Influence of sample surface on thermomagnetic effects in Fe40-Ni60

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It is shown that the thickness of the sample, i.e., its surface-to-volume ratio, affects the outcome of thermomagnetic treatments on Fe40-Ni60. The existence of surface effects is proved by the differences of hysteresis loops observed on samples of various thicknesses annealed under identical conditions. Surface influence is further checked by etching-off equivalent surface layers after a given annealing, and comparing the resulting loop changes: these are found to depend markedly on the initial thickness of the sample. The enhancement of thermomagnetic effects on samples with larger surface-to-volume ratios is qualitatively explained by observing that a distribution of directional anisotropy energy probably affects the loop shape more effectively if induced in a surface layer, since it is correlated with domain patterns intrinsically more complex than in the bulk.

1. Introduction

As is known, the effect of thermomagnetic treatments on magnetic properties of alloys characterized by directional order, depends on several factors: alloy composition, magnetization state during treatment, temperature and time of annealing, cooling rate, atmosphere. All these factors have been extensively studied, in particular for Fe-Ni alloys [1]. However, the influence of another factor, the sample surface-to-volume ratio, does not, so far, seem to have been taken into account, but for a brief mention by Zaikova *et al* [2].

In this paper it is shown that this factor plays a rather important role in affecting the outcome of a given thermomagnetic treatment in Fe40-Ni60.

2. Experimental

Toroidal samples (o.d. 35, i.d. 28 mm) of different thicknesses (6, 3, 1 and 0.5 mm) were machined from a vacuum-melted Fe40-Ni60 ingot. Samples were then polished, electrochemically etched and annealed at 1300°C for 24 h in H_2. Grain size was of the order of 0.5 mm. All further thermomagnetic treatments were performed at 450°C in N_2 for times of 4 h. In order to ensure identical heat-treatments, all samples were always annealed together.

Annealings were performed in one of the following conditions: (a) demagnetized state; (b) saturated state; (c) remanent induction state. Results are summarized in Figs. 1 to 3, each figure showing four loops (one for each thickness), traced at room temperature by means of an electronic hysteresigraph [3]. Fig. 1 shows low field loops after annealing in the demagnetized state: all loops show a constricted region, whose amplitude increases for decreasing sample thickness. Fig. 2 shows saturation loops after annealing under magnetic field: loops differ because of coercive field H_c, which is larger for thinner samples. Fig. 3 shows loops after annealing on remanence: all samples are characterized by square type loops, which show a marked increase of H_c and decrease of remanence for decreasing sample thickness.

Since samples differed only because of thickness, these results clearly point to the influence of sample surface on the outcome of any thermomagnetic treatment. This was further checked by electrochemically etching-off a surface layer after a given annealing, and comparing the resulting loop changes on various samples: these changes were found to depend markedly on the initial thickness of the sample. A typical example, for samples annealed on remanence, is shown in Figs. 4a and b. Fig. 4a refers to a 0.5 mm thick
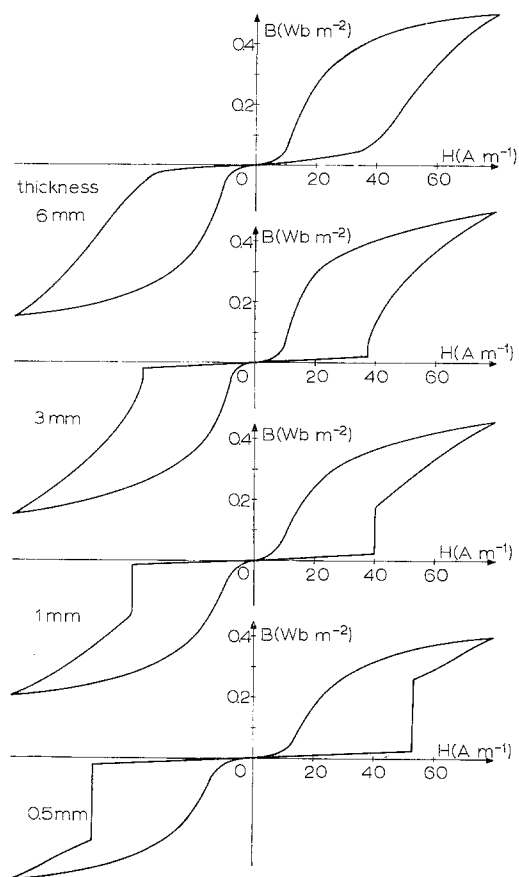
Figure 1 Hysteresis loops of specimens of different thickness after annealing in the demagnetized state.

sample, and shows the drastic variations of loop shape obtained by etching-off 0.05 mm. Fig. 4b refers to a 6 mm thick sample: loop changes obtained also by etching-off 0.05 mm are in this case very small, and even a 10% reduction of total thickness, percentually equivalent to the one of Fig. 4a, does not yield comparable changes of loop shape.

These effects are further enhanced on even thinner samples (0.2 mm), whose results are, however, not reported here, since the different preparation technique (lamination instead of tool working) may in principle affect the comparison.

3. Discussion

The dependence of magnetic properties on material thickness was already well known [2, 4-6] and attributed to convenient surface effects. The present results show, however, the existence of surface effects also on thermo-

magnetic phenomena. These effects may be qualitatively explained by observing that a distribution of directional anisotropy energy, K_u, is induced by a given heat-treatment according to the domain pattern characterizing the sample during annealing. Mazzetti *et al* [7] showed that strong mutual interactions among different Bloch walls characterize a given domain pattern after induction of a matching distribution of K_u. Because of these interactions, the blocking action exerted on Bloch walls by the induced anisotropy energy is in principle stronger when a more complex domain configuration is stabilized by the annealing. On the other hand, domain patterns on sample surface are certainly characterized by a smaller spacing of 180° walls and by a larger density of 90° closure domains [2, 5, 6], than bulk domain distributions. Therefore, an anisotropy energy distribution induced in a surface layer, can be expected to affect the loop shape more effectively than the one induced in an equivalent bulk layer. This effectiveness is enhanced on samples with larger surface-to-volume ratios, and this accounts for the results of Figs. 1 and 3. It must be emphasized that, although other factors may affect loop shape because of different sample thickness [2, 5, 6], the present results must essentially be accounted for in terms of surface induced anisotropy energy. This is clearly proved by the etching experiments, which yield the formation of new domain patterns characterized by little or no correlation with the old K_u distribution. Therefore, a loss of memory of the previous annealing results, which, for equivalent reductions of thickness, is more pronounced on samples with larger surface-to-volume ratios (Figs. 4a and b). Also the loop differences observed after annealing under technical saturation conditions (Fig. 2) must be attributed to a large extent to the different influence of the K_u distributions correlated with some residual surface domain pattern present during heat-treatment. In fact by etching-off comparable surface layers, one observes in this case an increase of H_c which is larger on samples of smaller initial thickness.

It was further verified that, in all cases, the etching procedure does not affect the material in any other way, since by repeating the identical annealing which preceded the etch, the same starting loop is obtained.

In conclusion it was shown that in Fe40-Ni60 the thickness of the sample, i.e., its surface-to-
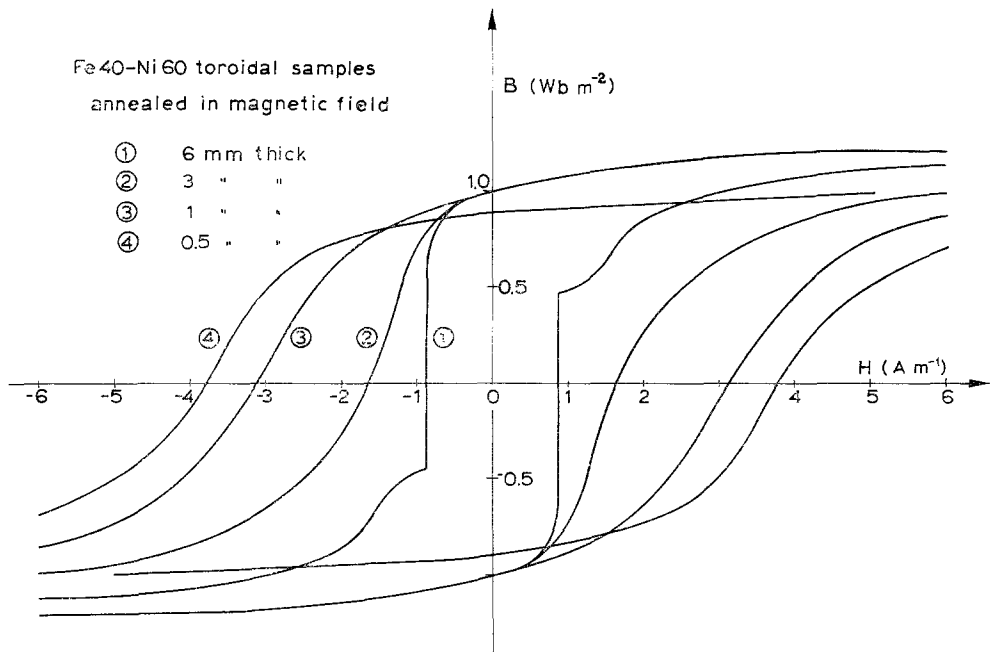
Figure 2 Hysteresis loops of the same specimens of Fig. 1 after annealing under magnetic field.
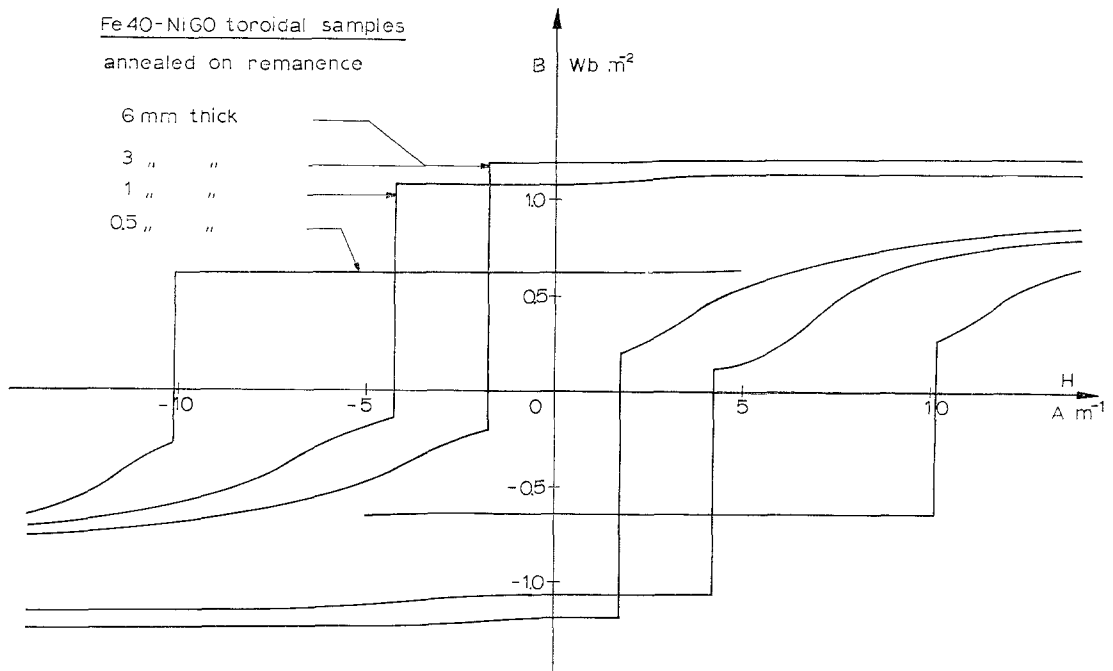

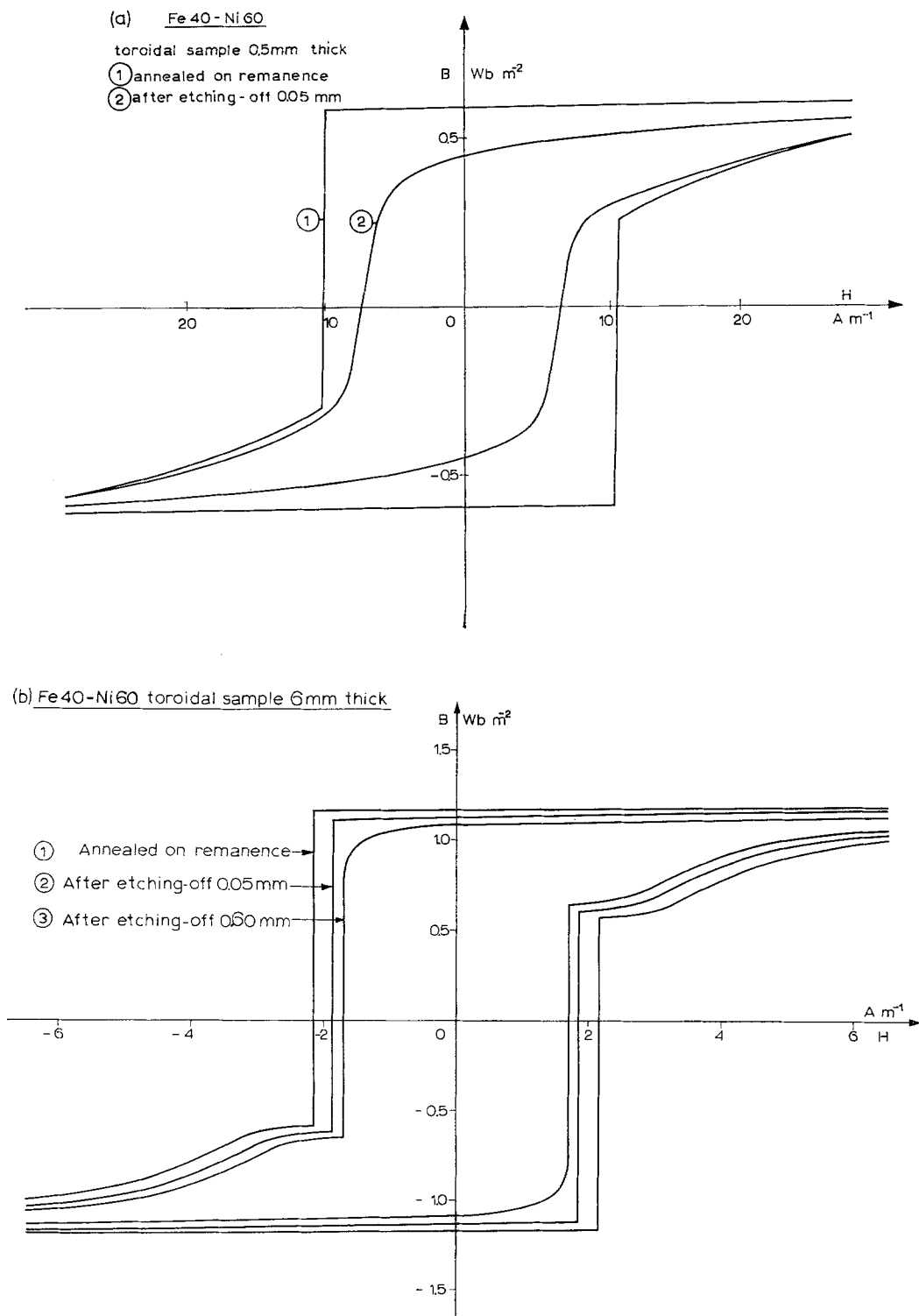*Figure 3* Hysteresis loops of the same specimens of Fig. 1 after annealing on remanence.

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Figure 4 Comparison of etching effects on samples of different thickness. (a) 0.5 mm thick sample: loop 1, after annealing on remanence; loop 2, after etching-off 0.05 mm. (b) 6 mm thick sample: loop 1, after annealing on remanence; loop 2, after etching-off 0.05 mm; loop 3, after etching-off 0.60 mm.

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volume ratio, must be included among the factors which control the outcome of any thermomagnetic treatment. It is likely that this conclusion holds also for other alloys characterized by relevant directional ordering phenomena.

Finally it must be pointed out that these surface effects should be taken into account also in measuring magnetic viscosity phenomena. The viscosity field induced by annealing in the demagnetized state can, in fact, be measured as the half-width of the constricted region of the resulting loop: as shown by Fig. 1 this width markedly depends, all other conditions being identical, on the sample surface-to-volume ratio.

Acknowledgements

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