Magnetic Properties of Single Crystals of Silicon Iron

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The magnetization curves for the [100], [110] and [111] directions of single crystals of iron containing 3.85 percent silicon were obtained from single crystal specimens cut in the form of hollow parallelograms so that the sides of each specimen were parallel to the tetragonal, digonal or trigonal axes, respectively. This method avoids the errors due to demagnetizing fields, inherent in previous measurements on single crystals. In addition to the well-known anisotropy at magnetizations above half of saturation, the data show for the first time considerable anisotropy at low magnetizations. A maximum permeability of 1,380,000 by far the highest ever reported for silicon iron, was observed in the [100] specimen after careful annealing. The magnetic anisotropy constants K_1 and K_2 were obtained from the magnetization curves and from torque measurements on a disk cut parallel to a (110) plane.

INTRODUCTION

THIS article describes three different ways of determining the ferromagnetic anisotropy constants K_1 and K_2 (see Eq. (1) below) of 3.85 percent silicon iron. This particular material was chosen because it is particularly easy to obtain in it very large single crystals of high permeability. The first way was to obtain the magnetization curves for the [100], [110] and [111] directions of a single crystal and then adjust the theoretical magnetization curves calculated according to the Akulov-Heisenberg theory to the experimental curves. A new method of measuring the magnetic properties of single crystals was employed. Single crystal specimens were used which formed closed magnetic circuits so that the specimens could be wound with primary and secondary windings and tested in the regular manner of testing toroidal specimens. The second method of evaluating the anisotropy constants was to measure the areas between the magnetization curves of these same specimens and then calculate the anisotropy coefficients from these areas as explained below. This method is, of course, essentially the same as the first. The third way of evaluating the coefficients was by means of torque measurements on a singe crystal disk. A disk was cut so that its plane was parallel to the (110) crystallographic plane. All three directions of interest lie along diameters of such a specimen. The disk was tested in a torsion magnetometer[1] and the theoretical torque curve which depends upon the anisotropy coefficients, was adjusted to fit the experimental curve.

[1] H. J. Williams, Rev. Sci. Inst. **8**, 56–60 (1937).

METHOD OF GROWING CRYSTALS

Large crystals of silicon iron were produced by melting silicon iron in an atmosphere of pure hydrogen and permitting the melt to solidify very slowly. Silicon iron containing 3.85 percent silicon was melted in an alundum boat 10 cm square and 2 cm deep. The furnace had an alundum muffle with a molybdenum winding and high grade alundum powder was used for external heat insulation. The melt was cooled at the rate of approximately 8°C per hour until it had completely solidified. The temperature gradient of the furnace was such that crystals grew across the melt resulting in a number of elongated crystals in the ingot. Some of these were approximately 1 cm wide, 2 cm deep, and extended in a somewhat irregular manner nearly 10 cm across the ingot. After etching it could be seen that these elongated crystals had tended to grow along a [100] crystallographic direction.

PREPARATION AND DESCRIPTION OF SPECIMENS

The approximate orientation of a (100) plane in a crystal was determined by naked eye observations, under a single source of light in a dark

TABLE I. *Dimensions of crystal specimens.*

DIRECTION OF SIDES	PLANE OF SPECIMEN	MEAN LENGTH OF SIDES (cm)	AREAS OF CROSS SECTION (cm²)
Tetragonal	(100)	1.72, 1.23	0.0697
Digonal	(100)	1.49, 1.17	0.0670
Trigonal	(110)	0.77, 0.77	0.0489

room, sticking a small mirror to the crystal so that it reflected a ray of light parallel to the rays reflected from etch planes. Since these are (100) planes of the crystal the plane of the mirror then lies parallel to a (100) plane. A flat surface was cut on the crystal parallel to the plane of the mirror. The orientations of this surface and of the axes lying in it were determined more accurately by means of x-rays and it was then possible to set the piece in a machine so as to grind any desired section to within half a degree or less of the desired orientation. It is possible to cut a parallelogram from a slice, properly located, so that each side of the parallelogram is parallel to any one of the three crystallographic directions, [100], [110] and [111] which are of most interest. The single crystal specimens were thus cut so that each may be described as a strip of metal forming a closed parallelogram having its sides parallel to a certain crystallographic direction.

Great care was taken in cutting the crystals to avoid straining them. After cutting, the specimens were etched to remove the strained material on the surface and then they were annealed in an atmosphere of hydrogen. They were etched again after annealing and since there were no signs of recrystallization, it was thought the specimens had not been unduly strained in earlier processes. Three magnetic test specimens of this sort were cut from the ingot. The first specimen had each side parallel to a tetragonal axis [010] or [001], the second specimen had each side parallel to a digonal axis, [011] or [01$\bar{1}$]. In each of these specmens the plane of the parallelogram was (100). The third specimen had each side parallel to a trigonal axis [1$\bar{1}$1] or [1$\bar{1}\bar{1}$], the plane of the parallelogram being (110). Table I gives the dimensions of the specimens, and the plane of the parallelogram.

The specimens are shown in Fig. 1.

Magnetic Tests

Magnetic data were obtained by winding each specimen with a primary and secondary winding and then testing with a Haworth fluxmeter.[2] Previous tests on single crystals have usually been made on ellipsoids or short rods and have the disadvantage that at low fields the effective magnetizing field is the difference between two relatively large quantities, the applied magnetizing field and the demagnetizing field of the specimen, so that small effective fields cannot be determined accurately. Therefore, the initial part of the magnetization curve up to approximately half the saturation value cannot be determined accurately nor can the residual induction be measured. The method described in this article eliminates this disadvantage. On the other hand the new method has the disadvantage that the flux at the corners may go in other crystallographic directions than those intended. In the specimens used these cross-corner distances are short in comparison to the total length of path and the cross-sectional area is slightly[3] greater at the corners.

Magnetization curves for the three specimens after annealing at 900°C for an hour and then reannealing at 1300°C for an hour are shown in Fig. 2. The curve for the [100] direction has a higher permeability than the other two curves. The maximum permeability is 624,000, the residual induction is 13,400 gauss and the coercive force is 0.028 oersted. The maximum permeability for the [110] direction is 64,000, the residual induction 10,400 gauss and the coercive force 0.043 oersted while the maximum permeability for the [111] direction is 19,300, the residual induction .2130 gauss and the

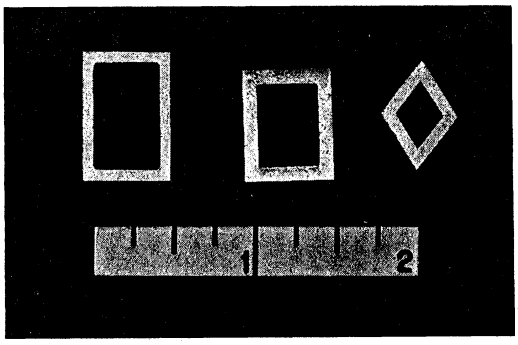

FIG. 1. Single crystal magnetic test specimens of 3.85 percent silicon iron. The first specimen has each side parallel to a tetragonal axis, the second specimen has each side parallel to a digonal axis and the third specimen has each side parallel to a trigonal axis. Scale in inches.

[2] F. E. Haworth, Bell System Tech. J. 10, 20–32 (1931).

[3] If the corners were rounded so as to give a uniform cross-sectional area the material cut away would amount to only about one percent of the total.

coercive force 0.11 oersted. Fig. 3 shows the permeabilities as functions of the induction. It should be noted that the [100] curve has not only a higher maximum permeability but also has much higher permeability at high flux densities. There are no points on the [100] curve for values of the induction between 2000 and 8000 gauss. This is because the magnetization curve is nearly vertical over this range. This gives a straight line when the permeability is plotted as a function of the induction.

The data of Figs. 2 and 3 show considerable anisotropy for values of magnetization less than half of the saturation value. This has not been previously observed nor has it been predicted by theory. However, a simple analysis shows that it should be expected. In this analysis the magnetization curves for the [110] and [111] are derived from the experimental curve for the

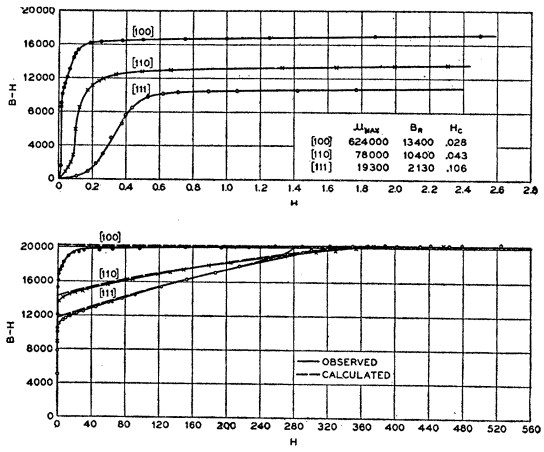

FIG. 2. The observed and calculated magnetization curves for the [100], [110] and [111] directions of single crystals of 3.85 percent silicon iron.

[100] by assuming that when H is applied along the [110] or [111] the component of H along a [100] will produce a magnetization in that direction given by a [100] magnetization curve; the projection of this value of I on the direction of the applied H gives the magnetization in that direction.

The [100] specimen was reannealed at 1300°C for two hours more and then tested. The maximum permeability was 1,180,000. Then it was heat treated at 600°C for three hours while a magnetic field of 10 oersteds was applied. The maximum permeability was then 1,030,000.

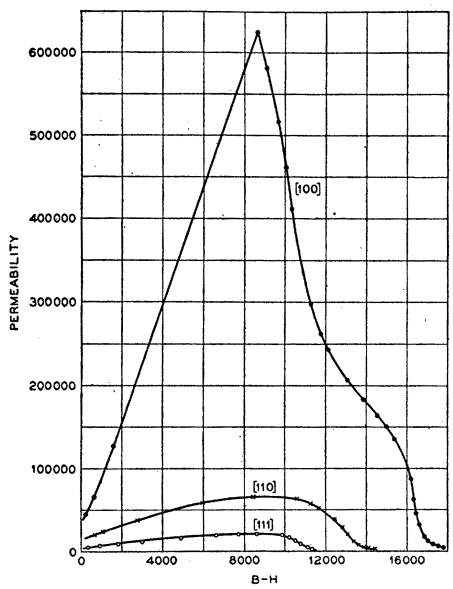

FIG. 3. Permeability *vs.* $B-H$ for the [100], [110] and [111] directions of single crystals of 3.85 percent silicon iron.

During this low temperature anneal the surface of the specimen became slightly oxidized so the specimen was etched to remove this coating. After etching the maximum permeability was 1,380,000. The permeability induction curves are shown in Fig. 4.

CONSTANTS BY FITTING CURVES

When a crystal is magnetized to saturation the expression for the energy of magnetization is to three orders of approximation,

$$E=K_0+K_1(S_1^2S_2^2+S_1^2S_3^2+S_2^2S_3^2) +K_2(S_1^2S_2^2S_3^2). \quad (1)$$

In this expression K_0, K_1 and K_2 are coefficients and S_1, S_2 and S_3 are the direction cosines of I_0, the saturation value of the local intensity of magnetization, with reference to the cubic axes. If the direction of magnetization is along a cubic axis the second and third terms in the expression are zero and so the energy of magnetization along a cubic axis is equal to K_0. To magnetize the crystal in any other direction, we may first magnetize the crystal along a cubic axis and then rotate the I_0 vector into the desired position. The second and third terms express the energy required to rotate the I_0 vector from a cubic

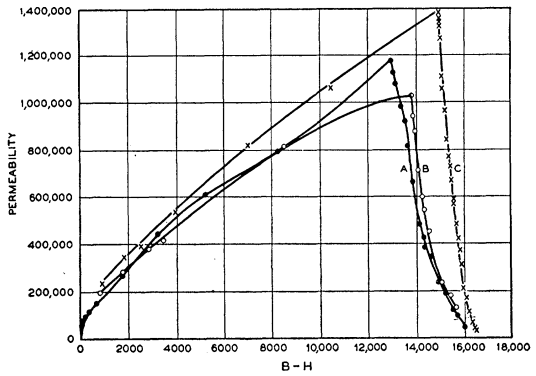

FIG. 4. Permeability *vs.* $B-H$ for the [100] direction. *A*, After heat treating the specimen at 900°C for one hour and 1300°C for three hours. *B*, After further heat treatment at 600°C for three hours in the presence of a magnetic field. *C*, After etching.

axis into any other position. The energy necessary to rotate I_0 is also equal to $-\int_0^{\alpha_0} L d\alpha$ where L is the torque and α_0 is the angle between the nearest cubic axis and the final position of I_0. It is assumed that the axis of L remains fixed. From this it is apparent that

$$L=-dE/d\alpha. \tag{2}$$

Let H make an angle α_0 with the nearest direction of easy magnetization. Then as H is increased I_0 will rotate from a position of easy magnetization and α, the angle between I_0 and the nearest direction of easy magnetization, will approach α_0. The equilibrium condition for a given value of H is that the torque due to the applied field is equal and opposite to the torque due to the forces of anisotropy which tend to keep the domains lined up in a direction of easy magnetization. The former is equal to the product of the normal component of I_0 and H, and the latter is equal to $dE/d\alpha$.

Equating these and solving for H gives

$$H=\frac{dE/d\alpha}{I_0 \sin(\alpha_0-\alpha)}. \tag{3}$$

The component of I_0 parallel to the applied field is given by the relation

$$I=I_0 \cos(\alpha_0-\alpha). \tag{4}$$

Eqs. (3) and (4) give the theoretical magnetization curves[4] for a single crystal. These curves intersect the I axis at $I_0 \cos \alpha_0$. The value of H required to saturate a material in the [110] direction is equal to $2K_1/I_0$, which gives a value of K_1. The values of K_1 and K_2 determined in this manner for the crystals of silicon-iron, are given in Table II.

CONSTANTS FROM AREAS

The energy required to magnetize a crystal to saturation in any direction $[hkl]$ is equal to

$$\int_0^{I_0} H_{hkl} dI_{hkl}.$$

Equating this to the expression for the energy given in Eq. (1) and then subtracting the energy of magnetization for the [100] direction from that for the [110] direction and the energy for [100] direction from that for [111] direction gives

$$E_{110}-E_{100}=\frac{K_1}{4}=\int_0^{I_0} H_{110} dI_{110}-\int_0^{I_0} H_{100} dI_{100}, \tag{5}$$

$$E_{111}-E_{100}=\frac{K_1}{3}+\frac{K_2}{27}$$

$$=\int_0^{I_0} H_{111} dI_{111}-\int_0^{I_0} H_{100} dI_{100}. \tag{6}$$

These equations give the anisotropy constants in

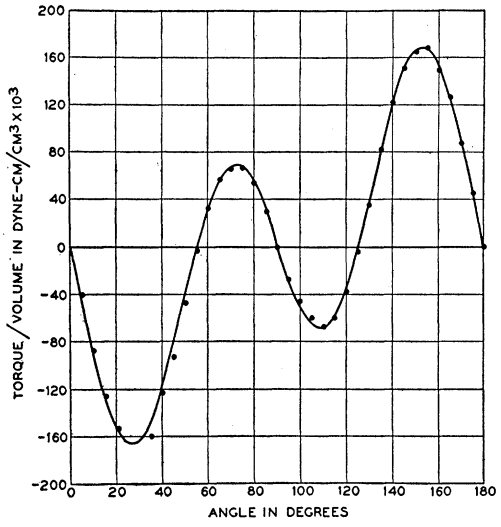

FIG. 5. Torque curve for a single crystal disk of silicon iron in the (110) plane, taken with an applied field of 5800 oersteds. The line is drawn according to Eq. (7) with $K_1=287{,}000$ and $K_2=100{,}000$.

[4] For a recent discussion of the theory of magnetization and torque curves involving K_1 and K_2, see R. M. Bozorth, Phys. Rev. **50**, 1076 (1936).

terms of the area between the magnetization curves. Values are given in Table II.

Constants from Torque Measurements

A third method of determining anisotropy constants is by means of torque measurements. A single crystal disk 1.77 cm in diameter and 0.222 cm thick was cut so the plane of the disk was parallel to (110). Torque measurements were made on this disk by means of a torsion magnetometer. Using the relation $L=-dE/d\alpha$ and substituting the direction cosines for a vector lying in the (110) plane gives the following expression for the torque.

$$L=-K_1(2\sin 2\alpha+3\sin 4\alpha)/8 \\ -K_2(\sin 2\alpha+4\sin 4\alpha-3\cos 6\alpha)/64. \quad (7)$$

Fig. 5 shows the theoretical and experimental torque curves. K_1 and K_2 were determined by trial to make the theoretical curve fit the experimental curve as closely as possible.

Table II. *Values of anisotropy constants.*

Method	K_1 (erg cm^{-3})	K_2 (erg cm^{-3})
Measurement of areas between magnetization curves	272,000	150,000
Fitting theoretical magnetization curves to the experimental curves	280,000	100,000
Torque measurements	287,000	100,000

The values of K_1 and K_2 determined by the three methods are given in Table II.

I wish to thank F. E. Haworth for the careful x-ray determination of the crystal orientations, and R. M. Bozorth for valuable discussions.

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The Viscosity of Air

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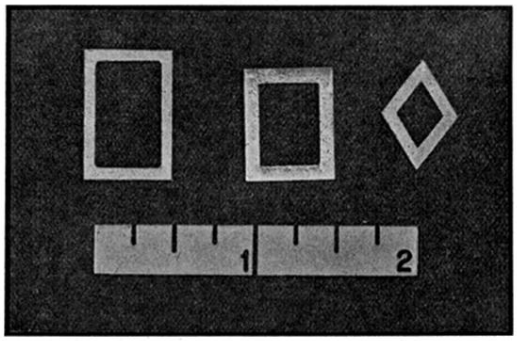
The viscosity of air has been remeasured with the rotating cylinder apparatus used by Day and Bleakney. All of the constants have been redetermined, and especial attention has been given to the corrections to the simple theory. The value obtained at 22°C is $\eta=1.8243\pm0.0045$ c.g.s. units. This raises the oil drop value of e to 4.796 e.s.u. It is shown in the appendix that a correction must be applied for the opening between the suspended cylinder and the guard cylinders, and also a correction must be applied for the moment of inertia of the air carried around by the cylinder when determining its moment of inertia. Neglect of these corrections has introduced some additional uncertainty into other work.

THE viscosity of air has become of special interest within the last few years since the suggestion of Shiba[1] that an error in the adopted value of this quantity was responsible for the discrepancy between the values of e determined by the oil drop and the x-ray methods. The work of Harrington[2] was supposed to have established its value with the necessary precision, but Shiba concluded from an examination of other determinations that this precision might have been considerably overestimated. Because of this interest and the presence in this laboratory of a rotating cylinder apparatus suited to this purpose it seemed worth while to make a complete redetermination of the viscosity. Although since the work was started, about a year ago, the very careful work of Kellström[3] and a preliminary note from Bearden have appeared and have confirmed Shiba's supposition, it may be still of interest to have an independent determination.

It is not necessary to enter into a detailed description of the apparatus since it has been previously described.[4] No essential changes have been

[1] K. Shiba, Sci. Papers Inst. Phys. Chem. Res. Tokyo, **19**, 97 (1932).
[2] E. L. Harrington, Phys. Rev. **8**, 738 (1916).
[3] G. Kellström, Phil. Mag. **23**, 313 (1937); J. A. Bearden, Phys. Rev. **51**, 378 (1937).
[4] R. K. Day, Phys. Rev. **40**, 281 (1932); W. M. Bleakney, Physics **3**, 123 (1932).



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