Powder Patterns on Ferromagnetic Crystals

K. J. SIXTUS General Electric Company, Schenectady, New York (Received March 5, 1937)

The regular patterns which, as Bitter has found, form on ferromagnetic single crystals from a suspension of magnetic iron oxide have been studied on large crystals of 3.5 percent silicon-iron. Three different types of patterns were observed at low, medium, and high fields, respectively. The direction of Type I and Type II patterns is determined by the crystal orientation; that of Type III is always nearly normal to the applied field. Type I patterns were found only after polishing or otherwise straining an annealed sample, and the line spacing stayed constant (except at H=0) as the field was increased. We conclude that surface strains

F^{OR} the study¹ of "Bitter" patterns² a suspension of iron oxide $(\gamma - Fe_2O_3)$ with a particle size of 0.5×10^{-4} cm in Nujol—a viscous mineral oil—was used. Here the particles do not have the mobility exhibited by colloidal particles such as used by L. W. McKeehan and W. C. Elmore.³ Once a pattern has formed, it stays nearly fixed under changed external conditions; it has to be stirred or replaced for a new pattern to develop.

The crystals were prepared in the form of disks or strips by recrystallization from silicon iron strips with 3.5 percent Si. The results on only three out of a total of ten studied are given here. The patterns on these three appeared more clearly than on the rest and it can be seen from Table I which gives their orientation that in all of them a (100) plane lies nearly in the surface.

The field was produced by an electromagnet for the short samples, by a coil for the long ones. The values of field strength given below are not corrected for demagnetization. The fact that the pattern remained "fixed" after its development in the field allowed the removal of the sample from the coil and its observation under the microscope. are essential in producing this type of pattern. Type II patterns were little affected by stress, and the lines multiplied when the longitudinal or normal field component was increased. Certain dissymmetries of the lines and their behavior near a hole in the sample were studied. It is concluded that the Type II lines are formed at the intersections of magnetic "sheets," lying in dodecahedral (110) planes, with the surface. Type III patterns form near surface inhomogeneities where the magnetic flux is forced to cross the surface and where the magnetization accordingly has a normal component.

DIRECTION OF PATTERNS

It was found independently by S. Kaya⁴ and by the author¹ that three different types of patterns existed in the different ranges of the magnetization curve, and that these types had different directions. On the first steep part of the magnetization curve patterns (Type I) form along intersections between the surface and those (100) planes most nearly perpendicular to the surface (Fig. 1). If the surface also lies nearly in a (100) plane as in our case there are two possible line directions. It depends on the polishing and field direction which set will appear; in some cases both are observed together. Between the first bend of the magnetization curve and the bend at reaching saturation, lines (Type II, Fig. 3a, c) form at intersections between (110) planes and the surface. Ordinarily only that set

TABLE I. Orientation of the crystal axes with respect to a coordinate system in the crystal surface. The suffixes y and x refer to the angles with the y and x axes, which lie in the sample surface at right angles to each other; the field is always applied parallel to the y axis.

Dimensions: Crystal No. 1. $1.55 \times 0.050 \times 2.4$ cm³; crystal No. 70. $1.35 \times 0.056 \times 5.0$ cm³; crystal No. 7N. $1.55 \times 0.054 \times 25.0$ cm³.

	[001] AXIS		[010] AXIS		[100] AXIS	
Crystal No.	α_y	α_x	β _y	β_x	Υy	γ_x
1 70 7 <i>N</i>	-85 88 86	$-74 \\ 79 \\ -68$	$31 \\ -10 \\ -26$	$ \begin{array}{r} 62 \\ -80 \\ -71 \end{array} $	$-60 \\ 79 \\ -71$	$-{}^{33}_{-15}_{-30}$

⁴ S. Kaya, Zeits. f. Physik 89, 796 (1934); 90, 551 (1934).

¹ Most of the experiments described here were reported at a meeting of the American Physical Society in February, 1934. An abstract of that paper appeared in Phys. Rev. 45, 565 (1934). A few experiments were made shortly after this meeting. Since a continuation of the work appears not possible in the near future, the data forming the original paper are given here together with some additional results.

² F. Bitter, Phys. Rev. **38**, 1903 (1931); **41**, 507 (1932). ³ L. W. McKeehan and W. C. Elmore, Phys. Rev. **46**, 226 (1934); **46**, 529 (1934).



FIG. 1. Type I pattern on crystal No. 70 at 5 oe. Due to inhomogeneous field distribution, several Type II lines appear on the lower part of the figure. The white lines are polishing scratches. (Magnification $15 \times$).

of lines appears whose direction is given by the (110) plane most nearly perpendicular to the field. Beyond the saturation bend we observe bands of irregular length and appearance whose direction is very closely perpendicular to the applied field (Type III, Fig. 3b).

Spacing

The spacing of the Type I pattern was fairly constant in different places on any one sample and it stayed constant over the Type I field range except in 0 field where the number of lines doubled (cf. McKeehan and Elmore³). On different samples the range of spacings found lay between 5 and 30×10^{-3} cm. This spacing is considerably higher than that observed by McKeehan and Elmore which was about 2×10^{-4} cm; it is probable that such a fine structure can only be detected with extremely fine suspensions.

Some remarkable properties were observed with the Type II pattern. Bitter² has shown that on nickel crystals the lines obviously multiply by splitting. The division of a line into two new ones at certain field values rather than the development of new lines appears to be the mode of multiplication in our case also, but the process is somewhat different from that observed on nickel. On the Fe-Si crystals the lines show a strong



FIG. 2. Type II patterns taken at the same place on crystal No. 1 after successive increases in field. (Magnification $48 \times$.) The Type II pattern on crystal No. 1 was observed in a field range between 20 and 160 oe.

tendency to have, at any field, a uniform spacing. Thus Fig. 2 shows clearly in several instances how the addition of a new line "pushes" the neighboring line away in order to equalize the line spacing.

Bitter has also pointed out² that on reapplication of a given field the lines appear in new places. We found in addition that the entire process of line multiplication is irreversible. If the field is reduced from any value in the Type II range, the number of lines does not change until this pattern disappears at the first bend. Another effect which illustrates the irreversibility of the process of line production is the following. If a field value in the Type II range is reached by increasing the field from the Type I range, many of the lines extend over a large part of the surface, as indicated by the photograph (Fig. 3a). If, however, the same field value is reached by a field reduction from the Type III range (Fig. 3b), the lines are short and less uniform in direction (Fig. 3c).

The magnetization curve of the long crystal No. 7N is given in Fig. 4, together with the spacing-field dependence of the Type II pattern. The magnetization curve seems to contain several sharp "kinks," connected by straight lines. These were first reported by W. Gerlach, and have later been either confirmed or doubted by



 $\longrightarrow H$

FIG. 3. *a*; Pattern (Type II) on crystal No. 70 at 36 oe. *b*; Pattern (Type III) after the field was raised to 90 oe. *c*; Pattern (Type II) after field reduction from 90 to 35 oe. (Magnification $15 \times$.) The Type II field range on crystal No. 70 lay between 8 and 80 oe.

other workers.⁵ The field dependence of the line spacing is definitely a discontinuous curve, indicating that line multiplication takes place at certain critical field values. The relation between the sudden slope changes in the magnetization curve and the changes in line spacing is here merely suggested, because the accuracy of the present measurements does not allow definite conclusions. There is additional evidence for the suggested relation in that the descending branch of the magnetization curve, though lying very close to the ascending one, has no breaks, while the line spacing during field reduction stays constant.

EFFECT OF STRAIN

Some preliminary tests were made on crystal No. 70 which was subjected to bending so that the upper surface could be put either under tension or compression. The Type I lines, which under compression ($\epsilon = -5 \times 10^{-4}$) and also with zero strain formed at an angle of -80° with the field direction, switched to the other possible direction, at $+10^{\circ}$ from the field direction, when tension ($\epsilon = 5 \times 10^{-4}$) was applied.⁶ The line spacing became more irregular with stress but was of the same general order $(15 \times 10^{-3} \text{ cm})$. The direction of the Type II pattern was the same $(+56^{\circ})$ in all three cases; the spacing, at a given field, showed a variation only in the case of compression (from 20 to 7×10^{-3} cm). It has to be added that with the Type I pattern the line changes were reversible when the stress was applied or removed, which, however, was not the case with Type II pattern. On application of pressure the lines multiplied; if now the stress was released without changing the field, the lines (after replacing the powder suspension) stayed in the same place although they were weaker than before. Only after the field had been reduced to 0 and returned to the previous value would the spacing return to its old value. The Type III pattern was not affected by stress.

The experiments were repeated using the long crystal No. 7N under pure tension, which offered two advantages. The strain could be determined

⁵ See discussion by L. W. McKeehan, Trans. A.S.M.E. 111, 11 (1934).

⁶ A similar effect was observed by N. Akulov and S. Raewsky, Ann. d. Physik [5] 20, 113 (1934).



FIG. 4. Magnetization curve of crystal No. 7N (not corrected for demagnetizing effect) and line spacing (Type II) vs. field relation. The vertical arrows point to sharp breaks in the magnetization curve.

more accurately, and a magnetization curve with a definite relation to the pattern could be obtained. The corresponding compression case was not investigated. Fig. 5 shows that only the steep part of the magnetization curve of crystal No. 7N is affected by tension. It should be added that a previous curve was retraced when the tension was released to the old value, indicating that the strain had been elastic. Judging from the magnetization curves, we should expect the most profound changes in the Type I pattern. This was confirmed, for with a stress of 100 kg cm^{-2} the lines which ordinarily formed at $+70^{\circ}$ had already switched their direction to an angle of -24° . Tension also affected the time interval between application of powder suspension and completion of the pattern. With 100 kg cm⁻², the lines could be seen clearly after less than 10 minutes, whereas it took more than one-half hour for them to appear without applied stress. The strain corresponding to 100 kg cm^{-2} was measured roughly and found to be about 8×10^{-5} . The tension was kept below 250 kg cm^{-2} to avoid plastic deformation, and at that value the pattern was as clear as at lower values.

The Type II pattern throughout the range of stress did not change materially, except that it

became a little less clear at higher tension. It was observed, in addition, that under tension the Type II pattern did, with increasing field, appear already below the first bend in the magnetization curve, contrary to the behavior without stress.

Considering the decisive effect which elastic tension has on the Type I pattern, it is not surprising that the direction and manner of polishing



FIG. 5. The first part of the magnetization curve of crystal No. 7N with different values of tension.

should also influence this pattern. Kaya⁴ has already given proof of this influence in very painstaking experiments and we give only a short account of our corroborating evidence. The long crystal No. 7N was polished with emery paper (000) and then annealed for one hour at 850°C in pure dry hydrogen. There was practically no change in the reflecting power of the surface before and after the anneal, indicating that surface contamination, if there was any, could only be very slight. The crystal after this treatment showed no Type I pattern, whereas Types II and III appeared as clear as ever. Half of the strip was polished with (000) emery paper parallel to the long axis of the sample and then a set of clear $+70^{\circ}$ Type I lines could be produced on the polished part.

Polishing affects a surface in two ways, both of which are possible causes for the formation of a pattern. It removes surface material and also introduces cold work effects in the outer layers of the remaining material, consisting in crystal reorientation and strains. If the first effect were the essential one, etching, which will remove any possible surface contamination, would also result in pattern formation. A solution of picric acid in alchol and also a mixture of hydrochloric and nitric acids were applied in different places on the unpolished part, but no pattern would form there. If, however, strains were introduced by subjecting the crystal to tension, the $+70^{\circ}$ lines appeared at about 100 kg cm^{-2} on the unpolished section, persisted at higher tension, and vanished again below 100 kg cm⁻².

The unavoidable conclusion to be drawn from



FIG. 6. Copper film thickness, at which the Type II pattern disappears, and spacing on crystal No. 1.

our experiments seems to be that surface strains, as caused by polishing or by applied tension, are a necessary condition for the formation of a Type I pattern. We observed no effect of polishing in the case of Type II and Type III patterns. This had to be expected from the absence of an effect of elastic tension on these patterns.

INCIDENTAL OBSERVATIONS

The cause for the formation of powder lines is the presence of poles in the surface. The following tests were undertaken to obtain more information about their strength in the Type II range. An indirect, qualitative method was employed. The crystal was covered with nonmagnetic material in successive layers and at intervals the range of field strength was determined in which the pattern could be observed. Copper was selected as nonmagnetic material because it could easily and uniformly be applied by an electroplating process and its thickness found from the increase in weight. The points on Fig. 6 were obtained in the following way: After plating to a certain thickness, which is plotted as ordinate, the sample was subjected to a magnetic field. The field strength was raised in successive steps, allowing sufficient time at any step for the powder to settle, and the minimum and maximum field values were noted at which a pattern could just be discerned. The experimental points were connected by the line a in Fig. 6. The result shows that the poles which formed at the boundary surface between Si-Fe and Cu, for example, in a field of 100 oersteds were not strong enough to attract powder in distances greater than 2.7×10^{-3} cm above that surface. The dashed line a' connects the experimental points determined at decreasing field values.

The pole density per unit area, σ , which is proportional to the component of magnetization normal to the surface may be roughly calculated with the data of Fig. 6. The force F acting on a pole m in distance a from a line of poles of line density ρ is $F=2\rho m/a$. Now $\sigma = \rho/b$ where b is the line spacing and therefore $\sigma = aF/2mb$. F is constant at the threshold and m is approximately constant along curve a which makes $\sigma \sim a/b$. Fig. 6 shows that the lines multiply as long as this ratio rises, a process which, however, is not reversible.

Two phenomena were in most cases associated with the Type II pattern which, on account of their appearance, we shall name "fringes" and "beards." The former can be clearly seen on Fig. 3a, where each line shows a sharp straight boundary towards the left and is jagged towards the right. The direction of these "fringes" is fixed with regard to the crystal; it is the same all over the surface and stays the same when the field direction is reversed. On the opposite face of the crystal the fringes point in the opposite direction. Incidentally, the fringes can also be seen on some of Kaya's photographs.⁴ They assumed a definite direction with respect to that dodecahedral plane whose intercept with the surface determined the line direction, forming on that side of a line where the dodecahedral plane made the smaller angle with the surface (Fig. 8). No fringe was observed on crystal No. 1, where the (110) plane made a right angle with the surface.

"Beards" were first observed on small holes drilled through the crystal. Fig. 7a shows that in the neighborhood of such a disturbance the Type II pattern forms quite unsymmetrically as regards line spacing. On one side of the hole the spacing is the same as on the rest of the crystal, but on the other side the line spacing is smaller and the total amount of powder deposited is much greater. On the reverse face of the crystal the beard pointed always in the opposite direction. At first it was thought that a local distortion might have caused this asymmetry, but it soon became evident that, like the fringes, it was related to the crystal orientation of the sample. In comparing four different crystals, it was found that the direction of the beard was dependent on the angle which the $\lceil 001 \rceil$ axis made with the axis of the hole. The beard formed on that side of a hole away from which the [001] axis through the hole was inclined, as shown in Fig. 8. The beard persisted in a field range where ordinarily only the bands of Type III would appear. Fig. 7b, taken in a high field, shows besides the beard a heavy symmetrical deposit. The latter was the clue for the explanation of Type III patterns which will be discussed later. The hole caused no disturbance in the Type I pattern.



FIG. 7. Patterns near a hole on crystal No. 70. a; in medium field. b; in high field. (Magnification $15 \times .)$

DISCUSSION

It appears as if the powder patterns could give us valuable information about the actual process of magnetization in crystals supplementing the picture derived from the statistical theory of this process given by Akulov and others. The patterns seem to furnish visible evidence for the existence of elementary magnetic districts made audible in



FIG. 8. The direction of "beards" caused by a hole and of "fringes" forming on lines, for different crystals.



FIG. 9. Explanation for the Type III pattern.

the Barkhausen effect. The evidence, however, is confined to the surface of the material, which especially as regards magnetic properties—may behave entirely different from the bulk.

The Type I pattern, according to our results, is due to a strain condition at the surface. The ordering effect of applied tension or of polishing is needed to make the magnetic districts line up in a block structure suggested by the Type I pattern. In the case of applied tension, this structure may, of course, extend through the whole crystal. It appears likely that the fine structure pattern as observed by McKeehan and Elmore which has been found so far only on highly polished surfaces can be classed together with our Type I pattern.

The Type II pattern has a definite relation to the normal component of induction which appears at the bend of the magnetization curve and vanishes at saturation. The line spacing decreases, though irreversibly, as this component increases. This component has not been actually measured, but our contention is borne out by conclusions drawn from several experiments: 1. If a field normal to the surface is applied, the number of lines increases. 2. If a sample in a longitudinal field is compressed, the number of lines increases. Since the magnetostriction is positive in the Type II range, compression will result in an increase in the normal component of magnetization. 3. The increase in line number on one side of a hole (beard in Fig. 7a) is due to an increase in the vertical field component on that side. 4. The lines multiply as long as σ (see above) rises. The Type II pattern may be only a surface phenomenon but its independence of surface conditions makes it likely that it reveals a condition present throughout the crystal. The observed pattern is in accord with the assumption that the regions magnetized at large angles to the surface are localized within the crystal in "sheets" parallel to (110) planes. This was also demonstrated in a model made of a razor blade held between two brass blocks. In a suitable field a line with fringe formed along the edge of the blade which was very similar in appearance to a Type II line.

The explanation for the Type III pattern was first reported in February, 1934 and has independently been indicated in Kaya's paper. It becomes evident in a sample in which a small hole has been drilled (Fig. 7). At and near saturation the local magnetization throughout the sample is practically parallel to the total magnetization, or, in different terms, the lines of force are parallel to H as long as the material is homogeneous. If, however, an obstacle such as a hole is present, the lines of force which can not be "compressed" any more will mainly be "pushed" away in a horizontal plane. Some are "pushed" upward and cross theiron-air boundary near the hole, forming poles on the surface along a line which goes through the hole and is perpendicular to the induction (Fig. 9). A rigorous mathematical solution of this problem is not possible on account of the dependence of permeability on magnetization.⁷ The "obstacle" need not be a hole; it may be represented by any region in which the permeability is lower than in the rest of the material, such as inclusions or

⁷ R. Richter, Elektrotechnik. u. Maschinenbau 51, 285 (1933).

regions with high local strains in or near the surface. It is evident that the Type III lines are not confined to single crystal samples. Polycrystalline material will exhibit them, particularly if it is subjected to a uniform stress which creates a direction of preferred orientation. Thus the bands, which were observed on stressed and twisted wires⁸ and which were found to lie approximately perpendicular to the direction of induction, are another form of Type III lines.

Discussions with Dr. I. Langmuir and Dr. L. Tonks have been of great help to the author in the progress of this work.

⁸ K. J. Sixtus, Phys. Rev. 44, 46 (1933).

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PHYSICAL REVIEW

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The Damping of Torsional Oscillations in Quartz Fibers

G. A. DOWNSBROUGH* Rutgers University, New Brunswick, New Jersey (Received September 21, 1936)

The results obtained by previous workers have been qualitatively confirmed, but it was demonstrated that many of the perplexing features were characteristic of the material used to mount the quartz fibers, and not of the fibers themselves. When fused quartz joints were substituted, these features were eliminated, and in addition the damping became very much smaller than in any previous work. As a result, it was also necessary to investigate energy losses hitherto unimportant; several were found capable of affect-

INTRODUCTION

LET a body whose moment of inertia is I execute torsional oscillations about the axis of a quartz fiber whose moment of torsion is T. If the oscillations are damped by a resisting couple proportional to the angular velocity, the equation of motion is

$$I(d^2\theta/dt^2) + b(d\theta/dt) + T\theta = 0 \tag{1}$$

with the solution

$$\theta = \theta_0 e^{-bt/2I} \sin \left(2\pi t/P\right),\tag{2}$$

where P is the period. Since b is very small for

ing the results, but in the final arrangement they were all insignificant. On the assumption that the damping is a purely viscous phenomenon, it is shown that the product of the period of oscillation and the logarithmic decrement should be a constant of the material. This was experimentally verified over a small range of periods; the value obtained was 1.3×10^{-6} . The coefficient of viscosity for quartz, derived from this, is 2.2×10^{6} g/cm sec.

quartz, P is given to a high degree of approximation by

$$P = 2\pi (I/T)^{\frac{1}{2}}.$$
 (3)

The damping of this oscillatory motion is most conveniently measured by the logarithmic decrement δ , defined as the natural logarithm of the ratio of successive amplitudes; thus

$$\delta = \frac{bP}{2I} = \frac{1}{n} \log_e \left[\frac{\theta_0}{\theta_n} \right] = \frac{P}{t_n} \log_e \left[\frac{\theta_0}{\theta_n} \right], \quad (4)$$

where t_n is the time for the amplitude to change from θ_0 to θ_n . Experimentally it is much simpler to measure t_n than to count n, the number of oscillations.

If we assume that the force dissipating energy

^{*} Now with the Research Laboratories of Johns-Manville Corporation, Manville, N. J.



FIG. 1. Type I pattern on crystal No. 70 at 5 oe. Due to inhomogeneous field distribution, several Type II lines appear on the lower part of the figure. The white lines are polishing scratches. (Magnification $15 \times$).



FIG. 2. Type II patterns taken at the same place on crystal No. 1 after successive increases in field. (Magnification $48\times$.) The Type II pattern on crystal No. 1 was observed in a field range between 20 and 160 oe.







FIG. 7. Patterns near a hole on crystal No. 70. a; in medium field. b; in high field. (Magnification $15 \times$.)