

MAGNETIC AFTER-EFFECT MEASUREMENTS ON METASTABLE SAMPLES¹⁾

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Magnetic after-effect (MAE) measurements have been carried out on samples structurally not stable in the investigated temperature range. It is shown, how irreversible MAEs induced by structural changes can be experimentally separated from reversible relaxation processes. General principles of ensuring an adequate interpretation of the MAE measurements on metastable samples are also given.

ИЗМЕРЕНИЯ МАГНИТНОГО ПОСЛЕДЕЙСТВИЯ НА МЕТАСТАБИЛЬНЫХ ОБРАЗЦАХ

В работе приводятся результаты измерений магнитного последдействия на образцах, которые в исследуемой области температур структурно нестабильны. Показано, как можно экспериментально отделить измерения необратимого магнитного последдействия, обусловленного структурными изменениями, от измерений процессов обратимой релаксации. Приводятся также общие принципы, обеспечивающие соответствующую интерпретацию измерений магнитного последствия на метастабильных образцах.

1. INTRODUCTION

The measurement of the magnetic after-effect (MAE) is a very sensitive tool for the investigation of mobile lattice defects in ferromagnetic materials.

The interaction of these defects with domain walls results in a stabilization of the latter, causing a time dependent permeability after demagnetization. This "accommodation" of lattice defects with respect to the domain walls is in general thermally activated, following the Arrhenius law. In this case, MAE measurements can be easily evaluated to give the activation parameters of the lattice defects. The evaluation is based in general on the following assumption: besides the MAE itself,

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the sample is in thermal equilibrium. This is a rather critical hypothesis, since samples with increased defect concentrations are seldom stable in the whole investigated temperature range. In favourable cases, however, the MAE and the phase transition appear in different temperature ranges. Some years ago, a MAE was detected in the temperature range of a metallurgical phase transition, i.e. the precipitation of Fe_8N_2 in a Fe-N alloy [1, 2]. This investigation allowed to formulate some general principles of such processes. In the present study, the effect of structural instability on the MAE spectrum was investigated on additional crystalline iron samples. The most interesting experimental aspects will be reported here.

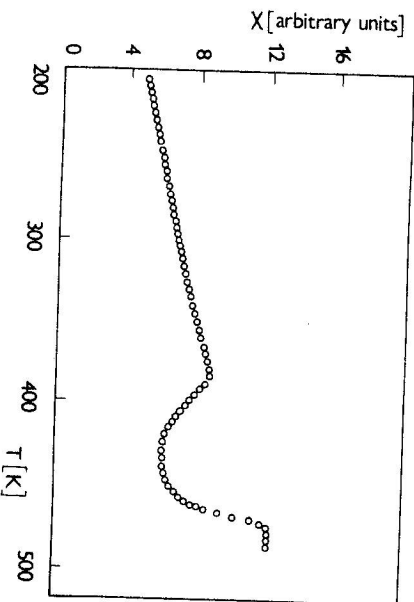


Fig. 1. Temperature dependence of the initial susceptibility of an as-quenched Fe—N sample.

II. EXPERIMENTAL TECHNIQUES

II. 1. Sample preparation and heat treatments

Specimens from high purity (Johnson and Matthey) iron were cold-swaged to 1 mm ϕ and stress-relieved annealed in pure hydrogen at 1000 K. Nitrogen or carbon were introduced by annealing the specimens during 36 h in H_2 +1—3% NH_3 or CH_4 at 773 K or 1000 K, respectively. After annealing the samples were quenched to room temperature and stored in liquid N_2 till the time of the measurement in order to prevent uncontrolled precipitation. Prior to nitriding, some iron samples were cold-worked at room temperature by plastic straining in an Instron machine up to 2%.

II. 2. MAE measurements

The MAE has been investigated in terms of time and temperature dependence of the initial susceptibility, $\chi(t, T)$, after demagnetization, using a very sensitive ac

measuring technique [3]. The experimental results are represented by isochronal relaxation curves of the reluctivity $\Delta\chi(t_1, t_2, T) = \chi(t_2, T) - \chi(t_1, T)$ where $r(t, T) = 1/\chi(t, T)$. In our measurements, $t_1 = 1$ s, $t_2 = 2, \dots, 180$ a.

III. EXPERIMENTAL RESULTS AND DISCUSSION

III. 1. Influence of precipitation and redissolution on the MAE spectrum

Fig. 1 shows the initial susceptibility of a Fe-80 wt ppm N sample. The pronounced susceptibility minimum at 430 K is due to the precipitation and redissolution of Fe_8N_2 , as proved by various independent experimental methods [1, 2].

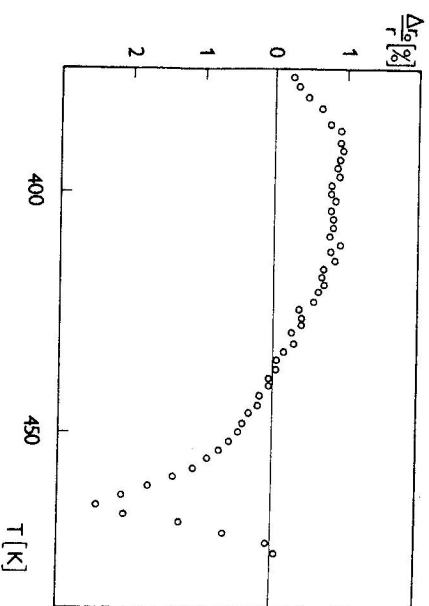


Fig. 2. Time and temperature dependence of the relative initial reluctivity of the same sample, without demagnetization, $t_1 = 1$ s, $t_2 = 180$ s.

In Fig. 2 a time-dependent measurement is shown on the same sample with $t_1 = 1$ s and $t_2 = 180$ s, but without any demagnetization of the sample. From a comparison of the results presented in Fig. 1 and Fig. 2, the practical and theoretical difficulties of MAE measurements in this temperature region can be recognized.

The process of precipitation is accompanied by a time dependent decrease of the susceptibility due to a pinning of the domain walls at the precipitates. This decrease of the susceptibility induces a so-called positive MAE during a time-dependent reluctivity measurement. The re-dissolution of the precipitates — as the reversed process — results in a time-dependent increase of the susceptibility and gives rise to a negative irreversible MAE.

Since these processes correspond — within given temperature ranges — to irreversible structural changes, they develop independently of any preceding demagnetization and consequently differ from reversible relaxation phenomena in the common sense. They could be observed as well in any static experiment, e.g. by a magnetic balance.

III. 2. Control of sample stability: separation of reversible and irreversible effects

As shown in Fig. 1 and Fig. 2, irreversible structural changes can be revealed by measuring without demagnetization. The problem of the reproducibility of the sample's state can be eliminated by controlling the stability during isothermal relaxation measurements performed in succession with and without preceding demagnetization.

From a series of such measurements the experimental results can be corrected to give the true reversible relaxation.

III. 3. Typical experimental results

III. 3. 1. Fe-N

By measuring on a "stabilized" sample — which had been annealed in order to allow all N to precipitate — the positive irreversible changes can be eliminated, so that in this case the true reversible effect can be well evaluated. As described in detail elsewhere, the reversible MAEs are due to relaxation of N atoms in the strain fields of coherent precipitates [1, 2].

III. 3. 2. Fe-C

By measuring on an as-quenched alloy, reversible and irreversible effects can be detected as well. In contrast to Fe-N the reversible effect disappears in the stabilized — precipitation annealed — alloy. This may be explained most probably by the loss of coherency of the carbide precipitates, which results in the breakdown of the strain field.

III. 3. 3. Cold-worked Fe-N alloys

Fig. 3 shows the complete set of measurements obtained on a sample which had been cold-worked prior to nitrating. Below 400 K, data measured with and without demagnetization agree rather well, indicating that only irreversible processes are

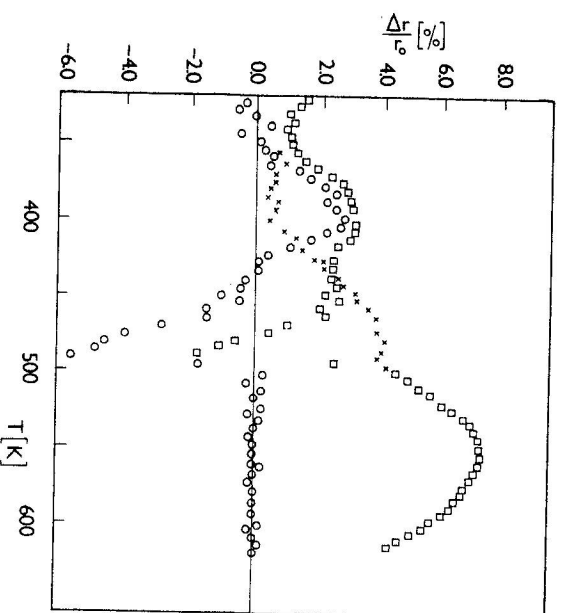


Fig. 3. MAE spectrum of an as-quenched Fe-N sample, cold-worked prior to nitrating, measured with and without demagnetization, $t_1 = 1$ s, $t_2 = 180$ s. \circ without demagn., \square with demagn., \times correct true MAE.

It is beyond the scope of this paper to give an unambiguous interpretation of this complicated process.

IV. CONCLUSIONS

1. In order to obtain adequate interpretations of experimental results, the structure and stability of the samples should be carefully controlled.
2. If possible, MAE measurements should be carried out on samples which are structurally stable in the investigated temperature region.

present in this temperature range. These irreversible processes are supposed to be induced by the precipitation of N, most probably on dislocations. Between 420—500 K, the re-dissolution of these precipitates takes place. Above 500 K, only a reversible, dislocation-induced relaxation process is present. After correction for the negative structural changes, a "shoulder" is obtained on the low temperature side of this dislocation peak. In a pure iron sample, the dislocation relaxation takes place at lower temperatures, and no "shoulder" can be detected. It is not easy to decide, whether the presence of a shoulder indicates an additional independent process, or results only from dislocation relaxation which is partially suppressed due to the presence of precipitates.

3. Since elastic properties are also structure sensitive — internal friction measurements may also be a valuable tool for the corresponding investigations. Unfortunately, no such data have been reported so far; but the control of the sample stability, e.g. by measuring both components of the complex modulus, should give additional information on these problems.

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