Magnetic Measurement of Low Temperature Recovery after Plastic Deformation

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THE recovery of magnetic properties of plastically deformed metals has been a subject of investigation for many years.[1, 2] It seemed interesting to perform additional experiments for the following reason.

It has been shown recently that the recovery of physical properties does not proceed continuously, but stepwise,[3] every step corresponding more or less to one activation energy. Several suggestions have been advanced to ascribe certain steps of recovery to the disappearance of specific types of lattice defects which are generated either by plastic deformation or by high energy particle irradiation.[4]

It has been generally assumed that the recovery at low temperatures, as measured, for instance, by the decrease of resistivity is due mainly to the annihilation of point defects such as vacancies in the lattice. The lack of response of hardness and other mechanical properties to a low-temperature anneal has contributed in making this assignment.

The low-temperature recovery of magnetic properties which by virtue of the magnetoelastic energy may be very strain sensitive has, to our knowledge, not been considered in this connection.

In the preliminary experiments to be reported in this paper we have tried to answer the following questions. Does the recovery of the magnetization curve (commutation curve) of nickel, plastically deformed at liquid nitrogen temperature reflect the recovery behaviour of the density, the resistivity, the release of energy as observed above room temperature in the work of Clarebrough, Hargreaves and West [5]? Do we find recovery magnetically at temperatures far below room temperature? Does the whole commutation curve recover in the same way by low-temperature anneal, or are special parts of it particularly affected?

Experimental

Nickel strips, kindly supplied by H. J. Meerkamp van Embden of the Philips Research Laboratories at Eindhoven were used. These strips were 24 cm. long, 2 cm. wide, 0·01 cm. thick. One batch which was analysed, contained 0·1 per cent. Co, 0·05 per cent. Mg and 0·03 per cent. Cu as main impurities. Prior to the measurement the material had been annealed *in vacuo* at about 1050° C. The structure after that treatment was polycrystalline, the orientation of the crystals was at random. The soft strips were inserted into and kept during the whole of the investigation in a slot within a bronze rod in order to avoid unwanted deformation as much as possible. While in the slot the strips were plastically drawn at − 196° C.

The recovery measurements were done ballistically against a reference strip in a similar rod which during the whole time of the investigation was kept in liquid nitrogen. This reference strip had been deformed before the measurements began and subsequently annealed three hours at 300° C. Because of this set-up the recovery as the difference in magnetization after

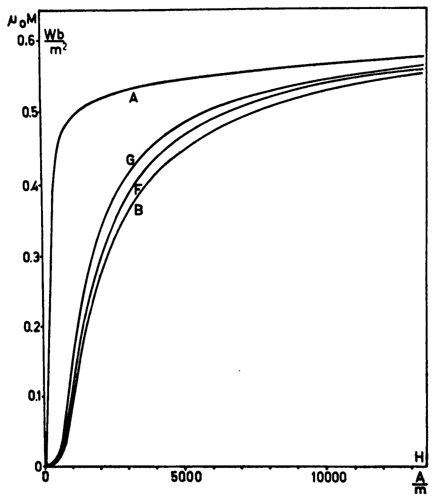

FIG. 1. Magnetization curves of nickel, measured at — 196° C.

A. Undeformed material.
B. After plastic deformation at — 196° C, 5·40 per cent. elongation.
F. Material as in B after recovery successively for about 3 hours at — 90° 20°, 120° and 200° C.
G. Material as in F after about 3 more hours at 300° C.

different thermal treatments (Fig. 2) is better known than the course of the magnetization curve. All measurements were done at — 196° C. The recovery at — 90° C occurred by placing the bronze rod with the specimen in a bath of propane kept at that temperature. Recovery at temperatures above room temperature occurred by annealing *in vacuo*.

EXPERIMENTAL RESULTS

Some of the results of measurement are given in Figs. 1 and 2. Apparently recovery occurs already at the lowest annealing temperature of — 90° C. We do not consider this recovery to be a thermally activated process as a whole for the following reason. In a set of experiments—not reproduced here—a specimen, of which the magnetization was measured at — 196° C, was (a) annealed for a short time at — 90° C, (b) subsequently annealed at this temperature for three hours, and (c) subsequently annealed at — 90° C for a short time again. The change of magnetization by the treatments (b) and (c) was equal, though smaller than that due to treatment (a). This indicates that in this case recovery is due at least partly to the stresses set up in the annealing operation : insertion of the specimen in the propane bath and in the liquid nitrogen respectively.

The stresses probably cause unanchored dislocations to move. This possibility must be kept in mind when considering analogous recovery experiments of other physical properties. At 20° C a short anneal after a preceding three-hour anneal had almost no effect.

The recovery of the magnetization curve becomes intense between 200° and 300° C. This matches with the measurements of the Australian workers.[5]

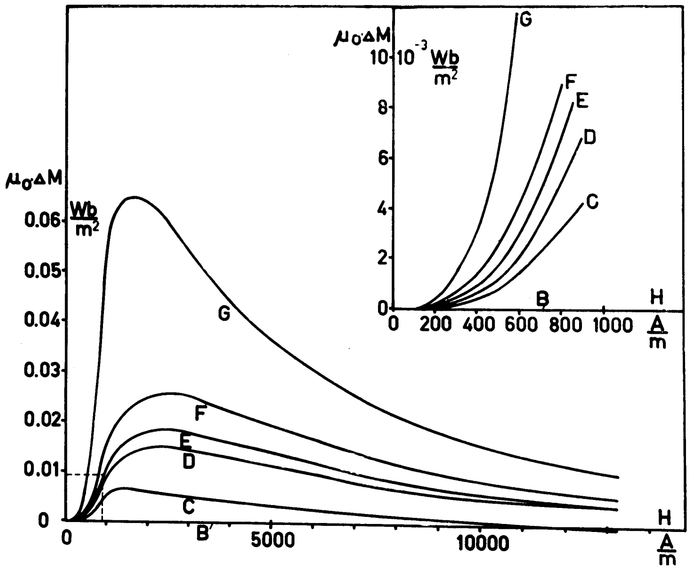

FIG. 2. Recovery of magnetization of nickel after plastic deformation at − 196° C, 5·40 per cent. elongation. All measurements at − 196° C.

B. Unrecovered material.
C. After recovery during about 3 hours at − 90° C.
D. Additional recovery at 20° C (about 3 hours).
E. Additional recovery at 120° C (about 3 hours).
F. Additional recovery at 200° C (about 3 hours).
G. Additional recovery at 300° C (about 3 hours).

In this temperature interval they find a hump in the rate of release of stored energy which they ascribe to the disappearance of vacancies. In accordance with these authors we find no recovery of mechanical hardness in this temperature region. We measured microhardness on a strip which had had about the same plastical deformation as the strip used for magnetic measurements, at − 196° C. For the soft strip we found at room temperature : H.V. = 68·3. The hardness of the drawn strip after a short stay at room temperature was 104 ; after three more hours at room temperature, 105 ; after three additional hours at 150° C, the room temperature hardness was 105, and after three more hours at 300° C, 104.

The figures show that the low-temperature recovery of the magnetization is especially pronounced at higher field strengths. The insert of Fig. 2 indicates that no low-temperature recovery occurs of the initial permeability. The increase of induction due to anneal compares with the decrease due to deformation in the region of saturation. Our field strengths were too small to make extensive measurements in this region, which has been treated theoretically by W. Fuller Brown and Néel [6] and experimentally by Dietrich and Kneller.[7]

Discussion

As has been said in the introduction, it would be desirable to define the nature of the defects disappearing at low temperatures. There is one clue : the initial permeability does not change at all at first, while recovery of the magnetization curve occurs for all higher field strengths, more so in the region of irreversible wall movement. We propose tentatively as explanation that pile-ups of dislocations disappear or disperse by low-temperature recovery. They give large, localized stress fields, which do not hamper the greater part of the domain surface present in low fields, but catch walls moving over large distances during irreversible movement.

Apparently the pile-ups can be made to disappear both by the application of external stress and by thermal activation. It may be that the point defects envisaged by the Australian workers [5] play a roll in the latter process.

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