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Stress level in Finemet materials studied by impedanciometry

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In this work, a study of the stress relief in Finemet ribbons, $Fe_{73.5}Cu_1Nb_3Si_{16.5}B_6$, as a function of the annealing temperature is presented. The as melt-spun samples are amorphous and become partially crystallized after annealing at appropriate temperatures. For temperatures $T_A \geqslant 480\,°C$ the samples are nanocrystalline, with a microstructure composed by α-$Fe_{1-x}Si_x$ $(x \sim 0.2)$ crystallites (10 nm average diameter) embedded in an amorphous matrix. Nanocrystallization, associated with stress relief effects, improves the soft magnetic properties of this kind of material. The stress level was quantified using magnetorestriction (measured by SAMR), magnetoelastic anisotropy, and domain wall energy data obtained from impedance spectra measurements. A reduction of the internal stress from 15 to 0.2 MPa was verified when comparing the as-cast to the sample annealed at 580 °C. Improvement of the magnetic softness of the samples was also followed by the increase of the domain wall and magnetization rotation contributions to the overall effective permeability.
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The nanocrystalline iron-based $Fe_{73.5}Cu_1Nb_3Si_{16.5}B_6$ alloys (Finemet) are well known for their excellent soft magnetic properties,[1] such as low coercivity (H_c), high effective permeability (μ), and small saturation magnetorestriction (λ_s). Produced by melt spinning, the as-cast samples are amorphous, evolving to a nanocrystalline phase under thermal treatment at proper temperatures $(T_A \sim 580\,°C)$. The absence of long-range structural order and crystalline anisotropy partially explain the soft magnetic properties of these samples in the amorphous phase. On the other hand, the nanocrystalline samples are composed of ferromagnetic α-FeSi nanocrystals embedded in an amorphous matrix of FeSiB. Grain size (typical diameter 10–15 nm) and relative composition are dependent on the annealing temperature and time. Despite the magnetocrystalline anisotropy on the FeSi nanocrystals, the grains are randomly oriented in such a way that the main contribution to the magnetic anisotropy arises from the magnetoelastic energy. Substantial stresses arise during the melt-spinning process. These stresses are nonuniform, leading to a local magnetoelastic energy. The associated average anisotropy constant can be conveniently treated as $K_u = 3\sigma\lambda_s/2$, where σ and λ_s are the average values of the local stress level and saturation magnetostriction over the considered volume. This anisotropy can be partially removed by annealing.

The main idea of this work is to quantify the internal stress relief, after thermal treatment, by measuring λ_s and determining K_u. This last parameter was measured through the domain wall energy (Σ), by using a method described in a previous work[2] based on the knowledge of μ_{DW}, the domain wall (DW) contribution to the effective permeability.

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The samples, ribbons with $70 \times 2.5 \times 0.034\ mm^3$, were annealed in vacuum at temperature (T_A) of 100, 480, and 580 °C during 1 h in order to reduce the internal stress level and to promote structural modifications discussed above. In Fig. 1, the evolution of M_s and λ_s as a function of T_A are shown. As seen from this figure, while M_s maintains roughly the as-cast value, as T_A is increased, λ_s is strongly reduced and changes its signal for the sample annealed at 580 °C. The total λ_s measured for each sample is the weighted average value between λ_s^{am} and λ_s^{cryst} of the amorphous and nanocrystalline volume fractions, respectively.[3] For these samples the amorphous phase has positive magnetostriction, while it is negative for the α-FeSi nanocrystals. Annealing induces the growth of the crystallized portion of the sample (the FeSi crystals), thus the samples having their total λ_s value modified as shown in Fig. 1.

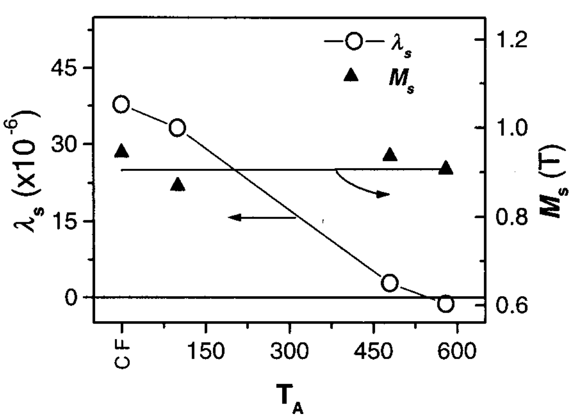

FIG. 1. Evolution of M_s and λ_s as a function of annealing temperature for the $Fe_{73.5}Cu_1Nb_3Si_{16.5}B_6$ samples.

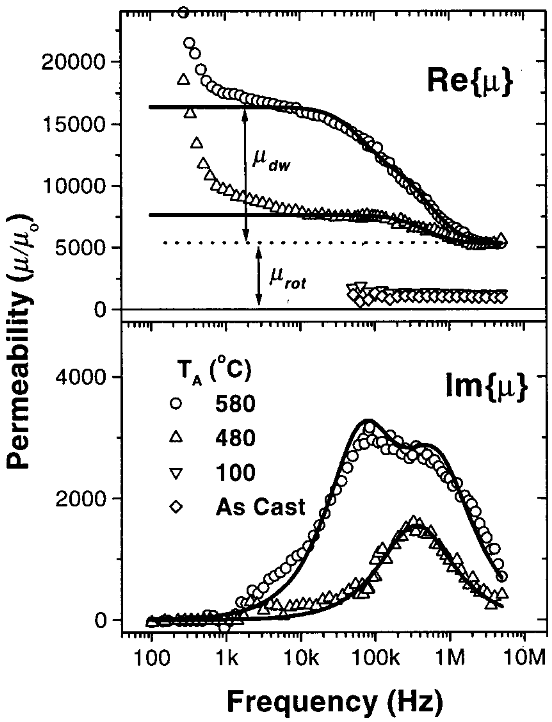

FIG. 2. Frequency spectra of the permeability for $Fe_{73.5}Cu_1Nb_3Si_{16.5}B_6$ samples as derived from impedance measurements. At the Im{μ} only curves for the annealed samples at 480 and 580 °C are presented.

The transverse permeability (μ) is also strongly affected by T_A due to its effect on both internal stress level and structural modifications. In Fig. 2, $\mu(f)$, as derived from impedance measurements[4] is presented. The solid lines in the figure are fits made with a Debye-type dispersion relation for the DW motion.[4] From this kind of procedure it is possible to separate μ_{DW} and μ_{rot}, the contributions from the DW motion and the rotation to the effective permeability, respectively.

Comparing the μ values obtained for the annealed to the as-cast samples, it can be seen that while the samples with $T_A \leqslant 100\,°\mathrm{C}$ present almost no modifications on the value of μ, $T_A \geqslant 480\,°\mathrm{C}$ have its permeability value raised to ~8000 μ_0 ($T_A = 480\,°\mathrm{C}$) and ~16 000 μ_0 ($T_A = 580\,°\mathrm{C}$). It can also be seen from Fig. 2 that both μ_{DW} and μ_{rot} have their values enhanced by the annealing process. The Im{μ} for the sample annealed at 580 °C displays two peaks in the permeability spectrum, as shown in Fig. 2. This feature is associated with the presence of two sets of domains: main stripe domains with average width of 300 μm, in a transverse orientation with respect to the sample length, and regions with secondary domains about 20 μm wide, structured in a maze pattern. This type of domain structure has been previously observed in Finemet materials, as described in Ref. 5. The other samples probably do present this kind of domain structure, but the low μ value impedes the identification of the domain families. The average domain width were obtained from the $\mu(f)$ spectra by fitting through a function that takes into account μ_{DW}, its corresponding relaxation frequency, and the sample thickness, using the domain width as the fitting parameter.[6,7]

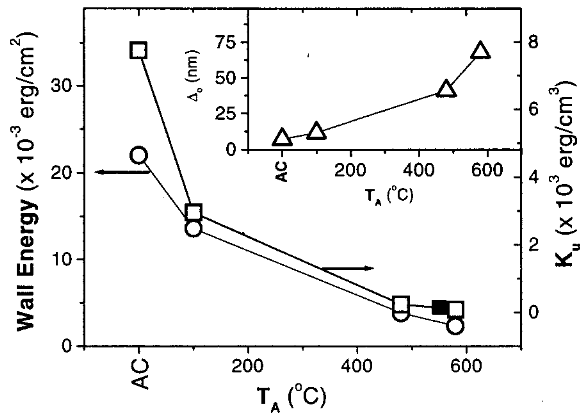

FIG. 3. Domain wall energy, uniaxial anisotropy constant (K_u), and domain wall thickness (Δ_0; inset) evolution as a function of T_A.

The DW energy limits the DW distortion when a magnetic field (H) is applied to a magnetic sample. Also, the same field dislocates the DWs proportionally to the permeability and the field, $x(t) = \mu_{\mathrm{DW}} H(t) a / M_s$, where a is the average domain width. As μ_{DW} can be easily obtained from the $\mu(f)$ spectra, it is possible to determine the critical field associated to the critical wall displacement (x_c) where it becomes unstable, meaning that adjacent domains with parallel magnetization collapse, saturating the sample.[8] By working with the impedance technique,[2] the magnetic field associated to the probe current is considered to distort the DW. Therefore Σ can be obtained from the critical current I_c which is related to Σ by $I_c = 2\Sigma w / M_s b$,[9] with w and $2b$ representing the sample's width and thickness, respectively. In Fig. 3, the evolution of Σ with the annealing temperature is presented. It is clear from this figure that Σ is strongly reduced as T_A increases. As it is well known, for a material having uniaxial anisotropy, K_u, the domain wall thickness (Δ_0) and Σ are related by $\Sigma = 4\sqrt{AK_u} = 4\Delta_0 K_u$.[10] Considering the exchange parameter $A \sim 10^{-6}$ erg/cm,[11] K_u and Δ_0 were determined as a function of annealing temperature (see Fig. 3). In order to compare the obtained anisotropy data with results from the literature, the closed square in Fig. 3 correspond to the K_u

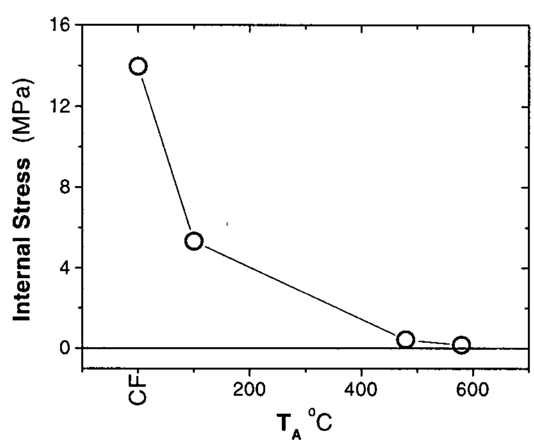

FIG. 4. Internal stress level as a function of T_A for the $Fe_{73.5}Cu_1Nb_3Si_{16.5}B_6$ samples.

determined by Yoshizawa and Yammuchi[12] for a similar sample. It can be observed from this figure that Δ_0 increases as function of T_A, accompanying the wall energy decrease.

As stated above, the main contribution to the anisotropy in these samples comes from the magnetoelastic interactions. This allows to estimate the stress relief promoted by the annealing from the uniaxial anisotropy. From Fig. 4 it can be seen that σ decreases (from 15 MPa, as-cast sample, to 0.2 MPa, $T_A = 580\,°C$) as T_A is increased. The σ values were determined using the magnetostriction of the amorphous phase and the anisotropy values shown in Fig. 3. Several of the soft magnetic properties exhibited by the annealed samples can be explained in terms of the observed stress relief.

In summary, in this work a study of the internal stress relief of Finemet samples, based on impedance measurements, is presented. Improvement of the soft magnetic properties, as verified by the permeability increase as a function of T_A, can be explained in terms of the reduction of the magnetic anisotropy promoted by the stress relief.

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