



Improving soft magnetic properties in FINEMET-like alloys. A study



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ABSTRACT

An approach to improve the soft magnetic properties of nanocomposite FINEMET-like alloys is presented by the study of the theoretical behavior of the coercive field with the crystallized fraction, $H_c(v_{cr})$, curves where the total anisotropy is taken into account. These curves can exhibit a minimum value that should be used for a proper design of soft magnetic alloys, consisting in matching that value with the optimal crystallized fraction of the alloy. For this, the effect of Si, Ge, Co and Al on the magnetic properties on the material phases and on its microstructure was analyzed while a good relationship between theoretical results and data in the literature was found. Small amounts of Ge and/or Al can improve the magnetic properties of FINEMET while the addition of Co deteriorates the coercive field as is predicted by the theory. Simultaneous addition of two or more solute elements to the α -Fe crystals was also evaluated and discussed. Results indicate the importance of knowing the effect of the alloying element on the crystalline magnetostriction constant and on the crystallized fraction of the microstructure.

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1. Introduction

Since its development, soft magnetic nanocrystalline materials have attracted great attention and much effort has been made by researchers in order to improve their soft properties, for example, with the addition of elements that increase the saturation magnetization and permeability, and reduce the coercive field and the magnetic losses. A small grain size, of the order of tens of nanometers, is the key to their good properties and, often, the addition of these elements was conducted for this purpose neglecting other important features. In a previous work [1], we have discussed the parameters to design nanocrystalline soft magnetic materials such as FINEMET, NANOPERM, HITPERM and NANOMAT types and the effect of some alloy elements on them. In order to do this, we have modeled the theoretical behavior of the coercive force, H_c , with the crystalline fraction, v_{cr} , of the nanocomposite materials taking into account some material parameters, like the grain diameter, D , the magnetocrystalline constant, K_1 , the crystalline and amorphous magnetostriction constants, λ_{am} and λ_{cr} , respectively, the internal stresses, σ_i , and v_{cr} , which act in magnetocrystalline and uniaxial anisotropies according to models in literature. In the case of FINEMET™ alloys (typical composition $Fe_{73.5}Si_{13.5}B_9Cu_1Nb_3$), they present a clear minimum in the $H_c(v_{cr})$ curve that can be exploited to soften the material. In this work, we go deeper into previous research focusing our attention on FINEMET-like nanocrystalline systems, analyzing the origin of this minimum, studying the

magnetic effect of some solutes on the crystalline phase (compiling several charts from literature) and the behavior of the magnetostriction constant of the amorphous matrix with the crystallized fraction, which improving the results obtained in [1]. The theoretical results are first compared with a whole series of a $Fe_{73.5}Cu_1Nb_3Si_{22.5-x}B_x$ alloys from literature validating the model, and the effect of Ge, Al, Co and Ge + Al on the α -Fe(Si) phases are discussed. The results give new tools for a proper soft magnetic nanocrystalline alloy design.

2. Material and methods

Our studies are based on an average anisotropy model that takes into account the random anisotropy model for the magnetocrystalline anisotropy [2] and the uniaxial anisotropies such as magnetoelastic and field-induced ones. A greater understanding of the subject can be found in [2,3] and a brief summary in [1]. Here, we write the equations used in the present work and the corresponding constants without going into details. It is known that the coercive field, H_c , depends on the total anisotropy, $\langle K \rangle$, and the magnetic polarization, J_s , as:

$$H_c = p_c \cdot \langle K \rangle / J_s \quad (1)$$

with p_c equal to ~ 0.2 for our materials. Before defining $\langle K \rangle$, we must study the anisotropies acting in our materials. The most significant are the magnetocrystalline, K_1 , and magnetoelastic, K_{Me} , ones, as field induced anisotropies are negligible or not present in the current studied material. In the random anisotropy model, K_1 is average as:

$$\langle K_1 \rangle = \beta \cdot v_{cr} \cdot K_1 \cdot (D/L_{ex})^{3/2} \quad (2)$$

where β is a constant estimated in ~ 0.5 and L_{ex} the correlation length of the ferromagnetic exchange to be defined later. With respect to the magnetoelastic anisotropy, we have that:

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$$Ku_{el} = 3/2 |\lambda_s| \cdot \sigma_i \quad (3)$$

where σ_i is the internal (mechanical) stresses and λ_s is the effective magnetostriction constant of the nanocomposite material that can be expressed as:

$$\lambda_s = v_{cr} \cdot \lambda_{cr} + (1 - v_{cr}) \cdot \lambda_{am} \quad (4)$$

being λ_{am} and λ_{cr} the magnetostriction constants of the amorphous and nanocrystalline phases, respectively, and v_{cr} the crystallized fraction.

The total anisotropy, $\langle K \rangle$, can be calculated as the average:

$$\langle K \rangle = \sqrt{\beta^2 \langle K_1 \rangle^2 + Ku_{el}^2} \quad (5)$$

The correlation length is then defined as $L_{ex} = \varphi (\beta \cdot A_{cr} / \langle K \rangle)^{1/2}$ with the constant $\varphi = 1.5$ and the exchange stiffness, A_{cr} , set in $0.6 \cdot 10^{-11}$ J/m, as it was reported in experimental results [2]. Eq. (5) becomes self-consistent and it can be solved by iteration. Introducing Eq. (5) in Eq. (1) we can obtain the behavior of H_c with v_{cr} , $H_c(v_{cr})$.

3. Calculation

3.1. Nanocrystalline phase considerations

FINEMET-like nanocomposite alloys are formed by α -Fe(Si, Si + M) solid solution nanocrystals, with M equal to one or more elements as Al, Ge, Co, among others, surrounded by a Fe-rich amorphous matrix. The total solute, Si + M, content of the nanograins range approximately from 15 to 22 at.%. The effects of the mentioned solute elements on the magnetic properties on Fe crystals are shown in Fig. 1. The elements Si, Al and Ge reduce J_s of the

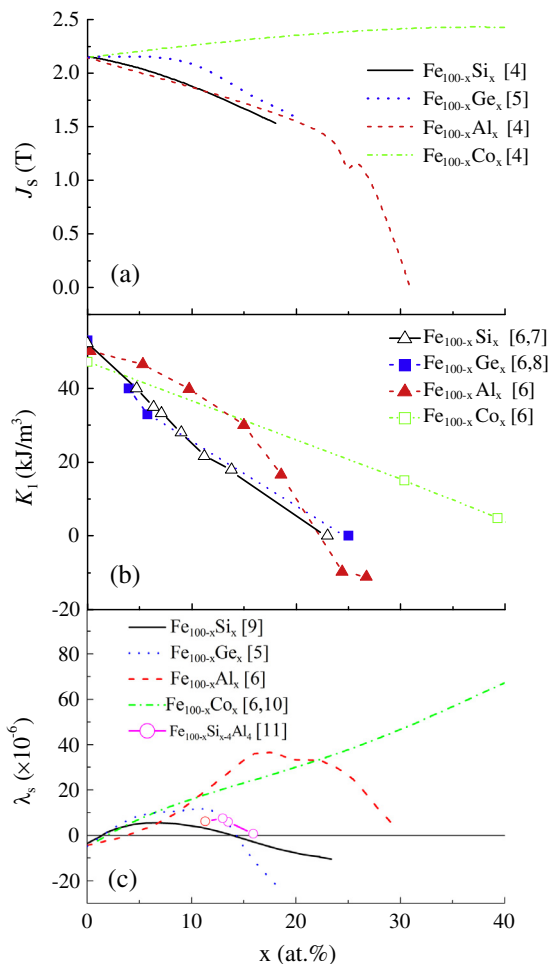


Fig. 1. Effect of Si, Ge, Al and Co solute elements on some magnetic properties of the α -Fe phase. Dependence on solute content of: (a) J_s , (b) K_1 and (c) magnetostriction constant. Data obtained from references indicated in graph legends. (See above-mentioned references for further information.)

Fe crystals in approximately the same way up to $\sim 20\%$ of solute content, while Co increases it and it has a maximum at $\sim 40\%$ of Co content (Fig. 1a). Also, all of them provoke a decrease in K_1 constant of the Fe crystals (Fig. 1b). This fact is important because, between certain limits, the soft magnetic properties of the nanocomposite material become more and more independent on grain size as $\langle K_1 \rangle$ is reduced (see Eq. (2)). Finally, Fig. 1c shows the way these solutes change the magnetostriction constant, λ_{cr} , of the Fe crystals, which affects the magnetoelastic anisotropy Ku_{el} (Eq. (6)). When $\langle K_1 \rangle$ is reduced (either by reducing D or K_1 of nanocrystals) Ku_{el} governs the total anisotropy and it can be reduced via the magnetostriction balance of both phases; as the amorphous matrix is a Fe-rich phase, it has a positive λ_{am} value and, in order to be balanced, we must have a negative λ_{cr} . In our examples of Fig. 1c, this condition is satisfied by a free-solute Fe crystal (not a FINEMET alloy) or by Fe crystal containing more than $\sim 15\%$ of Si or Ge. Alloying Fe with Al or Co will not satisfy this condition.

The effect of the internal stresses, σ_i , has also to be computed in the value of Ku_{el} since a total balance of the effective magnetostriction constant, λ_s , is not always possible, as we will see later on. Ribbons obtained in the as-quenched state have a high stress level reported in 30–15 MPa [12,13] that are reduced with subsequent heat treatment until the first stages of nanocrystallization (low v_{cr}), where there is an increase in σ_i due to the grain nucleation and a relatively low annealing temperature. At optimum annealing temperature with a relatively high v_{cr} , stresses can be released up to 2.5–0.2 MPa. A further increase in v_{cr} may result in a new increase in σ_i either due to a growth in the lattice mismatch in grain boundaries and/or to higher differences in densities between the crystalline and amorphous phases.

In addition, the v_{cr} plays a critical role in soft magnetic properties through the balance of magnetostriction constants of the nanocomposite material. The v_{cr} can be controlled in several ways, for example, it increases by introducing solutes to the α -Fe crystal, by replacing one solute element for another (like Si by Ge or Al) or by reducing B content.

3.2. Amorphous phase considerations

With respect to the saturation magnetization and the magnetostriction constant of the remnant amorphous matrix, for our calculus we can consider, on the one hand, that the saturation magnetization increases directly with its iron content (for

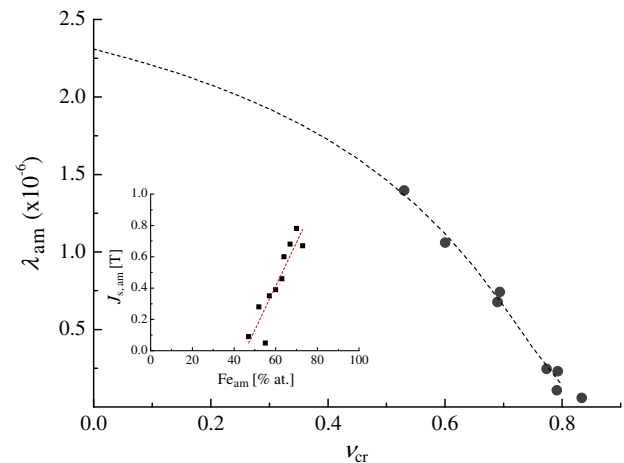


Fig. 2. Evolution of the magnetostriction constant, λ_{am} , of the amorphous remnant phase with the crystallized fraction, v_{cr} , obtained from Eq. (7). Inset, behavior of the saturation magnetization, J_s , of the amorphous phase of similar composition of the remnant matrix with its Fe content [14].

example, data of J_s vs. %Fe in the matrix obtained from [14] is plotted on inset of Fig. 2). On the other hand, the iron content in the matrix responds to the balance equation:

$$Fe_{am} = (Fe_{tot} - Fe_{cr} \cdot v_{cr}) / (1 - v_{cr}) \quad (6)$$

where Fe_{am} (Fe_{cr}) is the iron content of the amorphous (crystalline) phase and Fe_{tot} is the total iron content of the alloy. The magnetostriction constant of Fe-rich amorphous metal was found to vary with the square of its magnetization saturation with the following expression [15]:

$$\lambda_{am} = \sigma^2 \cdot 1.23726 \cdot 10^{-9} \quad (7)$$

where σ is the specific magnetization (in emu/g). Therefore, λ_{am} will decrease with v_{cr} since the matrix is depleted on iron as the crystalline fraction increases. Taking into account Eqs. (6) and (7) we found the following expression for λ_{am} :

$$\lambda_{am} = A + B((Fe_{tot} - Fe_{cr} \cdot v_{cr}) / (1 - v_{cr}))^2 \quad (8)$$

where A and B are two constants. In Fig. 2, we fitted experimental data of λ_{am} vs. v_{cr} using the J_s data of the remnant amorphous phase (converted to λ_{am} with Eq. (7)) given by Herzer in [16] for some FINEMET alloys type $Fe_{73.5}Cu_1Nb_3Si_{22.5-x}B_x$ ($x = 5-15$); we obtained the values of $9.12 \cdot 10^{-7}$, $4.356 \cdot 10^{-9}$, 71.4 at.% and 86.6 at.% for A , B , Fe_{tot} and Fe_{cr} respectively. These two last values are in good agreement with the at.% content of these alloys. Moreover, Eq. (8) predicts a $\lambda_{am} = 2.3 \cdot 10^{-5}$ at $v_{cr} = 0$, almost the reported value for the FINEMET alloy in amorphous state [17].

3.3. Magnetic coercivity and anisotropy curves

For a given FINEMET alloy, the chemical composition of nanograins, its grain diameter and the internal stresses change as the heat treatment progress until the completion of the crystallization of α -Fe(Si) nanocrystals (i.e., primary crystallization). For validating the model, is enough to fix all these parameter to their expected value for this stage of primary crystallization. For studying intermediate stages, the corresponding value must be set. Effects of these changes of chemical composition in the amorphous matrix are already contemplated with Eq. (8). Therefore, in Fig. 3, we reported the $H_c(v_{cr})$ theoretical behavior for some nanocomposite alloys consisting on α -Fe(Si) nanocrystals with Si content from 0 to 24 at.%. For each nanocrystal chemical composition, it was taken into account its K_1 and λ_{cr} values (Fig. 1), and for each v_{cr} , the corresponding λ_{am} value from Eq. (8). Also D and σ_i were set arbitrary at 13 nm and 1 MPa respectively. The first curve in Fig. 3a, when %Si = 0, is not for a FINEMET-like alloy, as the solute contain is null (indeed, the curve could correspond to a NANO-PERM-like alloy) and neither the following up to %Si = 14, but they were plotted to a better comprehension of these questions. The first theoretical curve of a FINEMET in our example is the one with %Si = 16. From then on, we can see that the curves present a minimum value in H_c , pronounced more rapidly with the silicon content. As we can see, all minima take place in the range from $v_{cr} = 0.55-0.63$.

In Fig. 4, we plotted separately the two components $\beta\langle K_1 \rangle$ and Ku_{el} , of the total anisotropy, $\langle K \rangle$, of Eq. (5), to understand the origin of this minimum. Depending on crystal composition, it may result from the balance of magnetocrystalline and magnetoelastic anisotropies (Fig. 4a), or only from the balance of negative and positive magnetostriction of the crystalline and amorphous matrix, respectively (Fig. 4b). The latter occurs when the contribution of the magnetocrystalline anisotropy becomes negligible. Bearing this in mind, we can decide which parameters to act on to improve the soft magnetic properties.

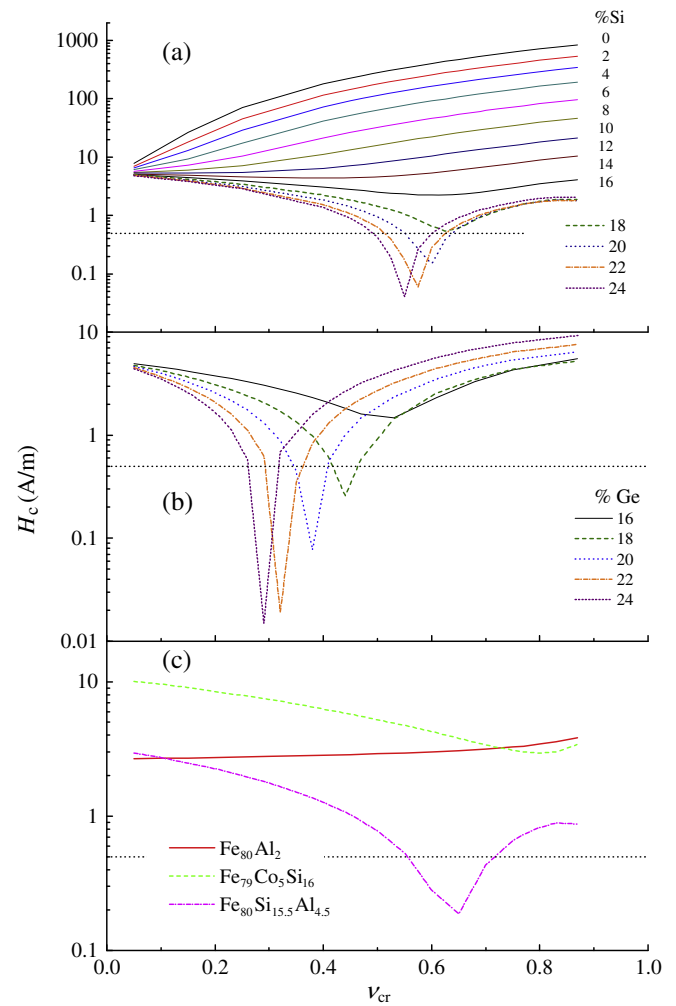


Fig. 3. Theoretical behavior of the H_c vs. v_{cr} for: (a) Fe-Si-B-Nb-B system, (b) Fe-Ge-Cu-Nb-B system, and (c) for Fe-Al-Cu-Nb-B, Fe-Si-Co-Cu-Nb-B and Fe-Si-Al-Cu-Nb-B systems.

4. Comparison of model predictions to published data

Results of these studies in the Fe-Si-B-Cu-Nb system were compared with some experimental data in [16] that reports the values of H_c, v_{cr}, D and %Si on α -Fe nanocrystals, which is necessary data for our calculation. In Fig. 5, H_c experimental data was plotted versus its respectively v_{cr} values, and it was found that they are well contained within two theoretical $H_c(v_{cr})$ curves with σ_i between 1 and 0.2 MPa. These curves were calculated using the corresponding data of %Si on nanocrystals, D and v_{cr} for each sample and limiting the internal stresses to the above indicated values. As it can be seen, curve in Fig. 5 resembles the theoretical ones in Fig. 3, were %Si on nanocrystals, D and σ_i were kept constants as v_{cr} increases. This indicates a good agreement between the model prediction and the experimental data.

The addition of Ge in FINEMET-like alloy was the subject of several works (see for example [18–20]). Although its effect in the magnetic properties of the α -Fe crystals are similar to the ones of Si (Fig. 1), only FINEMET alloys with minor addition of Ge can achieve soft magnetic properties comparable or better than FINEMET alloy [21]. In order to understand this, we studied the $H_c(v_{cr})$ theoretical curves from a Fe-Ge-B-Cu-Nb nanocrystalline system ($D = 13$ nm and $\sigma = 1$ MPa) shown in Fig. 3b. In these cases, the minima are placed at lower v_{cr} values with respect to the Fe-Si-B-Cu-Nb system (i.e., from $v_{cr} = 0.29$ to 0.52) since α -Fe(Ge) phase

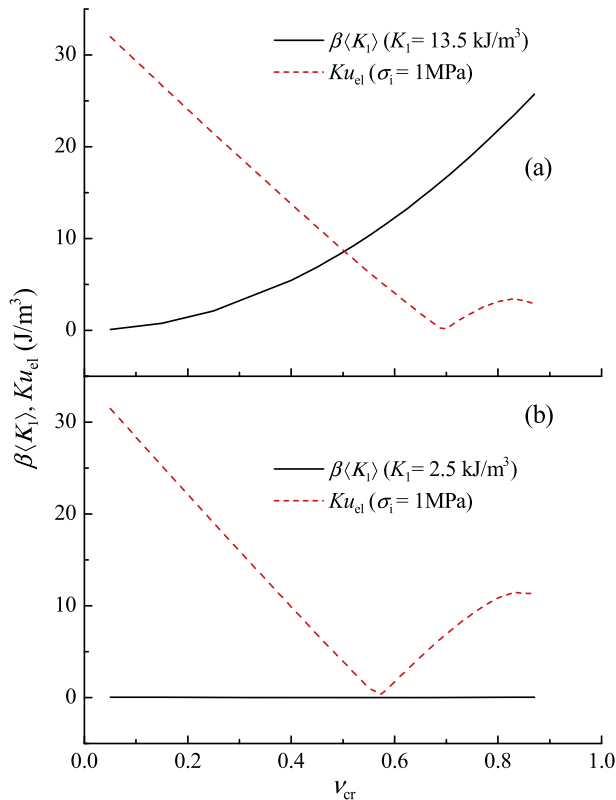


Fig. 4. Total anisotropy components $\beta\langle K_1 \rangle$ and Ku_{el} , as function of v_{cr} for (a) $K_1 = 13.5 \text{ kJ/m}^3$ and (b) $K_1 = 2.5 \text{ kJ/m}^3$.

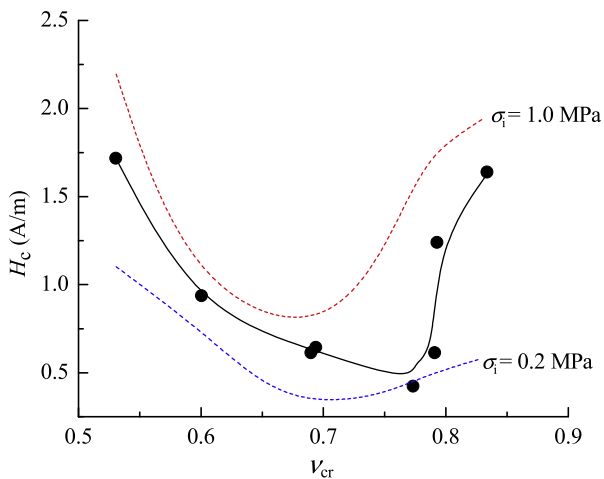


Fig. 5. H_c experimental data vs. v_{cr} and theoretical prediction curves for σ_i 1 and 0.2 MPa. The studied samples were for system $\text{Fe}_{73.5}\text{Cu}_1\text{Nb}_3\text{Si}_{22.5-x}\text{B}_x$ with $x = 14, 12, 10, 9, 8, 7, 6, 5$ and $v_{cr} = 0.53, 0.60, 0.68, 0.68, 0.77, 0.79, 0.79, 0.83$ respectively.

has a lower λ_{cr} constant than the $\alpha\text{-Fe}(\text{Si})$ one for solute content $>15\%$. However, the effect on the microstructure of adding Ge to the FINEMET system is to increase the v_{cr} with respect to Si addition [22], and therefore, the v_{cr} moves away from the $H_c(v_{cr})$ minimum; this is the reason why the Fe–Ge–B–Cu–Nb system does not improve the soft magnetic properties of FINEMET. This result can be compared with data in [19] where two alloys of the Fe–Ge–B–Cu–Nb system were investigated with $v_{cr} = 0.75$ and 0.83 , respectively, and $\sim 18\%$ Ge of content in the $\alpha\text{-Fe}(\text{Ge})$ nanograins having their optimum H_c values in 7 and 12 A/m, respectively (for a better fit, $\sigma = 2 \text{ MPa}$ can be proposed).

5. Discussion

The previous analysis of the addition of Si or Ge to the $\alpha\text{-Fe}$ nanocrystals indicates that the main problem to reach the soft magnetic properties of the nanocrystalline alloy is the mismatch between the minimum of the $H_c(v_{cr})$ curve and the optimal value of the crystallized fraction of the annealed alloy, being the former in lower v_{cr} values than the later. To reach an agreement between these two parameters, one can control the optimum crystalline fraction (for example, decreasing the Fe content and/or increasing B content) or move the minimum of the $H_c(v_{cr})$ towards higher v_{cr} values by increasing λ_{cr} (with the addition of solutes in the $\alpha\text{-Fe}$ nanocrystals).

For example, when Co or Al are added to the $\alpha\text{-Fe}$ crystal, both reduce the K_1 , but the crystalline phase has a positive λ_{cr} value, which is the same sign as the matrix one and, consequently, $\lambda_s = 0$ is never reached. This results in an increase in the H_c value with solute content like in the cases of HITPERM (Co solute). In Fig. 3c, we plotted three examples of $H_c(v_{cr})$ curves for alloys containing nanocrystals of $\alpha\text{-Fe}$ with Al, Si + Co, and Si + Al as solutes. In the first case, we calculate the curve for the $\text{Fe}_{78}\text{Al}_4\text{Nb}_5\text{B}_{12}\text{Cu}_1$ alloy of Ref. [23] considering the chemical composition of the crystalline phase $\text{Fe}_{80}\text{Al}_{20}$ as can be inferred from its lattice parameter. The curve has no minimum and the H_c increases with v_{cr} , indicating that it is not possible to optimize this alloy with a compensation of λ_s . The minimum reached for this alloy in literature is $H_c \sim 2.5 \text{ A/m}$. The second case refers to the $\text{Fe}_{68.5}\text{Co}_5\text{Si}_{13.5}\text{B}_9\text{CuNb}_3$ alloy of Ref. [24] (for calculating the curve, we estimated a positive $\lambda_{cr} = 6 \cdot 10^{-6}$ for the bcc $\text{Fe}_{79}\text{Co}_5\text{Si}_{16}$ phase and, $\sigma_i = 2 \text{ MPa}$ for the $v_{cr} = 0.80$ of this alloy). As expected, the simultaneous additions of Si + Co give rise to intermediate H_c values between that of FINEMET and HITPERM. The curve presents a minimum at $v_{cr} \sim 0.80$, which coincides with the actual v_{cr} value given in literature. According to our results, this alloy is already optimized with a $H_c = 3 \text{ A/m}$. In the third case, we simultaneously added Si + Al and the effects were similar to that of adding Si + Co: a reduction in K_1 and an increase in the λ_{cr} value with respect to the same solute content of pure Si. In Fig. 3c, the calculated $H_c(v_{cr})$ curve for a $\text{Fe}_{73.5}\text{Si}_{11.5}\text{Al}_2\text{B}_9\text{Nb}_3\text{Cu}_1$ nanocrystalline alloy of Refs. [1,25] is presented. In this case, we estimated a negative $\lambda_{cr} = -5.56 \cdot 10^{-6}$ extrapolated from data in [11] (see the curve $\text{Fe}_{100-x}\text{Si}_x\text{Al}_4$ in Fig. 1c) for nanocrystals composition $\text{Fe}_{80}\text{Si}_{15.5}\text{Al}_{4.5}$. The minimum of $H_c(v_{cr})$ curve is shifted towards higher values of v_{cr} . On the microstructure, the effect of adding Al to a FINEMET composition results in an increase of the v_{cr} ; in this case, the addition of 2% Al shifts the v_{cr} values in ~ 0.05 ($v_{cr} = 0.68$), with respect to the FINEMET with $\text{Fe}_{80}\text{Si}_{20}$ nanocrystals composition. [1]. Both effects, the increase in the λ_{cr} and in the v_{cr} , would give rise to an improvement on the magnetic properties, as results in [25,26].

Finally, in [21], we have obtained soft magnetic properties on some Fe–Si–Al–Ge–Nb–B–Cu nanocrystalline alloys indicating that with the simultaneous addition of Si + Al + Ge on $\alpha\text{-Fe}$ crystals it is also possible to achieve a good correspondence between the minimum of the theoretical $H_c(v_{cr})$ curve and its crystallization fraction of alloys.

6. Conclusions

The effect of Si, Ge, Al, and Co solute addition on soft magnetic properties of FINEMET-like alloys was explained through the study of the $H_c(v_{cr})$ theoretical curves. An expression of the behavior of λ_{am} with v_{cr} was found in order to consider its effect on Ku_{el} with the evolution of the crystalline fraction of the nanocomposite material. $H_c(v_{cr})$ experimental data on Fe–Si–Cu–Nb–B and on Fe–Ge–Cu–Nb–B systems and alloys containing $\alpha\text{-Fe}(\text{Al})$,

α -Fe(Si + Al) and α -Fe(Si + Co) nanocrystals were compared with the theoretical prediction resulting in a good agreement. The softest magnetic properties are given when a good coincidence between the minimum of the $H_c(v_{cr})$ curve and the optimal crystalline fraction is done. This can be achieved either by adjusting the crystallized fraction, v_{cr} , or increasing the nanocrystals magnetostriction, λ_{cr} , by proper selection of adding elements. The simultaneous addition of various solute elements in the α -Fe crystals can be a good strategy to improve the magnetic properties. Also, a large crystalline fraction ($v_{cr} \sim 0.75$ – 0.85) can cause residual stresses of few MPa which deteriorate the soft magnetic properties.

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