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A novel method determining longitudinally induced magnetic anisotropy in amorphous and nanocrystalline soft materials

F. Mazaleyrat,* Zs. Gercsi, and M. Lécivain

SATIE, ENS de Cachan, 61, avenue du Président Wilson 94235 Cachan cedex, France

L.K. Varga

RISSPO, Hungarian Academy of Science,

POBox 49, 1525 Budapest, Hungary

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Abstract

In ferromagnetic amorphous and nanocrystalline soft magnetic alloys the induced magnetic anisotropy plays a fundamental role in the hysteresis behavior but , due to the elongated shape, it can be measured only if K_U is perpendicular to the sample long axis. In order to measure the longitudinal induced anisotropy, an original method derive from known thin layers measurement techniques was used. Hysteresis loops shifted by perpendicular bias field were recorded for this purpose. Direct measurement of the longitudinal induced anisotropy in amorphous and nanocrystalline ribbons or wire without needing sample preparation is reported for the first time. Evidence of self-induced anisotropy is brought in a Fe-Co based nanocrystalline alloy.

*Electronic address: mazaleyrat@satie.ens-cachan.fr; URL: <http://www.satie.ens-cachan.fr>

In the early years after the discovery of Finemet, various experiments have been conducted in the aim of inducing uniaxial anisotropy (K_U) in nanocrystalline alloys. As it was expected from previous experiments with amorphous materials, uniaxial magnetic anisotropy was successfully induced in the direction of the field applied during nanocrystallization [1]. Herzer has shown that this phenomenon is related to the Fe-Si pair ordering during the crystallization stage [2]. The possibility of inducing anisotropy in Finemet by stress annealing was first attested by Glaser [3] and was proved to be transversal and strikingly strong. This last method can yield both transversal (positive) or longitudinal (negative) depending on magnetic and mechanical properties of both phases [4].

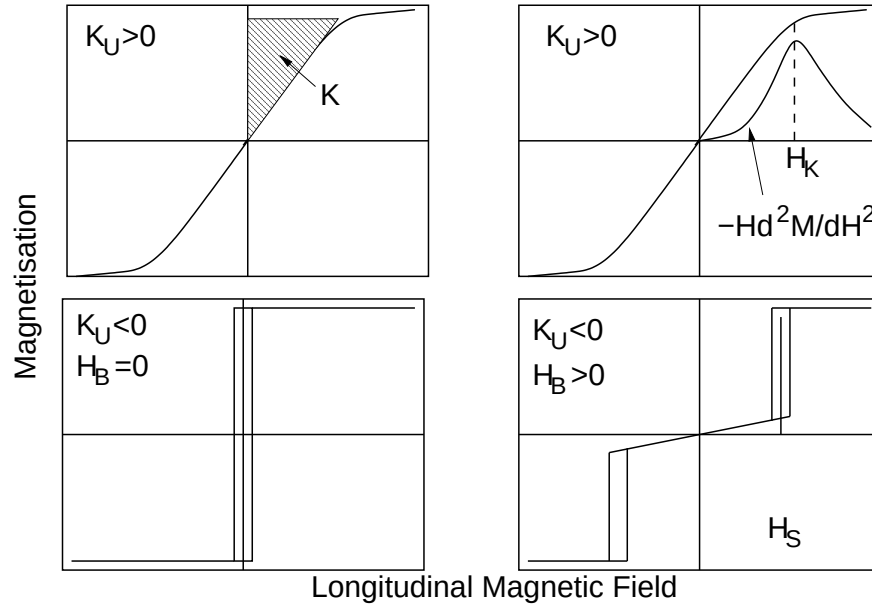


Figure 1: Various methods to determine uniaxial induced anisotropy

The transversal induced anisotropy can be evaluated by two different techniques. If the hysteresis loop is linear up to saturation, the magnetization occurs by rotation only and K_U can be easily deduced from the area on the left of the loop as depicted in Fig. 1 (top left box). However a more general method consisting in the computation of the second derivative of the return branch of the hysteresis loop was proposed in [5] and multiplying by the field yields the distribution of anisotropy fields [6] (see Fig. 1 top right box). The same result can be obtained in a more accurate way by a second harmonic detection induced by a small ac field superimposed to the dc field [7]. In the case of longitudinal induced anisotropy, the square hysteresis loop is clearly unsuitable for these evaluations because the magnetization reversal

Table I: Anisotropy fields measured by the present technique (except for $K_U > 0$)

Material	Sample	Annealing	$H_k(\text{A/m})$	$\mu_0 M_s(\text{T})$	$K_u(\text{J/m}^3)$	$H_c(\text{A/m})$
$\text{Fe}_{77.5}\text{B}_{15}\text{Si}_{7.5}$	A	As-cast	-44 ± 3	1.57	-35 ± 2	19
$\text{Fe}_{73.5}\text{Si}_{13.5}\text{B}_9\text{Nb}_3\text{Cu}_1$	F _L	Long. H-550°C/1 h	-66 ± 5	1.24	-41 ± 3	5.5
$(\text{Fe}_{90}\text{Co}_{10})_{73.5}\text{Si}_{13.5}\text{B}_9\text{Nb}_3\text{Cu}_1$	C	550°C/1 h	-68 ± 3	1.16	-40 ± 2	8.4
$(\text{Fe}_{90}\text{Co}_{10})_{73.5}\text{Si}_{13.5}\text{B}_9\text{Nb}_3\text{Cu}_1$	C _L	Long. H-550°C/1 h	-158 ± 2	1.16	-92 ± 1	8
$(\text{Fe}_{90}\text{Co}_{10})_{73.5}\text{Si}_{13.5}\text{B}_9\text{Nb}_3\text{Cu}_1$	C _T	Trans. H-550°C/1 h	$+86 \pm 3$	1.16	$+50 \pm 2$	5.8
$\text{Fe}_{90}\text{Zr}_7\text{B}_3$	N _S	Stress-600°C/1 h	-154 ± 6	1.63	-126 ± 5	36.5

takes place by a single large Barkhausen jump at $\pm H_C$. The coercive force in this case corresponds to the domain wall nucleation energy which is indirectly linked to K_U through the domain wall energy. However many other parameters are involved and finally K_U cannot be deduced from H_C . The torque method as well as bi-axial magnetic measurement can be applied but both requires circular samples which are difficult to cut from brittle ribbons. In addition, these methods are very sensitive to the shape of the sample. For this purpose, a method originally proposed by Weber et al.[8] have been adapted to long samples without needing sample preparation. The sweeping field (H) is applied along the easy axis which is longitudinal in this case. The hysteresis loop is obtained by integration of the pick-up coil voltage within the same axis. If a transversal bias field H_B larger than the anisotropy field H_K is applied, the hysteresis loop is flattened and practically anhysteretic until a critical value of H is reached at which the magnetization switch is shifted at $H_S \pm H_C$ (see Fig. 1).

The total magnetic energy depends on the angle φ between the magnetization and the measurement direction for a given angle θ between the easy axis and the measurement direction. When two perpendicular fields are applied and the easy axis is parallel to the measurement direction ($\theta=0$), we have:

$$E(\varphi) = K_U \sin^2 \varphi - \mu_0 M_S H \cos \varphi - \mu_0 M_S H_{\perp} \sin \varphi \quad (1)$$

In this case, there is 3 stable positions of the magnetization $\varphi = 0$ and $\varphi = \pm \pi/2$ and the magnetization switches from one position to another (at $H = H_S$) when :

$$E(0) = -\mu_0 M_S H = E(\pi/2) = K_U - \mu_0 M_S H_{\perp}$$

For a constant $H_{\perp}$, $E(0) = E(\pi/2)$ so the anisotropy constant is: $K_U = \mu_0 M_S (H_S - H_{\perp})$ and the anisotropy field is:

$$H_K = 2(H_S - H_{\perp}) \quad (2)$$

For circular samples with in-plane applied fields, K_U is deduced from a single split hysteresis loop because the demagnetizing factors are equal in both direction ($N_{\parallel} = N_{\perp}$), so H_K comes from eq. (2). In the case of long samples the longitudinal field is the applied one because of the negligible demagnetizing effect, but the perpendicular inner field is much smaller than the applied biasing field: $H_{\perp} = H_B - N_{\perp} M_{\perp}$. The magnetization can be substituted if the field is smaller or equal to the switching field: as there is no domain, permeability is basically due to a rotational magnetization process and thus depends linearly on the field: $M_{\perp} = \chi H_B$, so

$$H_K = 2[H_S - (1 - N_{\perp}\chi) H_B] = 2(H_S - \alpha H_B) \quad (3)$$

Of course the parameter α is highly dependent on the the demagnetizing factor which cannot be determined for most of samples with sufficient accuracy. This difficulty is evaded by using a plotting strategy: several hysteresis loops are recorded with various values of the biasing field. The linear plot of H_S as a function of H_B allows the determination of H_K by extrapolation at $H_B = 0 \Rightarrow H_K = 2H_S(0)$.

The experimental set-up, is composed of a 0.52 m long solenoid generating the longitudinal sweeping field in the homogeneous part of which the 7 cm long pick up coil and the sample are positioned and a perpendicular 0.3 m in diameter Helmholtz coils set. In our case this coils set is about 35 kg and H_B (20 kAm^{-1}) is sufficient only for the amorphous wire sample. Another set-up can pass through the deficient field problem, using an arrangement composed of two perpendicular iron cores. This device was already used for magnetostriction measurements by small angle magnetization rotation method [9]. The longitudinal field is known from Ampère's theorem, because $N_{\parallel}$ is strictly zero. In the other hand, the perpendicular field depends strongly on the position of the ribbon in the gap. This field is in a given position is $H_{\perp} = \sigma n I_B / d$, where σ is Hopkinson's leakage coefficient, if the yoke is far from saturation. Equation 3 becomes $H_K = 2(H_S - \alpha' I_B)$, where α' is eliminated by the plotting strategy.

Different type of nanocrystalline alloys were produced and annealed with or without longitudinal (800 A.m^{-1}) and/or transverse (10 kA.m^{-1}) fields or under a 100 MPa stress

according to Table I. A first important point is to verify the independence of H_S upon the maximum sweeping field H_{max} in particular is the sample is not fully saturated. For this purpose, several loops were recorded with a constant H_B and varying H_{max} . H_S was proved to be independent of H_{max} if the latter is sufficient to occur the switching of the magnetization. The relevance of this is illustrated in Fig. 2 where it is visible that the samples are not saturated for the largest bias fields. From these split loops, the values of H_S are extracted and plotted as a function of H_B in Fig. 3. The perfect alignment of points allows an accurate extrapolation to zero which give the values of H_K by a least means square linear fit with a remarkably low standard deviation. Table I summarizes the H_K and K_u values of selected materials.

The anisotropy associated to the single longitudinal core domain of amorphous $\text{Fe}_{77.5}\text{B}_{15}\text{Si}_{7.5}$ was measured with the present method. The result is a quite low value compared to the perpendicular anisotropy associated with the shell suggesting a quite small quenched-in tensile stress in the core (~ 2 MPa). The stress annealed Nanoperm alloy (N_S) exhibits a -150 J/m^3 anisotropy only which is very low compared to Finemet ($\sim +1000 \text{ J/m}^3$) annealed in the same conditions. This result suggest a weaker back-stress effect and open new investigation fields on stress induce anisotropy. Concerning the field annealed materials, the F_L sample exhibits a slightly higher value of K_U compared to transversal field annealing ($10\text{-}20 \text{ J/m}^3$) [10], which can be explained by a shape contribution. In contrast, relatively large K_U were induced in the Co doped alloy (C_L & C_T) as previously reported [11]. The fact that, in the longitudinal (C_L) and transversal (C_T) field annealed samples have not the same value of K_U (by opposite sign) may be explained partly by shape effect but also by the insufficient inner transversal field during annealing of C_T sample due to the demagnetizing factor ($N_{\perp} \gg N_{\parallel}$). A surprising effect is the splitting of the hysteresis loop of Co-doped sample annealed without field showing a notable long range uniaxial anisotropy comparable to that of F_L sample. This “self-induced” anisotropy is of great importance in the theory of random anisotropy model proposed by Suzuki et al. [12]. Indeed the change from the classical D^6 dependence of the coercive field to a D^3 law for some alloys (e.g. Nanoperm) was explained by the introduction of a long range K_U in the model which existence was never experimentally demonstrated. The origin of this self-induced anisotropy is not clear at all but two explanations are possible. A first one would involve the magneto-elastic origin linked to the different thermal dilatation coefficients of both amorphous and crystalline

phases but it should be a priori a short range contribution subject to averaging out. Another explanations should be the formation of a domain structure during annealing yielding high internal field and finally a long range field induced magnetic anisotropy [13]. It is worth noticing that this effect is not observed with Finemet which is not contradictory. Indeed, the Fe-Si crystallites are in the superparamagnetic phase during annealing [14] whereas it is probably not the case for Co-doped alloy due to the high Curie point of Fe-Co-Si phase [15].

The coercive fields are also reported in Table I. It is to notice that there are no clear relationship between the magnitude of the anisotropy fields and the coercivity of different materials. For instance, the results obtained with Fe-Co based nanocrystalline alloys suggest that the coercivity is basically linked to the magnetization process (rotation or domain wall displacement) and weakly to the magnitude of induced anisotropy.

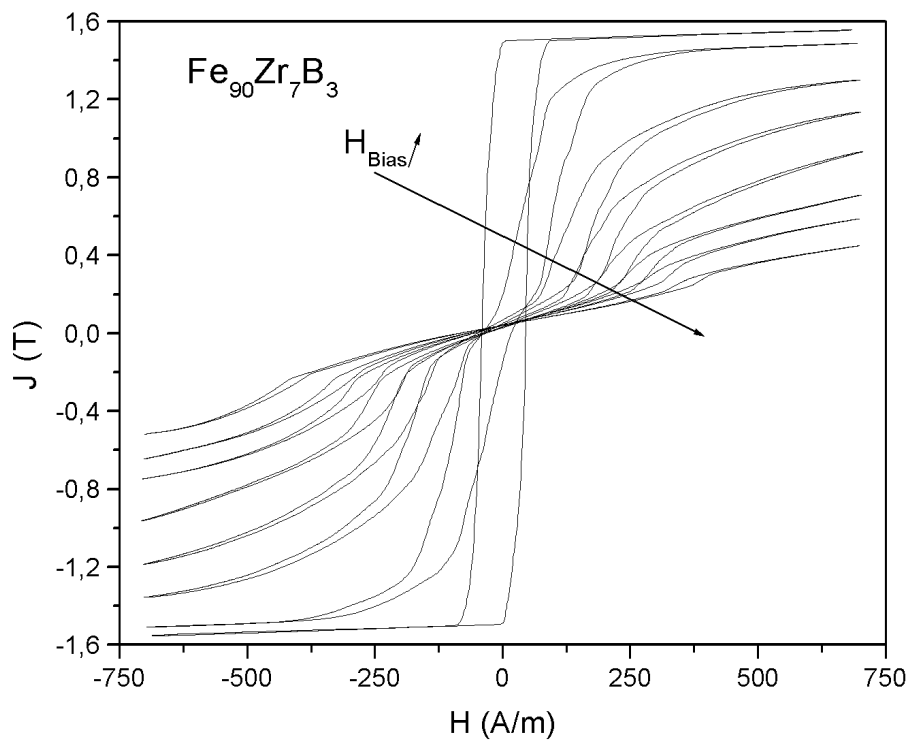


Figure 2: The shape of the hysteresis cycle as a function of increasing bias field

In conclusion, the approach of Weber [8] have been adapted to the determination of uniaxial magnetic anisotropy in anisotropic samples: the plot of $H_S = f(H_B)$ overcome the demagnetizing effect. This method allows for the first time to measure directly the longitudinal induced anisotropy in amorphous or nanocrystalline ribbons and wires. To the

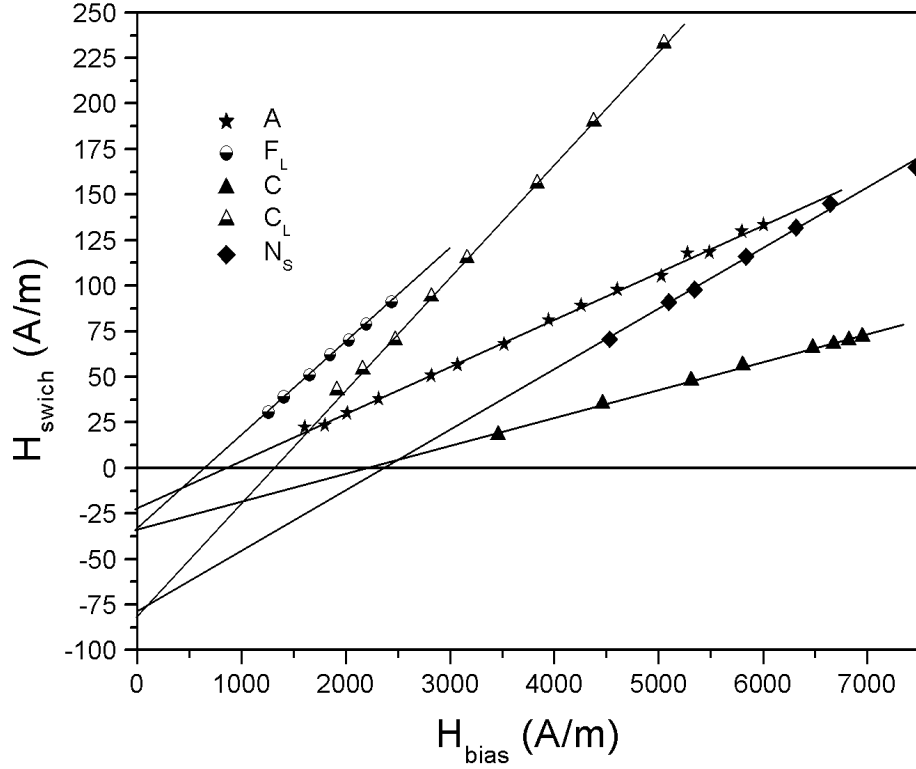


Figure 3: The switching fields plotted as a function of bias field. Extrapolation to $H_B=0$ yields $H_K/2$.

authors' knowledge, evidence of self-induced anisotropy in nanocrystalline samples is also reported for the first time.

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