SECONDARY MAGNETIC ANISOTROPY IN HITPERMS

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Abstract. FeCoNbB Hitperms without and with some Si show pronounced secondary magnetic anisotropy of the hard-ribbon-axis type if the surfaces differ from the ribbon interior. Precursor amorphous ribbons could bear some initial heterogeneity and ultrasonic cleaning prior to annealing caused a modest reduction of the secondary anisotropy indeed. Longitudinal field annealed heterogeneous samples do not show the expected general reduction of the hard ribbon axis but partly the opposite. As this effect is more pronounced when the alloy shows easier viscous flow at elevated temperatures, a creep-induced-like anisotropy is considered to overwhelm the feeble field-induced anisotropy. The obvious magnetoelastic anisotropy familiar with positively magnetostrictive materials is present as well.

1. INTRODUCTION

Materials produced of chemically reactive components (e.g., Fe) in a thin ribbon form which brings a large surface to volume ratio are often prone to various surface effects where surfaces differ from the rest of the ribbon (the interior) in some way. This trait we call macroscopic heterogeneity (MH) and it is frequently distinguished after a non-vacuum annealing. Different shrinkage of surfaces relative to the interior result in mutual forces: Surfaces exerting an in-plane biaxial compression (squeezing) on the interior produce characteristic hard-ribbon-axis (HRA) magnetic anisotropy in a positively magnetostrictive material [1,2]. At least this magnetoelastic effect is observed also on the Hitperms studied and shows a tilted loop in standard measuring geometry. Amorphous precursor ribbons could already bear a MH built during casting as well as due to certain handling practices or storage environment influences [3]. As the MH-induced anisotropy is still not well understood, an interaction with better characterized field-induced anisotropy was tried. The term “secondary” is here used for any magnetic anisotropy that is not explained as a direct consequence of composition, particular known atomic structure and sample shape.

2. EXPERIMENTAL

Amorphous ribbons of Hitperm- type composition (Table 1) have been prepared by planar-flow casting in air. Strips of 6 mm width, 15-20 µm thickness (it was determined from mass, density and the other dimensions) and 9 cm length were annealed in vacuum or in Ar ambience at 500 °C and 540 °C for 1 hour. Modest dc longitudinal field (550 A/m) was applied during Ar annealing to reference samples. Some ribbons were submitted to ultrasonic (35 kHz) cleaning prior to vacuum annealing. Hysteresis loops were recorded using a digitizing hysteresisgraph at standard ac (21 Hz) sinusoidal H excitation in Helmholtz drive coils. Magnetostriction coefficient $\lambda_s$ was determined from measurements on disk-shaped samples using a capacitive strain sensor [4], likewise the dilatation measurements on 7 cm long strips.

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Table 1. The composition and characteristic parameters of the Hitperms studied. $\lambda_s$ is the saturation magnetostriction coefficient of partly crystalline samples. Longitudinal thermal expansivity $\alpha$ is determined from dilatation curves (Fig. 2) between 20 °C and 120 °C at the start and at the end of the thermal cycle.

<table>
<thead>
<tr>
<th>Material</th>
<th>label</th>
<th>$\lambda_{\text{cryst}} \times 10^6$</th>
<th>$\alpha_{\text{amorph}} \times 10^4 [K^{-1}]$</th>
<th>$\alpha_{\text{cryst}} \times 10^4 [K^{-1}]$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe$<em>{61}$Co$</em>{20}$Nb$<em>7$B$</em>{12}$</td>
<td>Co$_{20}$</td>
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<td>7.3</td>
<td>11.7</td>
</tr>
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<td>Fe$<em>{54}$Co$</em>{27}$Nb$<em>7$B$</em>{12}$</td>
<td>Co$_{27}$</td>
<td>22.7</td>
<td>8.7</td>
<td>13.4</td>
</tr>
<tr>
<td>Fe$<em>{55}$Co$</em>{18}$Nb$_7$Si$<em>5$B$</em>{15}$</td>
<td>Si$<em>5$B$</em>{15}$</td>
<td>14.7</td>
<td>7.7</td>
<td>9.6</td>
</tr>
<tr>
<td>Fe$<em>{55}$Co$</em>{18}$Nb$<em>7$Si$</em>{10}$B$_{10}$</td>
<td>Si$<em>{10}$B$</em>{10}$</td>
<td>7.6</td>
<td>9.6</td>
<td>11.2</td>
</tr>
</tbody>
</table>

Fig. 1. Hysteresis loops after 1 hour annealing. Note different X-scales.
Fig. 2. The record of dilatation (elongation) at 5 K/min constant heating/cooling rate in Ar. Different stress was used to test the viscous flow capability of Si₅B₁₅. Whereas $\sigma = 5.5$ MPa just balanced the crystallization shrinkage, the doubled stress still kept a bit of flow at least down to 500 °C. The thinner Si₁₀B₁₀ clearly shrunk against 6.5 MPa. The cycles to 400 °C are to show that the structural relaxation is almost done after the first cycle and the non-crystalline ribbon shortens a bit even against $\sigma$.

3. RESULTS, DISCUSSION AND CONCLUSIONS

The ultrasonic cleaning appeared to reduce somewhat the HRA anisotropy component frequently observed also on vacuum-annealed samples. Thus the removable fraction of surface pollution also promotes the formation of the macroscopic heterogeneity and should be regarded as a part of an as-cast or initial heterogeneity. Ar-annealed samples have not been cleaned to allow for full “natural” heterogeneity. We recall here that no iron oxides were detected on the heterogeneous, partly crystallized samples [5].

Unlike the vacuum-annealed samples, standard loops tilted by the HRA anisotropy with poor approach to saturation characterize all the samples annealed in Ar (Fig. 1). The squeezing action of
the surfaces can be emulated by shrinking resin jacket [5]. Such a test has shown very similar loop response leading to an estimate of few tens MPa compressive stress $\sigma$ acting on the interior. Moreover comparing different Hitperms by this experiment show reasonable scaling of loop-determined magnetization work with the product $\lambda \times \sigma$. The point which is new with the Si-free Hitperms is, where the mutual force interaction in Ar-annealed samples comes from. Unlike Finemets and Si-containing Hitperms, the Ar annealing and ensuing crystallization increase the density of FeCoNbB Hitperms negligibly or not at all [5]. Thus even if preferred surface crystallization is observed, it is not so obvious what makes the surfaces to shrink more than the ribbon interior. Therefore dilatation (longitudinal dimension change) during a thermal cycle including partial crystallization was consulted. Experiments like these referred to by Fig. 2 and the $\alpha$ data in Table 1 show that the more crystalline material is indeed capable of larger expansion and shrinkage. A reasonable explanation would be a crystallization-induced increase of anharmonicity. The action of this mechanism is slightly different to the density increase at crystallization: If merely the larger shrinkage of surfaces serves the squeeze then it only starts to act as the temperature starts to decrease from the isotherm value whereas heterogeneously increasing density can generate macroscopic stress already during the annealing isotherm.

Field annealing is popular with Hitperms and one can expect it to coerce the tedious HRA anisotropy. In fact the field reduces HRA somewhat at 500 °C annealing (not shown) but higher temperatures gave results which look like the “two-knee” loops in Fig. 1. The possibility to explain this would be to consider the last finished anisotropy — the magnetostrictive one — to deteriorate so the field induced one. The obvious difference between Si-free Co$_{27}$ and Si$_5$B$_{15}$ Hitperms is hardly explained this way because the expansivity change (to produce $\sigma$) and the magnetostricton are both clearly smaller for Si$_5$B$_{15}$ and still the apparent field effect is larger here and opposite to what is expected (longitudinal field should cause an upright loop). The other difference is seen in Fig. 2: Si$_5$B$_{15}$ shows far easier viscous flow than Co$_{27}$. This flow can even be kept against the hampering effect of crystallization by few MPa more (see the doubled stress $\sigma$). Moreover if heterogeneous, Si$_5$B$_{15}$ can experience the mutual force interaction also at the beginning of isotherm. Thus it seems reasonable to consider a creep-induced-like anisotropy (“flow-induced” is appealing). When the surfaces keep squeezing the plastic interior, there is little chance to restore what has flown. Although we did not tested yet such anisotropy by a devoted annealing, if the easy axis is parallel to tension as in similar Hitperms [6], the observed hard axis is expected parallel to compression and this would strongly support the explanation based on field-modified creep-induced-like anisotropy.

Cleaning of precursor ribbons prior to annealing indeed reduces the MH-induced anisotropy, so it seems that a surface contamination also adds to an initial heterogeneity. Besides the obvious magnetoelastic anisotropy which produces the HRA contribution on a positively magnetostrictive heterogeneous ribbon with surfaces squeezing most in a cool ribbon, a second contributor can act namely in ribbons capable of viscous flow. It could be a creep-induced-like anisotropy which forms at elevated temperatures.

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