

shown. Another set of phenomena that requires further experimental investigation is that accompanying the transition from the ferromagnetic to the paramagnetic state. For some purposes it is sufficient to speak of a single critical temperature, but a closer inspection shows that in some substances, at any rate, the various properties change more or less continuously over a range of temperatures, and that there may even be two or three distinct critical temperatures quite close to each other. In Fig. 10 are plotted magnetization curves of nickel taken by Weiss and Forrer<sup>11</sup> near the Curie point. It would be of great interest to study these curves in relationship to the constant  $K$  of Eq. (4), whose behavior for iron is plotted in Fig. 6, and so to separate these effects which are due to the orientation of the regions as discussed above from those which are due to other mechanisms.

<sup>11</sup> P. Weiss and R. Forrer, *Ann. de Physique* 5, 153 (1926).

#### SOME NEW EXPERIMENTAL METHODS IN FERROMAGNETISM

*An address presented before the Symposium on Ferromagnetism at the meeting of the American Physical Society, Schenectady, New York, September 12, 1931*

BY S. L. QUIMBY

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WHEN your Committee honored me with an invitation to address this body on the subject "New Experimental Methods in Ferromagnetism," I accepted on condition that I be permitted to prefix this title with the adjective "Some." Taking advantage of this bit of forethought, I propose this morning to limit my remarks to a brief description of certain methods which have been devised for observing the magneto-elastic, mechanical and thermal properties of single crystals of nickel, as well as of polycrystalline specimens of the same substance.<sup>1</sup>

Attention should first be directed to the process used in refining the nickel.<sup>2</sup> The initial stage in this process is the preparation of a nickel chloride bath by dissolving Mond nickel in hot hydrochloric acid. Iron is then removed from the bath by precipitation of the hydroxide, and copper by electrolytic deposition on a rotating platinum cathode. Finally the pure nickel is deposited electrolytically on a stationary carbon cathode. This nickel is spectroscopically free from iron, cobalt and all other metallic impurities except a trace of copper too small to be detected by chemical means.

I shall now describe the apparatus used for the manufacture of single crystals of nickel. The specimens are desired in the form of rods 4 to 6 mm in diameter and 5 to 10 cm long.

The crystals are grown in a molybdenum-wire-wound vacuum electric furnace, following the method developed by Bridgman for the production of single metallic crystals. The novelty of the present arrangement consists in those features of design which permit the use of this method at a temperature of 1500°C.

Fig. 1 is a cross section silhouette of the furnace. On the outside is a fused quartz tube 5½ inches in diameter and 28 inches high. A clear quartz window is fused in at the top to permit the use of an optical pyrometer. The bottom is ground flat and makes a vacuum tight seal with the flat iron plate which forms the base of the furnace. Next is a radiation trap consisting of a tubular alundum furnace core 3 inches in diameter. Next is an alundum tube 2 inches in diameter and 24 inches long on the upper 9 inches of which are wound 80 turns of molybdenum wire. The diameter of the wire is 0.036 inches. Next is an alundum tube 1 inch in diameter and 10 inches long upon which are wound 90 turns of molybdenum wire. The space between these

<sup>1</sup> These methods have been developed through the cooperative activity of the following group working in the Physics Laboratories at Columbia University: Lewis Balamuth, W. T. Cooke, Fred Rose, Sidney Siegel, Clarke Williams, and Jerrold Zacharias. Detailed descriptions thereof will appear in due course in separate papers under appropriate authorship.

<sup>2</sup> F. A. Rohrman, *Transactions of the Electrochemical Society* 57, 325 (1930); 58, 403 (1930); 59, 359 (1931).

two tubes is filled with alundum powder to prevent arcing between neighboring turns of the inner heating coil. Last is the crucible, which is molded of a mixture of refractory cement and alundum grain. The crucible is mounted on an alundum rod, which in turn is mounted on an iron tube of  $\frac{3}{4}$  inch outside diameter closed at the lower end. This tube floats in mercury contained in a surrounding iron tube of 1 inch inside diameter. The mercury chamber is connected through a rubber hose with a reservoir for adjusting the initial height of the crucible, and through a glass tube with a stopcock. The aperture in this stopcock is plugged in such a manner as to form two small buckets of about 5 cubic mm capacity. When it is desired to lower the crucible out of the furnace the stopcock is rotated continuously by an electric motor acting

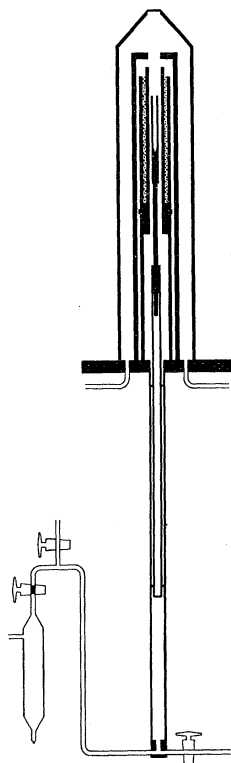


Fig. 1.

through a gear train. Each half revolution spills 5 cubic mm of mercury from the system into an evacuated chamber and lowers the crucible 0.01 mm. The crucible is lowered about 4 cm per hour. The nickel is premelted in a hydrogen furnace and cast into rods whose diameter is slightly less than that of the crucible. With this furnace single crystals of nickel are secured in the form of rods 4 to 6 mm in diameter and 6 cm long.

A Laue photograph yields the orientation of the cylinder axis in the crystal lattice by interpolation in the series of fifty face centered lattice patterns published by Majima.<sup>3</sup> In this manner the orientation can be determined with an accuracy of about one degree.

The first measurement which is taken upon the crystal is the variation of the coefficient of thermal expansion with temperature through the Curie point. As long ago as 1910 observations made upon specimens of very impure polycrystalline nickel showed that in the neighborhood of the Curie point the coefficient of thermal expansion varies with temperature in the

<sup>3</sup> Majima, Tokyo Institute of Physical and Chemical Research, Scientific Paper No. 111.

manner shown in Fig. 2.<sup>4</sup> The elegant method used by Colby cannot readily be adapted to a long cylindrical specimen. The present apparatus for the investigation of this phenomenon is mentioned here chiefly because it is an excellent example of the use which may be made of recent developments in the art of making and handling clear fused quartz. The apparatus sketched in Fig. 3 is composed entirely of clear fused quartz. One end of the specimen is fixed relative to the apparatus by virtue of its resting on a knife edge which engages with a tiny scratch in the specimen. The other end rests on a quartz roller 1 mm in diameter which in turn rests on a flat plane. Sputtered gold mirrors reflect into a telescope images of two scales placed five meters distant. These mirrors are rotated slightly with respect to each other about the roller axis to increase the range of the instrument. A third scale is reflected from a mirror on the front face of the mount and a fourth from a mirror under the knife edge. Observations on these scales

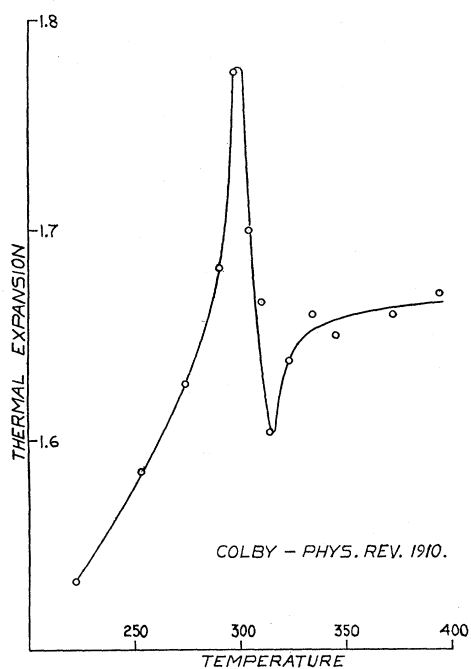


Fig. 2.

permit the evaluation of any shift of the apparatus or its mounting in the furnace. The apparatus is mounted on one end of a quartz tube 2 inches in diameter which is fastened at the other end and projects into the furnace.

Initial observations on the variation of the coefficient of thermal expansion with temperature in single crystals of pure nickel indicate that this quantity behaves near the Curie point in a manner similar to that previously reported for impure polycrystalline nickel.

The ordinary magnetostrictive change in length has been measured with this apparatus by using the central cylindrical portion of a long thin ellipsoid of revolution. When it is desired to make observations on short ellipsoids, a length of about 2 mm on each end is ground into a cylinder, and these cylinders rested on the knife edge and roller respectively.

The behavior of these quartz rollers is surprisingly good. Fig. 4 shows the design of a much more elaborate system constructed for the purpose of studying the variation of Young's modulus with stress, temperature, and magnetic field intensity in specimens whose form is a long thin cylinder or wire a mm or so in diameter. In specimens of this sort both the temperature and

<sup>4</sup> Colby, Phys. Rev. 30, 506 (1910).

the magnetic field are uniform only over a region some 10 cm in length near the center of the wire, and the problem is that of measuring the change in length under the applied stress of this portion alone. Two parallel rollers 2 mm in diameter and 10 cm apart rest upon a flat quartz plate and upon them bears the specimen under longitudinal stress. On the first roller there rests in addition a small triangular shaped quartz plate which has its upper surface ground flat. A third idling roller supports the rear end of this plate. The bearing surfaces of the plate on the rollers are two parallel polished quartz rods 1 mm in diameter fused to the plate. On top of the

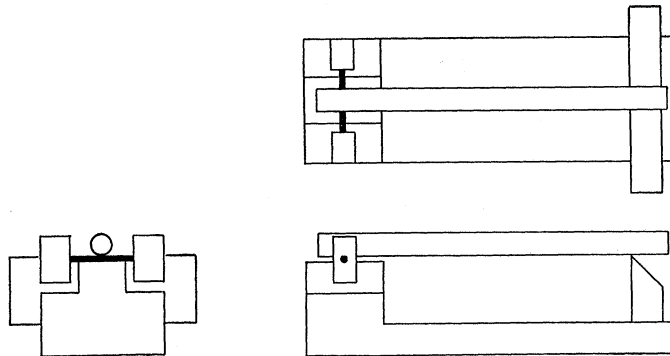


Fig. 3.

plate rests the roller carrying the mirrors. On the rear roller rests a block of roughly the same material as that under examination having quartz bearing surfaces as before. A metal rod of the same material screwed to this block leads forward and rests upon the mirror roller. If now the entire specimen be stretched by making one end fast and pulling on the other, then the rotation of the mirrors will be determined solely by the change in length of that portion of the specimen which lies between the rollers. Auxiliary mirrors as before permit the evaluation of any shift of the apparatus as a whole. The effect of thermal expansion is compensated sufficiently to keep

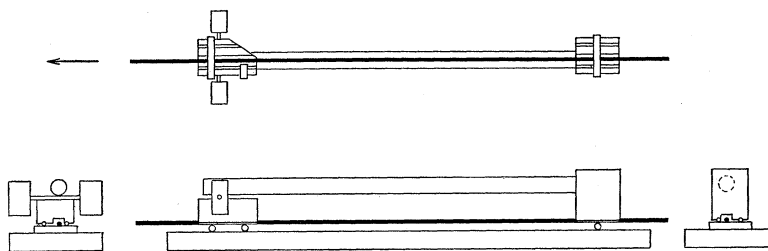


Fig. 4.

the scale images in view throughout a run by the device of making the upper bar of nearly the same material as that under examination. Fig. 5 gives an idea of the behavior of this rather complicated system. It shows the stress-strain relation for 8.6 cm of a nickel wire 1 mm in diameter when the applied load is altered between 1 kg and 3.5 kg. The lines are drawn straight. The lower line gives the effect of increasing load and the upper line the effect of decreasing load. The cause of the relative shift, which represents a linear displacement of about 1000 atomic layers, has not yet been determined. The obvious explanation, that of elastic fatigue, is not the correct one as the shift varies erratically from run to run.

I now return to a description of further experiments upon single crystals of nickel. It is desired to measure the variation with temperature through the Curie point of Young's modulus in the direction parallel to the axis of the cylindrical specimen. The method about to be de-

scribed requires the experimental determination of the frequency of free longitudinal elastic vibration of the cylinder. This being known, the velocity of sound in the direction of the cylinder axis is given by the relation

$V = f_N \times \lambda$ , where  $\lambda = 2 \times \text{length of cylinder}$ , and Young's modulus for this direction by the relation  $V = (E/\rho)^{1/2}$ , where  $\rho = \text{density of specimen}$ . The crystal is sawed in half, the ends ground flat, and the two halves cemented to a plate of piezoelectric quartz 0.4 mm thick. An electric axis of the quartz is parallel to the cylinder axis. The specimen is then suspended by and in electrical contact with two very thin wires, and these wires are connected to an oscillating electric circuit which establishes across the quartz an alternating potential difference of variable frequency. Accordingly the quartz, which is thus situated in a sinusoidally varying electric field, suffers a sinusoidally varying piezoelectric strain which is transmitted to the nickel rods. The result is that the entire system is set into forced longitudinal elastic vibration. Under these circumstances the electric current which flows into the little condenser with quartz for dielectric is determined in part by the ordinary electric polarization associated with the field and in part by the polarization associated with the stress in the quartz due to the

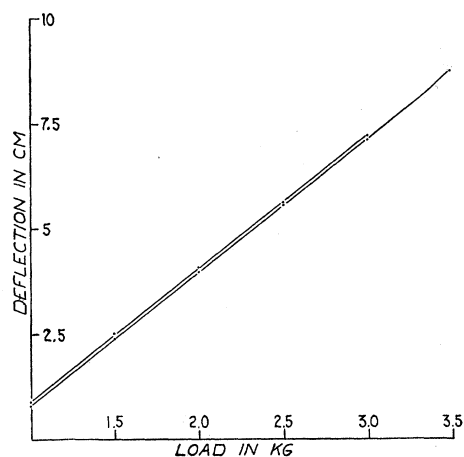


Fig. 5.

mechanical vibration. At a certain value of the impressed frequency the amplitude of vibration attains a maximum value, that is, the quartz-nickel system resonates. In the neighborhood of this frequency the current passes in succession through a maximum and a minimum value.<sup>5</sup> The resonance frequency of the system may be calculated from the observed values of current and frequency corresponding to these maximum and minimum currents. With this quantity and the known elastic data for the quartz plate, the desired free period of the specimen alone can readily be computed.

The foregoing considerations apply with only numerical modification to a nickel-quartz oscillator constructed by cementing a rod of quartz of rectangular cross section to one end of a single rod of nickel. In this case the quartz is so cut that the optic axis and an electric axis both lie in a plane perpendicular to the cylinder axis. The electric axis is perpendicular to a pair of opposite faces of the crystal and the electrodes are made by coating these faces with gold leaf.<sup>6</sup>

These composite piezoelectric oscillators can also be used to measure the coefficient of rigidity. For the system may be excited to torsional vibration by using a quartz rod of circular cross section and four quadrantal electrodes, or by using a quartz rod of different cut.<sup>7</sup> A knowledge of Young's modulus and the coefficient of rigidity affords a complete description of the

<sup>5</sup> Cf. Van Dyke, Proc. Inst. Radio Eng. **16**, 742 (1928).

<sup>6</sup> Quimby, Phys. Rev. **25**, 558 (1925).

<sup>7</sup> Giebe and Scheibe, Zeits. f. Physik **46**, 607 (1926).

elastic properties of an isotropic medium. It is not as yet possible to measure in this manner the rigidity modulus for single crystals of nickel. It can be shown that the free period of torsional vibration of crystalline rods depends upon the rigidity modulus alone only if the cylinder axis is oriented in certain directions in the crystal lattice. So far in the case of nickel no way has been discovered for controlling the orientation of the crystal axes when the crystal is grown.

Two experimental details of these methods require special mention. One is the nature of the cement which will cause nickel to adhere to crystalline quartz at a temperature of 400°C. The ends of the nickel rods are first thinly copper plated. The stick is then made under pressure, in a vacuum at a temperature of 600°C with copper borate as the adhesive.

The other detail is concerned with the way in which the frequency-current curves are obtained. The frequency difference between the maximum and minimum currents may be 100 cycles or less for an oscillator whose resonance frequency is 50,000 cycles. In order therefore properly to use this method a means must be provided for measuring easily and quickly the frequency of the oscillating electric circuit with an accuracy of 1 or 2 cycles in 50,000. For this purpose use is made of a piezoelectric clock and its auxiliary apparatus. A schematic of the method is given in Fig. 6. The frequency of an oscillating circuit is controlled by a piezoelectric quartz crystal mounted in a thermostat which assures a temperature constant to a hundredth

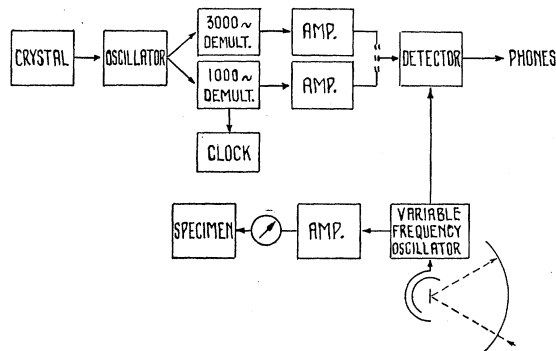


Fig. 6.

of a degree. Under these circumstances the frequency remains constant to a few parts in a million over a period of days and to a few parts in a hundred thousand over a period of years. The output of this circuit is used to control the frequencies of two demultiplying circuits. The latter are push-pull oscillators in which the natural frequency is determined by a resistance and a capacity. These circuits possess the peculiar property that if a high frequency alternating potential difference of a few volts be superimposed on the plate voltage then over a wide range of variation of their circuit constants they are constrained to oscillate at a frequency which is an integral sub-multiple of the superimposed frequency. In the system here depicted the piezoelectric oscillator frequency is approximately 51 kilocycles. One of the demultipliers is adjusted to oscillate at the 17th subharmonic and the other at the 51st subharmonic of this frequency. The latter drives a synchronous clock which is compared from time to time with the radio time signals from Arlington. Since the wave form of the demultipliers is extremely rich in harmonics, this arrangement affords standard frequencies good to one part in a million at intervals of 1000 cycles up to several hundred kilocycles.<sup>8</sup>

The variable frequency oscillator driving the specimen is coupled electrically to the output of the 1000 cycle demultiplier from which the audible harmonics have been filtered out, and the resultant beat note rectified and rendered audible in a pair of head phones. The frequency of this oscillator is controlled both by a tuning condenser and by a vernier condenser whose full scale value alters the frequency about 500 cycles. This vernier has a scale with 500 one millimeter divisions. Let us assume that the resonance curve which it is desired to obtain lies be-

<sup>8</sup> Hull and Clapp, Proc. Inst. Radio Eng., Feb. 1929, p. 267.

tween the frequencies 53,000 cycles and 53,500 cycles. The clock supplies the frequencies 52,000, 53,000 and 54,000. Thus the detector tube receives these frequencies together with the oscillator frequency, and its output contains the beat notes between the two. The frequency of the oscillator having been adjusted approximately to 53,000 by means of its rough calibration, the vernier is set at zero and the tuning condenser varied slightly until consonance is heard between two 1000 cycle notes. These are the beat notes between the oscillator frequency, 53,000, and the clock frequencies 52,000 and 54,000. The condition of consonance implies that the zero of the vernier corresponds to a frequency of exactly 53,000 cycles. Leaving the tuning condenser alone, the vernier is now altered until consonance is heard between two 500 cycle notes. These are the beat notes between the oscillator frequency of 53,500 and the clock frequencies of 53,000 and 54,000. Accordingly this position of the vernier corresponds to a frequency of exactly 53,500 cycles. Since, as was remarked, the size of the vernier and the distance of its scale are adjusted to give about 500 mm for 500 cycles, it follows that the frequency of the exciting oscillator may be determined over the range of the resonance curve to one cycle in 50,000.

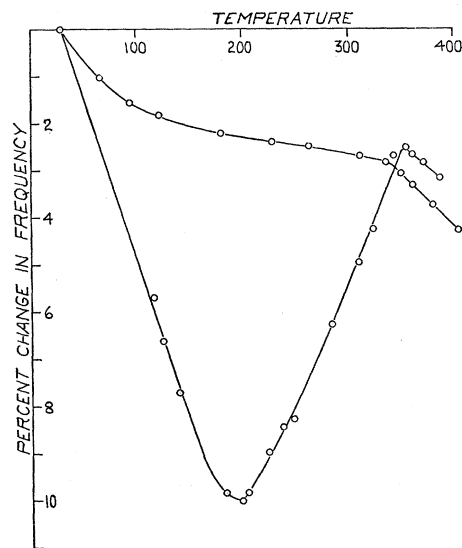


Fig. 7.

Fig. 7 shows the change in resonance frequency of two nickel rods as the temperature is raised from 23°C to 400°C. The lower curve is for a single crystal and the upper curve for a polycrystalline specimen. Attention is directed to the precision of measurement indicated by these curves. The resonance frequency may be measured with a precision of one tenth of one percent or better. Since Young's modulus varies as the square of the resonance frequency, this quantity may be measured with a precision of two tenths of a percent or better.

Unfortunately it is not possible to calculate Young's modulus from the observations on the lower portion of the single crystal curve with the aid of the simple formulae heretofore given. The reason for this is the fact that over this range of temperatures the internal friction of single crystal nickel increases to a point where it appreciably affects the resonance frequency of the specimen, i.e., the velocity of sound in the material. While this complicates the present investigation it is of considerable interest otherwise for the reason that there is a very intimate connection between magnetism and internal friction in ferromagnetic materials. It follows that the present method for studying Young's modulus demands a concurrent investigation of the variation with temperature of the internal friction in the material.

Fig. 8 is a schematic of an apparatus designed to measure the coefficient of internal friction of solids by direct determination of the decrement of longitudinal vibration in freely suspended

rods. The rod is excited to resonance at one of its harmonics piezoelectrically as before. The exciting crystal is then switched rapidly from the driving oscillator to an amplifier. The potential difference across the crystal produced by the piezoelectric charge, which is proportional in magnitude to the stress in the quartz and hence to the amplitude of vibration in the rod, is amplified and rectified. As the vibrations in the rod die out one obtains from the detector tube a logarithmically diminishing direct current whose decrement is twice that of the vibrations in the rod. This current is passed through a resistance and the consequent potential difference is impressed on the grids of two vacuum tube relays. The circuit of one of these relays appears

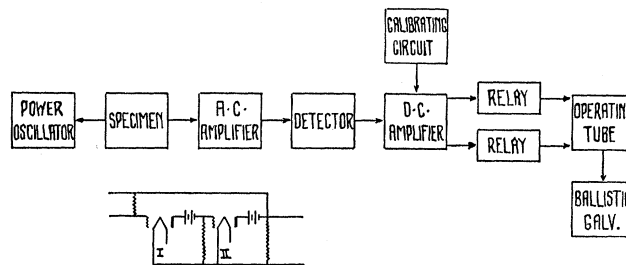


Fig. 8.

in the figure. This circuit possesses two stable states of current distribution. In state one current is flowing only in tube I and there is no measurable plate current in tube II: in state two current is flowing in tube II and there is no current in tube I. The system may be caused to pass from the first state to the second by applying a critical negative potential to the grid of tube I. The magnitude of this critical potential is subject to control. In the present apparatus one of these relays is set to operate at a voltage whose magnitude lies near the top of the decay curve just described, and the other relay to operate at a voltage which is  $1/e$  of this value. These relays control the grid potential of an operating tube in the plate circuit of which is a ballistic gal-

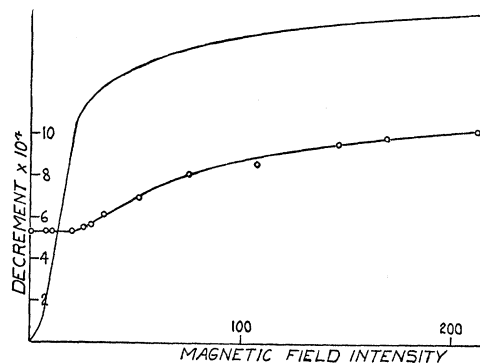


Fig. 9.

vanometer. Normally the plate current of this tube is made zero by a high negative grid bias. The operation of the first relay elevates the grid potential and allows current to flow through the galvanometer; the operation of the second relay restores the negative grid potential and stops the flow of current through the galvanometer. Thus current flows through the galvanometer; the operation of the second relay restores the negative grid potential and stops the flow of current through the galvanometer. Thus current flows through the galvanometer only during the time interval required for the detector plate current to diminish in the ratio 1 to  $1/e$ . Accordingly the ballistic galvanometer deflections are directly proportional to the time constant of the vibrating rod. The relay-galvanometer system is calibrated directly by replacing the



detector plate current by a logarithmically diminishing current in an electric circuit containing a standard resistance and a standard condenser.

Fig. 9 shows the variation with magnetic field intensity of the decrement of longitudinal vibration in a soft iron rod magnetized parallel to its axis. The curve upon which no points appear is the so-called "reversible" magnetization curve for the iron. It will be noticed that the internal friction remains constant over the entire steep portion of the magnetization curve and then increases to twice its initial value as the magnetization approaches saturation.

The magnitude of the coefficient of internal friction may also be obtained by measuring the electrical resistance of the piezoelectrically excited oscillator at resonance with an ordinary a.c. bridge. The bridge method, while more difficult to handle, has a wider range of application than the decremeter. The two methods supplement one another in a study of the behavior of these composite piezoelectric oscillators.

*Abstracts and Discussion of Papers presented at the Conference on Ferromagnetism at the meeting of the American Physical Society, Schenectady, New York, September 12, 1931*

#### BARKHAUSEN EFFECT: ORIENTATION OF MAGNETIZATION IN ELEMENTARY DOMAINS

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SINCE the postulate of the elementary domain by Weiss,<sup>1</sup> and the discovery of the discontinuous nature of the magnetization process by Barkhausen,<sup>2</sup> there have been many investigations attempting to throw more light on the nature of the elementary regions in each of which the magnetization changes as a unit. On the experimental side we are interested in knowing the sizes of the domains, the extent to which the domain is localized in space, the extent to which it is "saturated" in one direction, and the orientation of its magnetization vector before and after the change.

It has been found that all, or nearly all of the change in magnetization takes place in jumps, at least on the steeper portions of the hysteresis loop,<sup>3</sup> and that the volume of a domain varies but little with the condition of the iron, being sometimes thousands of times smaller, and sometimes thousands of times larger than a crystal.<sup>4</sup>

Mr. Dillinger and the writer have obtained some evidence that the elementary domain is a region well localized in space, within the whole of which the elementary magnets reverse. The following experiment is to the point. Two search coils are connected in series opposing to the amplifier. They are placed coaxially with the sample, equidistant from its middle. The distance between them is varied and the intensity of the Barkhausen effect is measured as a function of this distance. The differential effect increases rapidly with the distance between the coils until the distance is greater than about 3 cm. But the attainment of maximum at this distance is not due to the permanent change in magnetization over the 3 cm, but rather to the spread of eddy-currents for about that distance along the wire. This is proved by placing a small coil of wire carrying a small alternating current, around the middle of the specimen and making measurements similar to those of the Barkhausen effect just mentioned. The results,<sup>5</sup> plotted in Fig. 1, show that the measured spreading of a single disturbance due to an alternating field will account for the observed spreading of the Barkhausen discontinuities. As far as the data go they are consistent with a point source of the Barkhausen disturbance, but the accuracy

<sup>1</sup> P. Weiss, *Jour. de physique* [3] **8**, 542 (1899); *Phys. Zeits.* **9**, 358 (1908); P. Weiss and G. Foex, *Le Magnétisme*, Paris, 162 (1926).

<sup>2</sup> H. Barkhausen, *Phys. Zeits.* **20**, 401 (1919).

<sup>3</sup> R. M. Bozorth, *Phys. Rev.* **34**, 772 (1922). A similar result has also been obtained by F. Preisach, *Ann. d. Physik* (5) **3**, 737 (1929) with a different method.

<sup>4</sup> R. M. Bozorth and J. Dillinger, *Phys. Rev.* **35**, 733 (1930).

<sup>5</sup> Calculated from data already published (reference 4).