

## Ferromagnetic Anisotropy of Nickel-Iron Crystals at Various Temperatures\*

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Magnetization curves in important crystallographic directions at temperatures up to near the Curie points in small oblate spheroids cut from annealed face-centered cubic nickel-iron alloys with from 10 to 50 percent iron show a change in ferromagnetic anisotropy at about 24 percent iron. With less than this amount of iron the order of increasing difficulty for magnetization to saturation is, as in nickel at room temperature:  $\langle 111 \rangle$ ,  $\langle 110 \rangle$ ,  $\langle 100 \rangle$ . With more than this amount of iron the order is  $\langle 100 \rangle$ ,  $\langle 111 \rangle$ ,  $\langle 110 \rangle$ . A small decrease in the apparent demagnetizing factor of the spheroids with rising temperature was noted.

STUDIES of ferromagnetic anisotropy in solid solution alloys have already shown that the direction for easiest magnetization may change from one important crystallographic axis to another at room temperature as the composition changes. The first case of this sort to be reported occurs in the face-centered cubic alloys of nickel and iron. Lichtenberger<sup>1</sup> examined single crystal rods with their lengths parallel to a number of different directions in the crystal lattice. The number of specimens available in each composition was not large and the cross sections were not strictly uniform, so that the data scatter rather badly. He concluded that the directions for easiest magnetization changed from the form  $\langle 111 \rangle$  to the form  $\langle 100 \rangle$  as the iron content increased through 29 percent.

The second and third cases have been reported by Shih.<sup>2</sup> In body-centered iron-cobalt alloys, which he studied in this laboratory, he found the direction for easiest magnetization changed from the form  $\langle 100 \rangle$  to the form  $\langle 111 \rangle$  as the cobalt content increased through 42 percent. In face-centered nickel-cobalt alloys he found two transitions, from  $\langle 111 \rangle$  to  $\langle 100 \rangle$  at about 5 percent cobalt, from  $\langle 100 \rangle$  to  $\langle 111 \rangle$  at about 19.5 percent cobalt.

Lichtenberger's result has received partial confirmation from work by Burgers and Snoek,<sup>3</sup> who conclude from a change in the ferromagnetic

anisotropy of rolled polycrystalline sheets that the critical composition in the nickel-iron series occurs at 34 percent iron. In view of the uncertainties already noted in Lichtenberger's work a more careful examination of a few alloys in the critical range was undertaken here before the notes by Burgers and Snoek appeared and was continued with even greater interest thereafter.

The ferromagnetic anisotropy of nickel has been studied over a wide range in temperature by Honda, Masumoto and Shirakawa.<sup>4</sup> Although they did not comment upon the fact, their tables and curves show that the direction for easiest magnetization changes in this face-centered cubic metal from  $\langle 111 \rangle$  at room temperature to  $\langle 100 \rangle$  at temperatures of 200°C and higher. The technique of measurement with the pendulum magnetometer, which has been described elsewhere,<sup>5</sup> made it relatively easy to extend the range of investigation on nickel-iron alloys from room temperature nearly to the Curie points, which in this series are not higher than 570°C.

### SPECIMENS

Nickel-iron alloys of selected compositions were kindly furnished by the Bell Telephone Laboratories in the form of small rods. The original castings, rods about 2 cm in diameter, had been homogenized at high temperatures and rendered still more uniform in composition in the process of working. The pieces furnished were similar in quality to those used by Buckley,

\* Based on part of a dissertation presented to the Faculty of the Graduate School of Yale University in candidacy for the degree of Doctor of Philosophy.

<sup>1</sup> F. Lichtenberger, *Ann. d. Physik* **15**, 45-71 (1932).

<sup>2</sup> J. W. Shih, *Phys. Rev.* **46**, 139-142 (1934); **50**, 376-379 (1936).

<sup>3</sup> W. G. Burgers and J. L. Snoek, *Zeits. f. Metallkunde* **27**, 158-160 (1935); J. L. Snoek, *Nature* **137**, 423 (1936).

<sup>4</sup> K. Honda, H. Masumoto and Y. Shirakawa, *Sci. Rep. Tohoku Imp. Univ. [1]* **24**, 391-410 (1935).

<sup>5</sup> L. W. McKeehan, *Rev. Sci. Inst.* **5**, 265-268 (1934); L. W. McKeehan, R. G. Piety, J. D. Kleis, *Rev. Sci. Inst.* **7**, 494-497 (1936).

TABLE I. Data on spheroids ( $01\bar{1}$ ).  $\alpha$  is angle between rotation axis of spheroid and nearest axis of form  $\langle 100 \rangle$ ;  $\beta$  is angle between rotation axis and the plane (100);  $N$  is the demagnetizing factor necessary to make initial portion of  $I$ - $H$  curves at room temperature coincide with the line  $H=0$ .

Designation	Iron percent*	Nickel percent*	Diameter cm	Thickness cm	Volume cm <sup>3</sup>	$\alpha^\circ$	$\beta^\circ$	$N$
K10	10	90	0.3128	0.0284	0.001462	44	2	0.818
K30	30	70	0.3104	0.0259	0.001304	40	0	0.84
K35	35	65	0.3090	0.0256	0.001295	45	4	0.761
K50	50	50	0.3070	0.0276	0.001350	43	2	0.82

\* By synthesis. Analysis for nickel and iron requires more material than could be spared. Castings made in the same way, however, always gave nickel and iron within one percent of the intended values.

McKeehan and Cioffi<sup>6</sup> in studies of magnetization and magnetostriction in the nickel-iron alloys in the permalloy range. Impurities, by analysis, are principally sulphur, silicon and cobalt, amounting to about 0.15 percent.

Crystals a few millimeters in diameter and a few centimeters long were grown in magnesium oxide crucibles by the slow solidification process using a furnace similar to one described by Quimby.<sup>7</sup> After breaking away the crucible as carefully as possible the castings were annealed in hydrogen at atmospheric pressure at about 1100°C for one hour and slowly cooled. Laue photographs show but a small amount of residual lattice distortion.

The method of selecting crystals for study was similar to that described by R. G. Pietry<sup>8</sup> in connection with iron crystals. The only important difference lay in the occurrence of etch planes of forms  $\{111\}$  and  $\{100\}$  instead of  $\{100\}$  alone. Etching is more difficult, also, *aqua regia* being necessary for the more resistant compositions. After selection the process of forming an oblate spheroid in the desired orientation was that already described.<sup>5</sup> It was not generally feasible to cut the spheroid exactly as proposed, with the  $(01\bar{1})$  plane equatorial, and a method of describing the actual orientation of the equatorial plane is therefore necessary. Let  $\alpha$  be the angle between the rotation axis of the spheroid, which should be  $[01\bar{1}]$  and the nearest axis of form  $\langle 100 \rangle$ , which must be either  $[010]$  or  $[00\bar{1}]$ , and let  $\beta$  be the angle between the rotation axis

TABLE II. Work needed to magnetize.

Specimen	°C	10 <sup>4</sup> erg·cm <sup>-2</sup>			
		$W_{110} - W_{100}$	$W_{111} - W_{100}$	$K_1$	$K_2$
K10	14	-0.18	-0.33	-0.72	- 2.4
	150	-0.08	-0.15	-0.32	- 1.2
	300	0	-0.03	0	- 0.8
K30	14	0.17	0.17	0.68	- 1.5
	150	0.06	0.06	0.24	- 0.5
	300	0	0	0	0
	454	0	0	0	0
K35	14	0.36	0.22	1.44	- 7.0
	150	0.32	0.25	1.28	- 4.8
	298	0.24	0.20	0.96	- 3.2
	490	0.12	0.12	0.48	- 1.1
K50	14	0.83	0.43	3.32	-18.3
	150	0.69	0.52	2.76	-10.8
	300	0.46	0.36	1.84	- 6.8

and the plane (100). In a perfectly cut spheroid  $\alpha=45^\circ$  and  $\beta=0$ . It turns out that deviations of several degrees from the ideal values have a very small effect upon anisotropy in the equatorial plane because this passes through maxima and minima of the energy surface. The results can be corrected for such errors in cutting, as Shih<sup>2</sup> has pointed out in his latest paper, but this has not seemed necessary here.

Table I gives information about the spheroids used in the present work. The demagnetizing factor  $N$  is that observed to be necessary to make the initial portion of  $I$ - $H$  curves at room temperature coincide with the line  $H=0$ .

## RESULTS

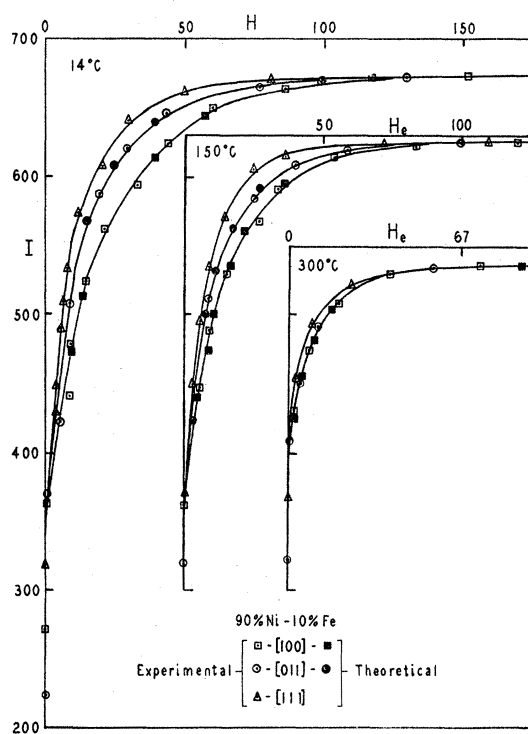
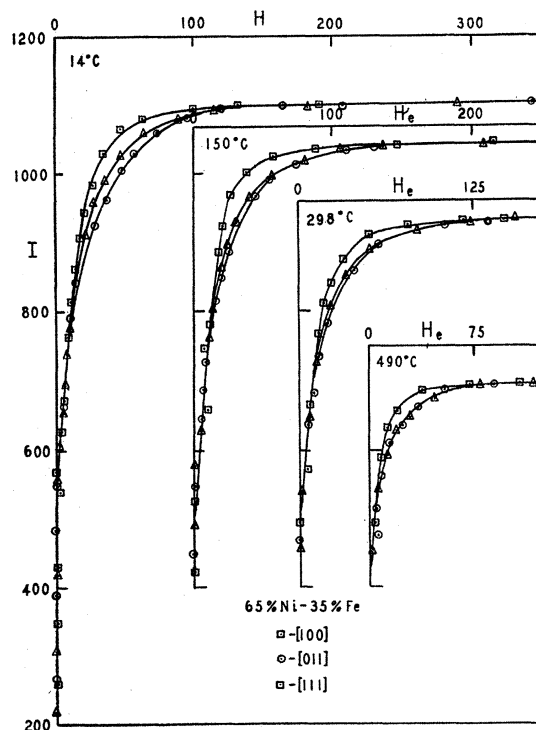
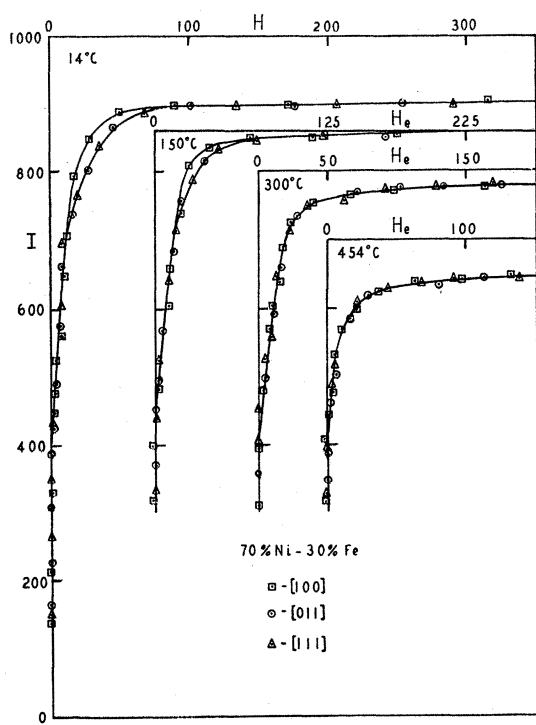
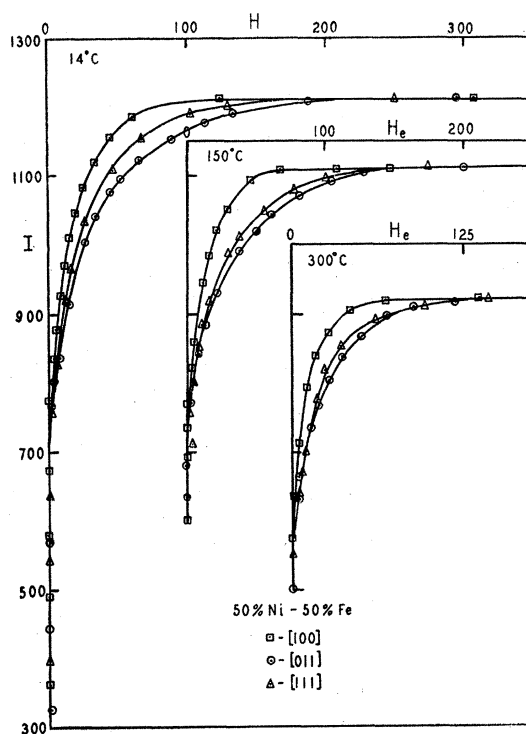
The method of measurement and the reduction of observations to the form of  $I$ - $H$  curves have already been described.<sup>5</sup> The results are presented in Figs. 1 to 4 and energy differences and energy coefficients are collected in Table II. For the method of computation and the meaning of symbols see the preceding paper by R. G. Pietry.<sup>8</sup> The most unexpected result is that the direction for easiest magnetization changes not from  $\langle 111 \rangle$  to  $\langle 100 \rangle$  as iron increases beyond a critical amount, but from  $\langle 111 \rangle$  to  $\langle 110 \rangle$ . The transition occurs as nearly as these data can indicate at 24 percent iron, as is seen from Fig. 5. This is to be compared with Lichtenberger's 29 percent and Burger and Snoek's 34 percent. (All compositions herein are in weight percent.)

In the case of specimen K10, which is most nearly like nickel in its anisotropy, Akulov's

<sup>6</sup> O. E. Buckley and L. W. McKeehan, Phys. Rev. **26**, 261-273 (1925); L. W. McKeehan, P. P. Cioffi, Phys. Rev. **28**, 146-157 (1926).

<sup>7</sup> S. L. Quimby, Phys. Rev. **39**, 345-353 (1932).

<sup>8</sup> R. G. Pietry, Phys. Rev. **50**, 1173-1177 (1936).

FIG. 1.  $I$ - $H$  curves for specimen K10.FIG. 3.  $I$ - $H$  curves for specimen K35.FIG. 2.  $I$ - $H$  curves for specimen K30.FIG. 4.  $I$ - $H$  curves for specimen K50.

method<sup>9</sup> of computing  $I$ - $H$  curves, which neglects  $K_2$ , is reasonably successful. Assuming  $I$ - $H$  along  $[111]$  as a basis, several points have been computed for  $I$ - $H$  along  $[011]$  and  $[100]$ . These are plotted on Fig. 1 and fall near the curves actually found.

One other peculiarity should be mentioned. As the temperature rises above room temperature the value of  $N$  which is apparently best at room temperature does not make the  $I$ - $H$  curve rise vertically from the origin. A smaller value of  $N$  is called for. The variation of  $N$  with temperature for a typical case is as follows:

Specimen	Temperature	$N$
K35	14°C	0.761
	150	0.759
	298	0.754
	490	0.749

This variation makes  $W_0$  uncertain, so that its values have not been presented in Table II. No satisfactory explanation for this behavior presents itself. It may be due to a variation in the thickness of a surface film magnetically very different from the interior. If so, the nature of this film and the factors upon which its thickness

<sup>9</sup> N. S. Akulov, *Zeits. f. Physik* **67**, 794-807; **69**, 78-99 (1931).

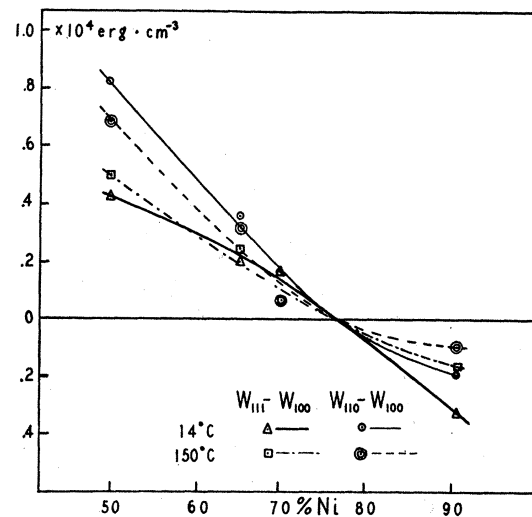


FIG. 5. Variation of energy differences with composition.

depends remain mysterious. A film of nonmagnetic material, everywhere of the same thickness, and thicker at higher temperatures, would change the apparent value of  $N$  in the observed manner.

It is with great pleasure that the author takes this opportunity to thank Professor L. W. McKeehan, who suggested this problem, for his advice and aid throughout the entire investigation.

## Electrical Resistivity of Single Crystals of Some Dilute Solid Solutions in Zinc

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Sets of single crystals were grown of binary alloys consisting of dilute solid solutions of Cd, Cu, Ag, Au, Ni and Fe, each in zinc. Electrical resistivities at 20°C are expressed in terms of principal resistivities,  $\rho_0$  and  $\rho_{90}$ , these values coming from the usual  $\cos^2 \theta$  ( $\theta$ =orientation) plot of measured resistivities of a set of crystals. The resistance increases in a given series of alloys with increasing concentration, in a nonlinear fashion. The initial increase in

resistivity (above that of pure zinc) in micro ohm-cm per atomic percent of solute is: Cd 0.94, Cu 0.4, Ag 1.1, Au 2.3, Ni 51.0, Fe 300.0. This is correlated with the nearness of the solute metal to zinc in the periodic table. The ratio of principal resistivities is slightly higher than that for the zinc crystal. Temperature coefficients decrease in such a way that Mathiessen's rule is satisfied (with a maximum deviation of 4 percent) for all the alloys.

### INTRODUCTION

THE resistivity and temperature coefficient of resistivity of dilute solid solutions of one metal in another have been studied frequently and the type of result to be expected is known at least qualitatively. Such studies in the past are apparently lacking for metals crystallizing in the nonregular system, probably due to the difficulty

of obtaining polycrystalline samples with grains oriented completely at random. It seemed worth while, therefore, to make a somewhat systematic study of single crystal samples, not only for the above reason, but because of the more fundamental nature of the approach using single crystals. Zinc was chosen as the solvent material. Binary alloys were made, each of zinc and some