Reprinted from

JOURNAL OF APPLIED PHYSICS VOLUME 42, NUMBER 11 OCTOBER 1971

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

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(Received 23 November 1970)

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 Ward[2] 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 Berger[3] and Villere[4] 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 Berger[5] 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 polycrystalline 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×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_c 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 least-squares curve-fitting procedure. At higher fields, the location of the curves was established by drawing smooth curves through the experimental points as measured.

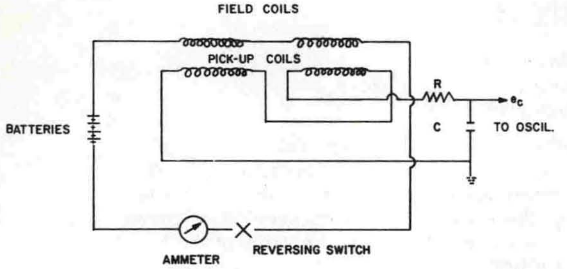

FIG. 1. Circuit diagram of apparatus.

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 °C, or in a quartz tube furnace under a dry-nitrogen atmosphere for temperatures above 100 °C. The temperature was held constant to within ±2 °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

$$M=M_s(1-a/H-b/H^2-\ldots),$$

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 Berger[5] 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.

The recovery of the shape of the normal magnetization 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.

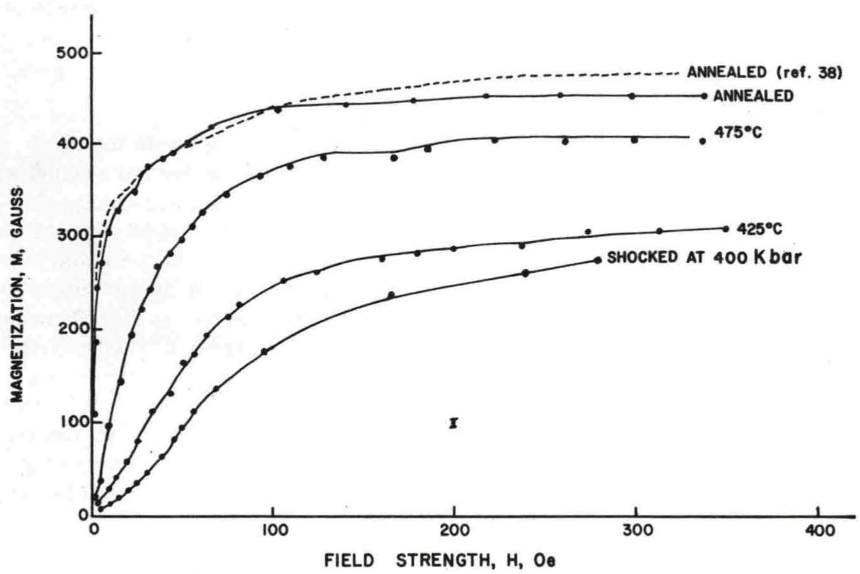

FIG. 2. Normal magnetization curves for nickel after shocking and annealing.

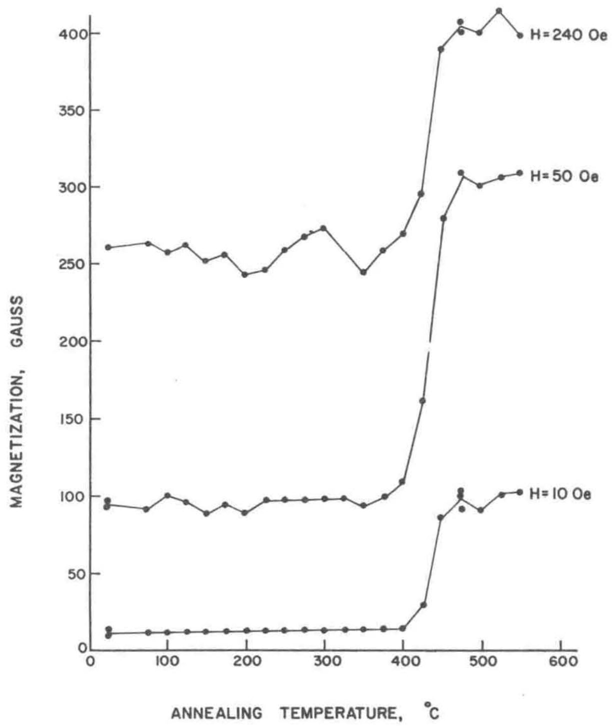

FIG. 3. Isochronal annealing curve for M at various H levels.

It can be seen from Fig. 2 that the initial slope of the magnetization curve is a region I parameter. This slope defines the initial susceptibility χ_0 which is related to the angle θ that the magnetization curve makes with the axes through the relation

$$\theta = \tan^{-1} \chi_0 .$$

The variation of θ with annealing temperature is shown in Fig. 4 where it can be seen that pronounced recovery begins at about 400 °C. However, in the annealing range between 25 and 400 °C there is a considerable amount of fine structure which may be related to low-temperature recovery processes. To examine this proposition, it was necessary to extract parameters which were clearly sensitive to low-temperature structure changes, i.e., point-defect processes. This was done as follows.

The ratio M/H is called the susceptibility χ and this parameter represents the relative increase in magnetic moment caused by the application of the magnetic field. Theoretically[7] it is found that this quantity is particularly sensitive to dislocation structure at high fields. Figure 5 shows the susceptibility curves for several samples. Characteristic of each susceptibility curve is the maximum susceptibility χ_m and the corresponding value of the field H_m. Figure 6 shows these parameters plotted as functions of the annealing temperature. These two curves show the desired sensitivity. For annealing temperatures between 25 and 400 °C, χ_m is nearly constant but H_m shows two minima. Between 400 and 475 °C both curves show significant change. Above 475 °C, the behavior is reversed with H_m remaining nearly constant and χ_m showing change.

The recovery of shocked high-purity polycrystalline nickel has been studied by Kressel[23] using standard resistivity methods. At a peak pressure of 330 kbar, the recovery spectrum above room temperature consisted of three well-defined annealing stages (stages III, IV, and V). Stage III had an activation energy of 1.1 eV and was centered around 88 °C. The activation energy for stage IV was found to be 1.5 eV and the recovery peak was centered around 260 °C. As shown in Fig. 6, this behavior correlates very well with the behavior found in the present study. The center of the low-temperature recovery peaks found by Kressel correspond exactly to the minima A and B.

Stage III and stage IV recovery are known to be associated with point-defect processes. Stage III is usually attributed to the migration of mobile interstitials to fixed vacancies.[24] However, some of Kressel's data suggest also the possibility of defect migration to dislocations. Stage IV recovery is interpreted as the annihilation of single vacancies.[25]

The recovery above 350 °C is caused by the rearrangement and annihilation of dislocations. Clearly, these processes affect all the magnetic parameters measured in this study. The points C and C′ in Fig. 6 suggest the interaction of dislocations with some type of point defect, probably vacancy clusters. However, there is some question to this interpreta-

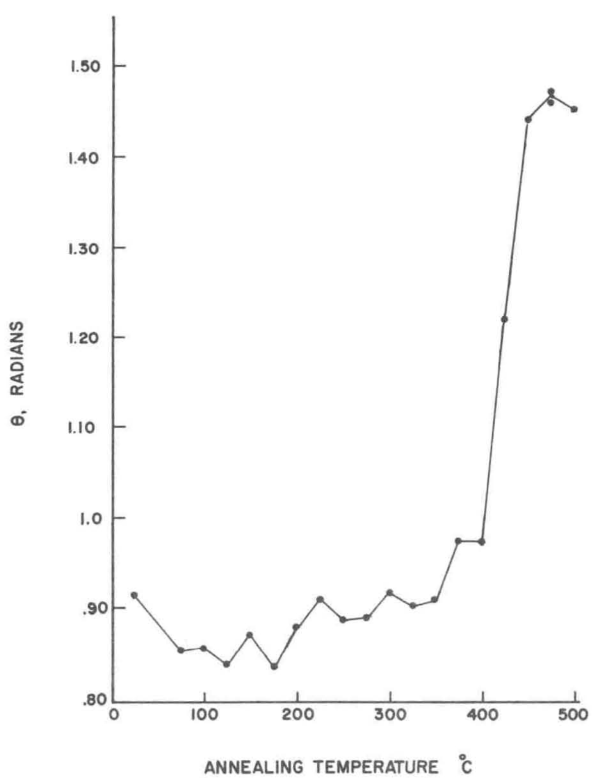

FIG. 4. Isochronal annealing curve for angle θ.

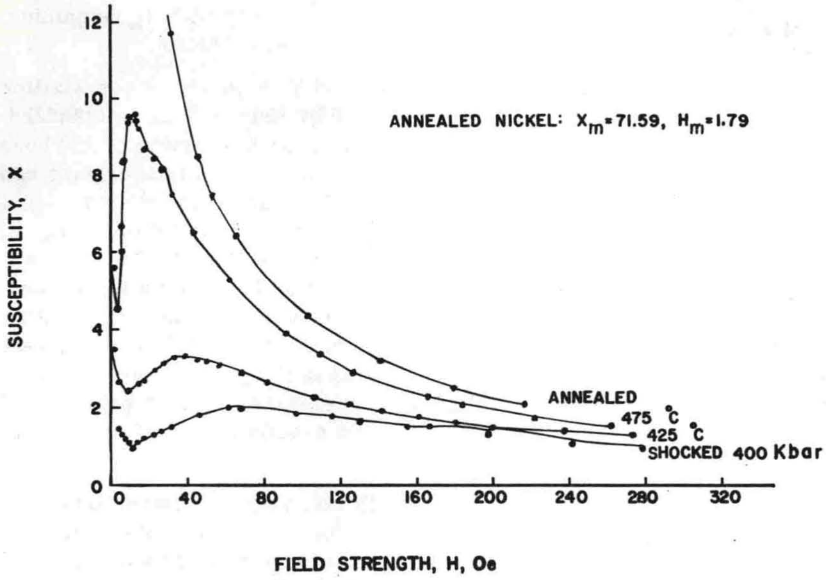

FIG. 5. Susceptibility curves for nickel after shocking and annealing.

tion because the temperature corresponding to these points is approximately equal to the Curie temperature for nickel. The effect of cycling through the Curie temperature is currently under study to clear up this point. The major recovery stage V should contain two regions which correspond to polygonization and primary recrystallization. In the present study the primary recrystallization phase was not resolved because the experiment did not extend to high enough temperatures. Annealing was discontinued because quenching stresses began to interfere with the measurements.

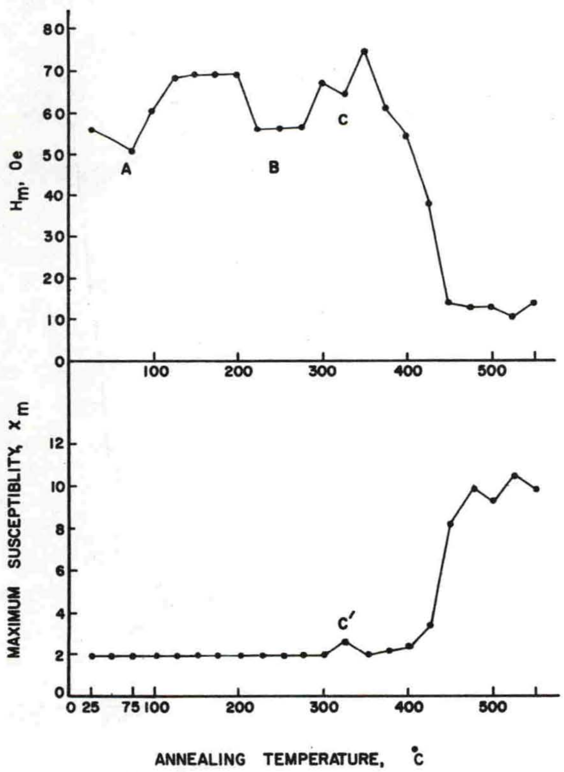

FIG. 6. Isochronal annealing curves for χ_m and H_m.

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