THE RICE INSTITUTE

DYNAMIC RESISTIVITY MEASUREMENTS

ON RAPIDLY-STRAINED COPPER

by

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A THESIS

SUBMITTED TO THE FACULTY

IN PARTIAL FULFILLMENT OF THE

REQUIREMENTS FOR THE DEGREE OF

MASTER OF SCIENCE

IN

MECHANICAL ENGINEERING

Houston, Texas
May 1960

ABSTRACT

Dynamic resistivity measurements were conducted on copper wires which were rapidly deformed in the plastic range. Of major interest were the instantaneous changes in the resistivity of the metal immediately after the straining was abruptly halted. Records of resistivity change versus time were obtained at three different temperatures. At each temperature, tests were conducted for varying values of strain in an attempt to deduce any dependence of the resistivity change on strain. It was observed that at each temperature, the resistivity decreased rapidly from a maximum value to an equilibrium value. The only useable results were those obtained at room temperature, and these are interpreted in terms of the diffusion of point imperfections to sinks, where they are annihilated.

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INTRODUCTION

In order to understand the motivation for this particular investigation, it is necessary to consider what happens to the lattice structure of a metal when it is strained. Formulating the problem with this in mind, it will then be indicated how measurements of the electrical resistivity give a picture of what is occurring within the metal.

In a well-annealed metal in an undeformed state, there probably exists [1] an initial dislocation density of approximately 10^8 lines per cm^2, which were created during the growth of the crystal structure. As a critical local shear stress within the material is reached, Frank-Read sources become operative and begin to emit dislocation loops[2]. Through interaction of these newly formed dislocations with the initial density, jogs are created in the dislocation lines (see discussion). A jog formed by the intersection of two screw dislocations can generate point defects. These are vacancies or interstitials, depending on the movement of the glissile screw dislocation.

The energy of formation of a vacancy is accepted[3] as being about 0.7 ev in copper, while that for an interstitial is roughly 5 ev. Thus one would expect that in a plastically deformed metal, the vacancy concentration would be substantially greater than the interstitial concentration.

At ordinary temperatures, point defects have sufficient thermal energy to be highly mobile in copper. Hence, as soon as the number of point defects formed exceeds the equilibrium concentration, they start recombining, or in general disappearing at sinks. The possible sinks are dislocations, grain boundaries, impurity atoms, and external sur-

faces. The most likely seem to be dislocations. Thus, if the strain-
ing were suddenly stopped, there would possibly remain a non-equilibrium
concentration of point defects present in the material which would still
be disappearing. One would like to determine the change in concentra-
tion of these point defects with time immediately after straining.
This problem forms the basis of this investigation.

Since the concentration of point imperfections cannot be measured
directly, attention is turned to some physical property of the metal
which depends on the presence of these defects. One of the most suit-
able quantities to measure is the electrical resistivity. Metals
possess electrical resistance because the electrons are scattered by
lattice defects such as chemical and physical impurities and phonons.
A metal with a perfect lattice would have no resistance[4]; thus, the
resistance can be regarded as a measure of the divergence from a per-
fect lattice structure, and hence a measure of the defect concentration.
It is more practical to speak in terms of resistivity than resistance,
because the former does not depend on the geometry of the specimen
whereas the latter does.

At ordinary temperatures, by far the greater part of the resistivity
is caused by scattering due to thermal vibrations of the lattice. At
low temperatures, the relative effect of lattice vibrations is slight.
In addition to this temperature effect, the resistivity also increases
with the number of structural defects, namely dislocations, vacancies,
interstitials, and foreign atoms. Since the number of the first three
of these imperfections increases with plastic strain, the resistance

likewise increases. Matthiessen's law, which states that the resistivity contributions arising from structural defects and from thermal vibrations are additive and independent, is applicable here.

From a calculation made by Jongenburger[5], the introduction of a single vacancy per cm^3 into an otherwise ideal lattice of copper would cause a resistivity increase of 1.53×10^{-21} microohm-cm; of a single interstitial atom per cm^3, about 5.7×10^{-21} microohm-cm. Also, the extra-resistivity associated with one centimeter of dislocation line per cm^3 is about 4×10^{-21} ohm-cm[13].

If the rapid deformation of a metal in the plastic region were suddenly stopped, the dislocation density, which is a function of strain, would remain essentially constant, and any "annealing" effect could be attributed primarily to the annihilation of point imperfections. If the deformation were sufficiently rapid, disappearance of point defects would still be occurring after the straining has stopped and instantaneous readings of the resistance should yield changes with time in concentration of point defects.

EXPERIMENTAL APPARATUS AND PROCEDURE

In order to effect a meaningful resistivity measurement, a high degree of care must be exercised in the design, set-up, and operation of the equipment to be used for such a measurement. Although this is common knowledge among those who have made such measurements, additional problems arise in an attempt to measure instantaneously small changes in the resistivity which occur very rapidly. Therefore, it is quite important that the apparatus used and the procedure followed in the investigation be spoken of in some detail.

The resistivity-measuring circuit is shown in Figure 1. It consists primarily of the following components: A Hewlett-Packard Model 200 CD oscillator was used to supply the current to the circuit at a given frequency. Transformer #1 was included in the circuit in an attempt to match as closely as possible the impedance of the specimen. Transformer #2 was of the shielded type and isolated the measuring circuit from the oscillator circuit; it also provided a 4:1 step-up of the specimen voltage. The amplifier was specially designed and built for the circuit. It is a high-gain, low hum, tuned amplifier with an optimum pass frequency of 5400 cps., which was the frequency at which all measurements were made. A large capacity 45 volt dry-cell battery was used with a 25-turn Helipot potentiometer as a suppressor, Figure 2. The output of the amplifier consisted of both a-c and d-c terminals. The d-c signal output was fed into a two-channel Sanborn recorder, where it was recorded permanently on one of the two channels. The a-c signal was monitored on an oscilloscope in order to observe any changes in the wave form of the output signal.

The temperature change of the specimen during and subsequent to straining was measured by means of a 30 gauge copper-constantan thermocouple. After amplification by a Sanborn model 150-1800 d-c preamplifier, the signal from the thermocouple was recorded on the second channel of the Sanborn recorder.

A picture of the tensile machine used for straining the specimen is shown in Figure 1. It consisted of a small motor and a series of reduction gears. The last of these gears drove a rack to which was connected a teflon holding piece which gripped one end of the specimen. The other end of the specimen was attached to a stationary teflon piece.

In order to stop the straining abruptly, a microswitch was placed on a support above the rack, which, when tripped, activated a relay. This reversed the motor and also activated another relay which disconnected the power to the motor. This combination of relays was such that the straining was halted very suddenly, with no measurable "coasting". Connections were made to enable remote operation of the tensile machine.

Polycrystalline copper wire of 24 gage was chosen for the test material. The primary reason for this choice was the abundance of investigations and literature concerning copper. To prepare the specimens, the copper wires were cut into pieces approximately 6" long, and a number of these were bundled together and placed in an annealing furnace. The annealing was done in vacuum, and the wires were kept at 550°C for one hour. The specimens were subsequently prepared as follows: Stakon terminal lugs were filled with solder and a wire was slipped into the solder; after solidification of the solder the copper wire was held

firmly to the terminal lug. This was done on each end of a length of wire. To ensure a firm grip on the copper wire, the excess length was wrapped about the remainder of the terminal lug in such a way that, upon connection to a teflon holding piece, additional gripping was provided.

The procedure followed in performing a test consisted of the following steps. First, a specimen was placed in the tensile machine by means of the teflon grips and screws, whereupon the cross-section of the wire was measured and an average value recorded. Next, the thermocouple was attached, proper care being taken to limit the amount of solder in the joint to a minimum. This is highly desirable for fast couple response. The current and potential leads were then soldered to the specimen near each end. The original length between potential leads was measured by means of an optical cathetometer which could be read to the nearest .001 millimeter. The signal from the amplifier was then suppressed to obtain a null position on the recorder. A Dewar flask was subsequently raised by means of a "Lab-jack" until the wire was surrounded by the testing medium. Another suppression reading was taken to determine the output of the amplifier with the specimen at the test temperature. Next, the thermocouple voltage was suppressed to give no input to the recorder at the start of a test. This suppression reading yielded the value of the testing temperature. The straining was then begun by means of the remote control device, and the resistance change of the wire was recorded as a function of time.

The calibration of the apparatus was carried out as follows: A dummy specimen was used with steps of resistance covering the range

likely to be encountered during all tests. The resistance between steps was measured accurately by means of a Kelvin double bridge. At the same time the output of the amplifier was suppressed to yield a reading on the 25-turn potentiometer. Thus, a plot of specimen resistance versus amplifier output was obtained. This calibration curve was used subsequently to obtain all values of specimen resistance (Figure 4).

RESULTS

According to Matthiessen's law the effects of temperature and lattice defects on the resistivity are additive. From the record of increase in temperature versus time, a curve of percentage change in resistivity due to temperature versus time can be drawn. A value of $0.0043 \ °C^{-1}$ for the temperature coefficient of resistivity at room temperature for copper was used. The resistivity record obtained on the recorder yields a plot of the total change in resistivity versus time. Subtracting the former from the latter gives the resistivity change due to point imperfections against time. The result of a typical test performed in air at room temperature is shown in Figure 5.

Several tests were also conducted at the temperature of liquid nitrogen. The results show that in this medium there is no noticeable temperature change. Moreover, there is only a very small, if any, decrease in resistivity immediately after straining.

The final series of tests was conducted at three different temperatures: 25°C, 0°C, and -80°C. All were performed in a medium of methanol. The data obtained at room temperature (25°C) were the only useful results; those obtained at the remaining two temperatures displayed too much scatter.

The total change in specimen resistance, including that due to change in geometry, was measured and plotted as a function of strain, Figure 6. This was done to make certain that the electrical equipment was functioning properly. As shown in the appendix(A), the relation between the total resistance change and the true strain should be $\Delta R \sim e^{2\delta}$, where ΔR = total resistance change and δ = true strain. When

plotted on semi-log paper, the curve should be a straight line with slope 2. This was done, and a slope of 2.07 was obtained using the data of Figure 6. This indicates that the electrical circuit was operating properly.

A plot of the resistivity change versus strain after deformation at room temperature is shown in Figure 7. An attempt was made to determine the functional relationship from this data, and it was concluded that it would be best represented by a polynomial expansion.

For a typical test at room temperature, the time needed for the resistivity to decrease from a maximum to an "equilibrium value" after straining was about 2 seconds.

DISCUSSION

Van Bueren[3] developed the following relation between strain and the number of point defects formed:

$$f_1 (\epsilon) = \frac{0.04}{b^3} \epsilon^2 \quad (cm^{-3}) \quad , \tag{1}$$

where $f_1 (\epsilon)$ = theoretical number of point defects formed per cm^3,

b = magnitude of Burgers vector, and

ϵ = plastic strain .

This applies to noble metals in which multiple glide occurs during plastic deformation, polycrystalline copper being one such metal. Upon examining the experimental observations of various authors on polycrystalline copper, van Bueren concluded that the resistivity-strain relations could all be represented quite accurately by the relation

$$\Delta\rho = .065 \epsilon^{3/2} \quad (\mu \; ohm\text{-}cm) \quad , \tag{2}$$

where $\Delta\rho$ = change in residual resistivity as a result of plastic strain.

Using a calculated value[5] for the extra-resistivity associated with point imperfections, (2) can be written

$$f_2 (\epsilon) = 4 \times 10^{19} \epsilon^{3/2} \quad (cm^{-3}) \quad ,$$

where $f_2 (\epsilon)$ = observed number of point defects formed per cm^3.

One would like to reconcile the theoretical and experimental results as given in (1) and (2). Suppose that the recovery of resistivity, which was observed to occur in a very short time, can be attributed to the disappearance of point imperfections. This suggests that there are a number of "lost" point defects present which have not previously been

detected in resistivity measurements by other investigators at room tem-
perature. If this number be denoted by $f(\epsilon)$, the experimental relation
(2) may be corrected by adding to it $f(\epsilon)$, so that the theoretical re-
lation

$$Kf_1(\epsilon) = f_2(\epsilon) + f(\epsilon) \tag{3}$$

will be satisfied. K in this expression is a number less than unity be-
cause some of the defects formed will have been annihilated before
counting has commenced. Thus,

$$f(\epsilon) = 2.5 \times 10^{21} K\epsilon^2 - 4 \times 10^{19}\epsilon^{3/2} \ (cm^{-3}) \ . \tag{4}$$

For a value of K = 0.2, a fair agreement with the data was obtained,
Figure 8. Thus it appears, from the small amount of unconfirmed data
obtained as a result of this investigation, that a square relation be-
tween the strain and the number of defects created does hold in poly-
crystalline copper.

Another calculation is of interest here. From equation (1), the
number of point defects created per cm^3 during a plastic deformation
of 10% is approximately 2×10^{19}. From the data of Figure 7, the num-
ber of defects annealing out for 10% strain can be computed, using the
value of extra resistivity associated with a single defect. Assuming
the decay to be due to annihilation of vacancies, the number corres-
ponding to a strain of 10% was found to be 2×10^{18} per cm^3. Thus, to
an order of magnitude, the number of "lost" defects is one-tenth the
total number thought to be formed during plastic deformation. This
fraction agrees favorably with the factor, K = 0.2, obtained above.

An attempt must now be made to justify the assumption that the
decay in resistivity after deformation can be explained in terms of dis-
appearance of point imperfections, mostly vacancies. The problem may

be approached from energy considerations first. Nicholas[6] makes the statement that the creation and subsequent annihilation of point imperfections could explain the large percentage dissipation of energy as heat during plastic deformation. Only a small portion($\simeq$ 5%) of the energy of deformation in copper is stored in the material [17, 18], and the remainder can be satisfactorily accounted for in terms of the annihilation of point imperfections. Thus it must be assumed that a number of point defects are created during the straining cycle in some manner. Since the energy of formation of an interstitial is much larger than that of a vacancy, vacancies will be created in preference to interstitials as a result of cold-work. Therefore, the picture can be simplified to one in which vacant lattice sites are formed in large numbers during cold-work.

There are several conceivable methods by which vacancies can be Produced during plastic deformation. Seitz[7] analyzes three possible methods of generation of vacancies. These are: (1) generation by local heating of the lattice in the immediate vicinity of the dislocation as it moves through the lattice and absorbs energy from the stress field; (2) generation by purely geometrical means when dislocations intersect to form jogs with the subsequent production of vacant sites; (3) generation in the vicinity of local lattice disturbances, such as large thermal pulses, etc. Of the three, Seitz concludes that (2) is the most likely to occur.

The formation of point defects by intersection of dislocations has been explained by Cottrell[1] as follows: Consider the crossing of two perpendicular screw dislocations:
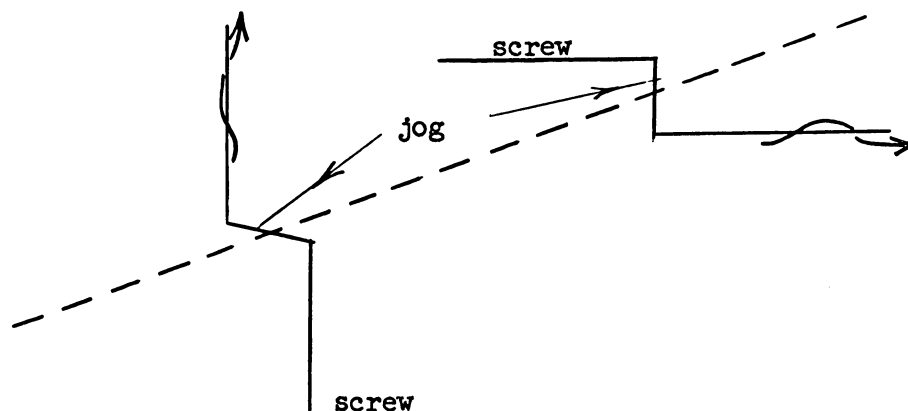
Upon intersection, two pieces of each dislocation are displaced by the magnitude of the Burgers vector of the screw which does the cutting. The jog created between the two pieces of each dislocation is an edge dislocation because it is normal to its Burgers vector, which lies along the screw axis. An edge dislocation can move easily in the plane containing its line and its Burgers vector. Thus, the jog can glide conservatively along the screw axis. In order that the screw dislocation move along with the jog, the edge dislocation must undergo a non-conservative motion, or motion out of its slip plane. Each jog is the end of an incomplete row of atoms along the edge of the half-plane of the dislocation, and this row may either disappear by forcing its atoms into interstitial positions, or grow as a result of gaining atoms from the lattice, in which case vacancies are generated by the jog. In a close-packed metal, such as copper, where the energy of an interstitial defect is greater than that of a vacancy, it is expected that vacancies are generated preferentially.

Seitz' method of point defect generation is similar to that of
Cottrell, but differs in that the moving screw dislocation, rather than
intersecting the stationary screw, bends or loops about the latter.
Eventually, opposite sides of the loop, being attracted to each other,
will come together on different slip planes to form the jog. Point
defects are then formed through non-conservative motion of jogs, as
above.

If a row of vacancies produced by either of the above methods
could break up into single vacancies and disperse as a result of local
heating of the lattice, an essentially geometrical method of creating
vacancies is obtained.

The following question might well be raised: does the high strain
rate used alter the above explanation of defect formation? Cottrell[8]
demonstrates that the plastic deformation mechanism and all accompany-
ing occurrences in copper are rather insensitive to the rate of strain.
From this, it is concluded that using a high strain rate in order to
facilitate observation of disappearance of vacancies is not only justi-
fiable, but actually desirable.

Having established a means of formation of vacancies, attention is
now turned to the annihilation of vacancies at room temperature. First,
it is pointed out that there does not exist in the literature a completely
satisfactory explanation for the disappearance of point defects after
formation. Nontheless, from experimental evidence[9], any point defects
in copper should be sufficiently mobile at room temperature to migrate
to sinks and dissipate their energy as heat. Several mechanisms for
vacancy disappearance have been proposed by various authors. Wintenburger[10]

and other investigators[14] propose that elastic interaction of vacancies and dislocations is responsible. Another group[15,16] proposes that vacancies group together to form clusters, which, in turn, diffuse to dislocations. For the purpose of further discussion, it will be concluded that vacancies, upon being created, will diffuse to a dislocation or other sink and be absorbed or otherwise eliminated. The diffusion of vacancies to sinks can be expressed by the equation

$$n = \nu t e^{-Q/kT} \tag{6}$$

where n = number of jumps made before disappearing,

ν = atomic vibrational frequency = 10^{13} sec^{-1},

t = time required to accomplish successful annihilation,

Q = activation energy of vacancy migration,

k = Boltzmann's constant,

T = absolute temperature.

From equation (6), the average number of atomic jumps made per vacancy before disappearing was calculated by using an activation energy, Q = 0.7 ev for vacancy diffusion and making use of the experimentally determined recovery time. From this calculation, a value of $n = 10^2$ was obtained. This should be compared with an expected value of 10^4 in cold-worked copper. Lomer and Cottrell[12], using the experimental results of other investigators, obtain calculated values of $10^0 - 10^2$ for n in cold-worked copper.

The problem of primary concern in this investigation was the measurement of any vacancy decay which occurred immediately after deformation had ceased. The above discussion is included in order to explain any phenomena occurring after deformation in terms of what

happened before the deformation stopped. Conversely, examination of the annealing kinetics should give some insight into the complex events accompanying plastic deformation.

Blewitt, et al.[11] found that different specimens of copper single crystals exhibited different initial resistivities, ρ o. They concluded from their resistivity measurements that the annealing kinetics of plastically deformed samples appear to depend on ρ_o. This could explain the scatter present in the data of Figure 7. On the other hand, Blewitt and co-workers worked at 4.2°K, and observed much smaller changes in resistivity than were observed in this investigation. Therefore, it is quite possible that this property cannot account for the scatter observed here.

The above discussion does not consider the effect of different temperatures of deformation on the results. It is not explained, for instance, how vacancies are lost at the low temperatures at which van Bueren[3] reports a $\epsilon^{3/2}$ relation for the number of defects formed.

CONCLUSIONS

A diffusion-type resistivity recovery process of short duration has been observed in copper at room temperature, which results when the strain rate is abruptly changed from a high value to zero. The magnitude of this resistivity change increases with plastic strain. This change can be explained in terms of vacancy migration and subsequent annihilation.

SUGGESTIONS FOR FURTHER WORK

First, the noise level in the resistivity-measuring circuit should be reduced as far as possible without sacrifice of sensitivity. Second, rather than employing a single strain rate as used in this investigation, a system in which the strain rate can be varied would be more desirable. Third, some type of temperature control should be incorporated into the apparatus. This would ensure complete uniformity in tests conducted at any given temperature. Finally, as suggested in the discussion of the results obtained here, the initial residual resistivity of each undeformed specimen should be determined. The determination of this property will require the use of very low-temperature media, such as liquid hydrogen or liquid helium. Any effect that this property has on the results of subsequent resistivity measurements can then be more readily determined.

APPENDIX

A. Variation of absolute resistance change with strain.

δ = true strain

R = resistance (ohms)

ρ = resistivity (ohm-cm)

ℓ = specimen length (cm)

A = specimen cross-sectional area (cm^2)

V = volume (cm^3)

Δ R = absolute change in resistance due to strain (ohm)

Assuming constant volume,

$$V = A\ell = constant$$

Then

$$R = \rho \frac{\ell}{A} = \frac{\rho}{V} \ell^2$$

$$\frac{R_2}{R_1} = \frac{\rho_2}{\rho_1} \frac{\ell_2^2}{\ell_1^2}$$

Since

$$\frac{\ell_2}{\ell_1} = e^{\delta}$$

$$\frac{R_2}{R_1} = \frac{\rho_2}{\rho_1} e^{2\delta}$$

Hence,

$$\frac{\Delta R}{R_1} = \frac{R_2 - R_1}{R_1} = \frac{\rho_2}{\rho_1} e^{2\delta} - 1$$

B. Sample calculation of amount of resistivity decay from the experimental data (specimen #137).

α = temperature coefficient of resistivity = $0.0043°C^{-1}$

ρ_o = resistivity of copper at 0°C = 1.56×10^{-6} ohm-cm

ρ_i = initial resistivity at the testing temperature (ohm-cm)

$\Delta \rho_T$ = resistivity change due to temperature (ohm-cm)

$\Delta \rho_D$ = resistivity change due to defects (ohm-cm)

$\Delta \rho$ = total resistivity change (ohm-cm)

Δ T = maximum change of temperature occurring in the specimen (°C)

$\Delta \rho$ corresponds to a voltage decrease of 33 mv (obtained from recorder calibration). The initial voltage input to the recorder was 6.94 v, which corresponds to a resistance of 3.18 milliohms through the calibration curve (Figure 4). The final voltage is thus 6.94 + 0.033 = 7.04 v, which corresponds to 3.21 milliohms. Since the geometry is not changed,

$$\frac{\Delta \rho}{\rho_i} = \frac{0.03}{3.18} = 0.0094 \quad \text{or} \quad 0.94\%$$

The temperature effect on the resistivity increase is found as follows:
ΔT corresponds to 0.0143 mv increase in thermocouple voltage which means a rise in temperature of 0.54°C. Hence,

$$\Delta \rho_T = \rho_o (\alpha \Delta T) = 0.00234 \, \rho_o.$$

And

$$\frac{\Delta \rho_T}{\rho_i} \triangleq \frac{\Delta \rho_T}{\rho_o} \frac{\rho_o}{\rho_i} = 0.00234 \frac{1.56 \times 10^{-6} \, \Omega \, cm}{1.72 \times 10^{-6} \, \Omega \, cm}$$

$$= 0.0021 \quad \text{or} \quad 0.21\%$$

Since $\quad \Delta \rho = \Delta \rho_T + \Delta \rho_D$,

Then $\quad \Delta \rho_D = 0.94\% - 0.21\% = 0.73\%$

This calculation assumes $\alpha = 0.0043 \, °C^{-1}$, and a testing temperature of 25°C.

SYMBOLS

δ - true strain

ϵ - engineering strain

b - Burgers vector magnitude

ρ - resistivity

l - specimen length

ν - atomic vibrational frequency

Q - activation energy for diffusion of a vacancy

K - Boltzmann constant

T - absolute temperature

t - time

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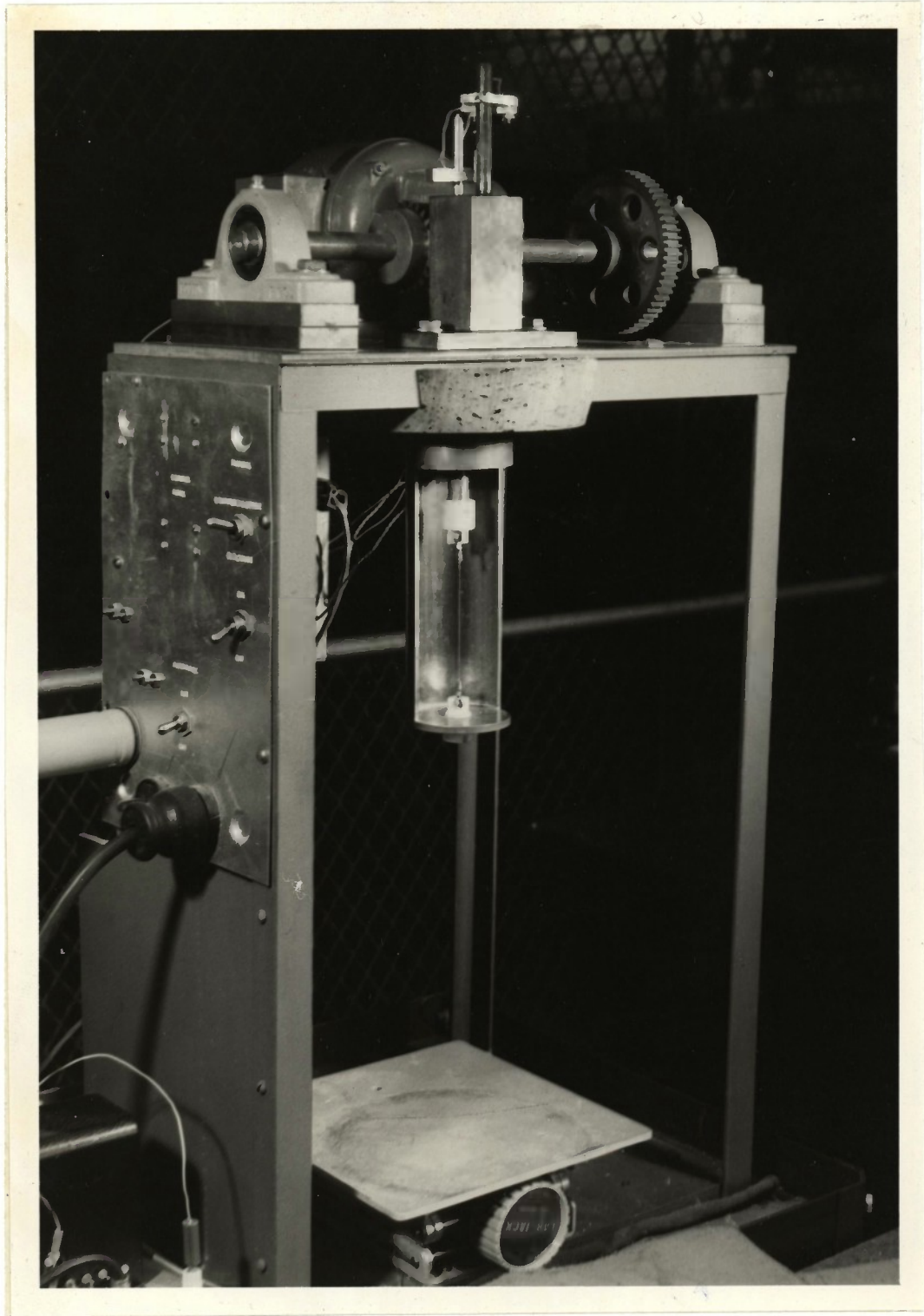
ACKNOWLEDGMENT

FIGURES

FIGURE 1. PHOTOGRAPH OF STRAINING MECHANISM
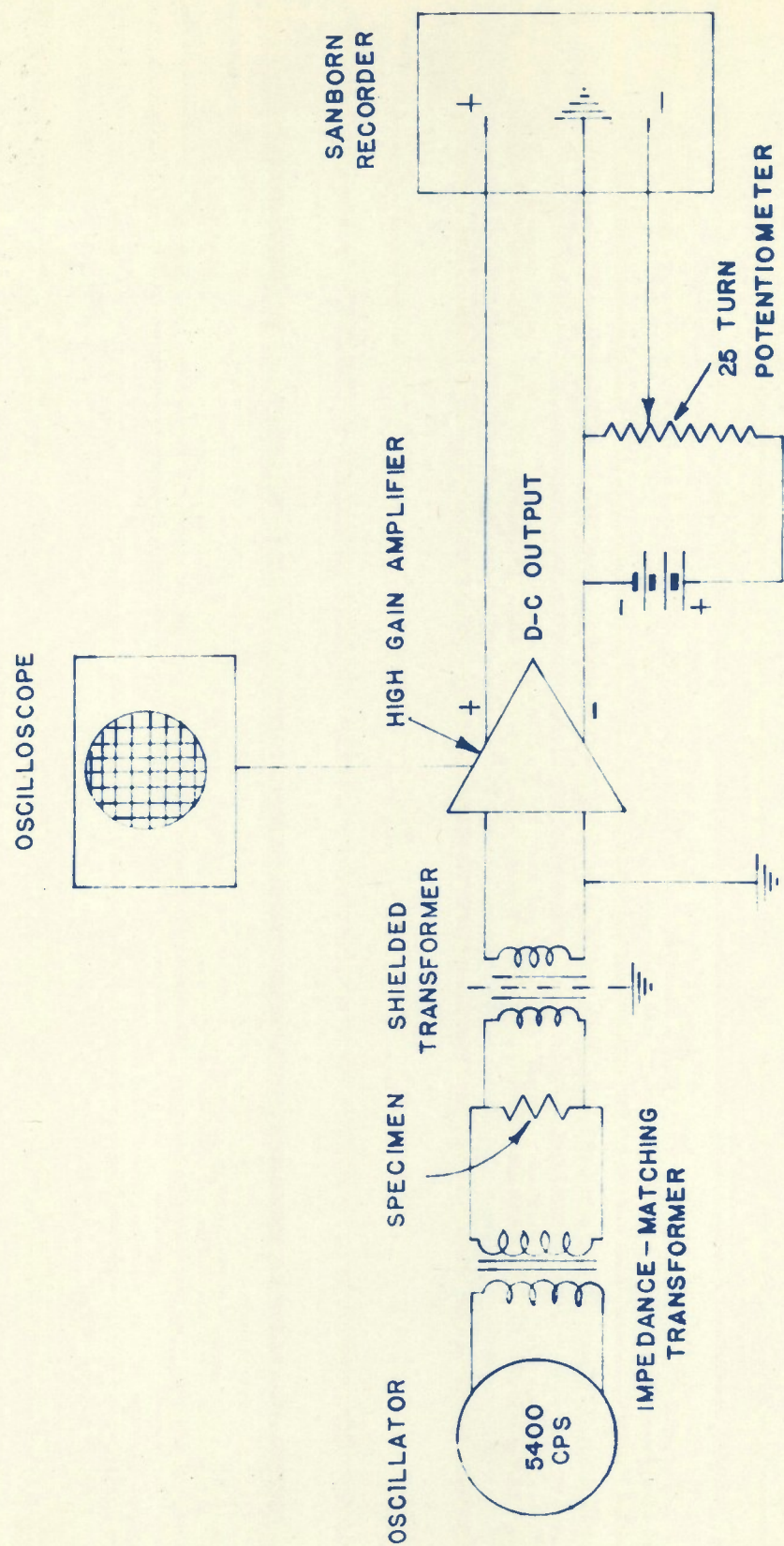
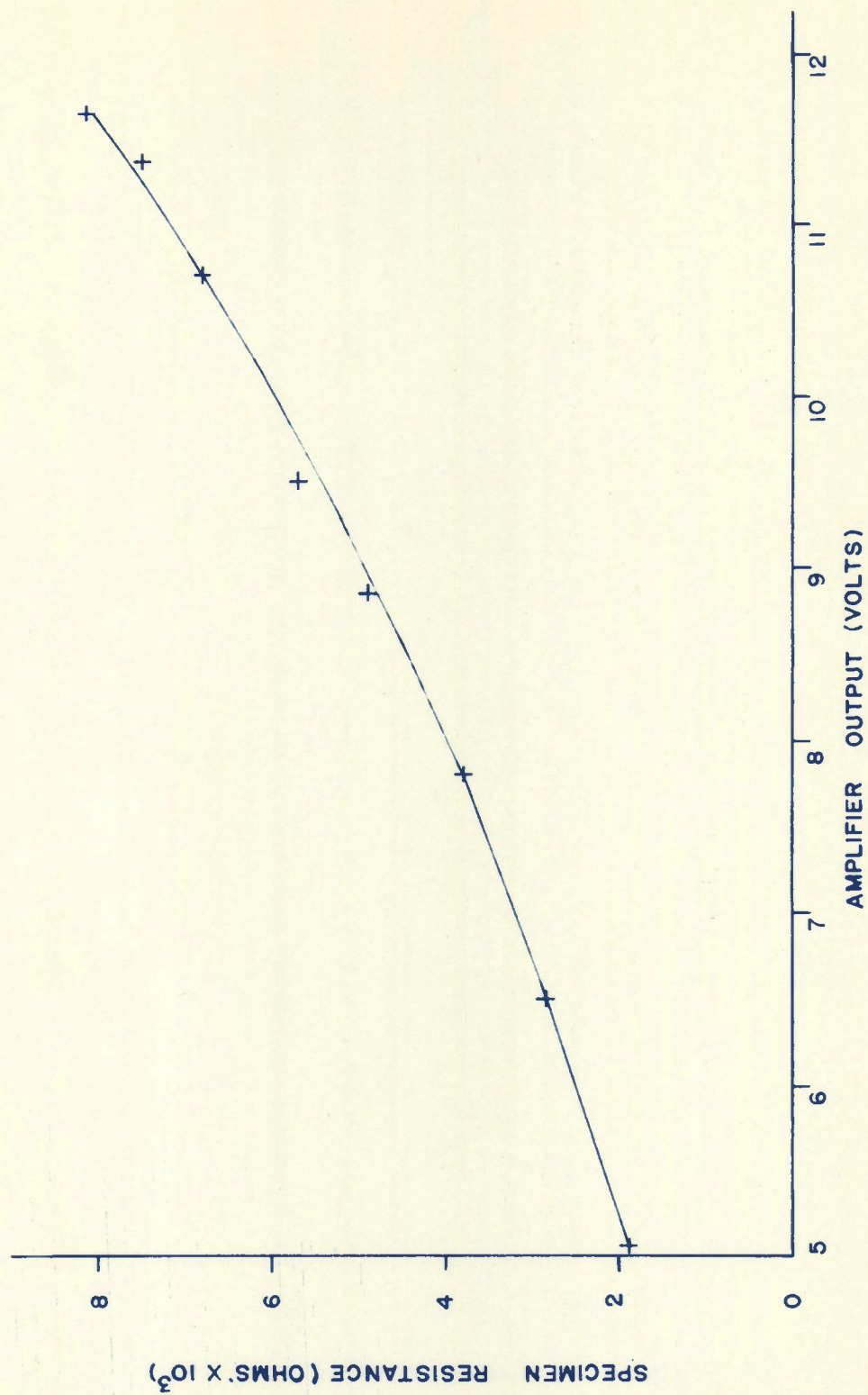FIGURE 2. RESISTIVITY CIRCUIT
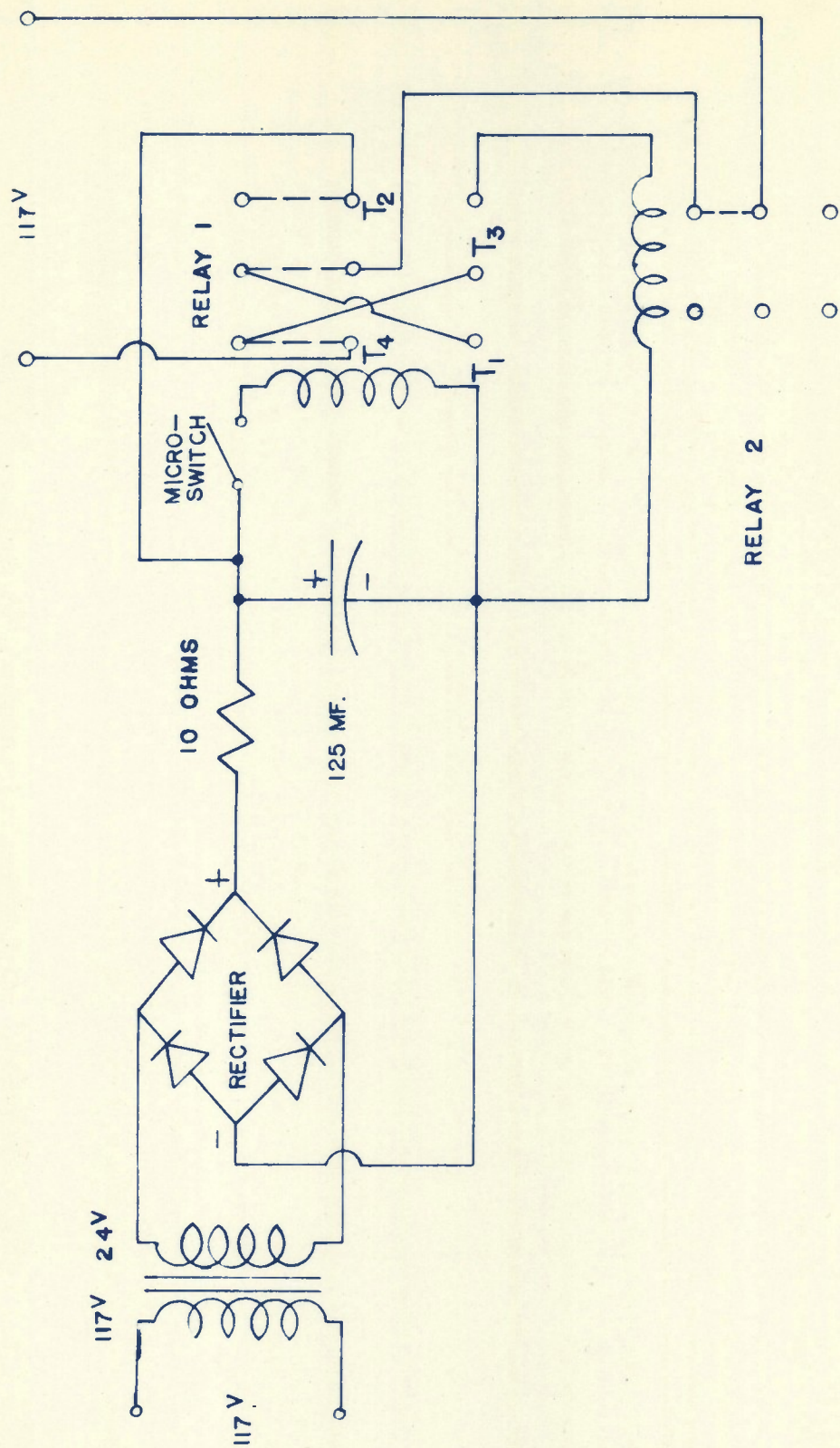
FIGURE 3.   STRAIN—HALTING CIRCUIT

AMPLIFIER OUTPUT (VOLTS)

SPECIMEN RESISTANCE (OHMS. X 10³)

FIGURE 4. CALIBRATION CURVE

RESISTIVITY CHANGE DUE TO TEMPERATURE
(% OF MAXIMUM)

CHANGE DUE TO TEMP. →

← RESISTIVITY (TEMP. CHANGE SUBTRACTED)

← TOTAL RESISTIVITY

TIME (SECONDS)

SPECIMEN RESISTIVITY (% OF MAXIMUM)
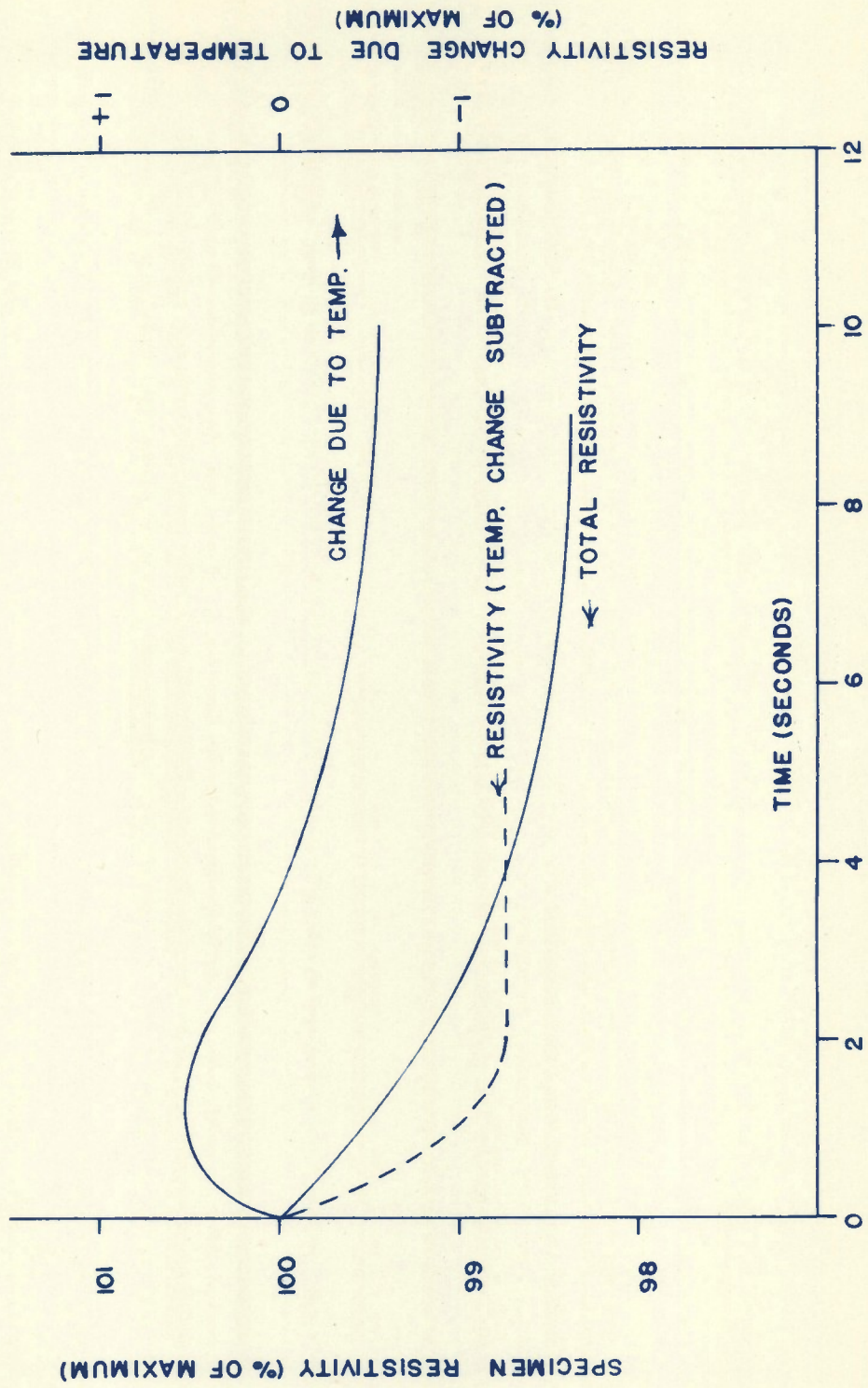
FIGURE 5. RESULT OF A ROOM TEMP —
ERATURE TEST

ROOM TEMPERATURE DATA

RESISTANCE CHANGE (%)

60

50

40

30

20

10

0

0   5   10   15   20   25

PER CENT STRAIN
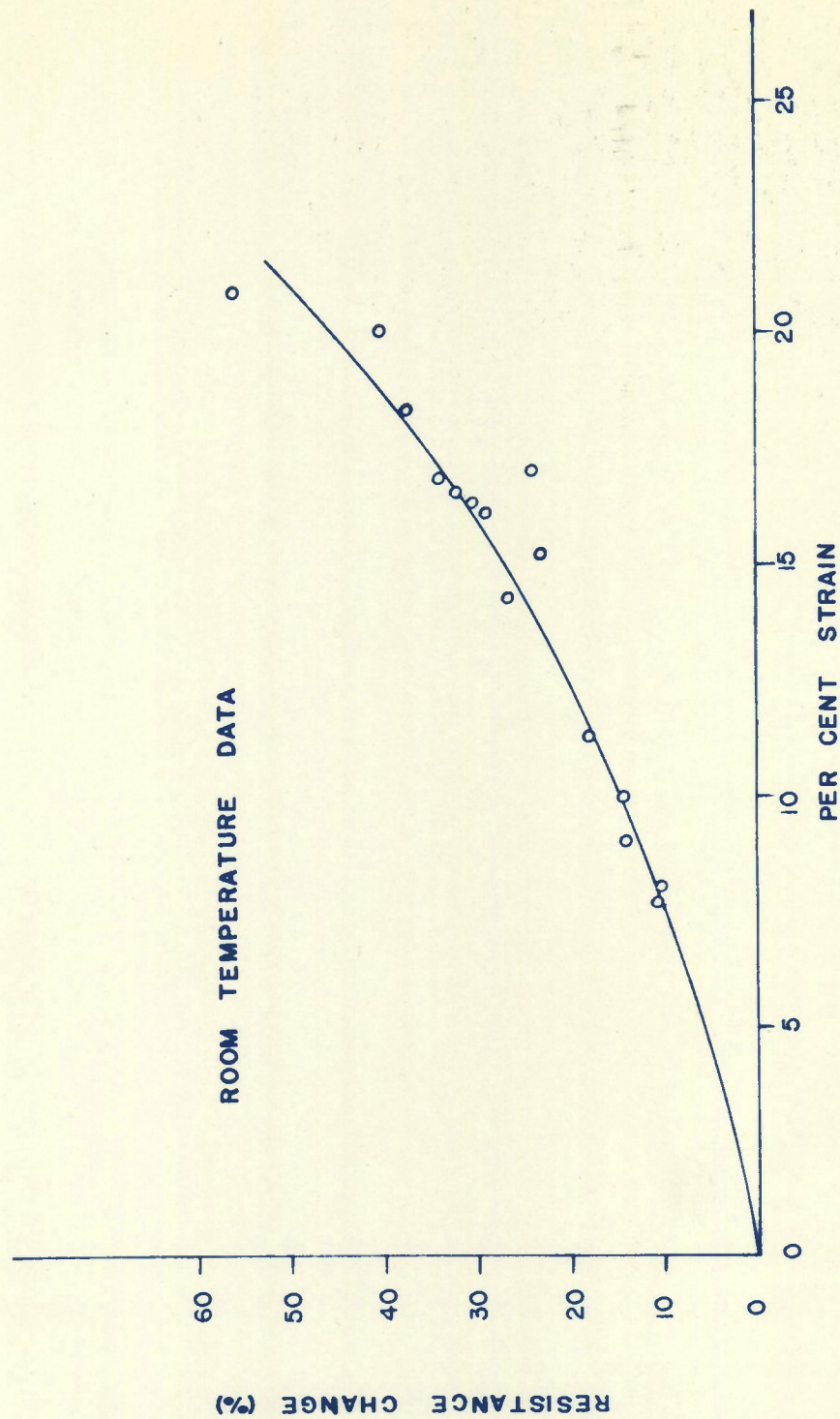
FIGURE 6. ABSOLUTE RESISTANCE CHANGE vs. STRAIN

ROOM TEMPERATURE DATA

DECREASE IN RESISTIVITY (% OF MAXIMUM)
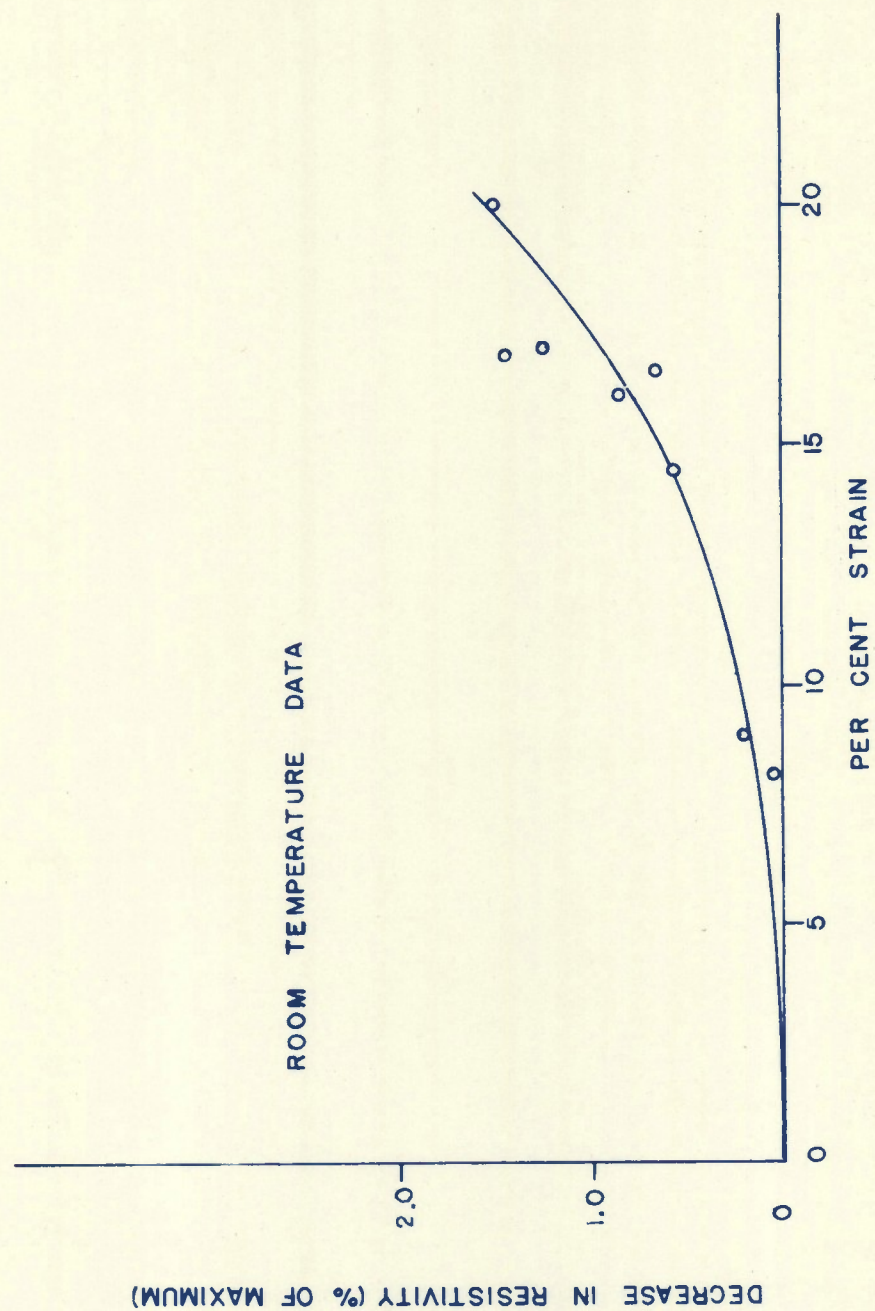
PER CENT STRAIN

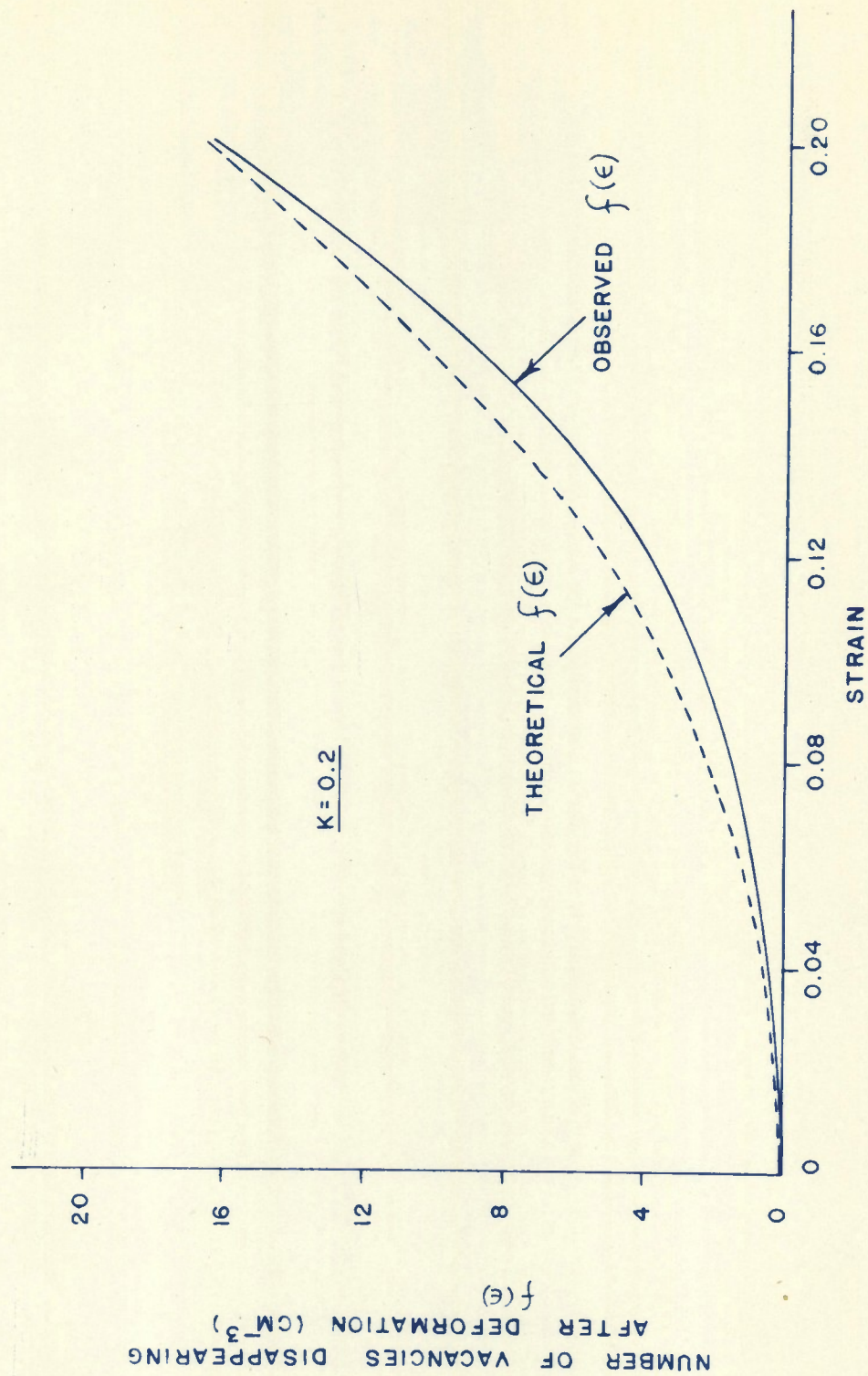FIGURE 7. DECREASE IN RESISTIVITY DUE TO DEFECTS
vs. STRAIN

FIGURE 8. COMPARISON OF CALCULATED & OBSERVED $f(\epsilon)$