious effect on the nature of the symmetry, although the shape of the loops changed. No tests were performed on unstrained crystals, so that the above observations apply to the prior strain range of $10^{-4}$ to $10^{-2}$. Due to this symmetry the reduced modulus line divided the loops into two nearly equal areas, so that

$$\text{Wirr} \approx 2W_T - \gamma_0 T_0$$ (8)

The general observations can be divided into the following topics:

(1). Decrement as a function of prior strain

(2). Stress strain relationships

(3). Decrement as a function of stress amplitude

(4). Decrement as a function of temperature

(1). Although no systematic investigation was made of the effect of prior strain and anneal time on the decrement, some significant data was obtained. Figure 8 shows the increase of the decrement with prior stain, at a constant value of stress amplitude. At a value of $\gamma_p = 2.8 \times 10^{-3}$ the decrement appears to have reached a maximum value. A slight decrease in the decrement was observed after a 16 hour anneal
at room temperature. At higher values of stress amplitude the annealing effect appears greater. No conclusion regarding this interesting result can be reached at this time. (This is due to the fact that in another annealing test at room temperature after a $\gamma_p = 0.68\%$, virtually no decrease in decrement was observed at values of $\tau_0$ from 70 to 20 psi over a period of 46 hours for crystal 42.) The decrement then increases with prior strain as before, although no apparent maximum is reached. As regards the hysteresis loops, the increase in decrement with deformation was a direct result of increasing non-linearity of the loading portion of the stress-strain curve with increasing prior strain. If the dislocation strain can be expressed by

$$\gamma_B = \rho b \bar{x}$$

where

$\rho$ = dislocation density in lines per sq. cm.

$b$ = Burger's vector

$\bar{x}$ = average distance over which the dislocations move,

then the dislocation strain is seen to be a direct function of dislocation density. The dislocation density is known to increase with plastic deformation$^{(6)}$, hence if $\bar{x}$ remains fairly constant then $\gamma_B$ will increase.

(2). An empirical relationship was found between the stress amplitude, $\tau_0$ and the strain amplitude, $\gamma_0$. Figure 9 shows the linear relationship between $\log \tau_0$ and $\log \gamma_0$. This
linearity was observed for all tests. For the relationship

$$\tau_o = c_3 \gamma_o^p$$  \hspace{1cm} (10)

developed constants $c_3$ and $p$ were determined. These values are reported in Tables III through VI. Errors in strain calibrations, of the order of 10%, entered into the determination of $c_3$. The errors in $p$ depended on the accuracy of the straight line through the data points. $p$ ranged from 0.710 to 0.978. $c_3$ had values from 0.40 to $11.0 \times 10^5$ psi. The adiabatic shear modulus of magnesium is about $2.6 \times 10^6$ psi. The instantaneous slope from equation (10) is given by

$$\frac{d\tau_o}{d\gamma_o} = pc_3\gamma_o^{p-1}$$  \hspace{1cm} (11)

which can be equated to the shear modulus. For a value of $p = 0.75$, and $c_3 = 10^5$ psi, $\gamma_o$ must equal $7 \times 10^{-7}$. Thus at very small values of strain, greater than $7 \times 10^{-7}$ in this example, the deviation from the adiabatic modulus is observed.

It is noted that the previous law includes elastic and plastic strain. That is $\gamma_o = \gamma_E + \gamma_P$, so that the previous calculation is only intended as a cut-off point for applicability of the law with respect to the assumption that there is a stress level below which dislocation bowing does not occur. In this measurement this stress level may be defined as that stress level below which no energy loss can be detected. In truth, there still remains out of phase plastic strain with respect to the elastic strain of the order
of $10^{-2}$ or less.

The deviation from the initial modulus is due to dislocation strain, $\gamma_B$. In the above example the dislocation strain contribution occurs at strain amplitudes of $7 \times 10^{-7}$. This corresponds to a stress amplitude of 2.4 psi.

The reduced modulus, $G'$, is given by

$$G' = \frac{\tau_0}{\gamma_o} = C \frac{1}{p} \frac{p-1}{p}$$

(12)

Since $p$ is always less than unity, $G'$ decreases with increasing stress amplitude. Again using $p = 0.75, C = 10^5$ psi and $\tau_0 = 50$ psi (e.g. Table VI, -102°C), the reduced modulus becomes $1.25 \times 10^6$ psi. Hence the apparent modulus decreases from $2.6 \times 10^6$ psi to $1.25 \times 10^6$ psi as the stress increases from 2.4 to 50 psi.

The total strain, $\gamma_o$, is the sum of the elastic strain and the dislocation strain. If $G$ is the true modulus, then the elastic strain is given by $\tau_o/G$. Hence the dislocation strain can be expressed as

$$\gamma_B = \gamma_o - \frac{\tau_o}{G} = \tau_o \left( \frac{1}{G}, -\frac{1}{G} \right)$$

(13)

The largest value of $\gamma_B$ observed was of the order of $2 \times 10^{-5}$. From equation (9) an estimate of the average distance moved by the dislocations can be made. For $b = 3.2 \times 10^{-8}$ cm., and $p = 10^8$ lines/cm$^2$, $\bar{x} \approx 6 \times 10^{-6}$ cm., which is of the order of 200 atomic spacings.

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No simple relation between $\tau_0$ and $\gamma_B$ could be found, except as expressed in equation (13). Figure 10 shows a typical plot of $\log \tau_o$ versus $\log \gamma_B$. Roberts (2), using zinc single crystals, found $\tau_o = k \gamma_B^n$. In this investigation the plots of $\log \tau_o$ versus $\log \gamma_o$ showed better linearity than those of $\log \tau_o$ versus $\log \gamma_B$.

(3). As previously reported an empirical relationship was found describing $W_{irr}$ and $W_T$ as functions of $\tau_o$ (See Figure 7). Thus:

$$W_{irr} = C_1 \tau_o^m$$

$$W_T = C_2 \tau_o^n$$

and values of $C_1$, $C_2$, $m$ and $n$ are given in Tables III through VI. $C_1$ and $C_2$ are expressed in psi. to the powers $1-m$ and $1-n$ respectively, and are dependent upon calibration errors. However, the ratio $C_1/C_2$ is independent of the calibration as explained before. Using equations (14) and (15) the decrement can be expressed as

$$\Delta_T = \frac{C_1}{C_2} \tau_o^{m-n}$$

$m$ is always greater than $n$ so that the decrement is an increasing function of stress amplitude. The ratio $C_1/C_2$ has units of $(\text{psi})^{n-m}$ so that the decrement is unitless. From the tabulated data, although the scatter is large, $C_1$ and $m$ are seen to be more temperature dependent than $C_2$ and $n$. $m$ had values from 2.44 to 4.88 with 2.65 being representative.
n ranged from 2.05 to 2.51, with 2.40 about average. Hence 
m-n was approximately 0.25 or 1/4, with a maximum of 2.46 
and a minimum of 0.11. C1/C2 varied from 0.20 to 0.00002(psi)^n-m.

The stress dependence of the decrement was also analyzed 
according to the Granato-Lucke (G-L) and the Teutonico-
Granato-Lucke (T-G-L) theories. These theories are briefly 
described in Appendix A and B, respectively. In the de-
velopment of these theories it is important to note the 
difference in the definition of the decrement as compared with 
equation (1).

\[ \Delta_1 = \frac{W_{rr}}{W_1} \quad (17) \]

Wirr is as defined before and W_1 is the strain energy den-
sity per cycle, given by the triangular area under the re-
duced modulus line in Figure 5. W_1 is hence given by 
1/2( T_o Y_o ). The only difference noted between \( \Delta_1 \) and \( \Delta_T \) 
is that \( \Delta_1 > \Delta_T \) since \( W_1 < W_T \).

For the G-L theory log \( (\Delta_1 \ T_o) \) versus \( T_o^{-1} \) should be 
linear. This linear relationship was observed twice, both 
times at -190°C. (See Figure 11). At other temperatures the 
scatter in the raw data was such that a straight line could 
be forced through the points, but by using plots of Wirr and 
\( W_1 \) versus \( T_o \) to determine \( \Delta_1 \), errors in planimetering were 
reduced with the resultant G-L plots showing curves that were 
consistently concave upwards. For the two fits at -190°C, 
the values of the slopes and the intercepts are shown in
Tables III and VI.

For the T-G-L theory a plot of $\log(\Delta_1 \tau_o^{1/4})$ versus $\tau_o^{-1/2}$ should be linear. At all temperatures, except $-190^\circ C$, this linear relationship was observed. Again the scatter in the raw data did not dictate a linear relationship, but after correcting as explained above the linear agreement was excellent. Typical curves are presented in Figure 12. Figure 13 shows the T-G-L analysis for one of the tests at $-190^\circ C$. The non-linearity is slight, but obvious, even from the raw data. The values of slopes and intercepts are presented in Tables III through VI, along with the related values computed from the theory, outlined in Appendix B.

The empirical relationships presented in equations (10) and (14) are in close agreement with the predicted dependence of the decrement on the stress amplitude according to the T-G-L theory.

Tables III through VI contain, in addition to previously described data, the time lapse between tests, the temperatures of the tests, and the maximum and minimum stress range over which the hysteresis loops were analyzed.

One additional interesting result is the stress amplitude necessary for dislocation bowing to occur as a function of temperature. Although subject to errors of the order of 25%, this variable was measured by taking the maximum amplitude available for which the loops appeared to have zero area,
i.e., retrace on loading and unloading. These values of stress, designated $\tau_B$, are presented in Tables III through VI. $\tau_B$ shows a definite tendency to increase with decreasing temperature. In all four cases it seems to reach a minimum value between $-60^\circ C$ and $0^\circ C$. $L_c$, in all the cases, was a minimum at the lowest temperatures and, in general, seemed to pass through a maximum value at the same temperature that $\tau_B$ was a minimum.

By assuming a value of stress amplitude and determining the decrement at that value over the range of temperature, using the plots of Wirr and $W_T$ versus $\tau_o$ obtained at each temperature, a curve of $\Delta_T$ versus temperature was obtained. Figures 14 through 17 show these curves for the four crystals tested. Since the stress rate was constant over the temperature range, each hysteresis loop associated with a particular stress amplitude has a certain period of formation. Thus, the curves drawn for different stress amplitudes correspond to curves of different frequencies. The temperatures associated with the peaks, or maxima, of these curves can be seen to be shifting towards higher temperatures as stress amplitude, and hence period, is decreased. In other words, increasing frequency causes an increase in peak temperature, in agreement with the phenomenological equation associated with relaxational mechanisms:

$$\omega = \nu_0 \exp[-Q/kT]$$

(18)
where

\[ \omega \] = applied frequency

\[ v_o \] = natural frequency

\[ Q \] = activation energy

\[ k \] = Boltzmann constant

\[ T \] = Absolute temperature

By finding the centroids of the areas under the curves, the peak temperatures for each curve, and hence each frequency, could be determined. The results were analyzed according to the predicted linear plot of \( \log \omega \) versus \( T^{-1} \). The slope of the line yields \( Q \) and the intercept at \( 1/T = 0 \) yields \( v_o \). In each case this linear relationship was found, and the values of activation energy and natural frequency were determined to be:

<table>
<thead>
<tr>
<th>Crystal</th>
<th>( Q ) (ev)</th>
<th>( v_o ) (sec(^{-1}))</th>
</tr>
</thead>
<tbody>
<tr>
<td>31</td>
<td>0.10</td>
<td>20</td>
</tr>
<tr>
<td>42</td>
<td>0.21</td>
<td>( 9 \times 10^5 )</td>
</tr>
<tr>
<td>61</td>
<td>0.49</td>
<td>( 3 \times 10^9 )</td>
</tr>
<tr>
<td>62</td>
<td>0.16</td>
<td>( 3 \times 10^3 )</td>
</tr>
</tbody>
</table>

In addition to the page of hysteresis loops versus stress amplitudes obtained for various temperatures for crystal 62, hysteresis loops were measured at a constant stress amplitude for three different cross-head speeds, hence three different frequencies. The decrement versus temperature curve obtained by directly varying the frequency in this fashion is shown in Figure 18. The result-
ing plot of log $\omega$ versus $T^{-1}$ is shown in Figure 19. The activation energy and natural frequency obtained are 0.33 ev and $4 \times 10^8$ sec.$^{-1}$, respectively.
DISCUSSION

Large hysteresis loops, and hence large values of decrement were obtained only after permanently straining the crystal to about 1%. The dislocation density, \( \rho \), increases quite rapidly with deformation through mechanisms such as the Frank-Read\(^{(2)}\) source. Since all quantitative descriptions of the decrement vary directly as \( \rho \) the decrement should be expected to increase with increasing deformation.

Since easy glide\(^{(8)}\) is observed in hexagonal crystals, the factor \( L_n \) in the G-L and T-G-L theories should remain constant with small strains.

Another simple mechanism described by Roberts\(^{(2)}\) can be used to explain the above observation. Initially, in zinc single crystals grown from the melt, a course dislocation network with long loops lengths is observed near the surface of a cylindrical specimen. The center contains a finer network with shorter loop lengths. These dislocations are assumed to be pinned by a distribution of impurities. Under an applied stress these dislocations may bow out until the stress necessary to break away from impurity atoms is reached. The stress necessary to activate a Frank-Read source is given by:

\[
\tau_a = \frac{\alpha Gb}{L} \tag{19}
\]

where \( G \) is the shear modulus, \( b \) the Burger's vector, \( \alpha \approx 1/2 \) and is assumed a constant, and \( L \) is the length of the dislo-
cation line. If the stress necessary to activate a source is less than the stress required for breakaway, then upon breakaway yielding of the specimen will be observed. If the freshly generated dislocations meet obstacles, such as tilt boundaries or oxide layers on the surface, pile-ups will occur. In this fashion the source may become hardened, requiring a higher stress for generation. However, the higher stresses may now be sufficient to cause breakaway of shorter loops from impurities without generating new loops. The initial micro-yield stress for crystal 61 was 28 psi, which suggests lengths, \( L \), of \( 1.5 \times 10^{-3} \) cm, are acting as sources. After a strain of \( \gamma \), the micro-yield stress increased to 50 psi. The effective length of generation then becomes \( 8 \times 10^{-4} \) cm. If breakaway from impurities can occur before this stress is reached then hysteresis will occur according to the G-L mechanism. For a stress amplitude of 25 psi, the decrement increased from approximately 0.04 to 0.12 over this deformation range.

The empirically developed equations (12) and (13) lack theoretical application. From equation (12), since \( p \) is less than unity, the reduced modulus becomes infinite as stress approaches zero. Theoretically, the reduced modulus should approach the elastic modulus as the stress approaches zero. Using \( G = 2.6 \times 10^6 \) psi, \( c = 105 \) psi, and \( p = 3/4 \), the value of stress below which the equation obviously does not apply is 5.7 psi.
Although Wirr and $W_T$, as given empirically in equations (14) and (15), both approach zero with stress amplitude, their ratio remains defined. $m$ is greater than $n$, and hence Wirr approaches zero more rapidly than $W_T$.

The stress amplitude dependence of the decrement as predicted by T-G-L, equation (3), appears correct over the temperature range observed. At $-190^\circ C$ the decrement seems to obey the relationship described by G-L, equation (2). Although it is not apparent from equations (2) and (3), T-G-L state that equation (3) should be equivalent to equation (2) at absolute zero temperature. In this light, it is not peculiar that the G-L relationship occurs at $-190^\circ C$ ($85^\circ K$).

According to the T-G-L concept, a hysteresis loop will not be observed until a dislocation breaks away from impurity pins and bows out. If the average distance between pins, $L_c$, decreases along the dislocation line, then the stress necessary for breakaway, $\tau_B$, will increase. As previously stated this concept was satisfied. From the tabulated data $\tau_B$ appears to be a minimum when $L_c$ is a maximum, and vice versa. Of course, the absolute value of $\tau_B$ depends on factors such as the interaction energy between dislocations and impurities, the type of impurities, the dislocation network within the crystal and the impurity content. For a test over the temperature range, though, all these factors stayed constant for each crystal, with just the value of $L_c$
changing.

This concept can explain the annealing effect observed. As fresh dislocations are generated the average value \( L_c \) should increase, since the concentration of pins on the new dislocations will be less than the average \( L_c \) before deformation. At a certain value of stress amplitude the dislocation strain becomes greater than before deformation. However, if the crystal is now annealed, impurities should migrate to the new dislocations and in effect decrease \( L_c \). With a smaller value of \( L_c \) the stress amplitude will have to be increased to give the same value of dislocation strain observed prior to annealing, for some of the dislocations will now require higher stresses for breakaway. In other words, at the same value of stress amplitude, fewer dislocations will break away after anneal than before, and a lower value of dislocation strain will occur. This has the effect of decreasing the area within the hysteresis loop, thus decreasing the decrement.

The equation governing the equilibrium number of impurities on the dislocations is given by

\[
\frac{b}{L_c} = C_o \exp\left[\frac{U_o}{KT}\right]
\]

(20)

\( U_o \) is the interaction energy between the impurity and the dislocation, and \( C_o \) is the atomic concentration. As temperature increases \( L_c \) should increase. However, as temperature increases the rate of diffusion will increase. The value of
Lc will be a balance between these two controlling factors. However, the diffusion coefficient and the interaction energy will be dependent upon the impurities involved. For the broad spectrum of impurities in this work a qualitative prediction of the dependence of Lc on temperature can not be made.

If Lc is assumed to be \(3.2 \times 10^{-5}\) cm. (e.g. Table IV), and \(C_o = 2 \times 10^{-4}\), at room temperature \(U_o\) will be 0.042 ev. \(U_o\) is extremely dependent on the value of \(C_o\). If \(C_o\) is \(10^{-5}\), then \(U_o\) would become 0.12 ev. From the spectrographic analysis given in Table II, it can be seen that the actual concentration of impurities is not accurately known. The calculated values of \(C_o\) were determined using the largest values of weight percent given. Although a direct comparison between magnesium and zinc is not correct, it is interesting to note that Roberts\(^2\) found values of \(U_o\) of the same magnitude in zinc crystals.

The maximum decrements observed were extremely large (0.30) compared with decrements reported in the literature\(^1\) (\(10^{-3}\) to \(10^{-2}\)). The implication is that 30\% of the energy input is lost through an internal dissipative mechanism. Caswell\(^{21}\) reports decrements of the order of \(10^{-3}\) in polycrystalline magnesium, with an internal friction peak at a temperature of 20°K.

The large decrements observed are believed due to the fact that large dislocation bowing and breakaway is measured in these experiments. The experiments by others are usually
of small plastic strain amplitudes and hence gross dislocation breakaway does not occur.

The results of the decrement versus temperature curves indicate a relaxation mechanism. The peaks occurred at $210^\circ K$ for crystals 31 and 61, $170^\circ K$ for crystal 62, and $150^\circ K$ for crystal 42. No relation between the peak temperatures and variables associated with different crystals could be found. The broad feature of the peaks is thought to be due to the superposition of several peaks, each associated with a different type impurity. Appendix C shows the activation energy determined by the broad peak shift should approximately represent the average of the activation energies associated with the individual peaks. Thus equation (18) can be used for calculating $Q$ and $v_0$. Varying the frequency indirectly by varying the stress amplitude resulted in low values of $v_0$, presented on page 28. The values of $Q$ and $v_0$ determined by directly varying the frequency seem to be the most reasonable. However, the main point is not the values obtained, but that relaxation peaks were observed.

The decrement increased directly with frequency as seen in Figure 18. T-G-L predict the decrement to vary inversely with frequency. In the light of a qualitative modification of the T-G-L theory which is suggested below, it is believed a relaxation loss will arise, hence the frequency dependence of the T-G-L theory can not be correct, but the decrement will show a maximum as a function of frequency.$^{(22)}$
T-G-L predict a non-symmetrical hysteresis loop, in that the unloading portion should be linear. Hysteresis loops measured in zinc by Roberts\(^{(2)}\) and those in this work are in direct disagreement with this prediction. To explain the symmetrical nature of the loops the following model is proposed: The concept of thermal and mechanical breakaway of dislocations from pinning points is assumed valid. In addition to impurities along the dislocation lines there is a scattering of impurities on the slip planes. If the dislocation breaks away at high velocities, of the order of the speed of sound, the kinetic energy associated with the dislocation is such that as it passes by the impurities scattered in the lattice it is just slightly perturbed. As the dislocation approaches the equilibrium bowed-out position, dictated by the stress amplitude, its velocity decreases. The dislocation then moves at a micro-creep rate, becoming pinned by the scattered impurities. As the stress is relaxed, line tension in the bowed dislocation must reach a value large enough to enable it to break away from the new pinning agents. In this fashion, the unloading portion of the hysteresis loop should be identical with the loading portion. Quantitative predictions from this model have yet to be worked out. This modification of the T-G-L theory should still give rise to the same type of stress dependence of the decrement provided the energy loss is negligible during micro-creep, which seems reasonable. A relaxation peak will then arise, which is not
evident from the T-G-L theory. For a simple relaxation type loss the magnitude of the decrement is not expected to rise when the frequency is increased to give a peak temperature shift to higher temperatures. The origin of this increase in decrement, as shown in Figure 19, is not known, but may be associated with the background and not the relaxation process.
The pinned dislocation concept of mechanical damping as presented by Teutonico-Granato-Lucke is supported by the following observations:

1. Proper stress dependence of the decrement
2. Stress necessary for initial dislocation breakaway is directly related to the concentration of pins along the dislocation
3. Shift to the stress dependence of decrement predicted by Granato-Lucke at low temperatures (85°K)

The two observations in disagreement with the Teutonico-Granato-Lucke theory are:

1. Symmetrical nature of hysteresis loops
2. Variance of the decrement directly with frequency and temperature

In light of these observations, it is believed that the model of pinned dislocations bowing out on the slip plane due to thermal and mechanical breakaway is a valid concept, and can be used to describe damping phenomena, provided the concept that the bowed out dislocation becomes pinned again before returning to its original position, so that a relaxation loss is expected.
SUGGESTIONS FOR FURTHER WORK

This investigator intends to further investigate hysteresis loops in controlled compositions of aluminum-magnesium alloys from liquid helium to room temperature. From such an investigation it is hoped to further clarify the phenomena of internal friction as regards temperature and impurities.
A. Outline of Granato-Lucke theory

In the Granato-Lucke theory the frequency independent loss, $\Delta_1$, is calculated by assuming the "bowed" dislocations cause strain in addition to the elastic strain. Prior to breakaway from impurity atoms, which pin the dislocation as described on page 2, the stress-strain relationship is linear. If the distance $L_c$ is constant throughout the network, then upon catastrophic breakaway from the pinning points a sudden increase in strain occurs with no increase in stress. Upon relaxing the stress, the line tension in the bowed dislocation returns it to its initial position. Thus the unloading portion of the predicted hysteresis loop is linear.

Using the analogy between a damped vibrating string and an edge type dislocation under an oscillating shear stress, they are able to solve the differential equation of motion. The applied stress is assumed to be $\tau_0 \cos \omega t$.

By calculating the dislocation strain associated with a distribution of loop lengths and assuming the dislocation collapses to its initial position in a linear fashion with stress, Granato and Lucke are able to calculate the area within their predicted hysteresis loop. Hence,

$$\text{Wirr} = \int_0^{\tau_0} (\gamma_2 - \gamma_1) \, d\tau$$

where $\gamma_1$ is the dislocation strain associated with the load portion of the loops and $\gamma_2$ is the dislocation strain associated...
ted with the unloading portion.

Assuming that the total vibrational energy per unit volume per cycle is \( \tau_o^2/G \), they then define the decrement as

\[
\Delta_1 = \frac{2W_{irr}}{\tau_o^2/G}
\]

Performing the required integration, the final result is

\[
\Delta_1 = \frac{4(1-\nu)}{\pi^3} \left( \frac{\rho L_n^3}{L_c} \right) \exp \left( -\frac{B}{\tau_o} \right)
\]

where

\( \nu \) = poisson's ratio

\( \rho \) = dislocation density (lines/cm²)

\( L_n \) = average length between nodal points (cm.)

\( L_c \) = average length between impurities (cm.)

\( b \) = absolute value of Burger's vector (cm.)

\( B = \frac{\pi f_m}{4 b L_c} \)

\( f_m \) = Cottrell binding force (maximum value)

The theory predicts a linear relationship between \( \log (\Delta_1 \tau_o^-1) \) and \( \tau_o^-1 \). The slope of the line is \( B \) and the intercept at \( 1/\tau_o = 0 \) is \( \frac{4(1-\nu)}{\pi^3} \left( \frac{\rho L_n^3}{L_c} \right) B \). Since \( L_c \), \( f_m \) and \( \rho L_n^3 \) are unknowns, equation (20) is used to solve for \( f_m \) and \( L_c \). This equation gives the concentration of impurities along the dislocation line. \( U_o \) is assumed equal to \( f_m b \). Thus, the experimentally determined slope \( B \) can be solved simultaneously with equation (20) for \( U_o \) and \( L_c \). The intercept can then be solved for \( \rho L_n^3 \). The determination of \( U_o \) from equation (20) is extremely temperature dependent, and, although the tempera-
ture of the test was known, it does not seem justifiable to use an equilibrium equation under non-equilibrium conditions. Thus, it is not known whether to use the temperature at which the test was run, or some higher temperature where an equilibrium concentration was established.

As an example of the above, taking $C_0 \approx 2 \times 10^{-5}$, and $T = 85^\circ K$, for the two tests performed, $U_0 \approx 0.04$ ev., and $L_c = 6.5 \times 10^{-6}$ cm. Larger values of $C_0$ give even smaller values of $U_0$ and $L_c$. The $U_0$ and $L_c$ values above are considered low by an order of magnitude, when compared with Roberts' data on zinc, and the $L_c$ values determined by the T-G-L theory in this work.

However, if $T$ is taken as $300^\circ K$, $U_0$ becomes 0.11 ev., and $L_c \approx 2 \times 10^{-5}$ cm. These values are now in the expected range. $\rho L_n^3$ then becomes $\approx 10^{-4}$, and for $\rho \approx 10^8$, $L_n \approx 10^{-4}$ cm.

From the above calculations the author feels that equation (20) cannot be used to solve for $U_0$ by using the temperature of the test.
B. Outline of Teutonic-Granato-Lücke theory \(^{(13)}\)

By assuming thermal breakaway from the pinning points, Teutonico-Granato-Lücke are able to modify the Granato-Lücke theory to give the following decrement:

\[
\Delta_1 = \left( \frac{1}{6} \right) (\gamma - 1) \frac{v_0}{\omega} \left( \frac{\pi}{1 + \log 2} \right)^{1/2} \left( \frac{2q \mu r}{T_0 b L_c^2} \right)^{1/4}
\]

\[\times \exp \left[ \frac{U_0}{kT} - (1 + \log 2) \left( \frac{8q \mu r}{T_0 b L_c^2} \right)^{1/2} \right] \]

where

\[
\gamma = \frac{L_n}{L_c}
\]

\[v_0 = \text{natural frequency of vibration}\]

\[\omega = \text{applied frequency of vibration}\]

\[h = \frac{L_c}{d}, \text{ where } d \text{ is a flexibility length giving}\]

the minimum distance along the dislocations of\n
points which move independently of one another\n
\[\mu = \frac{Gb^2}{2}\]

\[r_0 = \text{closest approach of impurities to dislocation center}\]

\[q = \text{constant relating Cottrell force to triangular force assumed by T-G-L,}\]

and the rest of the terms are as defined previously.

\[v_0 \text{ is given by:}\]

\[\frac{v_0}{c} \tan \left( \frac{L_c v_0}{2c} \right) = \frac{U_0}{\mu r_0^2} \] \(\text{(26)}\)

\[C \text{ is approximately equal to the velocity of transverse sound}\]

\[\text{-43-}\]
waves for both edge and screw dislocations.

Assuming, as do T-G-L, that \( \gamma-1 \approx 5 \), and \( r_o \approx b \), equation (25) can be reduced to

\[
\Delta_1 = \frac{A_2}{T_0^{1/4}} \exp \left( -\frac{U_o}{KT} - \frac{\Gamma}{T_0} \right) \exp \left( \frac{1}{2} \right)
\]

Hence at constant temperature, a plot of \( \log(\Delta_1 T_0^{1/4}) \) versus \( T_0^{-1/2} \) should be linear. The slope of the line is a direct measure of \( L_c \) and the intercept involves such terms as \( \nu_o, \omega, \rho L_n^2, \exp \left(-\frac{U_o}{KT}\right) \), and \( L_c \).

By again using equation (20), \( U_o \) can be determined. But, as described on page 34 and on page 42, the use of this equation is not justified.

The solution of equation (26) for \( \nu_o \) yields values of the order of \( 10^{10} \) cps. for all values of \( U_o \) from 0.05 to 0.20 ev., and \( L_c \) from \( 10^{-5} \) to \( 6 \times 10^{-5} \) cm., if \( C \) is assumed to be \( 4,600 \) M/sec. (20), \( r_o = b \), and \( \mu = 5.35 \times 10^7 \) ev./cm.

Since the T-G-L theory predicted the decrement to decrease with increasing frequency, and the opposite was observed, the intercept of equation (27) cannot justifiably be analyzed to yield values of \( \rho L_n^2 \).

The relation

\[
L_c = \frac{(7.2 \times 10^{-10})G^{1/2}}{\Gamma}
\]

was used to calculate \( L_c \). \( G \), in dynes/cm\(^2\), was corrected for temperature, and \( \Gamma \) is in \((\text{psi})^{1/2}\). \( L_c \) is given directly...
in cm. $b$ was assumed to be $3.21 \times 10^{-8}$ cm.\(^{(17)}\), and to remain constant with temperature.
C. Justification of the assumption that the activation energy determined from the peak shift of three superimposed relaxation peaks represents the average of the activation energies for each individual peak.

Assumptions:

1. The broad relaxation peak consists of three individual relaxation peaks.

2. All relaxation peaks can be described by \( \omega = \nu_0 \exp^{-Q/\kappa T} \)

3. \( \nu_0 \) is the same for all three processes.

Let two different frequencies, \( \omega_A \) and \( \omega_B \), be imposed on the system, and the temperatures of the individual peaks be \( T_1, T_2 \) and \( T_3 \) for \( \omega_A \), and \( T_1', T_2' \) and \( T_3' \) for \( \omega_B \).

If \( \omega_A > \omega_B \) then \( T_1 > T_1', T_2 > T_2', \) and \( T_3 > T_3' \). Hence:

\[
\omega_A = \nu_0 \exp^{-Q_1/\kappa T_1} = \nu_0 \exp^{-Q_2/\kappa T_2} = \nu_0 \exp^{-Q_3/\kappa T_3}
\]

\[
\omega_B = \nu_0 \exp^{-Q_1'/\kappa T_1} = \nu_0 \exp^{-Q_2'/\kappa T_2} = \nu_0 \exp^{-Q_3'/\kappa T_3}
\]

or

\[
\ln \left( \frac{\omega_A}{\omega_B} \right) = \frac{Q_1}{\kappa} \left( \frac{1}{T_1} \right) = \frac{Q_2}{\kappa} \left( \frac{1}{T_2} \right) = \frac{Q_3}{\kappa} \left( \frac{1}{T_3} \right)
\]

\[= \text{CONSTANT} = C\]

where

\[ \Delta \frac{1}{T_1} = \left( \frac{1}{T_1} - \frac{1}{T_1'} \right) \]

But the average peak shift is given by:

\[ \Delta \left( \frac{1}{T} \right) = \frac{1}{3} [ \Delta \frac{1}{T_1} + \Delta \frac{1}{T_2} + \Delta \frac{1}{T_3} ] \]

\[ = \frac{1}{3} \]
or
\[ \Delta \frac{1}{T_T} = -\frac{CK}{3} \left( \frac{1}{Q_1} + \frac{1}{Q_2} + \frac{1}{Q_3} \right) \]

Hence the total activation energy for the process is given by:

\[ Q_T = \frac{3Q_1Q_2Q_3}{Q_2Q_3 + Q_1Q_2 + Q_1Q_3} \]

Now if:

\[ Q_1 = Q_0 + \Delta_1 \]
\[ Q_2 = Q_0 + \Delta_2 \]
\[ Q_3 = Q_0 + \Delta_3 \]

So that

\[ Q_0 = \frac{Q_1 + Q_2 + Q_3}{3} - \frac{1}{3} (\Delta_1 + \Delta_2 + \Delta_3) \]

then

\[ Q_T = \frac{3Q_0^3 + 3Q_0^2(\Delta_1 + \Delta_2 + \Delta_3) + Q_0(\Delta_1\Delta_2 + \Delta_2\Delta_3 + \Delta_1\Delta_3) + \Delta_1\Delta_2\Delta_3}{3Q_0^2 + 2Q_0(\Delta_1 + \Delta_2 + \Delta_3) + \Delta_1\Delta_2 + \Delta_2\Delta_3 + \Delta_1\Delta_3} \]

But the initial assumption is that

\[ Q_T = \frac{1}{3}(Q_1 + Q_2 + Q_3) = Q_0 \]

So that \( \Delta_1 + \Delta_2 + \Delta_3 = 0 \)

Then

\[ Q_T = Q_0 + \frac{\Delta_1\Delta_2\Delta_3}{3Q_0^2 + \Delta_1\Delta_3 + \Delta_2\Delta_3 + \Delta_1\Delta_2} \approx Q_0 \]

Hence the average peak shift should approximately represent the average activation energy.
SYMBOLS

A - cross-sectional area of specimen, or area of gage plate
A₁ — intercept where \( \tau_0^{-1} \) goes to zero for a plot of log
\[ \Delta_1 \tau_0 \text{ versus } \tau_0^{-1} \]
A₂ — times \( \exp(-u_0/kt) \), intercept where \( \tau_0^{-1/2} \) goes to
zero for a plot of log \( \Delta_1 \tau_0^{1/4} \) versus \( \tau_0^{-1/2} \)
B — slope of line from plot of log \( \Delta_1 \tau_0 \) versus \( \tau_0^{-1} \)
b — Burger's vector magnitude
C — capacitance
C₀ — atomic concentration
C₁ — constant relating Wirr and \( \tau_0 \)
C₂ — constant relating \( W_T \) and \( \tau_0 \)
C₃ — constant relating \( \gamma_0 \) and \( \tau_0 \)
d — separation of gage plates
E — tensile modulus
fₘ — Cottrell force between an impurity atom and a dis-
location
G — shear modulus
G' — reduced shear modulus
K — dielectric constant
k — Boltzmann's constant
L — length of Frank-Read source
Lₙ — average distance between nodes of the dislocation
network
\( L_c \) - average distance between pinning points due to impurities along dislocation lines in the network

\( M_i \) - atomic weight of \( i \)th constituent

\( m \) - constant relating \( \text{Wirr} \) and \( \mathcal{T}_0 \)

\( n \) - constant relating \( W_T \) and \( \mathcal{T}_0 \)

\( p \) - constant relating \( \gamma_0 \) and \( \mathcal{T}_0 \)

\( P \) - tensile load

\( Q \) - activation energy associated with a relaxation peak

\( q \) - constant, equal to 3.07

\( T \) - temperature

\( U_o \) - Cottrell binding energy between an impurity atom and a dislocation

\( \text{Wirr} \) - irreversible work, or energy lost per cycle per unit volume, or area within a hysteresis loop

\( W_T \) - total vibrational energy density per cycle, or total area under the stress-strain curve

\( W_1 \) - reduced vibrational energy density per cycle, or area under the reduced modulus line

\( W_i \) - weight percent of \( i \)th constituent

\( \bar{x} \) - average distance a dislocation moves

\( \Delta_T \) - true decrement equal to \( \text{Wirr}/W_T \)

\( \Delta_1 \) - reduced decrement equal to \( \text{Wirr}/W_1 \)

\( \Gamma \) - slope of line from plot of \( \log \Delta_1 \mathcal{T}_0^{1/4} \) versus \( \mathcal{T}_0^{-1/2} \)

\( \gamma_p \) - permanent shear strain
\( \gamma_B \) - dislocation or bowing shear strain

\( \gamma_o \) - shear strain amplitude of hysteresis loop

\( \theta \) - angle between the normal to the basal plane and the cylindrical axis of the specimen

\( \phi \) - angle between the slip direction and the cylindrical axis of the specimen

\( \rho \) - dislocation density

\( \tau_B \) - shear stress necessary to cause dislocation bowing

\( \tau_o \) - shear stress amplitude of hysteresis loop

\( \tau_a \) - stress necessary to activate a Frank-Read source

\( \nu_o \) - fundamental frequency of a dislocation segment

\( \omega \) - frequency of stress oscillation
REFERENCES


ACKNOWLEDGEMENT

The author gratefully acknowledges Dr. John M. Roberts of the Department of Mechanical Engineering of the Rice University, without whose guidance, assistance and suggestions this work would not have been completed.

Helpful discussions with Dr. R. Azimow, and Dr. S. Okuda are also gratefully acknowledged.

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TABLE I
Spectrographic Analysis
(weight percent)

<table>
<thead>
<tr>
<th>Element</th>
<th>MP-1U</th>
<th>MS-2</th>
<th>MS-5</th>
<th>MS-8U</th>
<th>MS-10U</th>
<th>MP-12U</th>
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</thead>
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<td>0.0013</td>
<td>0.002</td>
<td>0.002</td>
<td>0.002</td>
<td>0.0024</td>
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<td>&lt;0.010</td>
<td>&lt;0.010</td>
<td>&lt;0.010</td>
<td>&lt;0.010</td>
<td>&lt;0.010</td>
</tr>
<tr>
<td>Cu</td>
<td>&lt;0.001</td>
<td>&lt;0.001</td>
<td>&lt;0.001</td>
<td>&lt;0.001</td>
<td>&lt;0.001</td>
<td>&lt;0.001</td>
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<tr>
<td>Fe</td>
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<td>0.0005</td>
<td>0.0008</td>
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<td>0.0033</td>
<td>0.0047</td>
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<td>&lt;0.001</td>
<td>&lt;0.001</td>
<td>&lt;0.001</td>
<td>&lt;0.001</td>
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<td>&lt;0.010</td>
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<tr>
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<td>0.003</td>
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<td>0.014</td>
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<td>C_o x 10^4</td>
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<td>1.27</td>
<td>2.08</td>
<td>2.54</td>
<td>2.53</td>
<td>5.75</td>
</tr>
</tbody>
</table>

Notes:
MP designates polycrystal, MS designates single crystal.
U denotes slightly less pure starting material.
C_o is atomic concentration and is determined as follows:

\[ C_o = \frac{\Sigma Wi/Mi}{W_{mg}/M_{mg} + \Sigma Wi/Mi} \]

where
Wi = weight percent of ith element
Mi = atomic weight of ith element
W_{mg} = weight percent (by difference) of magnesium
M_{mg} = atomic weight of magnesium
TABLE II

<table>
<thead>
<tr>
<th>Crystal</th>
<th>θ</th>
<th>ϕ</th>
<th>E&lt;sub&gt;calc.&lt;/sub&gt;</th>
<th>E&lt;sub&gt;obs.&lt;/sub&gt;</th>
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<td></td>
<td>°</td>
<td>°</td>
<td>psi x 10^-6</td>
<td>psi x 10^-6</td>
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<td>6.42</td>
<td>5.94</td>
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<tr>
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</table>

θ = the angle between the normal to the basal (0001) plane and the cylindrical axis of the crystal.

ϕ = the angle between the slip direction <1120> and the cylindrical axis of the crystal.

E = Tensile modulus (calculated and observed).
### TABLE III
Crystal 31

Stress rate = 2.42 psi/sec., prior strain = 1.2%
Maximum stress = 45.6 psi., minimum stress = 10.9 psi.

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<th>Time</th>
<th>Temp.</th>
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<th>$C_2 \times 10^{-7}$</th>
<th>$C_3 \times 10^{-5}$</th>
<th>$m$</th>
<th>$n$</th>
<th>$p$</th>
<th>$C_i/C_s \times 10^3$</th>
<th>$T_B$</th>
<th>Remarks</th>
<th>$\Gamma$</th>
<th>$L_s \times 10^5$</th>
<th>$A_0 e^{-U_0/\kappa T}$</th>
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<td>5.73</td>
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<tr>
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<td>0.72</td>
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<td>G-L*</td>
<td>-</td>
<td>-</td>
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</table>

*B = 113 psi

$A_1 = 51.6$ psi
<table>
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<tr>
<th>Time</th>
<th>Temp.</th>
<th>$C_1 \times 10^8$ (psi)</th>
<th>$C_2 \times 10^7$ (psi)</th>
<th>$C_3 \times 10^{-5}$ (psi)</th>
<th>$m$</th>
<th>$n$</th>
<th>$p$</th>
<th>$C_1/C_2 \times 10^2$ m-n-m</th>
<th>$T_B$ psi</th>
<th>Remarks</th>
<th>$\Gamma$ (psi)</th>
<th>$L_c \times 10^5$ cm</th>
<th>$A_2e^{U_0/4kT}$</th>
</tr>
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<tbody>
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<td>0.72</td>
<td>2.8</td>
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<td>Temp. (°C)</td>
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<td>C2*10^7_1-n (psig)</td>
<td>C3*10^-5 (psig)</td>
<td>m</td>
<td>n</td>
<td>p</td>
<td>C1/C2*10^2 (psig)</td>
<td>m-n</td>
<td>T_B (psi)</td>
<td>Remarks</td>
<td>Γ (psi)^1/2</td>
<td>L_c*10^5 cm</td>
</tr>
<tr>
<td>--------------</td>
<td>------------</td>
<td>---------------------</td>
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<td>---</td>
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TABLE VI
Crystal 62

Stress rate = 2.96 psi/sec., prior strain = 5%
Maximum stress = 70.9 psi., minimum stress = 15.8 psi.

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<th>C x10^7</th>
<th>C x10^-5</th>
<th>m</th>
<th>n</th>
<th>p</th>
<th>C1/C2 x10^2</th>
<th>m-n</th>
<th>T_b</th>
<th>Remarks</th>
<th>( \Gamma ) ( \psi )</th>
<th>L x10^5</th>
<th>A e ( -U_0/\kappa T )</th>
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* B = 108 psi
A_1 = 78.1 psi

-59-
Magnesium Crystal with Grips and Gage

Scale in inches

Figure 1
SCHEMATIC OF RECORDING CIRCUIT

PROXIMITY METER

LOW IMPEDANCE OUTPUT (0-1ma.)

UNKNOWN CAPACITANCE

110Ω

X-Y RECORDER

Y INPUT

X INPUT

SIGNAL FROM INSTRON LOAD CELL

FIGURE 2
Low Temperature Apparatus

A - Tri-Axial Cable To Proximity Meter
B - Capacitance Gage
C - Specimen

FIGURE 3
MODULUS CALIBRATION

T = 26 °C

STANDARD STEEL SPECIMEN
E = 30.2 x 10^6 psi
Area = 0.196 in^2
Gage Length = 1.00 in

MAGNESIUM CRYSTAL 31
E = 5.81 x 10^6 psi
Area = 0.167 in^2
Gage Length = 1.00 in

Load (Pounds)

Displacement (Micro inches)
(Meter Sensitivity 4-150)

Figure 4
HYSTERESIS LOOPS - CRYSTAL 42

T = 23°C

HISTORY: Prior Strain 6.6 x 10^-3 in/in
Annealed at 24°C. For 39 Days
Strain of 0.2 x 10^-3 in/in immediately
Before Testing
Stress Rate = \( \dot{T} = 2.32 \) psi/sec

Figure 5

Resolved Shear Strain

\( \gamma_B \)

\( \gamma_0 \)

Initial Modulus

\( \tau_0 \) to Shear Stress - \( \tau (\text{psi}) \)
$W_{irr}$ AND $W_T$ vs $\tau_0$

CRYSTAL 6l  \hspace{1cm} T = -50^\circ$C

Prior Strain = $5.1 \times 10^{-3}$ in/in
Stress Rate = $\dot{\tau} = 2.85$ psi/sec
100 Planimeter Units =
$72.7 \times 10^{-6}$ in.lbs./in.$^3$

Figure 6
Figure 7

Log \( W_{irr} \) AND Log \( W_T \) vs Log \( \tau_0 \)

CRYSTAL 62

Prior Strain = \( \gamma_p = 5.0 \times 10^{-2} \) in. L. N.2
Stress Rate = 2.96 psi/sec

\( T = -102{}^\circ C \)

\( \Delta \) = \( W_T \)

\( \Theta \) = \( W_{irr} \)

100 Planimeter Units = 0.731 \( \times 10^{-4} \) in./lbs/3
DECREMENT VS. PRIOR STRAIN
AT CONSTANT STRESS AMPLITUDES

CRYSTAL 61
T = 25°C

STRESS RATE = 2.85 psi/sec

FIGURE 8
LOG $\tau_0$ VS. LOG $\%$

CRYSTAL 62

STRESS RATE = 2.96 psi/sec
PRIOR STRAIN = $5.0 \times 10^{-2}$ in liq. $N_2$

FIGURE 9
LOG $\tau_o$ VS. LOG $\gamma_B$

- CRYSTAL 42, -50°C
  $\dot{\gamma} = 2.32$ psi/sec, $\gamma_p = 6.6 \times 10^{-3}$

- CRYSTAL 31, 26°C
  $\dot{\gamma} = 2.42$ psi/sec, $\gamma_p = 1.2 \times 10^{-2}$

FIGURE 10
LOG $\Delta \tau$ VS $1/\tau_0$

CRYSTAL 62
$T = -189.5°C$

STRESS RATE = 2.96 psi/sec.
PRIOR STRAIN = $5.0 \times 10^{-2}$ in LIQ. N$_2$

FIGURE II
LOG $\Delta_1 \tau_o^{1/4}$ vs $1/\tau_o^{1/2}$

CRYSTAL 42

Stress Rate = 2.32 psi/sec
Prior Strain = $6.6 \times 10^{-3}$

Data Obtained From $W_{irr}$ And $W_1$ vs $\tau_o$ Plots
LOG $\Delta_1 \tau_0^{1/4}$ vs $1/\tau_0^{1/2}$

CRYSTAL 62

$T = -189.5^\circ C$

Stress Rate = 2.96 psi/sec
Prior Strain = $5.0 \times 10^{-2}$ in Liq. N$_2$

○ Raw Data
● Data From $W_{irr}$ And $W_1$ vs $\tau_0$ Plot

Figure 13
DECREMENT VS. TEMPERATURE
CRYSTAL 31

Prior strain = 1.2 x 10^{-2}
Stress rate = 2.42 psi/sec.

○ $\tau_0 = 40$ psi
△ $\tau_0 = 30$ psi
● $\tau_0 = 20$ psi

Temperature °K

Figure 14
DECREMENT VS. TEMPERATURE
CRYSTAL 42

STRESS RATE = 2.32 psi/sec
PRIOR STRAIN = 6.6 x 10^{-3}

\( \Delta_T \)

\begin{align*}
\tau_0 = 70 \text{ psi} \\
\tau_0 = 50 \text{ psi} \\
\tau_0 = 40 \text{ psi}
\end{align*}

FIGURE 15
DECREMENT VS. TEMPERATURE
CRYSTAL 61

STRESS RATE = 2.85 psi/sec
PRIOR STRAIN = 5.1 x 10^{-3}

\( \Delta_T \)

TEMPERATURE °K

FIGURE 16
DECREMENT vs TEMPERATURE AT CONSTANT STRESS
CRYSTAL 62

○ $\tau_o = 50$ psi
△ $\tau_o = 45$ psi
● $\tau_o = 40$ psi
▽ $\tau_o = 30$ psi

Stress Rate = 2.96 psi/sec
Prior Strain = $5 \times 10^{-2}$ in Liq. N$_2$
Data Taken From $W_{irr}$ And $W_T$ vs $\tau_o$ Plots

Figure 17
Decrement vs Temperature

At Constant Stress Amplitude, $\tau_o = 48.6$ psi

And Varying Cross-Head Speed

CRYSTAL 62

○ Stress Rate = 2.96 psi/sec
△ Stress Rate = 7.4 psi/sec
● Stress Rate = 14.8 psi/sec

Figure 18
LOG PERIOD VS. RECIPROCAL TEMPERATURE
CRYSTAL 62

\[ \nu_0 = 4 \times 10^8 \text{ sec}^{-1} \]
\[ Q = 0.33 \text{ ev} \]

FIGURE 19