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TEMPERATURE DEPENDENCE OF
ANISOTROPY CONSTANTS OF AN
ORIENTED FE-SI ALLOY

Thesis for the Degree of M. S.
MICHIGAN STATE COLLEGE
Clyde Arthur Morrison
1951

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R. D. Spence

Major professor

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TEMPERATURE DEPENDENCE OF ANISOTROPY CONSTANTS
OF AN ORIENTED FE - SI ALLOY

BY

Clyde Arthur Morrison

A Thesis

Submitted to the School of Graduate Studies of Michigan

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I dedicate this thesis especially to Dr. Robert Spence, for without his advice and friendship it never could have been accomplished. I secondly wish to thank Charles Kingston for his untiring efforts in constructing the needed technical equipment.

Clyde Arthur Morrison

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I. INTRODUCTION

In single crystals of iron, nickel, and various substances, the magnetic properties depend on the direction in which they are measured. When a substance is found to exhibit these characteristics it is said to be magnetically anisotropic. The knowledge of the reaction of certain elements and compounds to a magnetic field is of special interest to manufacturers of certain types of magnetic and electromagnetic equipment. It is also of interest in science in the building of a more complete theory of magnetism.

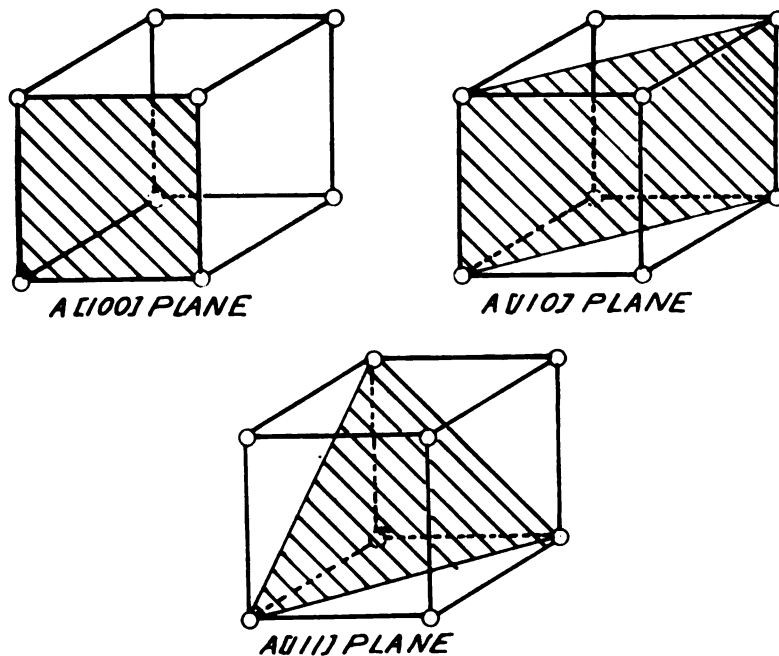


Fig. 1. Showing the three most common planes of a simple cubic crystal.

II. THEORY

The iron crystal is an excellent example of a substance exhibiting magnetic anisotropy. It is customary to express the

magnetization direction with respect to the crystallographic axis in Miller indices. These are the reciprocals of the intercepts of planes passed through various atoms in the elementary cell, as shown in Fig. 1. The $[100]$ direction would then be all directions perpendicular to a $[100]$ plane. Similarly, the $[110]$ and the $[111]$ directions would be perpendicular to $[110]$ and $[111]$ planes respectively.

In iron, experiment has shown the $[100]$ direction is the easy direction of magnetization, that is, less energy is necessary to bring the crystal to magnetic saturation along this axis than any other in the crystal. The intermediate direction is the $[110]$, and the direction of hard magnetization is the $[111]$. On the other hand, nickel crystals have their easy, hard, and intermediate directions of magnetization as the $[111]$, $[100]$, and $[110]$ respectively.

In a quantitative measurement and evaluation of the anisotropy constants the following expression is found to be the most convenient.

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

where K_0 is isotropic, K_1 and K_2 are the first and second anisotropy constants respectively. The α_i 's are the direction cosines of the magnetization vector with respect to a crystallographic axis. The terms in α_i^2 are missing, since

$$\alpha_1^2 + \alpha_2^2 + \alpha_3^2 = 1 \quad (2)$$

The terms in α_i^4 are missing, because

$$(\alpha_1^2 + \alpha_2^2 + \alpha_3^2)^2 = 1 = \alpha_1^4 + \alpha_2^4 + \alpha_3^4 + 2(\alpha_1^2 \alpha_2^2 + \alpha_2^2 \alpha_3^2 + \alpha_3^2 \alpha_1^2) \quad (3)$$

$$\frac{1 - (\alpha_1^4 + \alpha_2^4 + \alpha_3^4)}{2} = \alpha_1^2 \alpha_2^2 + \alpha_2^2 \alpha_3^2 + \alpha_3^2 \alpha_1^2$$

and, therefore, yield no terms differing from (1). In many cases it is unnecessary to carry the expansion beyond the terms involving

$\alpha_1, \alpha_2, \alpha_3$, since the coefficients of succeeding terms are very small and very difficult of measure experimentally.

A theory of magnetic anisotropy was first prepared by Mahajani.⁷ By assuming small, round, flat magnets, magnetized perpendicular to their plane at the lattice points of a body centered cubic lattice the magnets will be stable when the direction of magnetization of these magnets is the $[100]$ direction. Similarly, if the assumed magnets are bar shaped and their magnetization is in the long direction, the stable direction will be the $[111]$ direction if each such magnet is placed at a lattice point of a face centered cube. This explains qualitatively the reason for iron having the easy direction of magnetization as the $[100]$ and nickel the $[111]$.

Using a phenomenological quantum mechanical theory, Van Vleck¹⁰ calculated the anisotropy constant K_1 . The theoretical value agrees with experiment in magnitude and sign; it shows that K_1 should be temperature dependent. Due to dipole-dipole interaction the value for K_1 is $\frac{A^2}{10\lambda k T_c}$, and of the order of $\frac{A^2}{\lambda^2}$ if due to quadrupole-quadrupole effect. Where A is the spin orbit constant, T_c the Curie temperature, and λ is a quantity of the order of magnitude of the separation of energy levels caused by the interaction of the orbit with the crystalline field. However, the value of K_1 thus predicted, does not fall off as rapidly as found experimentally. Van Vleck suggests that the above relation be considered a function of temperature to be determined rather than the one chosen.

In this research, the values of K_1 are found at various temperatures from 25° centigrade to 450° centigrade; and from this we obtain an empirical temperature dependance for K_1 . Williams and

Bozorth¹³ have used an empirical form

$$K_1 = K_0 e^{-\alpha r^2} \quad (4)$$

for nickel, and a somewhat similar expression will be employed here for iron.

The above relations given for the anisotropy energy are, in a strict sense, applicable to only single crystals. However, in fabricated materials the separate crystals composing the material are usually oriented in a special way. This orientation is often determined by the method employed in heat treatment or in rolling and recrystallization of the stock. For example, in cold-rolled silicon steel sheets of the type used in the present work, the plane of the sheet coincides with the $[110]$ plane, and the $[100]$ direction is the direction of rolling.³ Whereas, cold-rolled iron has a 45° angle between the direction of rolling and the easy direction of magnetization.² The orientation of the crystals may be determined by the Von Laue spot diffraction of X-rays,⁶ or they may be determined by the comparison of curves obtained experimentally to those predicted for a crystal assumed to be in the same configuration.¹¹

Since there is a degree of orderliness in a polycrystalline material, a sample cut from this material should exhibit magnetic anisotropy. That is, the material should show a preferred direction of magnetization, and the relation for the anisotropy energy in terms of the crystallographic axis is still applicable. The relative magnitude of the anisotropy constants will, however, depend on the degree of orderliness.

The relation between the torque and the anisotropy energy is

$$\tau = - \frac{\partial E}{\partial \theta} \quad (5)$$

where θ is the angle, as measured in the plane of the sample, between

the magnetization vector and a crystallographic axis. The usual convention is to measure θ from the direction of easy magnetization. At sufficiently high field intensities (1000 - 2000 Gauss) the anisotropy energy is the only term in the total energy of a ferromagnetic which is angle dependent. An example where θ is measured in a [100] plane, the direction cosines are $\alpha_1 = \cos\theta$, $\alpha_2 = \sin\theta$, and $\alpha_3 = 0$. The torque is then

$$T = \frac{K_2}{2} \sin 4\theta \quad (6)$$

Similarly, for a sample where θ is measured in the [110] plane from the [100] direction.

$$T = \frac{K_1}{8} (2\sin 2\theta + 3\sin 4\theta) + \frac{K_2}{8} (\sin 2\theta + 4\sin 4\theta - 3\sin 6\theta) \quad (7)$$

Expressions for the torque of a sample in various planes are given by Bozorth.²

III. EXPERIMENTAL APPARATUS

In the experimental work to be described in this thesis, the anisotropy constant is determined from torque curves. Such curves are obtained by plotting the torque exerted by the field on the sample, versus the sample's orientation in the magnetic field. Once the experimental curve is plotted the constants K_1 and K_2 can be determined.

The instrument constructed to find the torque curves was a torison magnetometer, an instrument quite similar to the one constructed by Williams.¹² A torison fiber is calibrated so that the torque can be computed directly from a given deflection when the magnetic field is on.

The constant for the fiber was computed by a torison pendulum method. The wire used for the suspension was the same as in the magnetometer constructed by Kropschot,⁴ and the particulars of the

method can be found in his thesis. The constant was found to be in good agreement with the value given by Kropschot. The torque is given by

$$T = 7,257 \varphi \quad (8)$$

where φ is the angular separation, in radians, of the two indicators on the magnetometer

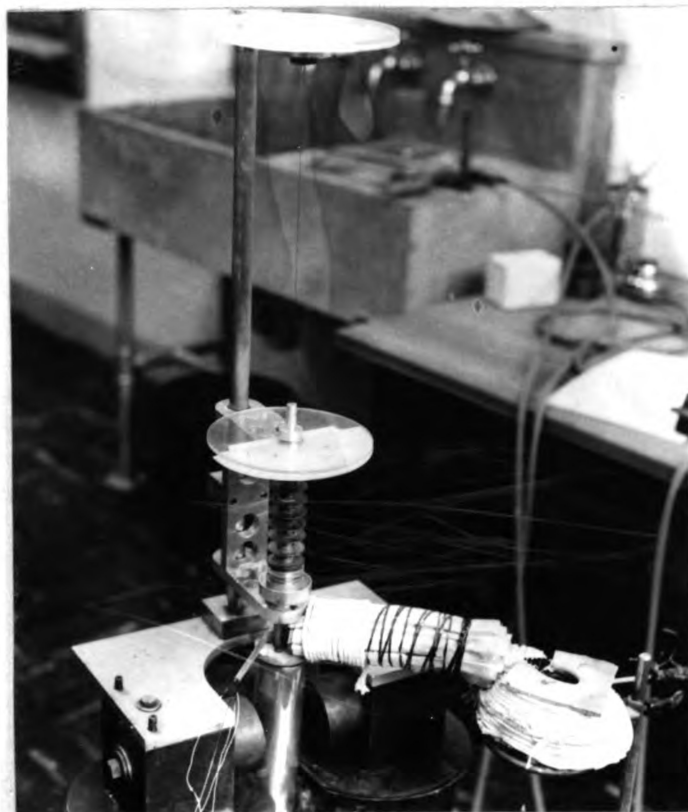


Fig. 2. The Torque Magnetometer

To obtain temperatures above room temperature in the sample, a coil was made of stainless steel tubing. The coil was wrapped with asbestos, and one end connected to a nitrogen tank. The other end was run through an asbestos washer into a vacuum flask surrounding the sample. Nitrogen was used since it does not react with the iron sample, and does not form a scale of any kind on the sample. The coil was wound cone shaped so that it could be heated most effectively by a torch applied beneath it. To obtain a temperature of 400° C.

in the sample, it was necessary to apply a pressure of about eight pounds per square inch from the nitrogen tank, and run the torch about medium heat. The heated gases were allowed to escape through a glass tube, running deep into the vacuum flask, on the opposite side of the asbestos washer. In order to keep the heat from reaching the suspension and changing its modulus of rigidity, a number of copper fins were added to the top of the sample holder as shown in Fig. 4. As a second precaution, an aluminum cup was added below the

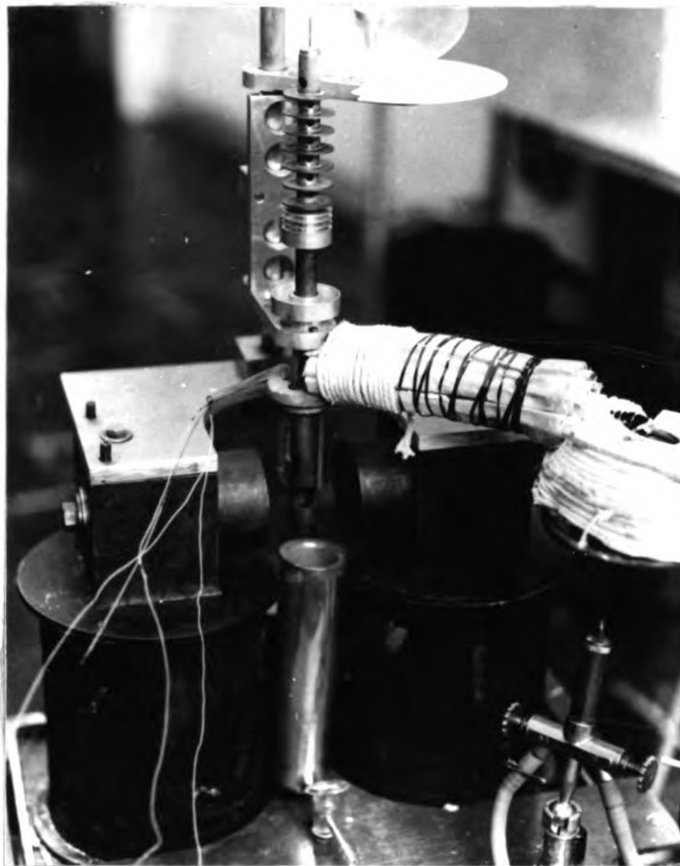


Fig. 3. Torque Magnetometer disassembled.

indicator dial in such a way that a smaller inverted cup fastened to the sample holder fitted down into it. This cup could be filled with a liquid to keep the heat from rising any higher on the sample holder. The addition of liquid was unnecessary as the copper fins kept the heat from reaching the suspension.

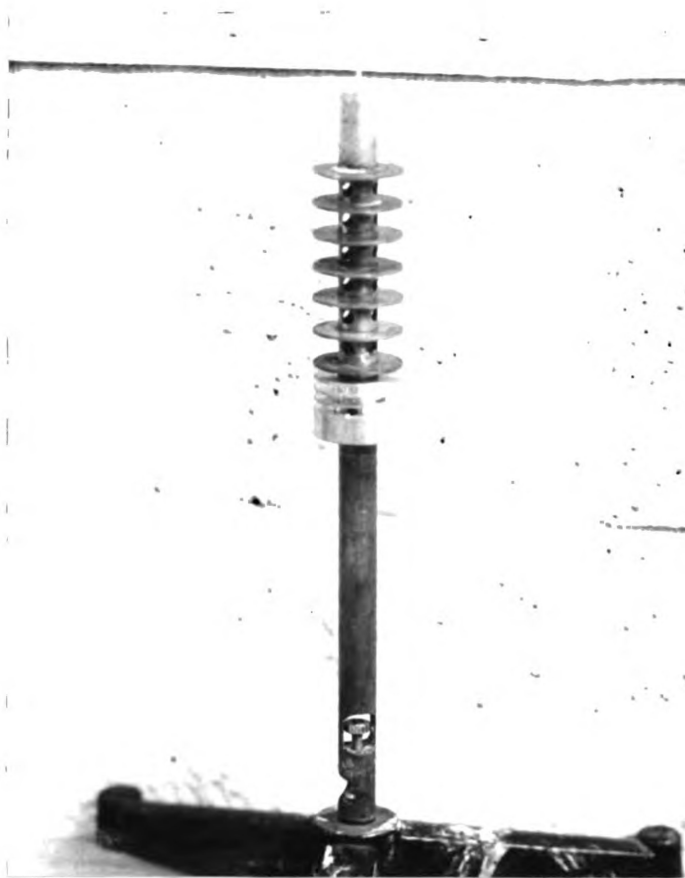


Fig. 4. Sample holder.

The temperatures were recorded by a copper Constantine thermocouple connected to a potentiometer. The thermocouple entered the flask through the glass tube used for an exhaust for the hot gas. After running through the glass tube the thermocouple was wrapped with a thin layer of asbestos and tied to the side of the bearing support about $1/16$ " from the sample. To determine the relation between the temperature of the sample and the temperature where the thermocouple was fastened, a second thermocouple was run through the glass tube and fastened into the sample holder. Heat was then applied to the sample holder by the method described above, and the temperature was recorded from the thermocouples simultaneously. The results showed that both thermocouples registered the same temperature

whenever the temperature reached a stationary value. To convert the e.m.f. from the thermocouple to temperature, a graph was plotted with data taken from the Handbook of Chemistry and Physics. This plot was checked by taking three standard points: the boiling point of water, the freezing point of lead, and the condensing point of sulphur. The three points were found to agree with the curve plotted from the handbook. The thermocouple connected to a potentiometer was then used to calibrate an automatic temperature recorder connected to a second thermocouple. A permanent temperature record of a number of readings was then obtained, and any sudden change in the temperature could be instantly detected.

IV. PREPARATION OF SAMPLES

The sample holder was constructed to accommodate samples about the size of a dime. These samples were prepared from a sheet of three per cent silicon steel called Silectron. The sheet was about .0260 cm. thick, so that all the samples prepared had a very small demagnetizing factor.² The method of preparation was quite similar to the method given by Mc Keehan.⁸ A rather large hexagonal piece of stock was cut and soldered to a $\frac{1}{4}$ " brass shaft. The shaft was then inserted in a collet lathe, and the sample turned down to the desired diameter. To insure uniform saturation within the material, the samples were cut into oblate spheroids. This was accomplished by fastening the brass shaft into a chuck mounted on a small electric motor. To take the rough edge off the sample, a file was held against the corners while the motor was running. A large ellipsoid was then drawn to scale on a piece of cross section paper, and pasted on the wall of a dark room. A mercury point source light was set behind the

sample so that the cross section of the sample was projected to the graph. Then, with the motor running, a fine abrasive was held against the sample until its cross section as projected just fit the ellipse. After a number of samples were prepared in this way they were removed from the brass shafts and the solder scraped off while still molten. A mild etching solution of nitric acid and distilled water was used to remove any excess waste left on the edges of the sample.

Since it is quite customary to express the anisotropy constants in terms of ergs/cm^3 , it is necessary to measure or compute the volume of each sample. This was done by finding the weight and dividing by the density of the stock. An average density of 7.61 gm/cc was used. The volumes of the samples were near 0.030 cc., which is too small to determine directly, to a very large degree of accuracy, by an experimental method.

V. HEAT TREATMENT

Before data was taken on the temperature dependence of the anisotropy constant an experiment was performed to see if any permanent change in crystalline orientation occurred when the material was subjected to high temperatures. It was thought that the material might be under strain. By heating to a high temperature for a period of time and controlling the cooling process these strains could be removed.

A number of Silectron disks were prepared for the heating process, and their torque curves were recorded from the magnetometer. After recording the torque curves, the samples two at a time, were inserted in a slot in a small square lava block. These samples were in contact with a thermocouple which entered through the back of the block. The block was then placed in an oven capable of reaching temperatures of

1500° C.

After subjecting the samples to temperatures ranging from 600° C. to 1100° C. for several hours, they were removed and their torque curves again recorded. The curves obtained were identical with those recorded before heating. Consequently, the anisotropy constants determined at a high temperature will be dependent on the temperature of the sample and not a permanent change due to internal strains.

VI. EXPERIMENTAL PROCEDURE

To obtain the torque curves above room temperature it is necessary to set the nitrogen tank pressure at a low value and wait until the temperature reaches a stationary value, which requires fifteen to thirty minutes. Since it is impossible to predict beforehand the exact temperature at which this will occur, the temperatures

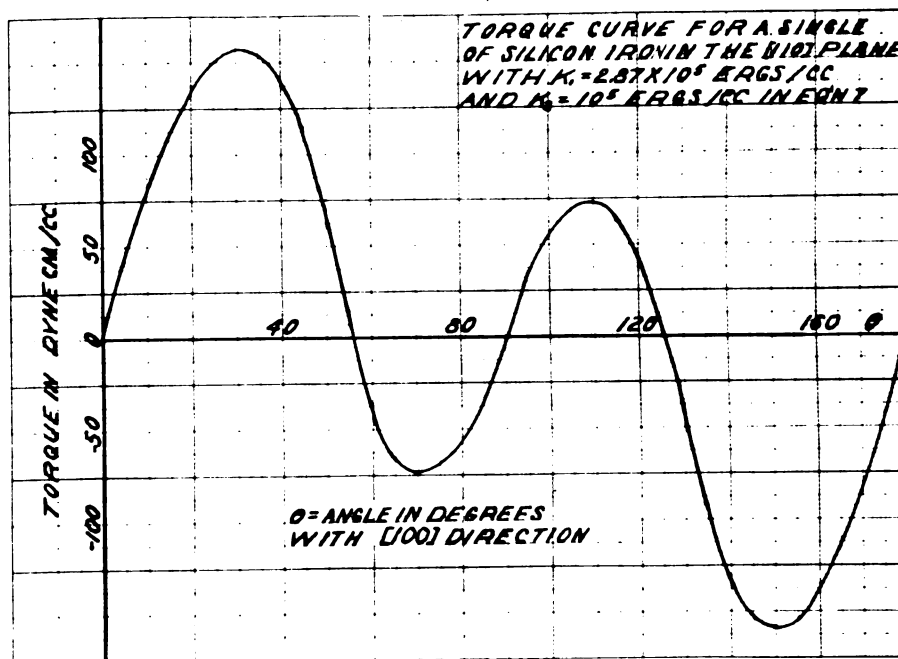


Fig. 5

appear on the graph with a certain degree of haphazardness.

To find the orientation of the sample a comparison was made of the torque curve obtained from the sample to one plotted using the theoretical expression for the torque on a sample in the $[110]$ plane.

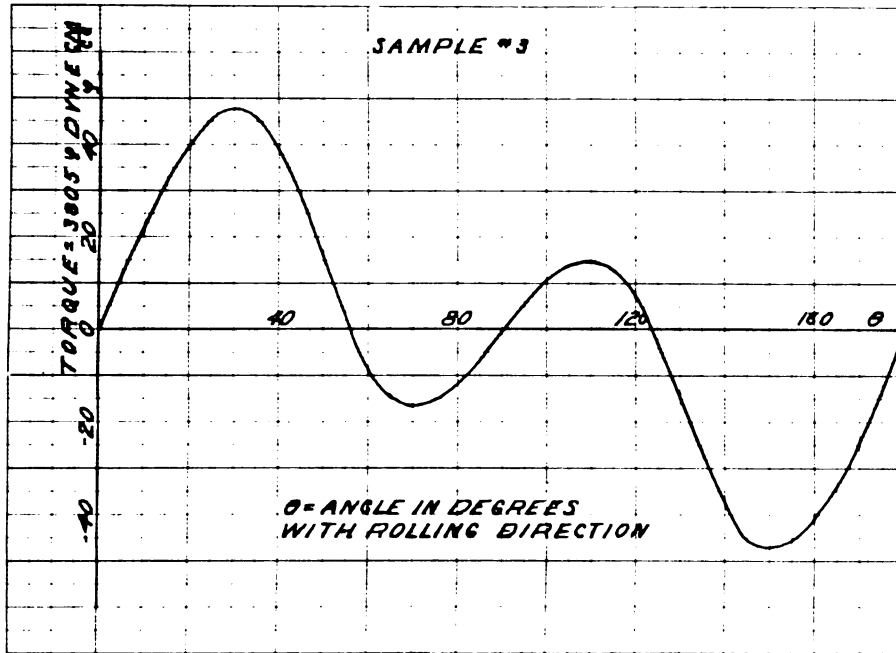


Fig. 6

From Fig. 5 and 6 it is clear that the sample lies in the $[110]$ plane. The curve obtained experimentally appears inverted with respect to those found in the literature, this is due to a rather arbitrary convention of choosing which indicator's reading shall be subtracted from the other. In all the curves plotted the top indicator was chosen positive for convenience giving curves similar to Fig. 5.

The expression for the torque on a sample in the $[110]$ plane was given in equation 7, and it is easily seen that

$$\left(\frac{\partial T}{\partial \theta}\right)_{\theta=0} = 2K,$$

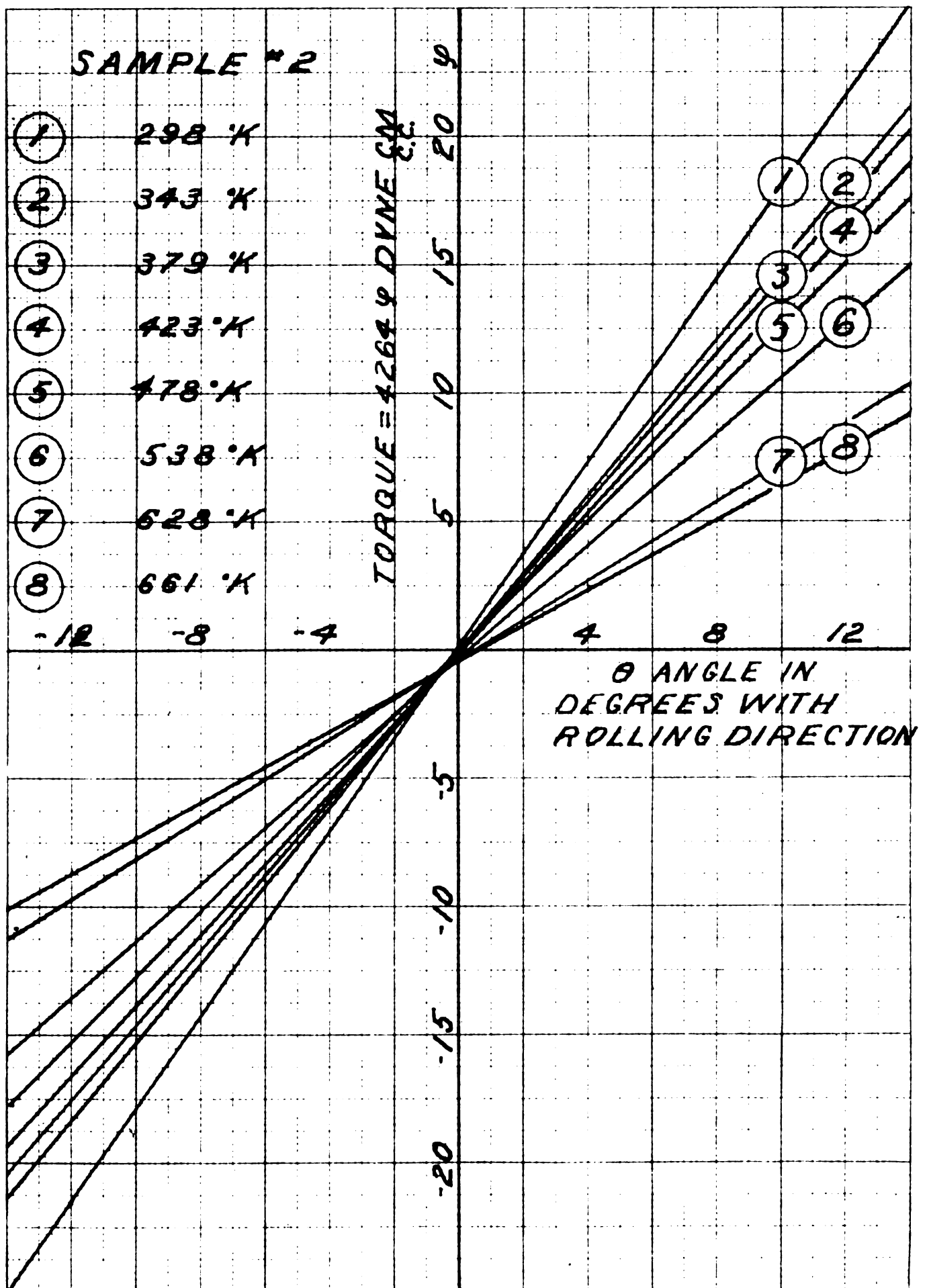
The torque curve is quite linear for some distance near $\theta=0$, and a small part of the curve can be expanded quite accurately in this region. The anisotropy constant K_1 can then be computed directly from the slope of the curve obtained. A value of K_2 can be determined from

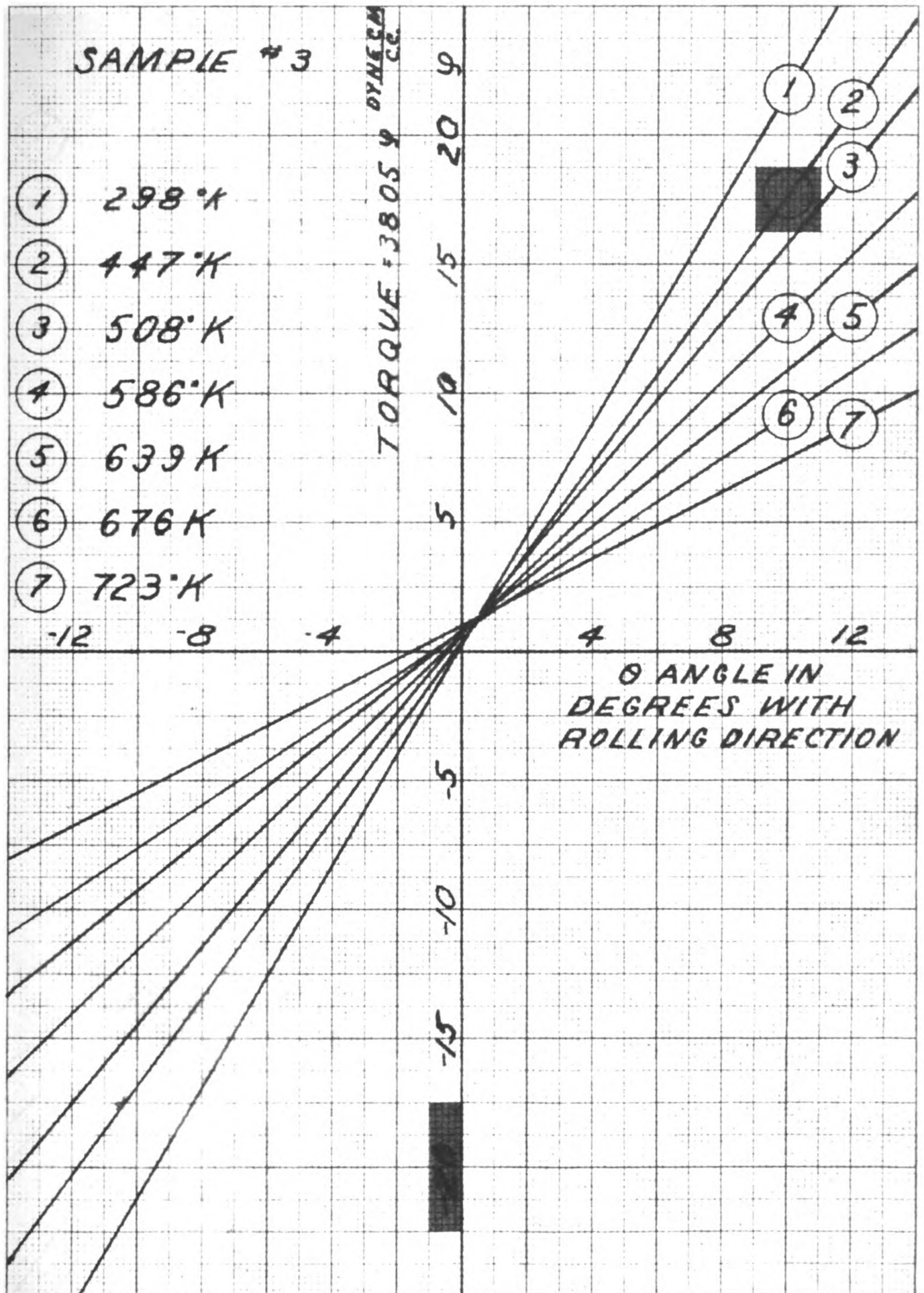
$$\left(\frac{\partial T}{\partial \theta}\right)_{\theta=\frac{\pi}{2}} = K_1 + \frac{K_2}{2}$$

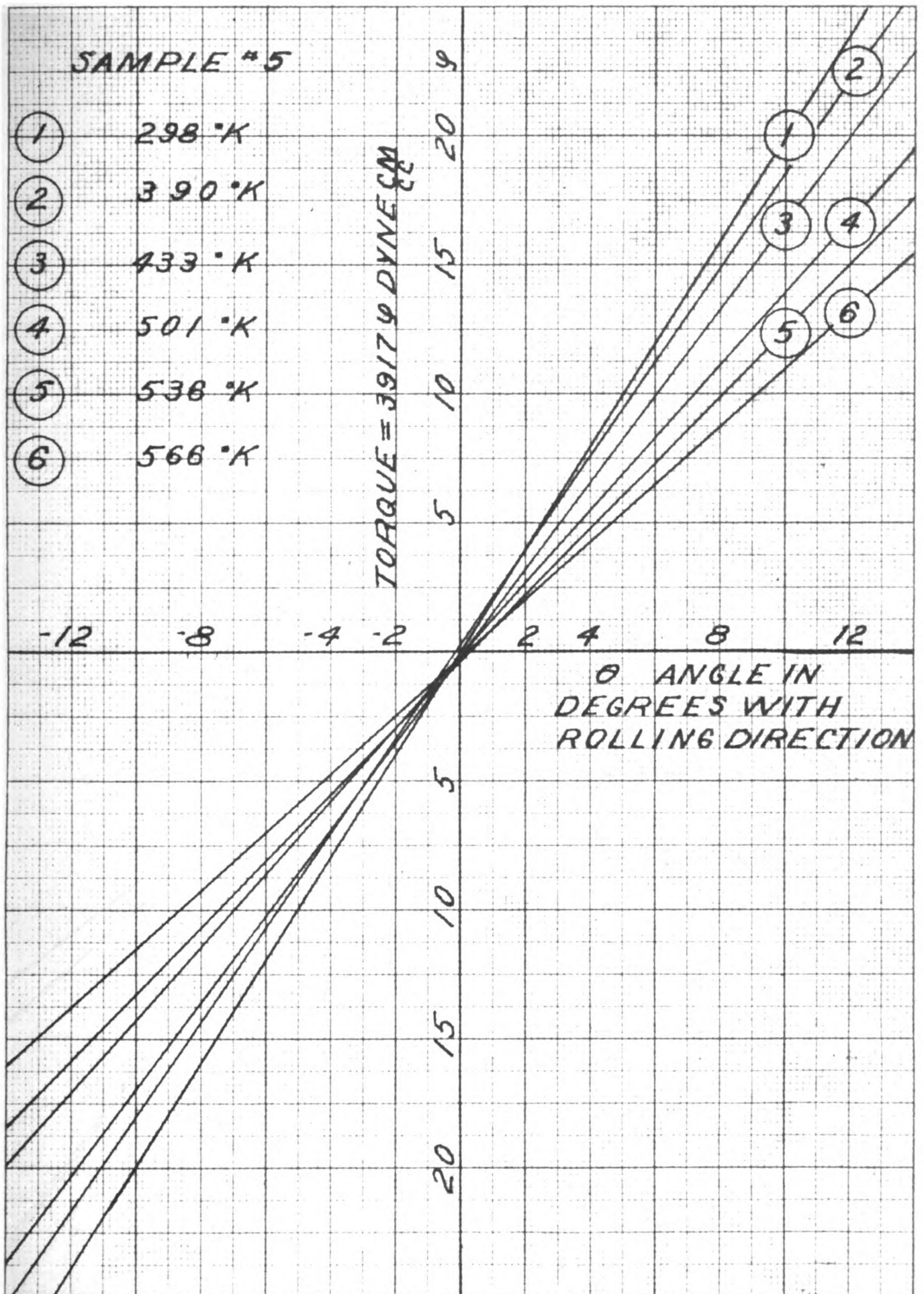
The curve can be expanded in the region $\theta=\frac{\pi}{2}$ and K_2 computed after a value of K_1 is determined. However, the region linearity is much smaller and the results for K_2 are very unreliable.

VII. Data

Following are some of the graphs extended about $\theta=0$ at various temperatures for the three samples of oblate spheroids of Silectron.



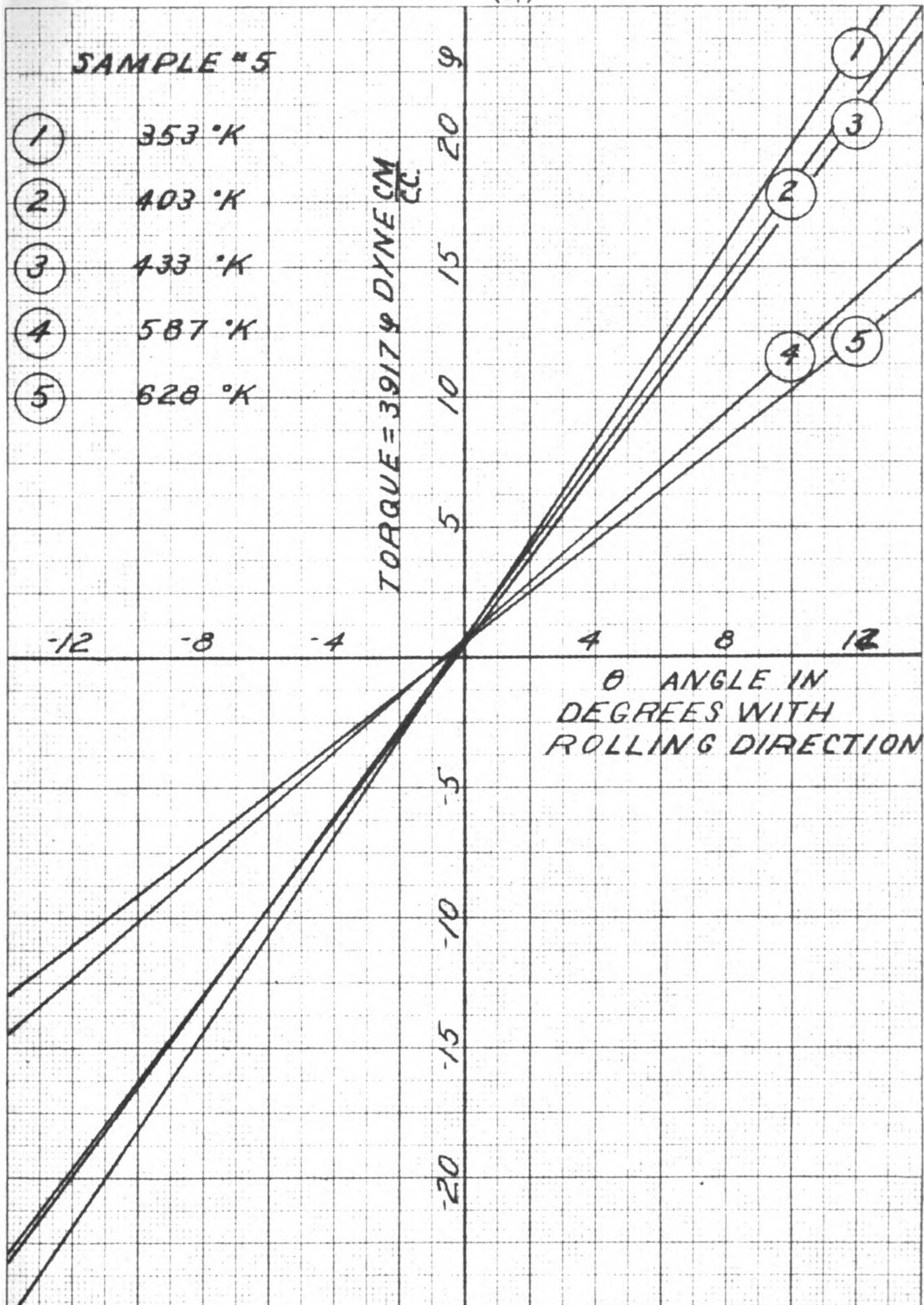




SAMPLE #5

- | | |
|---|--------|
| ① | 353 °K |
| ② | 403 °K |
| ③ | 433 °K |
| ④ | 587 °K |
| ⑤ | 628 °K |

TORQUE = 39179 DYNE CM

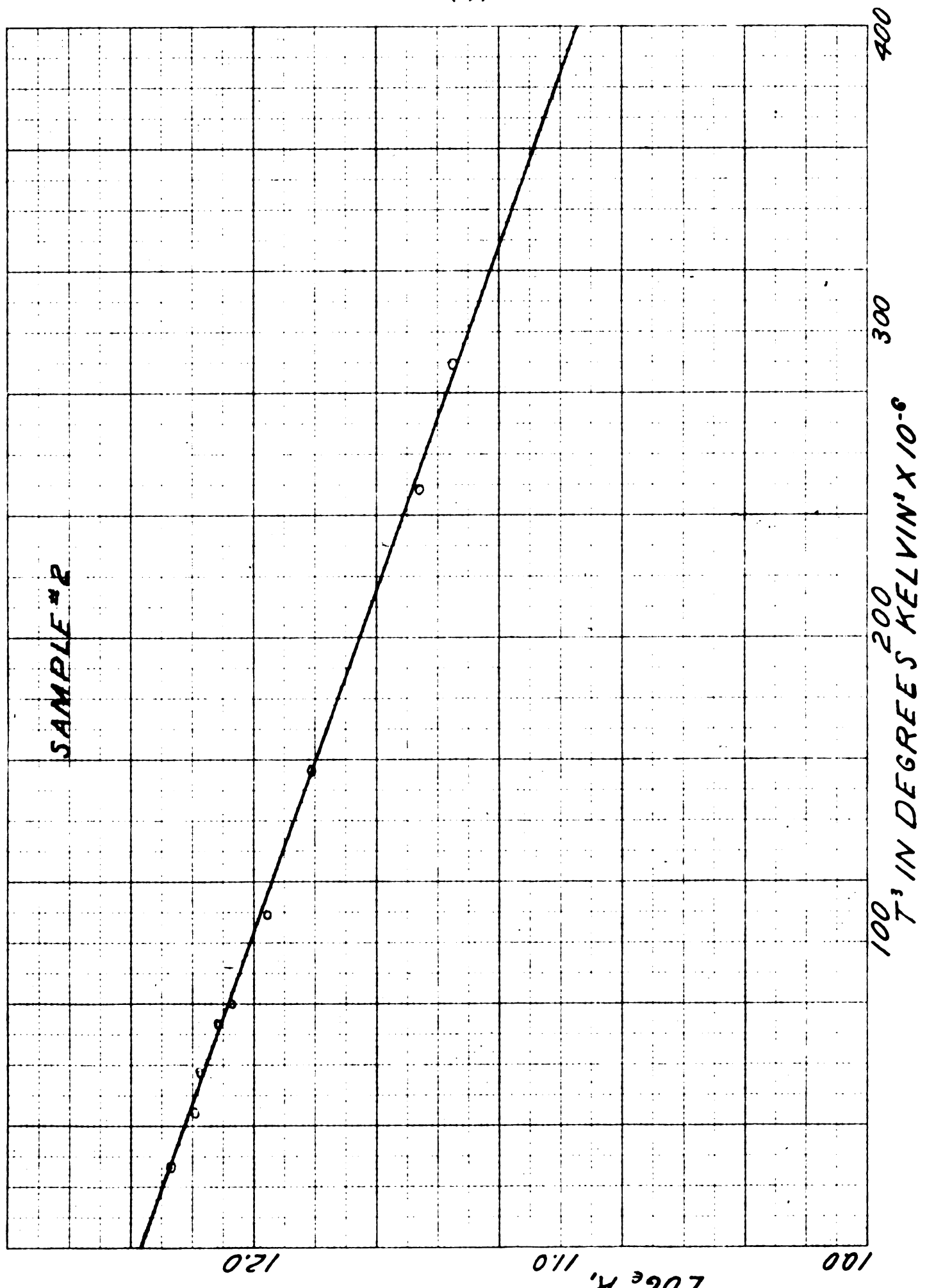


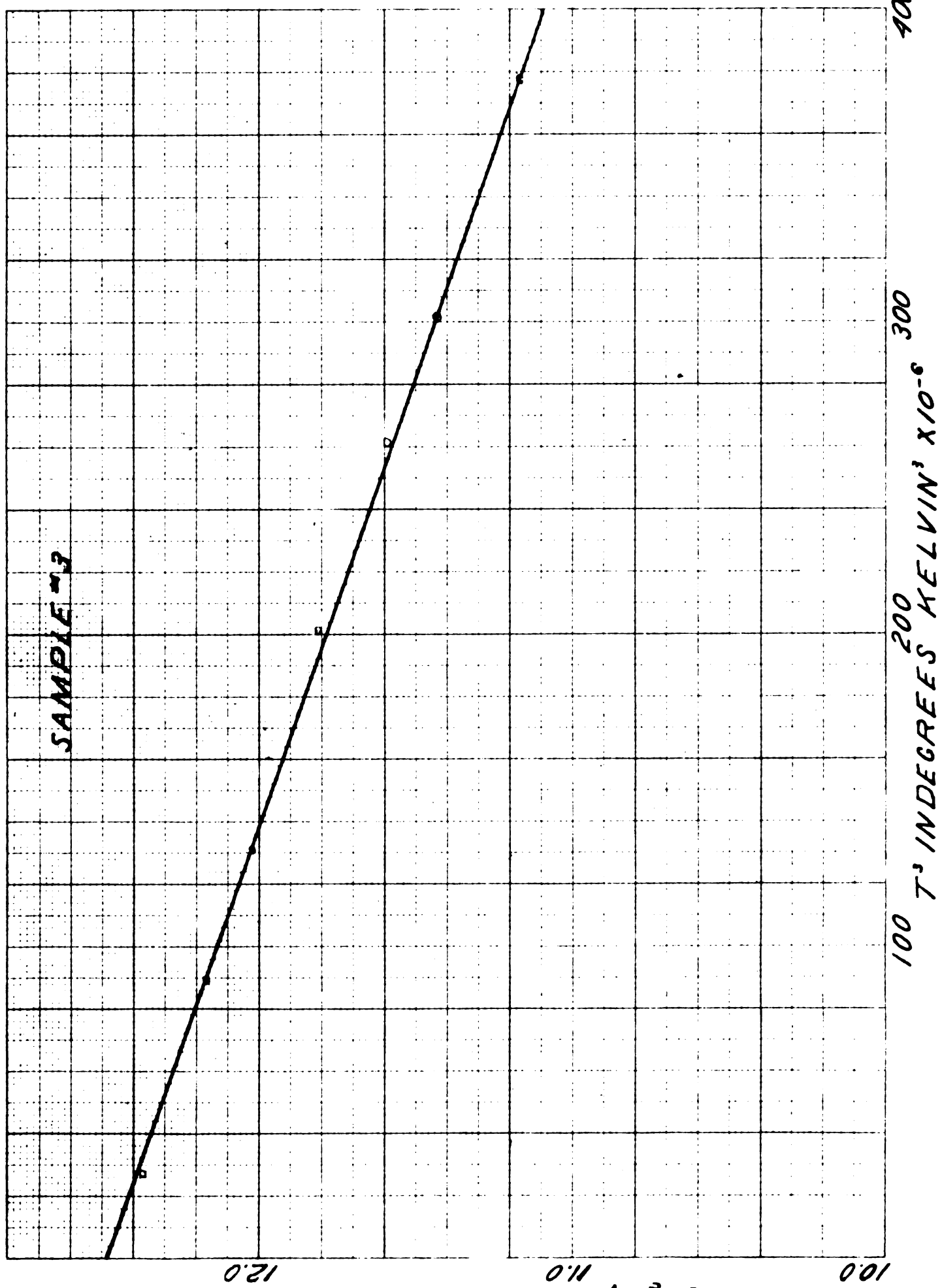
The values for K_1 computed from the preceding data are:

Sample #2		Sample #3	
K_1 ergs/cc x 155	T(degrees Kelvin)	K_1 ergs/cc x 10 ⁻⁵	T(degrees Kelvin)
2.121	298	2.35	298
1.98	343	1.92	447
1.94	379	1.66	508
1.88	423	1.35	586
1.56	478	1.09	639
1.34	538	.919	676
.951	628	.709	723
.847	661		

Sample #5	
$K_1 \times 10^{-5}$ ergs/cc	T(degrees Kelvin)
2.23	298
2.10	353
2.02	390
1.92	403
1.87	433
1.57	501
1.43	536
1.26	566
1.21	587
1.08	628

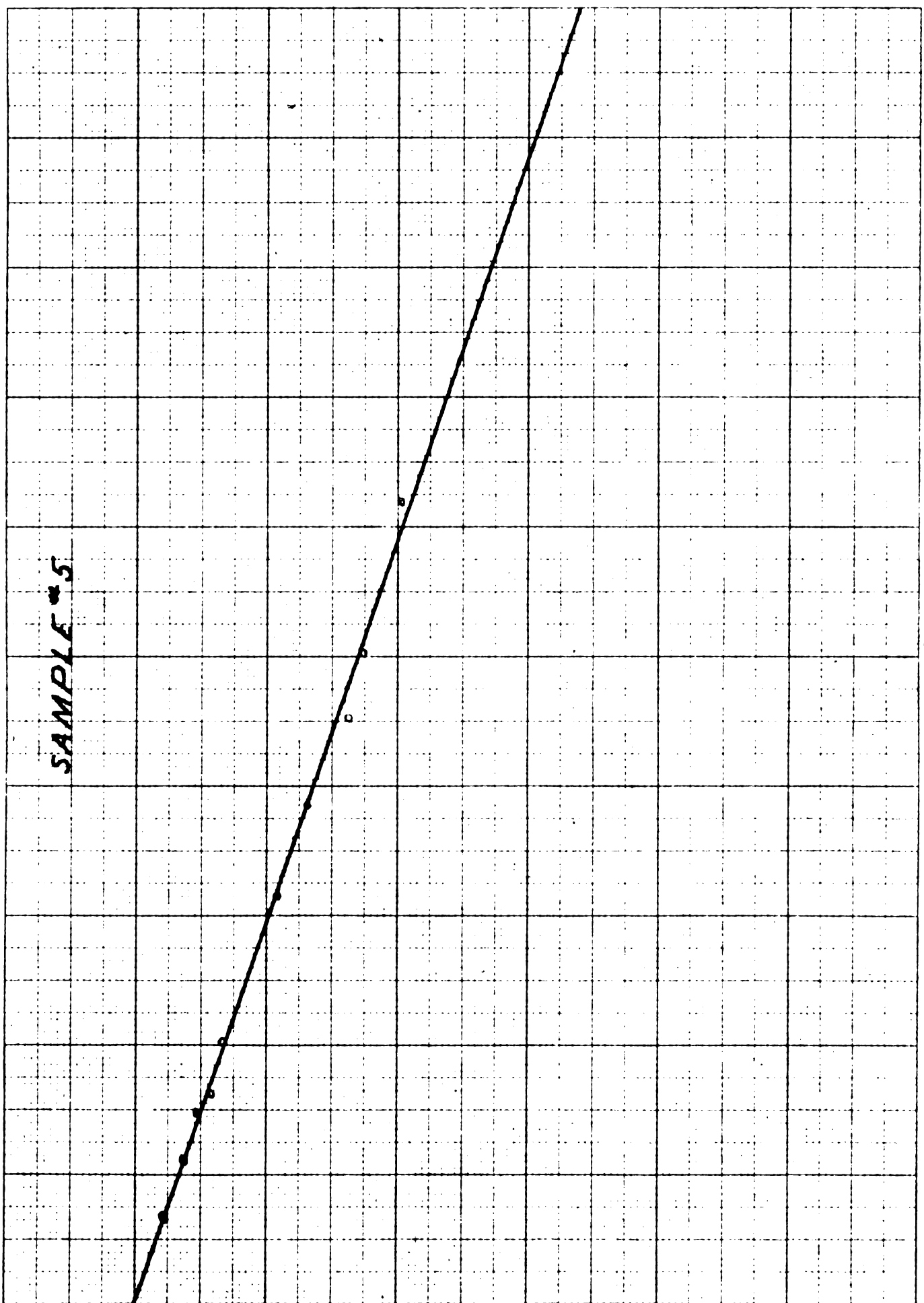
It was found more convenient to choose an empirical relation of the form $K_r = K_0 e^{-\alpha T}$ rather than the relation chosen by Williams and Bozorth.¹³ The following graphs are used to evaluate K_0 and α in the above expression.

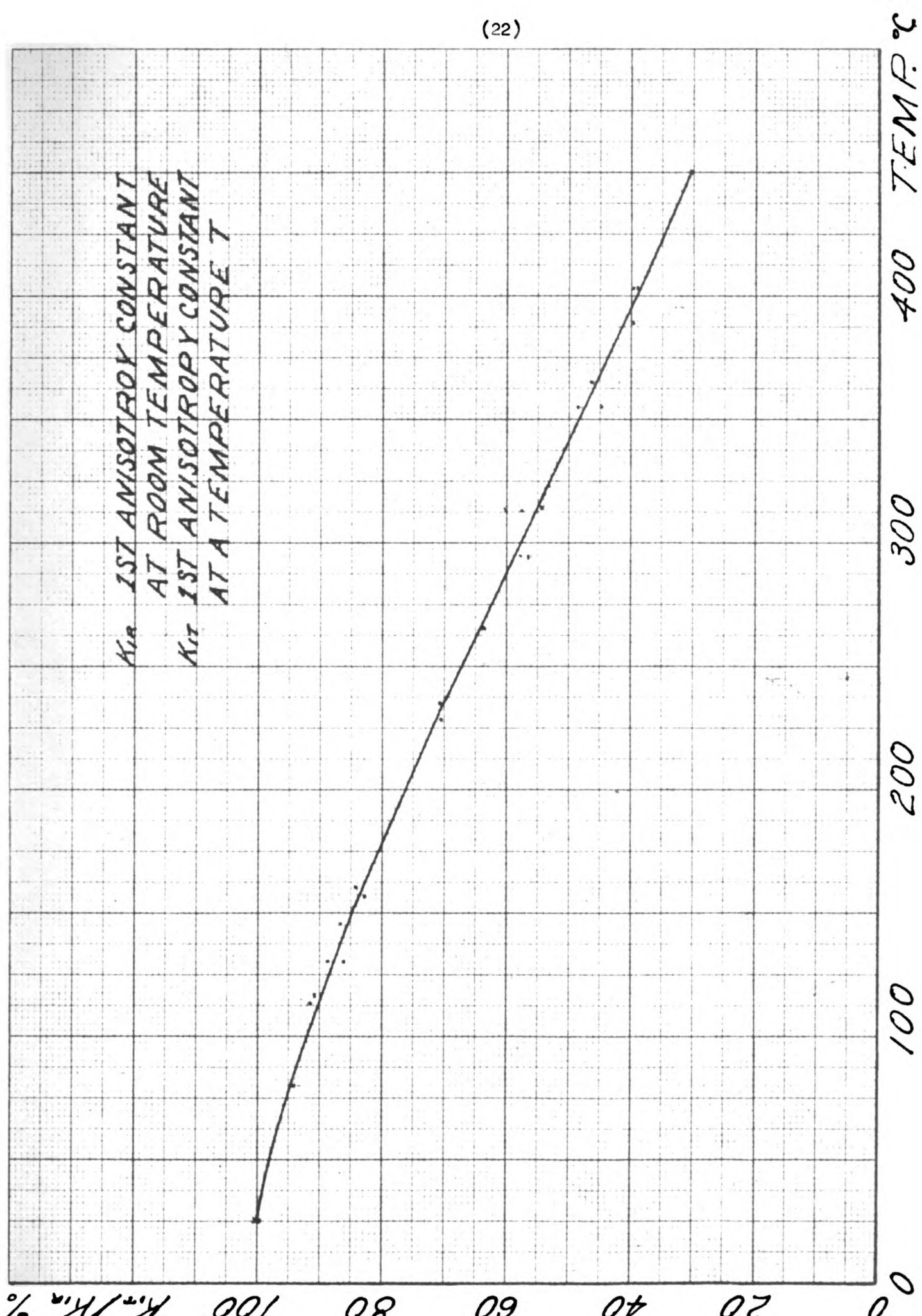




SAMPLE #5

100 T° IN DEGREES KELVIN'S X 10⁻⁶ 300 400





From the graphs of $1/n K_1$ versus T^3 the following values were computed:

Sample 2

$$K_{10} = 2.36 \times 10^5 \text{ ergs/cc}$$

$$\alpha = 3.58 \times 10^{-9} / \text{deg. Kelvin}^3$$

Sample 3

$$K_{10} = 2.63$$

$$\alpha = 3.48 \times 10^{-9} / \text{deg. Kelvin}^3$$

Sample 5

$$K_{10} = 2.43$$

$$\alpha = 3.40 \times 10^{-9} / \text{deg. Kelvin}^3$$

The results of the work can be summarized as follows:

1. The average value of K_1 at room temperatures is

$K_{10} = 2.23 \times 10^5 \text{ ergs/cc}$. The standard deviation from this value $\pm 0.11 \times 10^5 \text{ ergs/cc}$.

2. An average value for K_{10} of $2.47 \times 10^5 \text{ ergs/cc}$ with a standard deviation of $\pm .11 \times 10^5 \text{ ergs/cc}$.

3. The average value for α is $3.48 \times 10^{-9} / (^{\circ}\text{K})^3$ and a standard deviation of $\pm .07 \times 10^{-9} / (^{\circ}\text{K})^3$.

4. There is no permanent change in the anisotropy constants of Silectron when subjected to temperatures up to 1100°C . and allowed to cool rather slowly to room temperature.

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