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Isochronal Recovery of the Shape of the Normal Magnetization Curve of Nickel Shocked at 400 kbar

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The recovery of the shape of the normal magnetization curve of high-purity polycrystalline nickel shock loaded to 400 kbar has been studied using an isochronal annealing technique. It was found that gross shape recovery occurs after annealing at temperatures in excess of 400 °C. Lower-temperature annealing does not effect the shape of the normal magnetization curve significantly but lower-temperature annealing processes are reflected in the value of the field strength associated with the maximum susceptibility.

It is well known that the passage of a shock wave through a material alters many of the residual properties of the material.1 The effect of shock loading on the residual magnetic properties of ferromagnetic materials has received only limited attention. Kumar and Ward² found that the magnetic susceptibility of mild steel decreased by approximately 2% after shock loading to 115 kbar. In a more complete study, Rose, Villere, and Berger³ and Villere⁴ have studied the effects of shock loading on the normal magnetization curves and hysteresis loops for Armco iron. In their study, they found that all structure-sensitive magnetic properties changed in a systematic way with peak shock pressure. As peak shock pressure increased, the normal magnetization curve was observed to be shifted to higher fields and the hysteresis loop was observed to open. A similar effect of peak shock pressure has also been observed by Berger⁵ on shock-loaded high-purity nickel.

For the most part, residual shock effects can be attributed to the structural defects which are produced during the shock deformation process. Lattice defects are characterized by internal stress fields which have a pronounced influence on the magnetization of ferromagnetic materials. The effect of internal stress on magnetic properties has been theoretically treated using micromagnetics and phase theory. $^{6-10}$ The theoretical investigations have been supplemented by a number of experimental studies which determined the effects of defect formation on such magnetic parameters as initial and reversible susceptibility, coercive force, approach to ferromagnetic saturation, magnetocrystalline anisotropy constants, and others. 9 , $^{11-15}$

In this investigation an attempt has been made to study the defects occurring in shocked nickel by measuring the normal magnetization curve of a shocked nickel sample and monitoring the change in shape as a function of isochronal annealing temperature. In several investigations^{11-13, 15-18} the recovery of magnetization, reversible susceptibility, and coercive force has been studied and correlated with defect structure. This investigation is an extension of these works.

EXPERIMENTAL METHODS

Materials and Specimen Preparation

The material used in this investigation was poly-

crystalline nickel with the following nominal composition: 99.98% Ni, 0.02% Co.

The nickel was annealed at 600 °C for 6 h in vacuum. Part of the annealed material was set aside and the remainder was shocked at 400 kbar using the flying-plate technique described by Rose and Grace.¹⁹ Small-rod specimens approximately 6.6 cm \times 0.47 cm diam were machined from both the annealed and the shocked material. During machining, care was taken to prevent heating.

Magnetic Measurements

Measurement of the normal magnetization curve was accomplished using the circuit shown in Fig. 1. This device, a modification of the device developed by Hudson,²⁰ consists of two identical pick-up coils surrounded by identical field coils. The field coils are connected in phase, but the pick-up coils are connected in phase opposition. This results in equal and opposite induced voltages in the pick-up coils when the magnetizing field is changed by changing the current in the field coils. The measurement of the magnetic induction B as a function of the magnetizing field H for a particular sample can then be made by placing the sample in one of the pick-up coils and changing H. The emf appearing across the pick-up coils is fed to an integrating circuit so that a quantity e, is recorded, which is proportional to the total change in flux produced by a known change in H. If the change in H is made equal to H, the quantity e_c is proportional to B. The magnetization M is then calculated from the relation

$$M=(B-H)/4\pi.$$

There are some errors associated with the data used to establish the shape of the normal magnetization curves. The error associated with each point on the curve was estimated by repeatedly measuring the normal magnetization curve of an annealed nickel control sample. Five runs were made and the average scatter was ± 5 G. This scatter is significant at low fields where the rate of change of the magnetization with field strength is rapid. In this region, the location of the curve was established by a leastsquares curve-fitting procedure. At higher fields, the location of the curves was established by drawing smooth curves through the experimental points as measured.

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FIG. 1. Circuit diagram of apparatus.

The other curves obtained in this investigation are not individually measured but are established by taking points from the normal magnetization curve. Thus the uncertainty in their position is very small. In a number of cases, duplicate magnetization curves were run on the test sample after isochronal annealing at various temperatures. The results of these tests are shown on the appropriate figures by duplicate points and these points indicate the manner in which the experimental errors associated with the location of the normal magnetization curve effect their respective curves.

Annealing

The shocked nickel specimen was annealed either in water, at temperatures up to $100 \,^{\circ}$ C, or in a quartz tube furnace under a dry-nitrogen atmosphere for temperatures above $100 \,^{\circ}$ C. The temperature was held constant to within $\pm 2 \,^{\circ}$ C. The specimen was charged into the water or furnace at temperature and annealed for 5 min beginning when the specimen reached the annealing temperature. When annealing in the water, the time required for the specimen to reach temperature was negligible; when annealing in the furnace, the time required for the specimen to reach temperature was about 10 min. At the end of the annealing time, the samples were quenched in water.

EXPERIMENTAL RESULTS AND DISCUSSION

The normal magnetization curve for a well-annealed ferromagnetic material may typically be divided into three regions as described by Bozorth.²¹ Region I is a region of reversible domain boundary displacement which starts at the origin and increases linearly according to the Rayleigh relation

$$M = (\mu_0 - 1)H/4\pi$$

where $\mu_0 = (dB/dH)_{B=H=0}$. Region II is a region of irreversible boundary displacement. In this region dB/dH is large, sometimes reaching values of 10^6 . Region III is a region of reversible domain rotation which fits a relation of the type

$$M = M_{a}(1 - a/H - b/H^{2} - ...)$$

where M_s is the specific magnetization in an infinite field and the a, b's are constants.

Figure 2 shows the measured normal magnetization curves of fully annealed, shocked, and selected intermediate annealed specimens. Also shown in Fig. 2 is a curve for high-purity nickel published elsewhere.²² As can be seen, the literature curve and the measured curve are in excellent agreement.

Examination of Fig. 2 also shows that for shocked nickel, region I extends to much higher fields than annealed nickel and that a distinct region II is considerably reduced. This observation is consistent with that of Rose *et al.*³ for iron and Berger⁵ for nickel. The shape of the normal magnetization curve does not appreciably change with annealing temperature up to about 400 °C. Above 400 °C, the curve begins to show noticeable shape recovery.



tion curves is better illustrated in Fig. 3 where M is plotted against annealing temperature for various values of H. In this figure it is again apparent that significant shape recovery begins at about 400 °C.

The recovery of the shape of the normal magnetiza-

FIG. 2. Normal magnetization curves for nickel after shocking and annealing.