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MAGNETIC PROPERTIES OF NANOCRYSTALLIZED METALLIC GLASSES AT ELEVATED TEMPERATURES

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Abstract - In the present paper some of the magnetic properties of the nanocrystalline Fe-based magnets produced by an appropriate annealing of their metallic glass precursors are reviewed. These properties are discussed on the grounds of their characteristics measured at the elevated temperatures. It is shown that the effective magnetostriction these magnets display, results from the competition among two contributions of the opposite sign originating from the individual magnetic phases, crystalline phase and the residual glassy matrix in which the nanocrystallites are embedded. It is also shown that at certain conditions the magnets considered expose superparamagnetic behavior and that their isothermal magnetization characteristics can successfully be used to calculate the distribution of the particle volumes. Application of the recently invented new genetic algorithm method, a powerful tool to calculate these distributions is, finally, presented.

I. INTRODUCTION

A new class of magnetic alloys, the most common example of which is the Fe-based nanocrystalline magnets (invented in 1988 by Yoshizawa et al.[1] and well now known under their trade name FINEMET of the standard composition $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$) offer an interesting new possibility for tailoring superior soft magnetic properties comparable with those of permalloys or cobalt based metallic glasses but at much higher saturation induction (1.2-1.3T). Their attractive properties result from the fact that they contain a high volume fraction of ultrafine grains of bcc-Fe(Si) solid solution (or of DO_3-type structure) with a typical grain diameter of 10 nm, homogeneously distributed within the material volume. These grains are embedded within, and separated one from another by a portion of the amorphous phase (residual glassy matrix) from which they are formed by means of precisely controlled annealing of their metallic glass precursor produced in a form of a thin ribbon using conventional rapid quenching techniques. The origin of the excellent magnetic softness of these alloys, as it has been shown by Herzer [2], lies in that, that the local magnetocrystalline anisotropies are averaged out in a way similar to that in amorphous alloys [3] for grain sizes smaller than the ferromagnetic exchange length. It has been shown [4] that this mechanism is highly efficient for the considered nanocrystalline magnets, reducing the effective anisotropy by several orders of magnitude compared to that of the individual grain.

The superior soft magnetic properties, however, are also due to the simultaneously low or vanishing magnetostriction the examined materials show. This property is very important from the point of view of application because of the stresses usually induced when assembling magnetic components which might significantly degrade magnetic softness of their core material (via magnetoelastic interactions). The origin of low effective magnetostriction can be understood from the two phase nature of the material considering the composition of both, individual phases. Taking this point into account, Herzer [5] has described the resulting effective magnetostriction as a sum of the contributions coming from the corresponding phases weighted by their volume fractions. Since the bcc-Fe(Si) alloy with a high silicon content (nanocrystalline grains contain about 20 at%Si) exhibits negative magnetostriction [6], whereas, the residual amorphous matrix-positive (see e.g.[7]), the above description might give a correct order of magnitude of the effective magnetostriction observed in nanocrystalline alloys considered. A number of experiments recently performed [8,9,10,11] show that the proposed mechanism is, in principle, correct. In this paper the results of experiments performed at elevated temperatures on a series of samples diversified in respect of the content of the nanocrystalline phase [12] will primarily be discussed supposing they give a reliable answer regarding the nature of low effective magnetostriction in the considered nanocrystal-

line magnets.

The discussed materials can be classified as the granular solids, a class of materials which usually consist of fine metal particles embedded in an immiscible medium which may be isolating or metallic like in the case of the materials considered. A distinct feature of the material considered is, however ever, that both its phases are magnetic, the property which makes this material an unique one among the class of granular solids. Like other artificially structured solids, granular materials comprise complex structure on the nanometer scale and, what seems to be the most interesting feature of these materials, extra degrees of freedom owing to which the physical properties can be manipulated to achieve tailored materials for applications as well as for exploration of basic physical phenomena. These relevant extra degrees of freedom are the particle size and particle volume fraction, both easily controlled experimentally by process conditions (temperature and time of annealing). In the temperature range interesting from the view-point of applications (around ambient temperature) the materials considered demonstrate ferromagnetic properties and then their granular nature does not manifest directly from the magnetic characteristics. However, at elevated temperatures, higher than the Curie point of the residual amorphous matrix (Curie temperature of the glassy residual matrix is much lower than that of the crystalline particles), this granular character becomes clearly visible, manifesting itself in the magnetic characteristics of the material. It becomes particularly evident if the size of the particles is small enough to meet the single-domain requirements (several tens of nanometers for Fe) and, simultaneously, the particle volume fraction is sufficiently low to rule out magnetic interactions between particles. In this instance, superparamagnetic behavior should be expected. In the paper the properties of the nanocrystalline magnets of the type considered, observed in the range of elevated temperature, will be reviewed. Finally, it will also be shown how the isothermal magnetization characteristics can be exploited to attain an information on the structure of magnetic granular material with the help of a new numerical method based on the genetic algorithm.

II. TEMPERATURE CHARACTERISTICS OF MAGNETOSTRICTION

According to the hypothesis mentioned earlier [5], the effective magnetostriction in nanocrystalline magnets of the type considered can be expressed as a volumetrically weighted sum

$$\lambda_s^{eff} = p\lambda_s^{cr} + (1 - p)\lambda_s^{am}, \tag{1}$$

where p is the volumetric fraction of the crystalline phase, λ_s^{cr} and λ_s^{am} are the saturation magnetostriction coefficients for crystalline phase and residual glassy matrix, respectively.

Experiments recently reported and performed on a series of FeCuNbSiB-samples with different content of the crystalline phase show that the effective magnetostriction depends, indeed, roughly linearly on p. However, accepting such an approximation one must assume that both magnetostriction coefficients do not depend on the crystalline fraction. From the very nature of the crystallization process it is obvious that this assumption is, in reality, not satisfied since the larger crystalline fraction the greater the enrichment in niobium and boron of the glassy matrix. Consequently, the saturation magnetostriction of the matrix decreases with an increase of p. In the case of the crystalline phase it can, however, be assumed that this quantity does not really relies upon p since the content of silicon in the growing nanocrystals is only slightly dependent on the conditions of the annealing process (see e.g.[13,14]). Considering the above, the simplest form of the phenomenological dependence of the effective magnetostriction on the crystalline fraction, which should better reflects the real mechanisms responsible for this quantity, can be expressed as

$$\lambda_s^{eff} = p\lambda_s^{cr} + (1 - p)(\lambda_{s0}^{am} - kp), \tag{2}$$

where k is a constant which reflects the rate of a decrease of the glassy matrix magnetostriction with the crystalline fraction and λ_{s0}^{am} is the saturation magnetostriction coefficient of the metallic glass precursor (for $p = 0$).

In order to verify to what extend Eq.(2) describes satisfactorily the experimental results, an experiment has recently been performed [12] for a series of samples of the composition of their metallic glass precursor $Fe_{73.5}Cu_1Nb_3Si_{15.5}B_7$ (produced by Vacuumschmelze GmbH) diversified in respect to the crystalline fraction (p=0.14-0.8). The effective magnetostriction has been measured for these samples as a function of the elevated temperatures in the range from the room temperature up to 400°C, expecting that the experimental data obtained this way might give better insight into the nature of the mechanisms responsible for the effective

magnetostriction (for details of this experiment, see [12]). The temperature dependencies of the effective magnetostriction are shown in Fig.1 (Fig.1b shows, in an enlarged scale, the part of the characteristics presented in Fig.1a for very low values of magnetostriction).

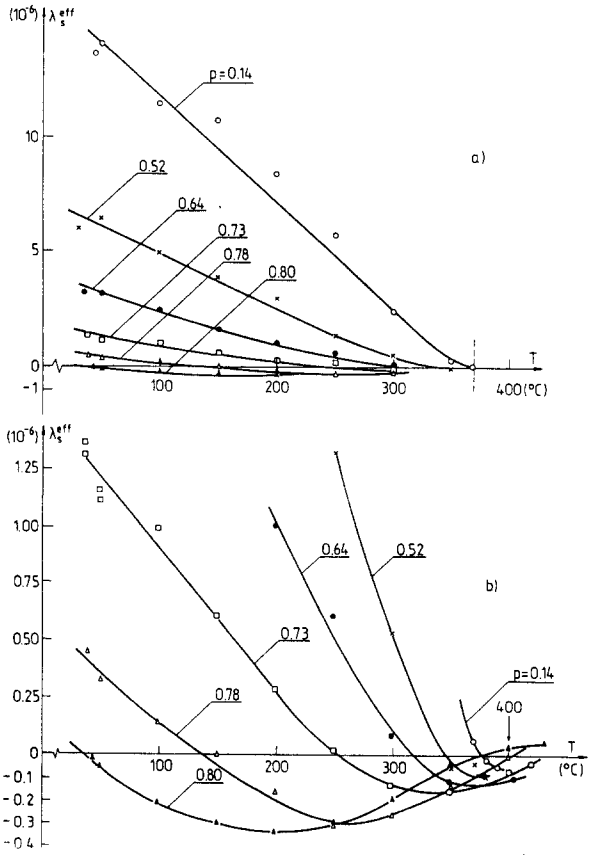

Fig.1. Temperature dependencies of the effective magnetostriction for a series of FeCuNbSiB samples with different fraction of nanocrystalline phase (a); part of the same characteristics as in Fig.1a but in an enlarged scale (b).

The dependence of λ_s^{eff} vs. p, shown in Fig.2, was plotted taking the points at 50°C for different values of the crystalline fraction from the characteristics given in Fig.1a. A fit of these data to Eq.(2) with the known value of $\lambda_{so}^{am} = 20.8\times10^{-6}$ (as measured for the as-quenched metallic glass precursor), gives $k = 6\times10^{-6}$ and $\lambda_s^{cr} = -3.9\times10^{-6}$, the latter of a quite reasonable value if compared with the data given in [6]. Taking into account the calculated value of the constant k, it can be demonstrated that the saturation magnetostriction of the glassy matrix decreases from its initial value for the as-quenched state up to 14×10^{-6} with an increase of the crystalline fraction within its available range of 0.14 - 0.8.

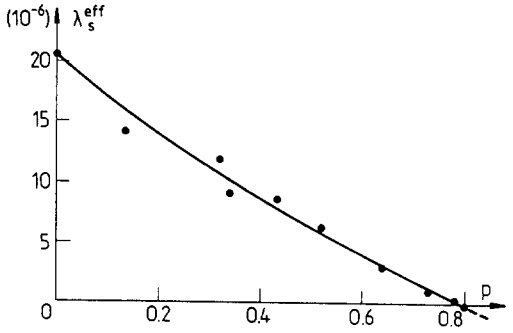

Fig. 2. Dependence of the effective magnetostriction versus the fraction of nanocrystalline phase [solid line is a fit to Eq.(2)].

A distinct feature of the characteristics shown in Fig.1b is that they are crossing the abscissa axis at the temperature which depends on the crystalline fraction, the result which is

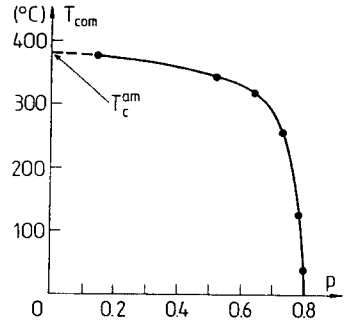

Fig. 3. Dependence of the compensation temperature of effective magnetostriction as a function of nanocrystalline phase fraction.

not surprising if one assumes that the operating mechanism is the weighted balance of two contributions (for some of these characteristics, as seen in Fig.1b, the second intersection for higher temperature appears also, but this effect will be discussed later). These compensation temperatures of the effective magnetostriction are plotted as a function of the crystalline fraction p (Fig.3). As seen from the dependence presented in this figure to obtain nanocrystalline material which would show vanishing magnetostriction in the vicinity of the room temperature, its metallic glass precursor should be annealed so as to produce relatively large amount of the crystalline phase. For commercial materials of the type considered, the

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content of this phase is in the range of 0.7 - 0.8, so that the requirement for low magnetostriction is satisfied.

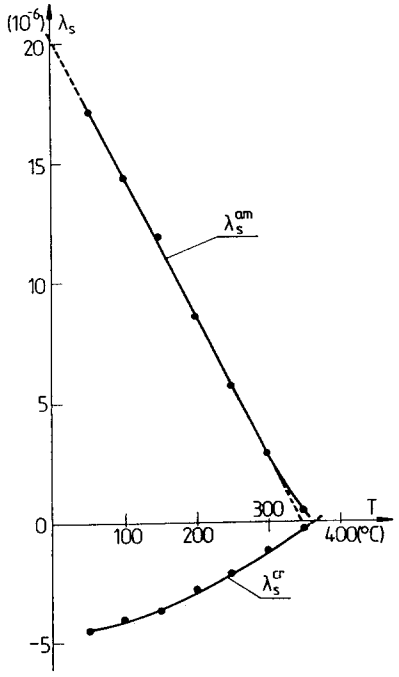

Fig. 4. Temperature dependencies of the saturation magnetostriction of individual phases, nanocrystalline and residual glassy matrix ones, calculated according to Eq.(2) from the characteristics presented in Fig.1.

The temperature characteristics of magnetostriction presented in Fig.1 were also used to separate the temperature dependencies of the individual contributions originating from both, the crystalline phase and residual glassy matrix (for details of the procedure used, see [12]). They are shown in Fig.4. As seen, the one for glassy matrix can fairly well be approximated by a straight line which, as expected, achieves zero-value at Curie temperature of the matrix (approx.350°C). The linearity of this dependence can be explained assuming that magnetostriction of the glassy matrix arises from a single-ion crystal field term which scales with a cube of the saturation magnetization (see e.g.[15]). Though, the assumed scaling is not quite correct (see e.g.[16]), nevertheless, it should be of a sufficient accuracy when analyzing this temperature dependence calculated basing on the phenomenological relationship. Therefore, taking into account that the temperature dependence of the saturation magnetization follows the relationship $M_s(T) = M_s(0)(1 - T/T_c)^{0.36}$ (see e.g.[2,17]), where T_c is the Curie temperature of the glassy matrix, and T-absolute temperature, it can be concluded that the temperature dependence of the residual glassy matrix magnetostriction should scale as $(1 - T/T_c)^{1.08}$ and, hence, can be approximated with good accuracy by a straight line.

As seen in Fig.4, the temperature characteristic of the crystalline phase magnetostriction is clearly nonlinear. Since to the best knowledge of the authors of the cited papers, there are no data available in the literature on the temperature dependence of magnetostriction for high silicon content Fe(Si) crystalline alloys, one can only speculate about the origin of this dependence. It seems that the most reasonable interpretation is that the considered dependence arises from the single-ion as well as two-ion (anisotropic exchange interactions) mechanisms (the latter being scaled with a square of the saturation magnetization), the occurrence of which is very likely in binary alloys (see e.g.[18]). However, better understanding of the behavior of magnetostriction of the crystalline phase requires to measure this quantity at the temperatures well above the Curie point of the glassy matrix. For the material studied, with Curie temperature of the matrix around 350°C, such an experiment was not possible because of the temperature limit of the measuring set-up used. It seems, however, that the use od samples with much lower Curie temperature of the glassy matrix could make it possible to complete the data. Such an attempt is actually in progress. It is, however, worth to noticing that the measurements of magnetostriction at the temperatures higher than the Curie point of the glassy matrix require much stronger external field to achieve the saturation state. It results from the fact that the nanocrystals are then decoupled since the matrix separating them becomes paramagnetic in this range of temperatures. An unexpectedly small magnetostriction (practically non-measurable) observed in this temperature range, as reported in [19], might probably be explained considering the above requirement.

The analysis presented above confirms it, in principle, that the effective magnetostriction of the considered nanocrystalline magnets results from the competition between two contributions coming from the individual magnetic phases existing within the material. However, this analysis appears to be oversimplified to a certain degree since a number of phenomena possible to occur and which may influence the magnetostriction have been neglected. For instance, the stresses generated in the material because of different thermal

expansion coefficients of both phases may alter the magnetostriction. Since the average size of the nanocrystals is very small, their surface to volume ratio can be large, and then the influence of the surface magnetostriction (observed in layered thin film structures [20]) may also be important. However, the experiments enabling to detect the above effects seem to be rather complex. With a view to the presented analysis which leads to the conclusions of a quite physical meaning, one may presume that the influence of the mentioned phenomena could have been of the second order of magnitude only.

III. MAGNETIZATION AT ELEVATED TEMPERATURES-MAGNETIC GRANULOMETRY TECHNIQUE

As mentioned above, in the range of temperatures higher than the Curie point of the glassy matrix, magnetic coupling between particles (nanocrystals) becomes the weaker the higher the temperature. The rate of this weakening depends markedly on the nanocrystalline fraction since the lower this fraction the larger the separation between the particles. The decoupling effect is well evidenced by the temperature dependence of the coercivity measured for the material considered. A dramatic increase of this quantity observed when the temperature exceeds the Curie point of the matrix (see Fig.5) is precisely due to the mentioned decoupling which causes a similar increase of the effective anisotropy

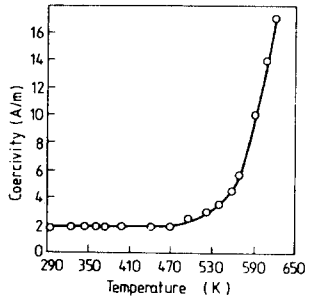

Fig.5. Coercivity versus temperature for typical FINEMET-sample (after Zbroszczyk [21]).

since the averaging process becomes then less efficient. When initially amorphous sample is annealed, the nanocrystals created within the bulk of the material grow with no preferential direction so that the easy directions of magnetization in the particles, are randomly distributed in the whole sample. At the elevated temperature, above the Curie point of the matrix, and if the nanocrystalline fraction is small enough so that magnetic interactions between particles can be neglected, the magnetic moment of the individual particle lines up with its easy axis. In this case, contrary, to the situation which occurs at lower temperature, the whole sample does not show collective magnetism. If now the sizes of particles are sufficiently small to satisfy the requirements for a single-domain state, the occurrence of superparamagnetism should be expected at the temperatures at which thermal energy exceeds magnetic energy of the particles. A number of experiments have shown that at the suitably selected conditions superparamagnetism occurs, indeed, in real samples. The existence of this magnetic phase in nanocrystalline magnets considered here, has for the first time been evidenced by Ślawska-Waniewska et al.[22] measuring the isothermal magnetization characteristics which superimpose and exhibit no hysteresis when plotted using the reduced coordinates (see Fig.6), thus, satisfying the well known operational criterion for the occurrence of superparamagnetism (see e.g.[23]). It

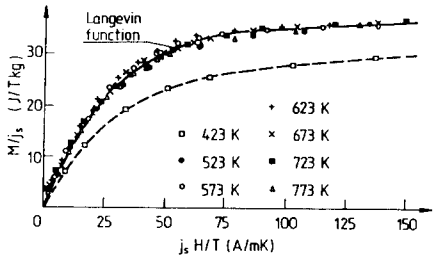

Fig.6. Superimposition of magnetization curves plotted in reduced coordinates [$j_s = M_s(T)/M_s(0)$] to demonstrate the superparamagnetic behavior of the nanocrystalline FeCrCuNbSiB sample with 18 vol.% of crystalline phase in the elevated temperature range (notice that the characteristic for 423 K does not superimpose with those for higher temperatures; T_c for residual glassy matrix is ~490 K and then the sample shows superferromagnetic behavior for the measuring temperature lower than this Curie point).

should, however, be recalled that the search for superparamagnetic behavior in samples of conventional metallic glasses has been undertaken long ago. For instance, Závěta et al.[24] have shown the occurrence of this behavior in an amorphous system $(Fe_x\ Ni_{1-x})_{80}P_{10}B_{10}$ being attributed to tiny iron-enriched clusters. Similarly, Hara et al.[25], studying fine-particle magnetism in the devitrified metallic glass $Fe_{43}Cr_{25}Ni_{20}B_{12}$, have also observed superparamagnetism which in this case was related to the very fine precitipations of a metastable bcc-phase in a paramagnetic matrix. The characteristics presented in Fig.6 were obtained for the sample of a metallic

glass with nominal composition $Fe_{66}Cr_8Cu_1Nb_3Si_{13}B_9$ (chromium was added to lower the Curie point of the residual glassy matrix) annealed so as to produce relatively small crystalline fraction ($p = 0.18$). A simple geometrical considerations shows that in this case the average separation between the particles is roughly of the order of the dimension of the particle itself (~ 10 nm, as estimated from magnetic measurements and confirmed by direct TEM-observations [22]). Since the range of the exchange field in metals is of the order of 1 nm (see e.g.[26]), and the dipolar interaction energy is then roughly equivalent to 120°C, as estimated [27], the occurrence of superparamagnetism at the temperatures higher than the Curie point of the matrix (~ 215°C) is not surprising. It has been shown quite recently that the superparamagnetic behavior is also observed in the samples of the standard FINEMET composition, if only the crystalline fraction is sufficiently small and the temperature of measurement is high enough [28]. The occurrence of superparamagnetism in the nanocrystalline magnets considered here, has additionally been confirmed by the results obtained from the measurements of the initial susceptibility [29] and also by Mossbauer experiments [30]. It is worth to noticing that for samples exhibiting relatively high content of the crystalline phase their isothermal magnetization characteristics, measured in the temperature range above the Curie point of the matrix, reflect typical so-called superferromagnetic behavior (this term, introduced by Mørup et al.[31], is related to the case when magnetic coupling between particles is too strong to be neglected and, hence, preventing superparamagnetic relaxations).

The studies of superparamagnetism in nanocrystalline materials may open new opportunities to investigate fundamental magnetism, but can also be utilized for practical purposes. For instance, since the superimposition principle is satisfied only for the particles which are chemically stable (their dimensions do not change with time), this property, important from the view-point of applications, can be tested for a variety of possible material compositions yielding nanocrystalline structure. Similarly, analyzing magnetization characteristics obtained in the temperature range, within which the material studied shows superparamagnetic behavior, the size of nanocrystals, as well as its distribution, can be calculated. This method being quite popular in the fiftieth (see [23] and the references therein) with a view of its application to magnetic granulometry, can, nowadays, be useful again for nanocrystalline magnets. It is well known that an analysis of the thermal equilibrium magnetization properties of an assembly of isotropic single-domain particles is analogous to the Langevin approach of atomic paramagnetism with the only difference that then one is dealing with a giant magnetic moment of the particle composed of many atomic moments (10^4-10^5) coupled ferromagnetically. Therefore, the isothermal magnetization characteristics for a superparamagnetic sample can formally be described by the familiar Langevin function. A fit of the experimental data to this function makes it possible to determine magnetic moment of the individual particle (assuming that all the particles are of the same volume) as well as the number of these particles in the sample. Knowledge of these values allows to calculate the particle volume and, furthermore, its diameter assuming a definite shape of the particle - spherical, for instance.

Application of the above procedure to the experimental data presented in Fig.6 gives the average particle diameter equal to 10 nm, the value which is in satisfactory agreement with that obtained from direct TEM-observations (for the details of this calculation see [22]).

However, in a real nanocrystalline material a certain distribution of the particle volumes (sizes) usually exists. In this case parts of the magnetization characteristics can be used to obtain various averages over the particle volume distribution. Since the initial susceptibility is sensitive to the larger particles present, whereas the approach to saturation is governed by the smaller particles, measurement of both types of averages yield information on the width of the distribution.

In order to take into account the existing distribution, the Langevin formula should, however, be modified (see e.g. [32]), expressing now the magnetization as a sum over all individual particles

$$M(T,H) = \sum_j m_j L(m_jH/k_BT)\, f(m_j), \qquad (3)$$

where m_j is the magnetic moment of the j-th particle, L is the Langevin function (k_B - Bolzmann constant and H - applied magnetic field), $f(m_j)$ - the distribution function of the particle magnetic moment directly related to the distribution of the particle volume (assuming that the magnetization of the particle is the same as in the bulk material).

Commonly accepted procedure when applying Eq.(3) is to assume an analytical model for the distribution function, most often of the log-normal type. However, the most serious drawback of this type of distribution is that it is unimodal, what may not always necessarily be true in practice. To

overcome this weakness, a new version of powerful genetic algorithm, modified to handle continuous variables in order to recover original distribution $f(m_j)$ in a form of smooth histogram, has recently been proposed by Gutowski [33]. The most important advantage of this new numerical method is that there is no need to assume *a priori* a specific distribution function when applying this technique to analyze experimental data.

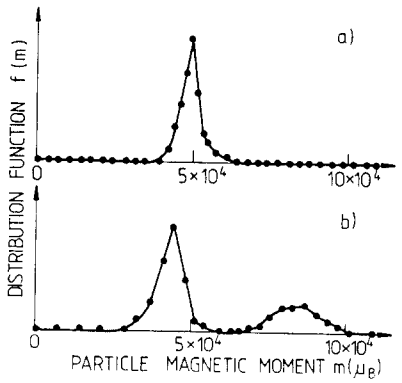

Fig.7. Distribution of the particle moment in the nanocrystalline FeCrCuNbSiB sample annealed at different conditions; (a) at 520°C for 20 min; (b) annealed once again at the same temperature for 10 min.

A persuasive example which demonstrates the strength of the mentioned new method are the calculated distributions of the particle magnetic moment obtained with the help of this technique for nanocrystalline sample (FeCrCuNbSiB) at first annealed at 520°C for 20 min ($p=0.18$), and next, after completing all the measurements, annealed once again at the same temperature but for 10 min [32]. The distribution calculated after the first annealing from the isothermal magnetization characteristics measured at the temperatures at which the sample displays superparamagnetism is presented in Fig.7a, whereas that obtained the same way after the second thermal treatment is shown in Fig.7b. The latter exhibits two distinct peaks. One of them (for lower magnetic moment) can be attributed to the nucleation of new crystallites, whereas the other, corresponding to the larger moment, results from the growth of crystals already present in the material after the first annealing. The occurrence of these two peaks in the calculated distribution function shows perfectly the great predominance of the invented method in comparison with the conventional ones. The example presented shows that apart from many other possible applications of the developed technique, the new genetic algorithm method is, indeed, a very powerful tool when applied to magnetic granulometry in order to study the crystallization processes.

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