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Determination of Magnetic Anisotropy of Single Crystals of Iron and Nickel by Means of the Torque Magnetometer

presented by

Joseph Mudar

has been accepted towards fulfillment
of the requirements for

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Robert D Spence
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Major professor

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Determination of Magnetic Anisotropy of Single Crystals of Iron and Nickel by Means of the Torque Magnetometer

BY

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A Thesis

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Joseph Mudar

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INTRODUCTION AND THEORY

It has been known for some time that the energy required to magnetize single crystals of ferromagnetic materials depends on the angle between the magnetization vector and the crystalographic axes.[1] This energy must have the same symmetry as the crystalographic lattice. Thus for a cubic lattice:

$$E = K_0 + K_1(\alpha_1^2\alpha_2^2 + \alpha_2^2\alpha_3^2 + \alpha_3^2\alpha_1^2) + K_2(\alpha_1^2\alpha_2^2\alpha_3^2 + \cdots) + \cdots \quad (1)$$

where the K_n's are called the anisotropy constants and the α_i's are the directional cosines of the magnetization vector with respect to the crystallographic axes. The terms containing K_1 and K_2 are the only terms of the series which are normally required to represent experimental results. In certain situations the term containing K_2 is often neglected.

This thesis reports the results of measurements of the temperature dependence of K_1 in single crystals of nickel and iron. The theory of the experiment has been discussed by Morrison[3] and therefore, only a brief summary of the theory will be given here.

If the magnetization vector lies in certain planes in the crystal the expression for the anisotropy energy is greatly simplified. In the (100) plane $\alpha_1 = \cos\theta$, $\alpha_2 = \sin\theta$, $\alpha_3 = 0$, where θ is the angle between the magnetization vector and an easy direction of magnetization. If the magnetic field is of sufficient strength to align the magnetization vector parallel to the magnetic field in a sample in which the magnetic field lies in the (100) plane, the torque which arises from the

anisotropy energy is given by:

$$L = -\frac{\partial E}{\partial \theta} = -\frac{\partial (K_1 \sin^2\theta \cos^2\theta)}{\partial \theta} = \frac{K_1}{2} \sin 4\theta \qquad (2)$$

This torque can be obtained by measuring the twisting of a fiber attached to the sample. The torque can therefore be expressed as $\eta\phi$ where η is a constant characteristic of the fiber and ϕ is the angle through which the fiber is twisted. Evaluating the derivative of the torque at $\theta = 0$ we get:

$$\frac{\partial L}{\partial \theta (\theta=0)} = \frac{\partial}{\partial \theta} \frac{(K_1 \sin 4\theta)}{2} (\theta=0) = 2K_1 \qquad (3)$$

also

$$\frac{\partial L}{\partial \theta (\theta=0)} = \frac{\partial \eta\phi}{\partial \theta (\theta=0)} = \eta \frac{\partial \phi}{\partial \theta (\theta=0)} \qquad (4)$$

therefore

$$K_1 = \frac{\eta}{2} \frac{\partial \phi}{\partial \theta} (\theta=0) \qquad (5)$$

where $\frac{\partial \phi}{\partial \theta}$ is the slope of the torque curve. If θ and ϕ are expressed in the same units the slope of the curve can be measured directly from an experimental torque curve on which ϕ is plotted as a function of θ. The experimental torque curves for nickel are shown in Figures (6) and (7) and the curve for iron in Figure (8). If the expression containing K_2 is neglected, K_1 in a (110) plane has the same expression as given in equation five.

EXPERIMENTAL APPARATUS AND PROCEDURE

The apparatus for obtaining the torque curves is essentially the same as used by Morrison. In order to improve the accuracy of the torque curves, a torque magnetometer was designed and built which utilized a rotating radar magnet. Although this magnet had a field of 2,000 oersteds, this was insufficient to insure the parallelism of the magnetization vector and the external magnetic field in the samples of the shape used in this experiment. This was shown by a shift in the maxima and minima of the torque curves of the (100) plane, causing the positive and negative slopes at $\phi = 0$ to differ by an amount in excess of the experimental error. Since it would be somewhat cumbersome to construct a rotating magnet capable of producing sufficiently large fields, the rotating sample method of obtaining the torque curves was adopted. The field used in this case was 8,000 oersteds and was supplied by a large electromagnet.

Owing to the various sizes, materials and crystallographic planes of the samples, it was necessary to use various diameters of fibers to obtain satisfactory torque curves. It was found that guitar strings served the purpose very well.

Although the method of heating was the same as described by Morrison, the technique of measuring the temperature of the sample differed. The copper-constantin thermocouple was placed in direct contact with the sample and rotated with it as shown in the section drawing in Figure (3). The e. m. f. was read, before and after the slope $\phi = 0$ was plotted, by attaching two clips from the potentiometer to the free ends of the thermocouple.

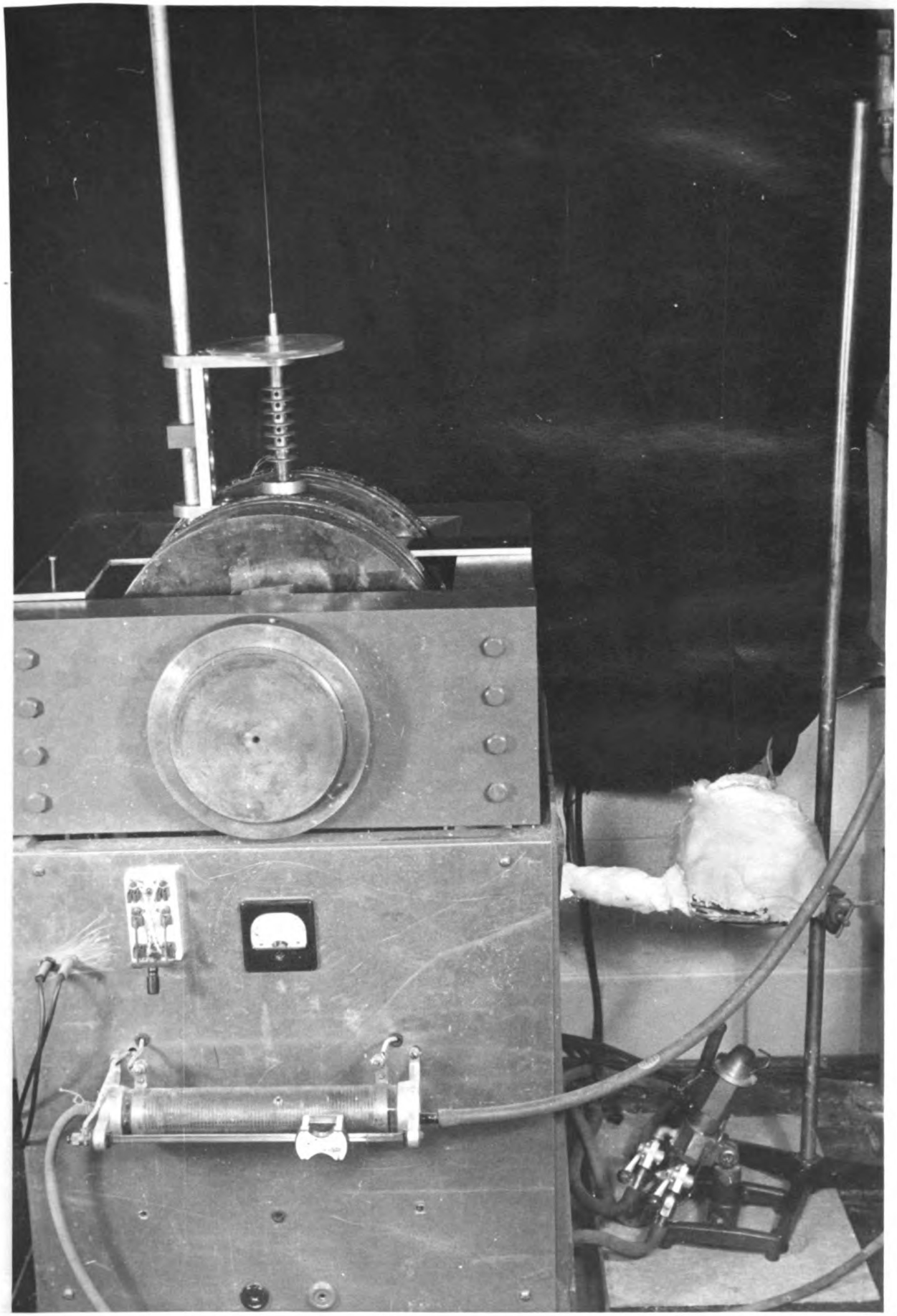
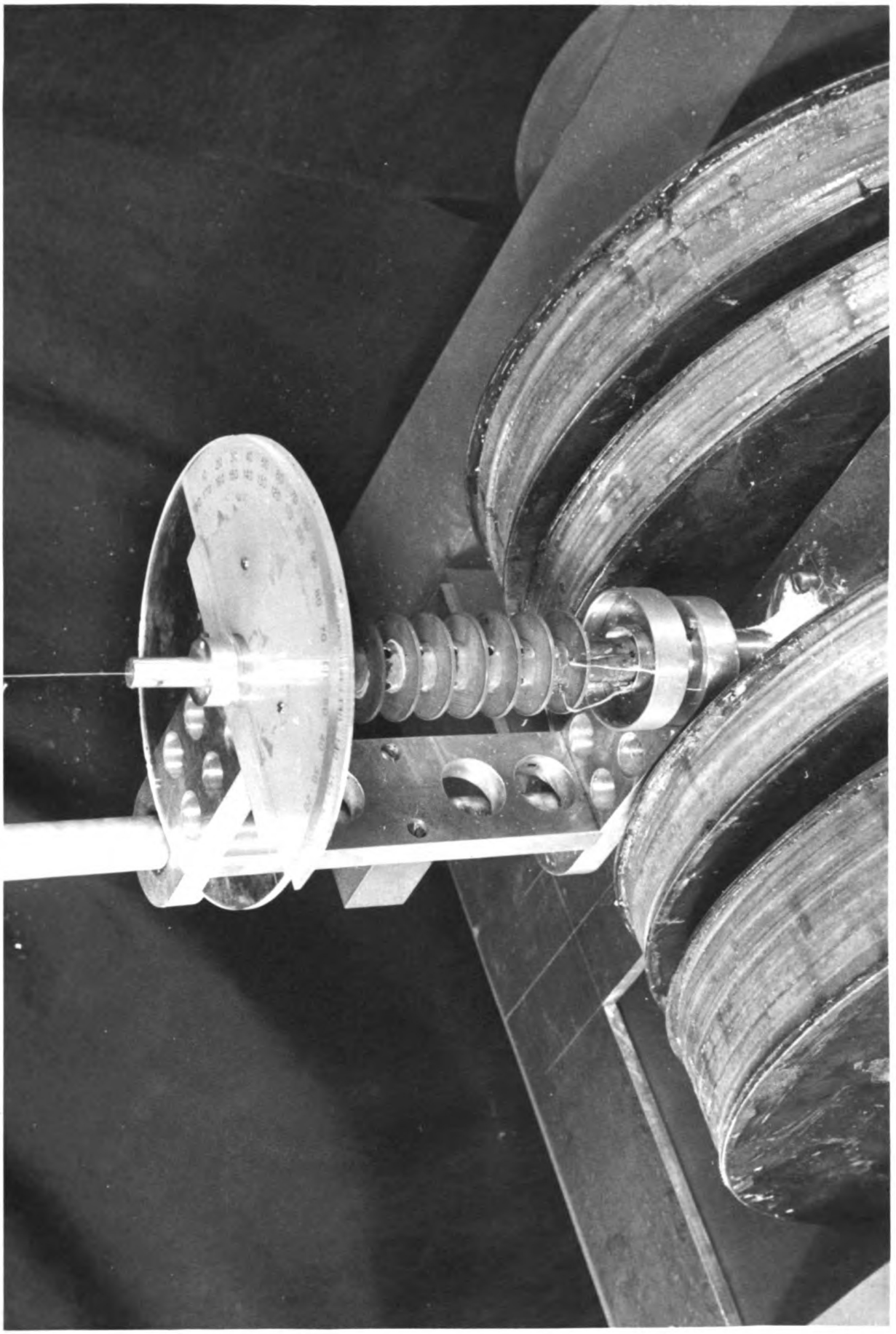


FIG. 1 THE TORQUE MAGNETOMETER



THE TORQUE MAGNETOMETER

FIG. 2

CROSS SECTION OF SAMPLE HOLDER

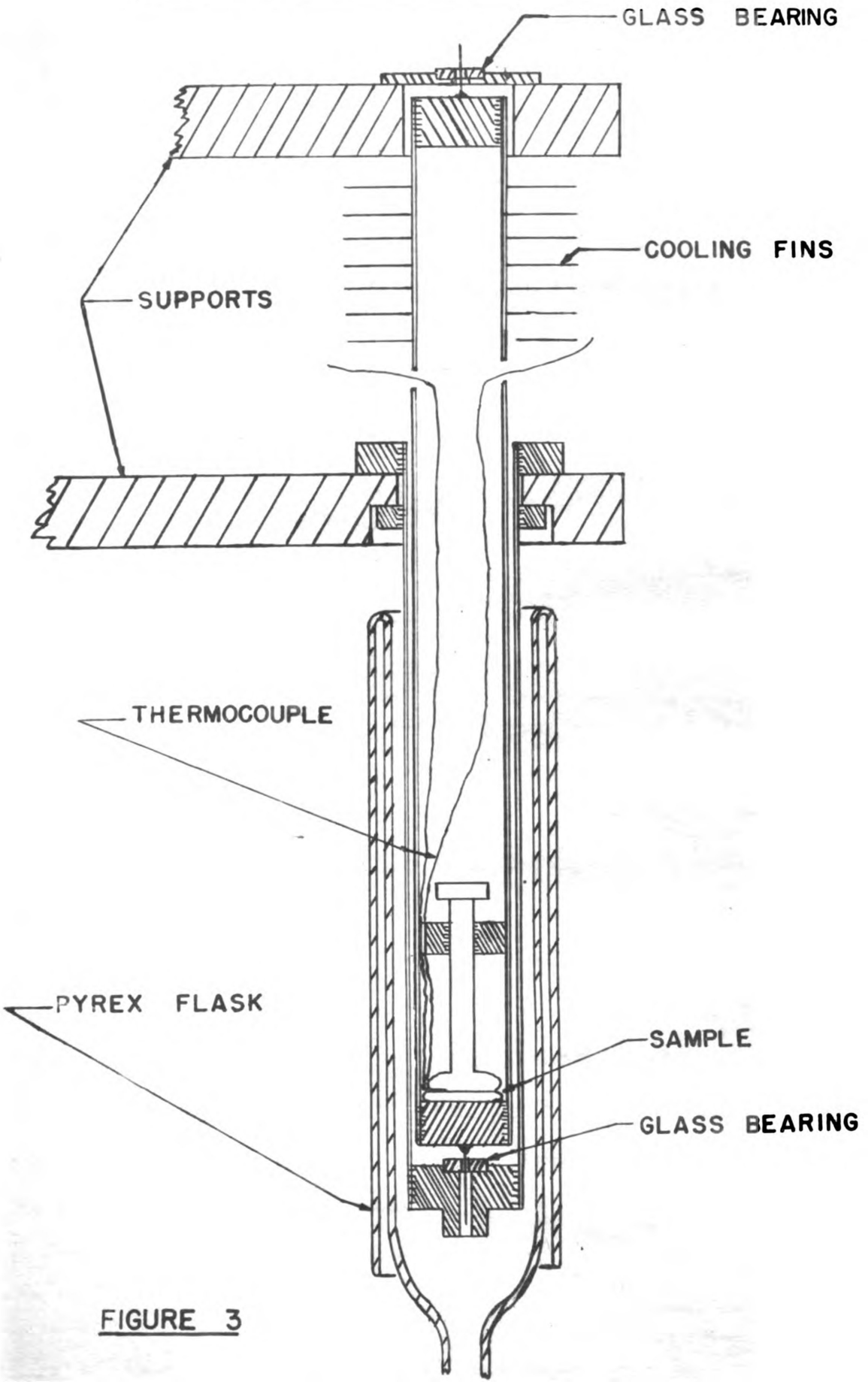


FIGURE 3

PREPARATION OF SAMPLES

Crystals of Armco iron which has a purity of 99.94 per-cent were obtained from Virginia Institute for Scientific Research in the form of rods three-eighths of an inch in diameter and about 4 inches long. When the rods were etched in a ten per-cent solution of nitric acid in water it was found that the rods contained many crystals, the largest of which was about two inches long. The crystals of nickel were obtained from Horizons Incorporated; Princeton, New Jersey, and were reported 99.50 per-cent pure by the supplier. They were in the form of rods one-half inch in diameter and about five inches long, the single crystal running the entire length of the rod.

From these rods it was necessary to cut out slices in the (100) or (110) plane. To do this it was necessary to find the orientation of the cubic lattice in the rod. It was relatively easy to find a rough orientation in the case of iron by observing the light reflected from the (100) planes. A more precise determination of the crystallographic orientation was made by the back reflection Laue X-ray technique. The crystal was clamped in a rod holder, the base of which was roughly normal to a collimated X-ray beam. A collimator was used which had an end diameter of one-fourth inch. A one-fourth inch hole was cut in the film holder and this slipped over the collimator. The Laue patterns obtained are in the form of intersecting hyperbolas as shown in Figures (4-a) and (5-a). The rod was then adjusted until the point of intersection coincided with the hole in the film. The reflecting plane, in this position, was then

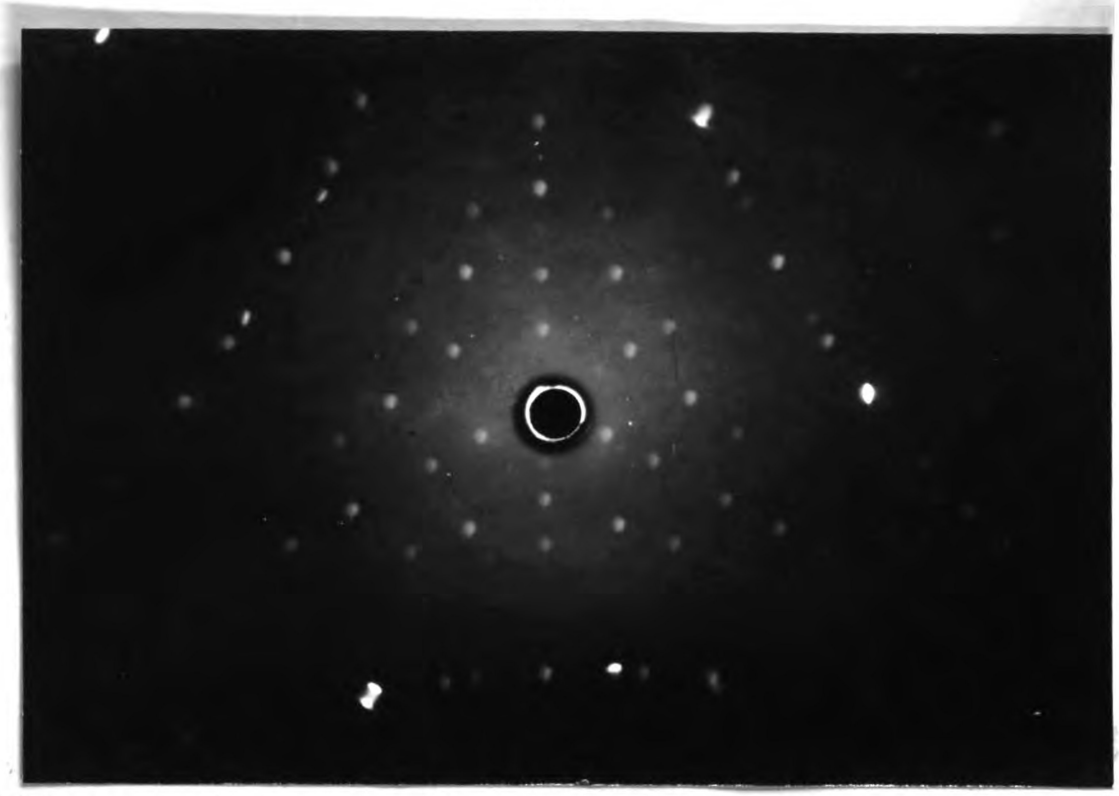
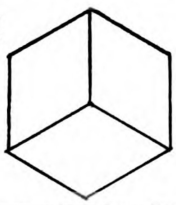


FIG. 4a



BACK REFLECTION

CUBE SHOWS LATTICE POSITION IN RELATION TO PATTERNS

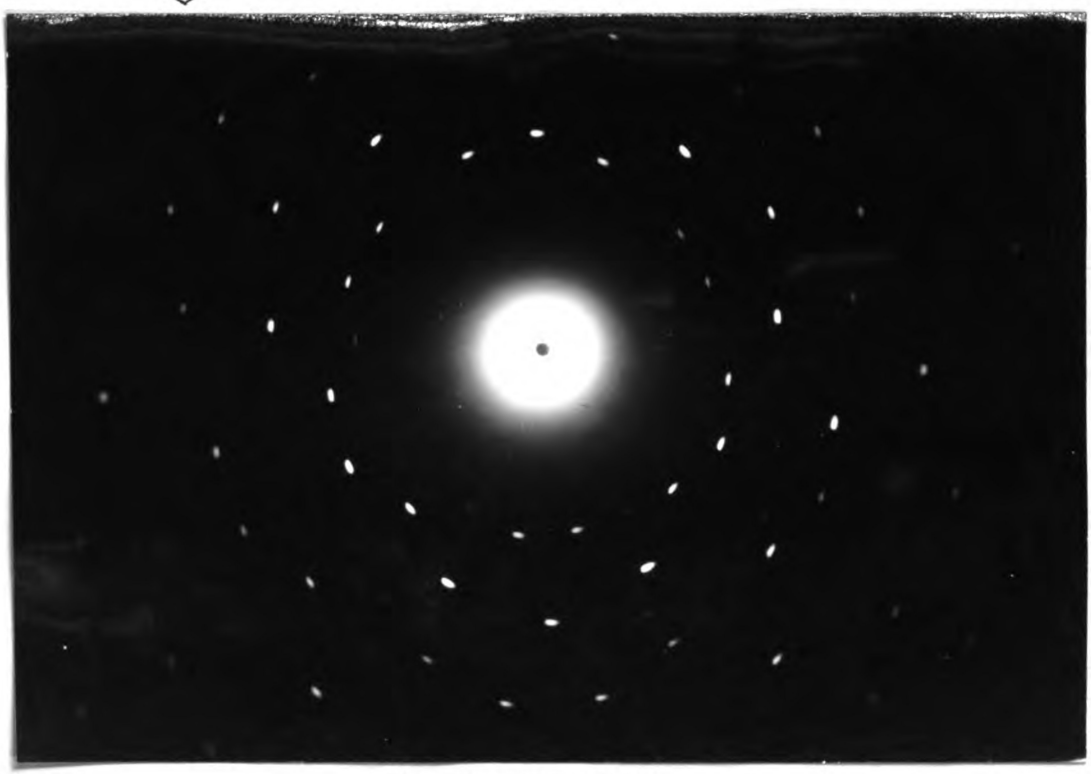


FIG. 4b

TRANSMISSION

LAUE PATTERNS OF IRON WITH THE (111) PLANE PARALLEL AND X-RAY BEAM NORMAL TO THE PAPER

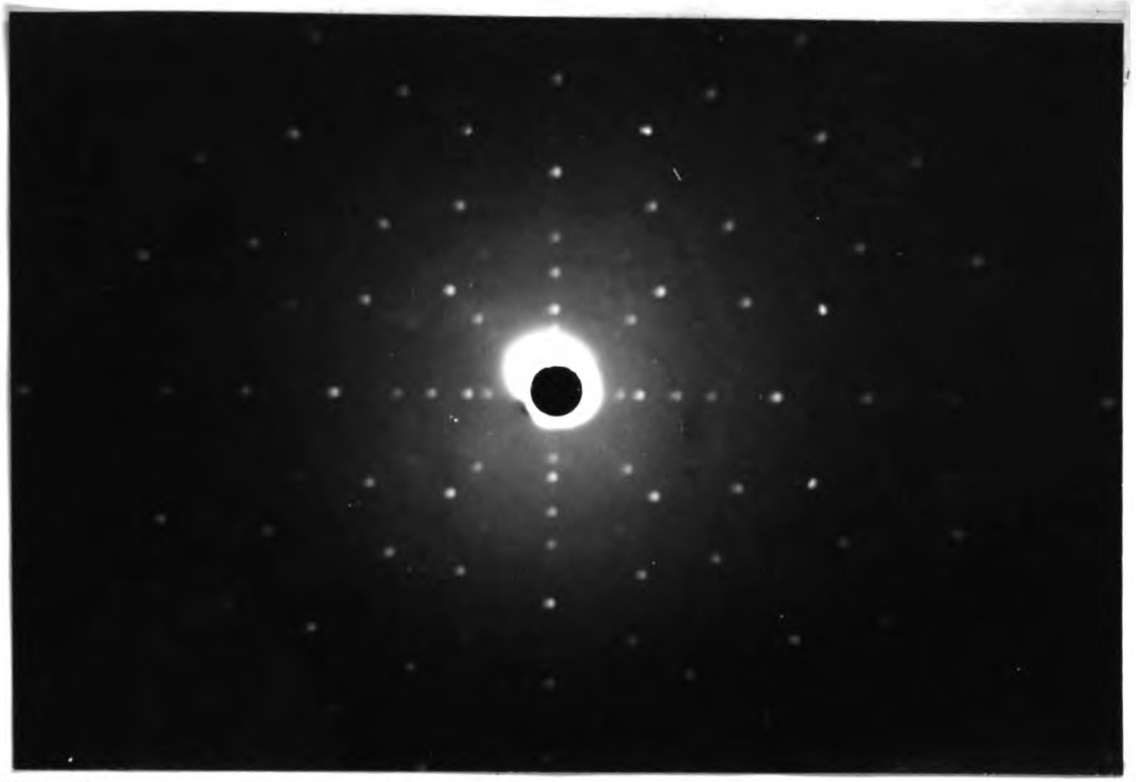
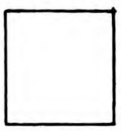


FIG. 5a



BACK REFLECTION

CUBE SHOWS LATTICE POSITION IN RELATION TO PATTERNS

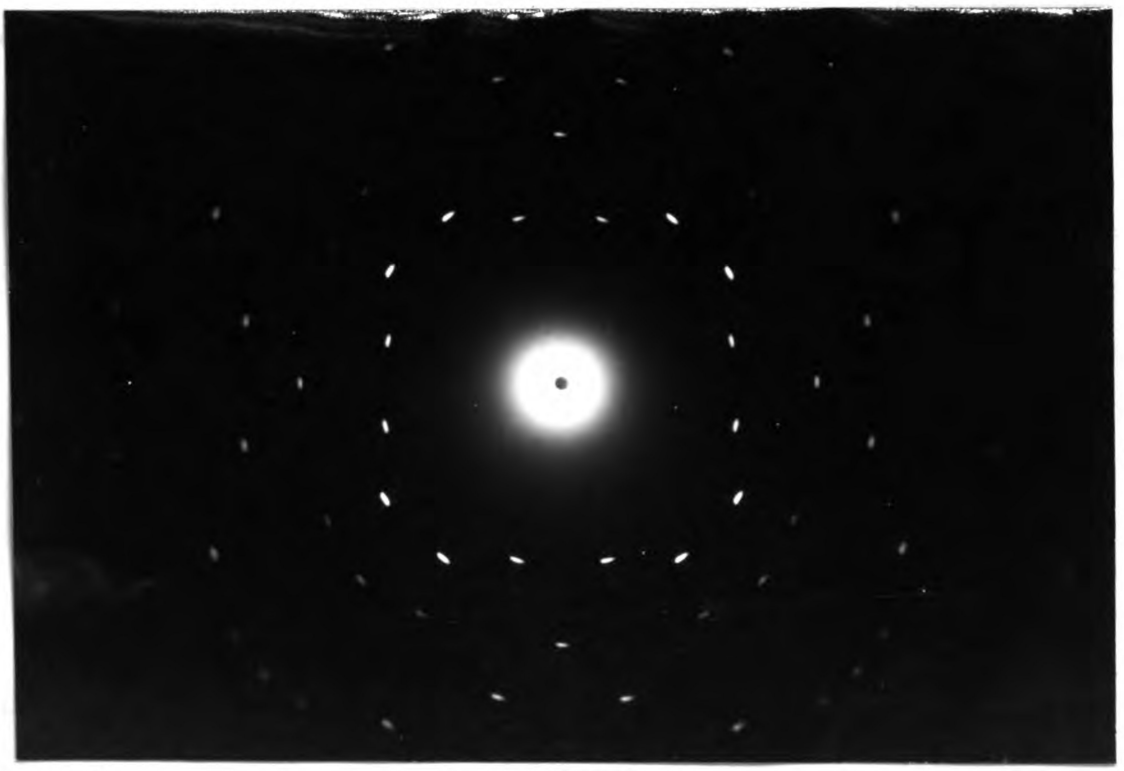


FIG. 5b

TRANSMISSION

LAUE PATTERNS OF IRON WITH THE (100) PLANE PARALLEL AND X-RAY BEAM NORMAL TO THE PAPER

also normal to the X-ray beam. The rod holder was now clamped in a Di-Met cutting saw and a slice of about .050 inch was cut out. A .020 inch abrasive wheel was used.

The orientation of the nickel crystal was somewhat more difficult due to the fact that no etching solution could be found which produced etch pits with any clarity. Therefore the X-ray back reflection orientation was basically by trial and error. The rod was adjusted until a point of intersection was coincident with the hole in the film; with the hope that the symmetry of spot patterns would be four, two, or three fold indicating a (100), (110), or (111) plane, respectively. Considering there are eight (111) planes, six (100) planes, and six (110) planes in a cubic lattice, the author was amazed at the number of planes which gave centered, intersecting patterns with no recognizable symmetry.

When a spot pattern was obtained which appeared to give the proper symmetry, the orientation was checked by rotating the crystal in a plane parallel to the lines of symmetry the proper number of degrees to bring other known planes normal to the beam. If these also gave the correct symmetry the crystal was uniquely oriented.

Since the rod holder was not accurately oriented with respect to the beam and since the process of transferring the rod from the X-ray unit to the saw introduced errors, a final orientation was made by Laue transmission patterns. To accurately position the sample holder normal to the X-ray beam, transmission pictures of cleaved rock salt were taken with the rock salt occupying the samples position. The sample holder was

adjusted until the spot patterns from the rock salt were symmetric about the central dot.

The samples were sanded with consecutively finer sandpaper and etched with nitric acid solutions to a depth of .005 inch. This removed any strains introduced by sawing. A sample was then placed in the holder, X-rayed, and the resulting pattern observed. If the spot pattern did not have all corresponding spots equidistant from the central dot, and in the (100) and (111) planes, equidistant from each other, as shown in Figures (4-b) and (5-b), the orientation was corrected by preferential sanding of one surface. Therefore, when the oriented side of the sample became parallel with the desired crystallographic plane the sample was wedge-shaped. This was corrected by sanding the other surface and checking with a micrometer until the two surfaces were parallel. The samples of iron were then about .010 inch thick and the nickel about .020 inch thick. In this manner samples were oriented which were accurate to within one degree.

The next task was to form the samples into approximate oblate spheroids. The first step was to solder the samples to the end of a brass rod. Woods metal, which has a low melting point, was used to eliminate any strains induced by excessive localized heating. The samples were then machined circular using a very sharp tool. An oblate spheroid was approximated by filing the corners with a fine toothed file and sanding with fine sandpaper while the sample was turning in a lathe. A final etch was given to remove the strained material introduced by the sanding.

RESULTS

The results of the experiment are summarized in the two graphs in Figures (9) and (10). The temperature dependence of K_1 in iron obtained in this experiment agreed very well with the values given by Honda, Kaya, and Masumoto.[2] The results for nickel, however, differ considerably from the values obtained by Honda, Masumoto, and Shirakawa.[2] They have indicated a positive value for K_1 in the temperature range from 100 to 300 degrees Centegrade whereas our results show K_1 approaching zero asymptotically and never taking on positive values.

Measurements of K_2 from samples in the (111) plane was attempted. In the iron the torque in this plane was so small that it was considered negligible. The nickel sample produced a torque curve of roughly the proper shape, $(\sin 6\theta)$, but the curve was erratic so that no quantitative results could be obtained.

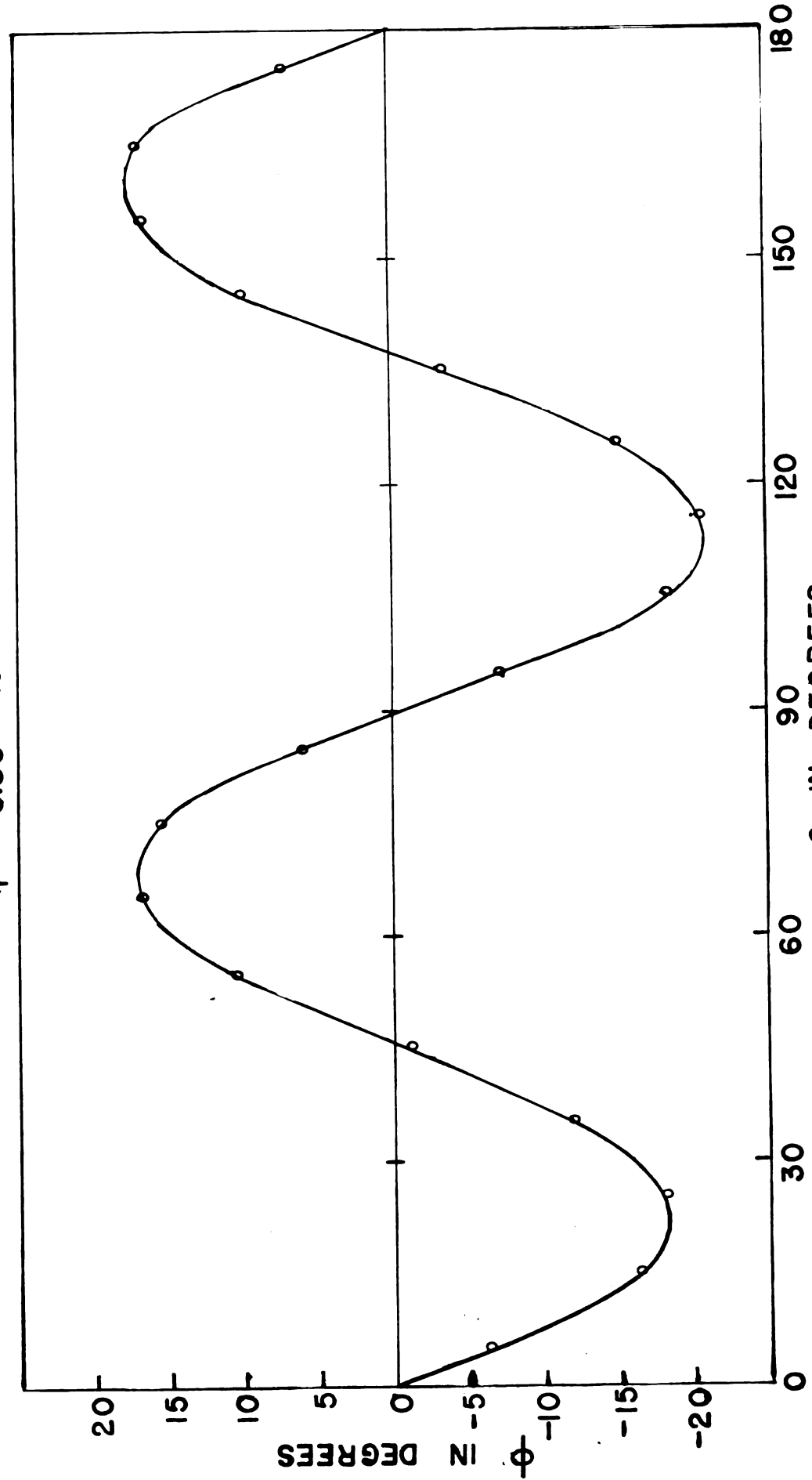

TORQUE CURVE OF .059 CC. SAMPLE OF NICKEL IN "100" PLANE
η = 3.83 x 10³ ERGS
φ IN DEGREES
θ IN DEGREES
20
15
10
5
0
-5
-10
-15
-20
0
30
60
90
120
150
180

FIGURE 6

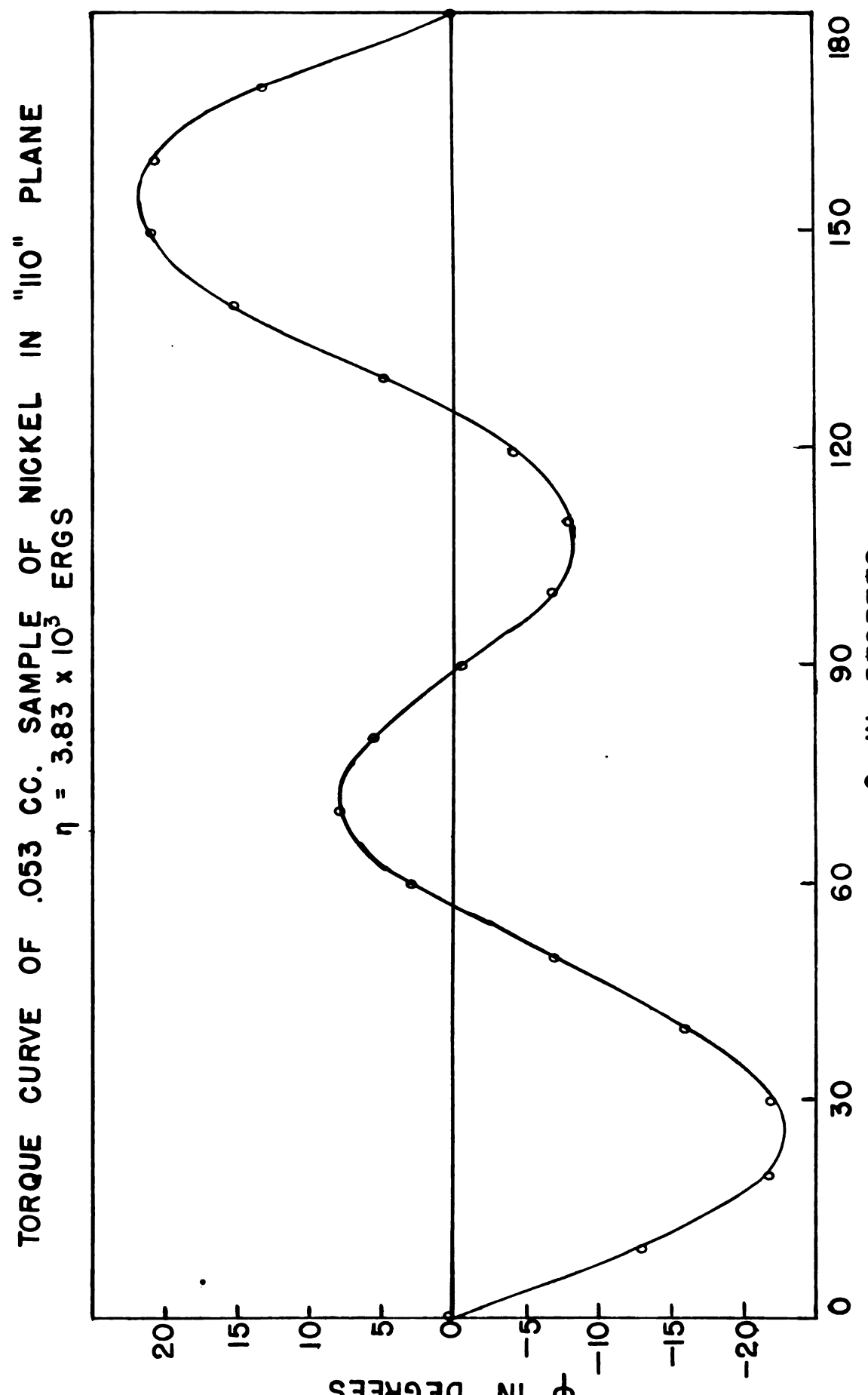

TORQUE CURVE OF .053 CC. SAMPLE OF NICKEL IN "110" PLANE
η = 3.83 x 10³ ERGS
φ IN DEGREES
20
15
10
5
0
-5
-10
-15
-20
0
30
60
90
120
150
180
θ IN DEGREES

FIGURE 7

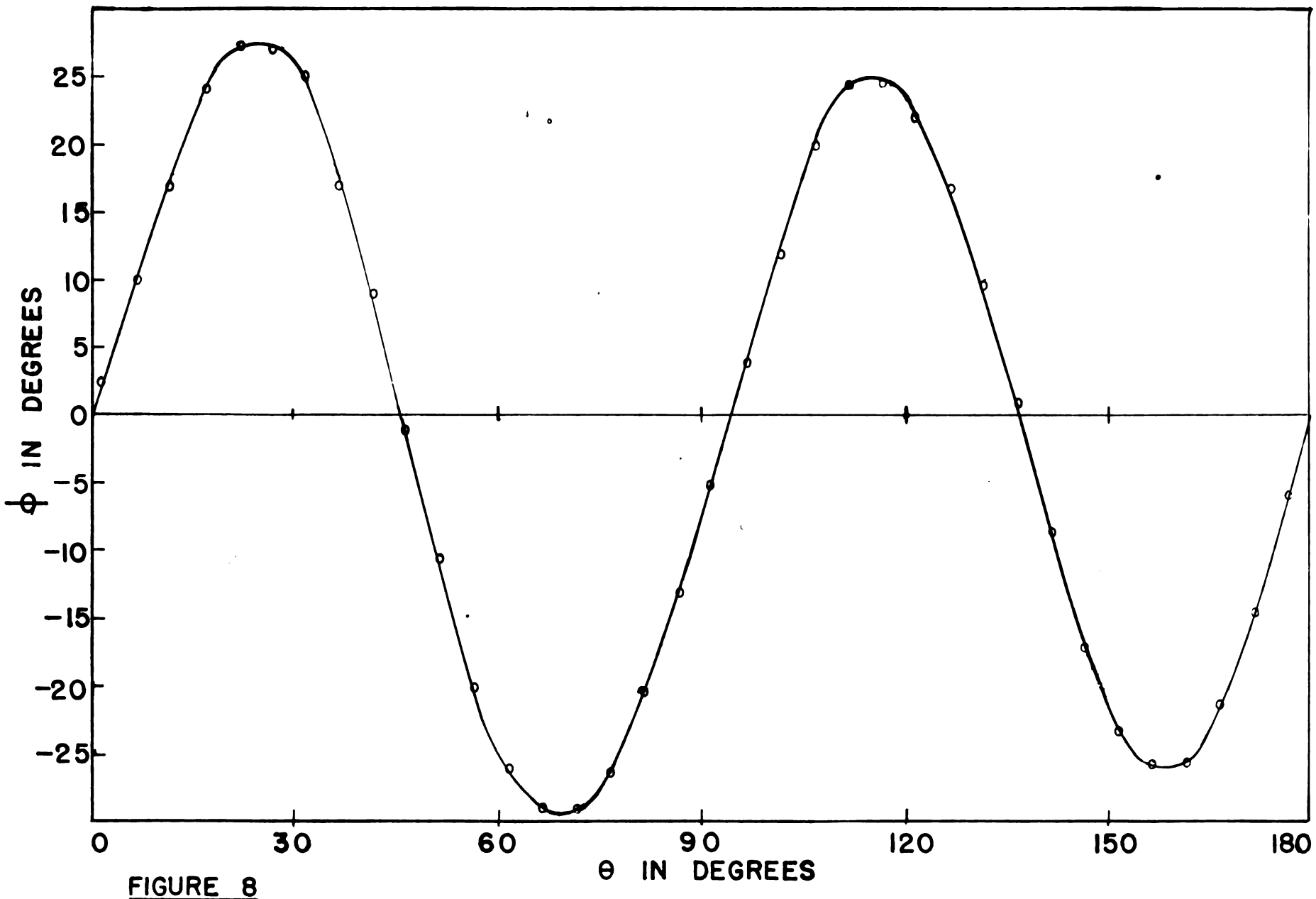

TORQUE CURVE OF .008 CC. SAMPLE OF IRON IN "100" PLANE
η = 3.83 x 10³ ERGS
25
20
15
10
5
0
-5
-10
-15
-20
-25
φ IN DEGREES
0
30
60
90
120
150
180
θ IN DEGREES

FIGURE 8

TEMPERATURE DEPENDENCE OF K_1 IN IRON SAMPLE IN "100" PLANE

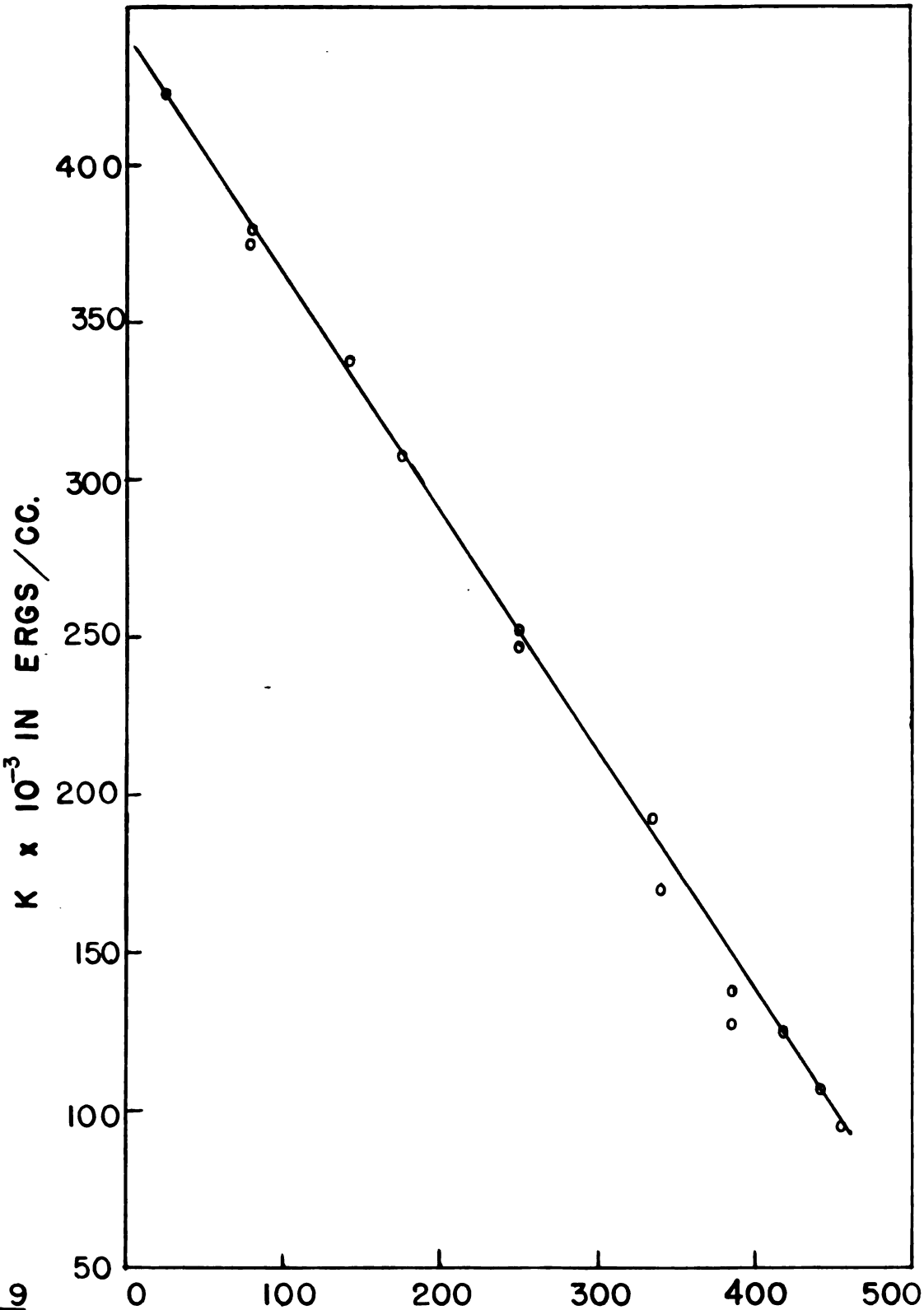


FIG. 9

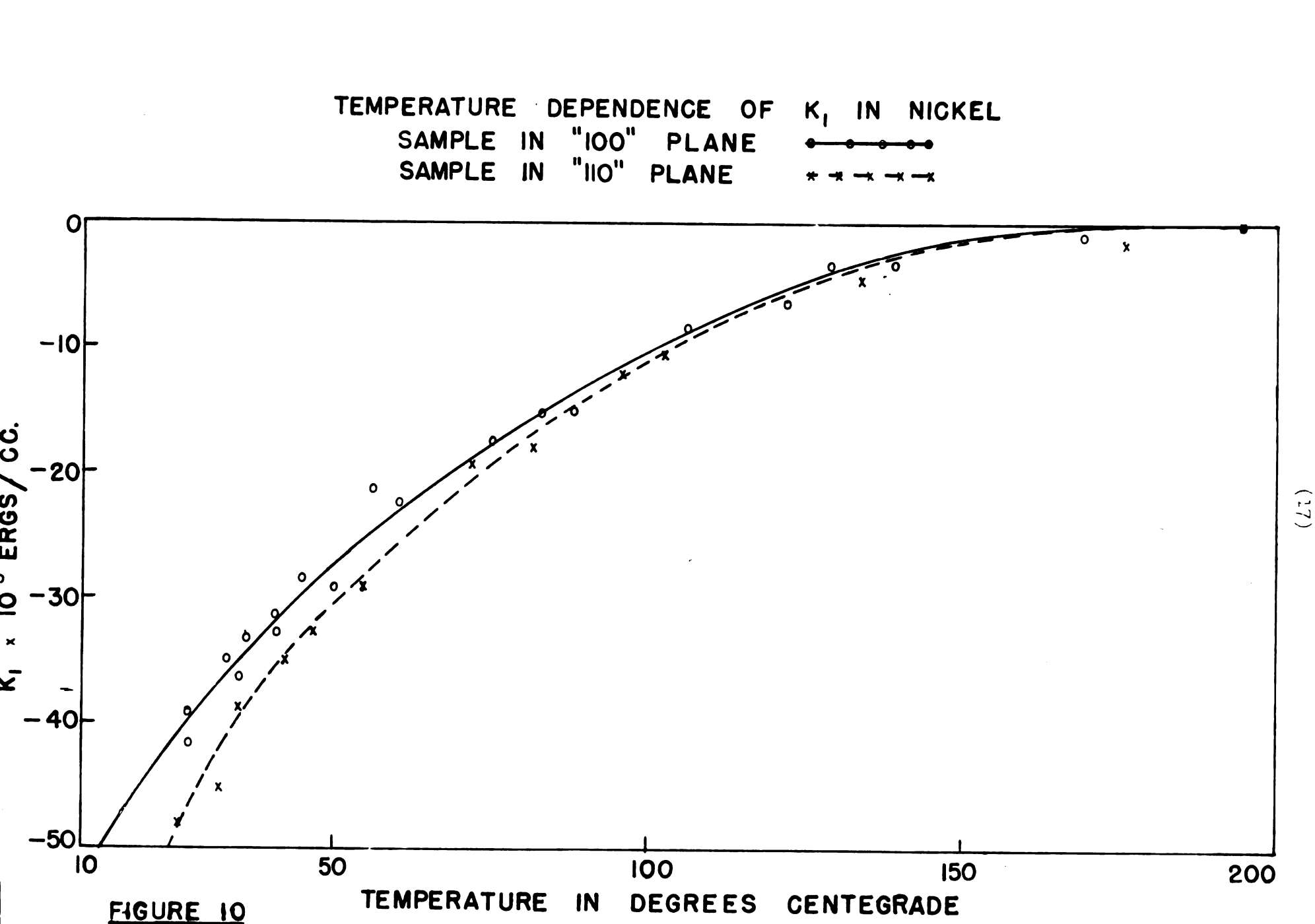

TEMPERATURE DEPENDENCE OF K_1 IN NICKEL
SAMPLE IN "100" PLANE
SAMPLE IN "110" PLANE
0
-10
-20
-30
-40
-50
$K_1 \times 10^6$ ERGS / CC.
10
50
100
150
200
TEMPERATURE IN DEGREES CENTEGRADE

FIGURE 10

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