Temperature, stress, and structural-relaxation dependence of the magnetostriction in $(Co_{0.94}Fe_{0.06})_{75}Si_{15}B_{10}$ glasses

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High-sensitivity measurements of the magnetostriction constant λ_s have been performed in the nearly zero magnetostriction $(Co_{0.94}Fe_{0.06})_{75}Si_{15}B_{10}$ metallic glass as a function of the stress and temperature after pulse-annealing treatments. A stress dependence of λ_s in this amorphous alloy is reported for the first time. At room temperature a slope of about -2×10^{-7} GPa^{-1} has been determined leading to a change in the sign of the magnetostriction constant in some circumstances. For determination of the thermal variation of λ_s, the temperature was changed by means of the alternating current flowing through the sample during the measurement, which was done by the small-angle magnetization-rotation (SAMR) method. The compensation temperatures, i.e., those temperatures at which the value of λ_s vanishes in going from negative to positive, were determined after isochronal annealing. In this way, the evolution of the single-ion and two-ion contributions to the magnetostriction was monitored during the structural relaxation. The ratio between the two contributions follows a chemical short-range-order kinetics. The stress dependence of the single-ion and two-ion contributions was deduced from measurements of the stress dependence of the magnetostriction at different temperatures.

I. INTRODUCTION

Magnetostriction in Co-rich amorphous alloys is a subject of growing interest.[1–5] Alloys having nearly zero magnetistriction at room temperature are suitable for technical applications, and are obtained by adding low amounts (e.g., 5–6%) of Fe or other metals to Co-based glasses.[1] For some of them, zero magnetostriction occurs at a "compensation temperature." This thermal behavior of the magnetostriction has been interpreted as a competition between two different contributions. A negative contribution which shows a temperature dependence close to the third power of the spontaneous magnetization, is related to a single-ion anisotropy in the uniaxial symmetry. The positive contribution depends on the temperature as the square of the spontaneous magnetization and originates from two-ion anisotropic exchange interactions.[2] The influence of composition of the one-ion and two-ion contributions to the magnetostriction has been thoroughly studied in $(Co_{1-x}Fe_x)_{75}Si_{15}B_{10}$, $[(Co_{1-x}(FeNi)_x]_{75}Si_{15}B_{10}$, and $(Co_{1-x}Ni_x)_{75}Si_{15}B_{10}$ alloys.[4–6] Magnetostriction in these compounds is intimately related to short-range order and is sensitive to structural relaxation. The influence of annealing on the magnetostriction constant (λ_s) at room temperature has been reported for $Co_{58}Fe_5Ni_{10}B_{16}Si_{11}$ and $(Co_{0.95}Fe_{0.05})_{75}Si_{15}B_{10}$ alloys.[7,8] In a recent study, the temperature dependence of λ_s was measured in $(Co_{0.95}Fe_{0.05})_{75}Si_{10}B_{15}$ after electric-current annealing.[9] No changes in the compensation temperature (T_{CO}) were observed, but this temperature was so high (320°C) in the as-cast glasses that the structural changes produced by the measurement itself could hide the effect of the annealing.

Very low values of λ_s at room temperature are equivalent to compensation temperatures near room temperature. Alloys with such characteristics are suitable for detecting small changes both in λ_s and T_{CO} after annealing. From these quantities, the single-ion and two-ion contributions to the magnetostriction can be obtained. That is, changes in T_{CO} during the structural relaxation can be used for clarifying the microscopic nature of the relaxation as well as the different interactions taking place between the magnetic atoms.

The aim of the present work was to detect the influence of the structural relaxation on the thermal variation of the magnetostriction constant in $(Co_{0.94}Fe_{0.06})_{75}Si_{15}B_{10}$. This alloy shows low values of λ_s in the $(Co_{1-x}Fe_x)_{75}Si_{15}B_{10}$ series in the as-cast state. Depending on the cooling rate during the quenching, as well as on small deviations from the nominal composition, λ_s values at room temperature in the as-cast state range from -2×10^{-7} to $+1\times10^{-7}$.

Corresponding compensation temperatures extend from about 150 °C to values below room temperature. These figures are, however, approximate because a strong dependence of magnetostriction on the applied stress has been found (see below).

The low values of T_{CO} in this alloy make it very suitable for studying the relaxation because the measurements of λ_s as a function of temperature after annealing does not involve any further relaxation and the changes observed are due only to the structural relaxation appearing during the annealing proces.

II. EXPERIMENTAL PROCEDURE

Amorphous ribbons about 0.55 mm wide and 21 μm thick were prepared by the single-roller quenching method. Details of the sample preparation are given in Ref. 10. As-cast values for the Curie temperature (T_C), the spontaneous magnetization (M_s), and saturation magnetostriction (λ_s) at room temperature are the following: $\mu_0 M_s = 0.85$ T, $T_C = 370$ °C, and $\lambda_s = -1.3\times 10^{-7}$. As mentioned above, these vaues are slightly different for different batches.

Annealing was performed by means of current pulses flowing through the samples. The experimental setup has been described elsewhere.[9–11] Stress and magnetic field can be applied simultaneously during the pulses, and the magnetization curves can be recorded. The temperature of the sample during the pulse was determined by comparing the value of the spontaneous magnetization with the values obtained at a well-known temperature in a conventional furnace. It was observed that the temperature of the sample, after the initial rise, stabilizes within about 2 sec. Annealing was performed with 10-sec pulses in order to insure an almost isothermal treatment.

Magnetostriction was determined by two different methods. One method uses the change in the inverse initial susceptibility (χ^{-1}) produced by a tensile stress (σ). The magnetostriction constant is then given by[12]

$$\lambda_s = -\tfrac{1}{3}\mu_0 M_s^2 (d\chi^{-1}/d\sigma) . \quad (1)$$

This method is very simple and sensitive, but it applies only to negative values of λ_s. If positive values are to be measured, a magnetic anisotropy perpendicular to the ribbon axis is needed in order to maintain χ^{-1} positive for some range of applied stress.

This restriction is overcome by using the small-angle magnetization rotation (SAMR) method. The principle of the method has been described previously.[13,14] The sample is subjected to a longitudinal bias field H_z and a tensile stress σ along its axis, and a small alternating transverse field H_y of frequency f. $H_y \ll H_z$ produces a small rotation of the spontaneous magnetization around its equilibrium position along the ribbon axis. In our case the transverse field is produced by an ac current, $f = 88$ Hz, flowing through the sample, whereby a voltage (V) of frequency $2f$ is induced in a pickup coil wound around the sample. Following[14] this one has

$$V = A{H_y}^2/(H_z + 2\lambda_s\sigma/\mu_0 M_s)^2 . \quad (2)$$

Here H_y is the transverse field at the ribbon surface: $H_y = I/2b$, I being the current, $2b$ the width of the ribbon, and A is a constant. The magnetostriction constant can be obtained by changing H_z and σ while keeping V constant. Under these constraints Eq. (2) gives

$$-3\lambda_s/\mu_0 M_s = (dH_z/d\sigma)_{H_y, V} . \quad (3)$$

Measurements of λ_s above room temperature can be easily performed by increasing the current through the sample.[15] The bias field must then be increased as to maintain constant the ratio H_z/H_y, in order to keep the rotation angle of the magnetization at a small value. Measurements have been done with current intensities ranging from 30 to 350 mA corresponding to temperatures between 20 and 250 °C.

This measuring method shows great advantages for monitoring the effects of the structural relaxation on the thermal dependence of the magnetostriction. Annealing and measurements can be performed *in situ* and very quickly. Furthermore, the sensitivity of the SAMR, as well as of the χ^{-1} method, is very high and magnetostriction coefficients of about 10^{-9} can be determined, in contrast to the strain-gauge method with a resoluton of about 10^{-7}.

III. EXPERIMENTAL RESULTS AND DISCUSSION

A. Preliminary measurements and stress dependence of λ_s

Room-temperature measurements of the magnetostriction constant were performed in as-quenched samples prior to any treatment. An anomalous variation of λ_s with the stress applied during the measurements was observed. For instance, two different samples showed a change of λ_s from low stress (100 MPa) values of zero and -3.5×10^{-7}, respectively, to high stress (1 GPa) values of -1.3 and -5.4×10^{-7}. The results obtained by the SAMR and χ^{-1} methods were mutually in agreement. Figure 1 shows curves for χ^{-1} [Fig. 1(a)] and SAMR [Fig. 1(b)] measurements after some annealing treatments performed under stress or under the influence of a magnetic field, with the aim to induce a magnetic anisotropy in the samples. Two different samples of the same batch were used in these measurements. In both cases, a change in sign is observed for λ_s as a function of the applied stress. From the curves in Fig. 1(a) it is evident that a perpendicular anisotropy has been induced. The magnitude of this anisotropy $K_\perp$ is related to the inverse initial susceptibility at zero applied stress χ_0^{-1} in the following way:[16]

$$K_\perp = \tfrac{1}{2}\mu_0 M_s^2 \chi_0^{-1} . \quad (4)$$

This anisotropy was stress induced in order to measure a positive magnetostriction constant by this method. The minimum in the χ^{-1} versus σ curves indicates, however, that λ_s passes through zero at this point and the positive slope for higher stress corresponds to negative values of λ_s. To the best of our knowledge, such a change in sign of the magnetostriction on the applied stress, has only been reported previously by Herzer[17] in a glass of similar composition.

For the samples in Fig. 1(b) a longitudinal, field-

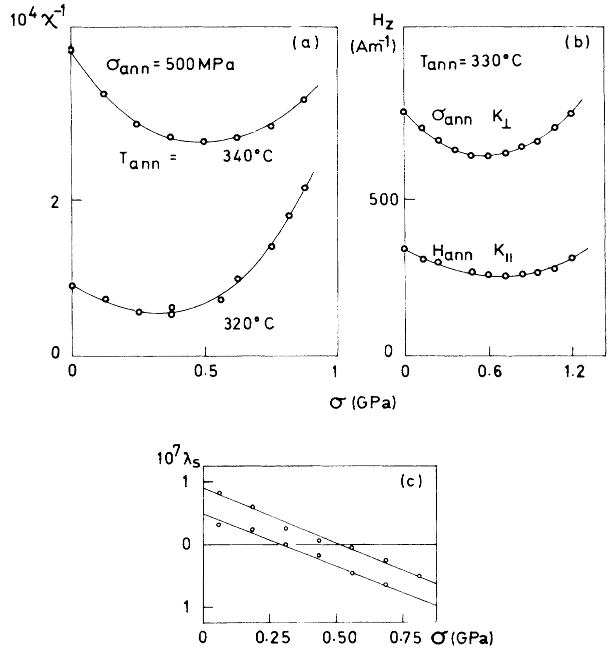

FIG. 1. Curvature in (a) the inverse initial susceptibility and (b) bias field indicates the stress dependence of magnetostriction in samples with stress- and field-induced anisotropy. In (c) the values of the magnetostriction constant deduced from the curves in (a) are plotted. A double point in (a) gives an idea of the dispersion in the measurements.

induced ($K_\parallel$) as well as a perpendicular stress-induced ($K_\perp$) anisotropy is present, and the same behavior for the magnetostriction can be observed. The change in the magnetostriction constant is almost linear with the applied stress in all cases. Figure 1(c) shows the values obtained from the curves in (a). A slope of -1.8×10^{-7} GPa^{-1} is obtained in this case. From the curves in (b) the slopes are -2×10^{-7} GPa^{-1} and -1.3×10^{-7} GPa^{-1} for $K_\perp$ and $K_\parallel$ respectively.

This dependence of λ_s on the applied stress is rather small, and it might hardly be observable except in low magnetostriction alloys and with relatively high applied stresses. Although the most correct definition of the magnetostriction constant should correspond to the zero-applied-stress value, the values of λ_s reported below have been obtained by averaging between 100 and 500 MPa unless otherwise stated. This is done in order to obtain a more accurate value for studying structural relaxation.

B. Influence of the structural relaxation on λ_s

Figure 2 illustrates the thermal dependence of λ_s on the successive isochronal ($t=10$ sec) annealing of a sample at different temperatures (T_{ann}). This sample was different from those of Fig. 1. As seen from Fig. 2, both the room temperature value of the magnetostriction and the compensation temperature exhibit a strong dependence on T_{ann}. This dependence is shown in Fig. 3. The more remarkable features of this dependence are as follows.

(i) No changes are observed below $T_{ann}=280\,°C$.

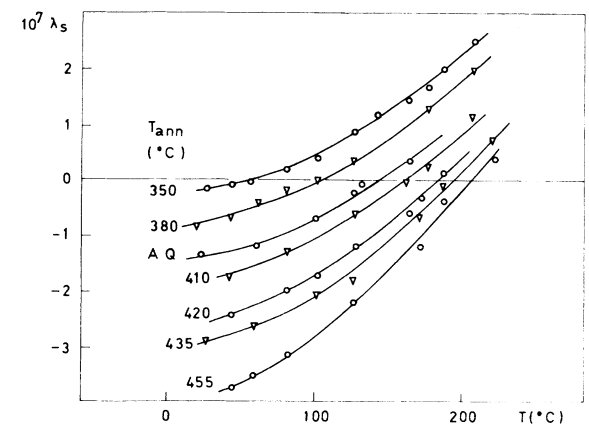

FIG. 2. Thermal evolution of the magnetostriction after 10-sec pulse annealing at the indicated temperatures. Compensation temperatures are shown to differ widely. AQ refers to as-quenched samples.

(ii) For low annealing temperatures (from 280 °C to 350 °C) λ_s increases towards positive values, while T_{CO} decreases down to 50 °C.

(iii) For high temperatures, i.e., $T_{ann}>350\,°C$ an opposite behavior takes place. However, two different regimes can be distinguished. Up to 390 °C a reversible behavior is

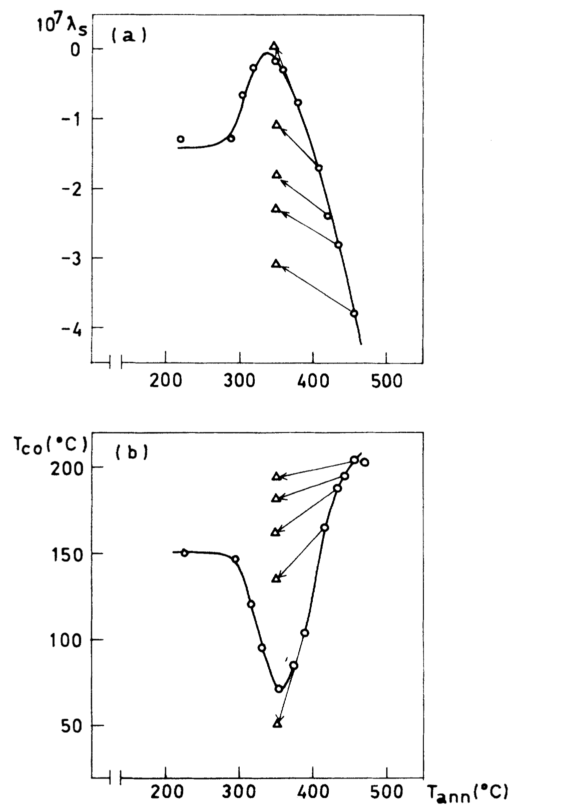

FIG. 3. Variation of (a) the room-temperature magnetostriction constant λ_s and (b) the compensation temperature T_{CO} upon structural relaxation. The triangles represent the values obtained by annealing at 350 °C after the first treatments.

recognized by further annealing at 350 °C. As seen in the figure the room temperature value of λ_s increases after successive annealings at 380 and 350 °C, and T_{CO} decreases, indicating that the equilibrium was not reached during the first 10 sec annealing at 350 °C.

For temperatures higher than 390 °C, a subsequent annealing at 350 °C shows that new irreversible processes have occurred, and the reversible mechanisms acting at lower temperature are no longer able to restore the previous values of λ_s and T_{CO}.

The results reported so far are in agreement with those obtained in $(Co_{0.95}Fe_{0.05})_{75}Si_{15}B_{10}$.[8] Moreover, λ_s for the latter alloy showed an initial increase and a final decrease, as a function of time during some isothermal treatments. This fact clearly indicates the existence of two different mechanisms contributing in opposite ways to the relaxation of λ_s.

A very similar kinetic behavior has been reported by Yokota *et al.*[18] for the electrical resistivity of $(Co_{0.525}Fe_{0.075}Ni_{0.4})_{73}(Si_{0.4}B_{0.6})_{27}$ alloys, and interpreted in terms of chemical and topological short-range-ordering[(CSRO) and (TSRO)] processes according to a model developed by Van Den Beukel and Radelaar.[19] At the first stages of the relaxation, CSRO increases because the equilibrium at the annealing temperature is more ordered than in the as quenched state. In our case, and for 10-sec annealing time, the equilibrium is reached by annealing at around 350 °C. For higher annealing temperatures CSRO decreases following the thermal equilibrium.

The high cooling rate after pulse annealing freezes this equilibrium, and a low-temperature annealing results in an increase of CSRO. If longer time or higher-temperature annealing is performed, irreversible processes contributing to an increase in the TSRO are activated. These processes are responsible for the irreversibility described above.

The structural units which give rise, upon ordering, to the increase of the magnetostriction constant should be the same as those giving rise to the induction of magnetic anisotropy by stress or field annealing.[20] If so, stress-induced anisotropy, which results from an high degree of order, will also be reflected in the magnetostriction. The latter is expected to increase with the anisotropy, if irreversible TSRO does not occcur during the treatment. This behavior is clear from the results given in Fig. 4. After stabilization by annealing at high temperature (400 °C), a sample is stress annealed at 365 °C under increasing loads. The induced anisotropy and the magnetostriction constant change in the predicted way, i.e., λ_s increases as the induced anisotropy does. This behavior may seem in contradiction with the applied stress dependence of λ_s displayed in Fig. 1, but the induced anisotropy is likely to originate from back stresses then acting in the opposite direction to the applied stress in Fig. 1. On the other hand, the temperature variation of $d\lambda_s/d\sigma$ (see Fig. 6 below) must be taken into account for explaining this effect. After further annealing without stress, initial values are restored, indicating that the TSRO has not been modified during the treatments. The values of λ_s at two different applied stresses, show that the dependence of λ_s on σ is also sensitive to changes in CSRO.

From a microscopic point of view, single-ion and two-

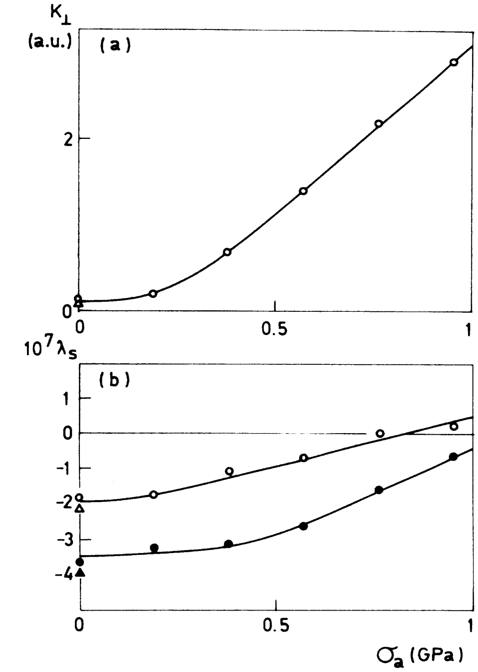

FIG. 4. (a) Stress-induced anisotropy and (b) change in the magnetostriction constant upon stress-annealing 10 sec at 365 °C under the indicated load. The samples were preannealed 10 sec at 400 °C Open symbols represent values of λ_s at 100 MPa and solid ones at 1 GPa. Triangles represent the values obtained after relaxation of the induced anisotropy.

ion contributions to the magnetostriction and magnetic anisotropy are present. If the temperature is not too close to the Curie temperature, the thermal evolution of λ_s is given by

$$\lambda_s(T)=\alpha J_s^3(T)+\beta J_s^2(T) , \quad (5)$$

where $J_s=\mu_0 M_s$ is the magnetic polarization. The coefficients α and β account for the contribution from the single-ion and two-ion mechanism, respectively. Fitting of the experimental values to Eq. (5) is done by plotting λ_s/J_s^2 against J_s, whereby straight lines are obtained from which α and β can be deduced. They are represented in Fig. 5 as a function of the temperature of annealing. Both coefficients show a minimum at 420 °C.[21] Their ratio (β/α) can also be obtained directly from the compensation temperature as

$$\beta/\alpha=-J_s(T_{CO}) . \quad (6)$$

Values obtained by the two methods are also shown in Fig. 5, and confirm that a maximum of chemical order is reached by annealing around 350 °C because the ratio between the two-ion and single-ion contributions is a maximum at this point. This annealing time and temperature roughly correspond to processes with an activation energy of 1.6 eV, which coincides with the lowest energy in the spectrum of induction and relaxation of magnetic anisotropy in stress-annealing treatments.[22,23] The relationship between CSRO, induced anisotropy and magnetostriction is, in our opinion, highly clarified by these measurements.

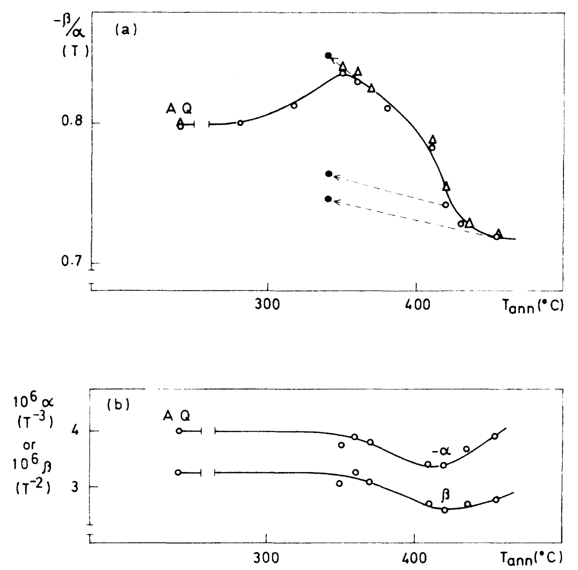

FIG. 5. Single-ion (α) and two-ion (β) contributions to the magnetostriction as a function of the annealing temperature are shown in (b). The upper part (a), shows the ratio $-\beta/\alpha$ obtained directly (triangles) and from the values of the compensation temperatures (circles).

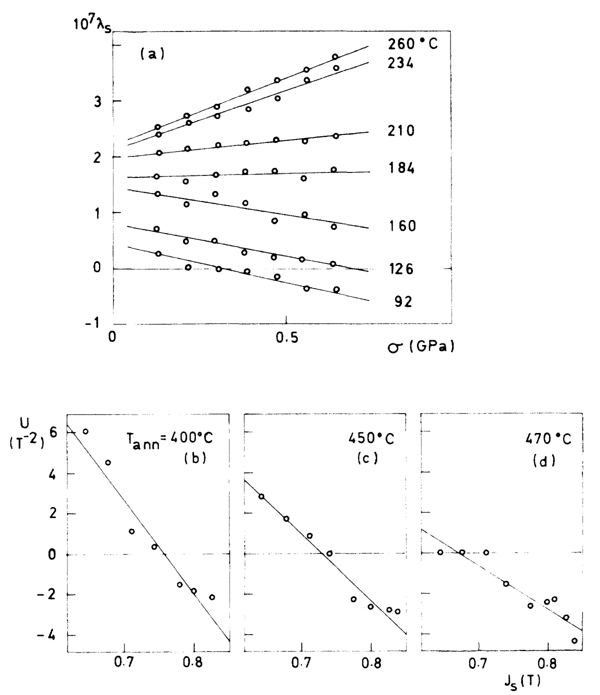

FIG. 6. Stress dependence of the magnetostriction at different temperatures (a) for a sample annealed 10 seconds at 400 °C. The lower parts (b), (c), and (d), shows the fit to Eq. (9) from which values of $d\alpha/d\sigma$ and $d\beta/d\sigma$ are deduced. The quantity plotted in the ordinate is $U=10^7 J_s^{-2}(d\lambda_s/d\sigma)$.

On the other hand, the separate evolution of the coefficients α and β is more difficult to explain and seems to be related to changes in TSRO.

C. Temperature evolution of the stress dependence of λ_s

Up to now only an average value of λ_s has been discussed, but the stress dependence of λ_s was also determined at different temperatures. Some measurements are shown in Fig. 6(a) for a sample previously annealed for 10 sec at 400 °C. The dependence of λ_s on σ remains approximately linear at all temperatures, but the slope $(d\lambda_s/d\sigma)$ changes from negative at low temperatures to positive at high temperatures. This behavior of $d\lambda_s/d\sigma$ is similar to the dependence on the temperature of λ_s itself. By taking the derivative of Eq. (5) one has

$$d\lambda_s/d\sigma=(d\alpha/d\sigma)J_s^3+(d\beta/d\sigma)J_s^2 \quad (7)$$

and the influence of stress on the single-ion and two-ion contributions to λ_s can be separated. This has been done in Fig. 6(b) by plotting $U=J_s^{-2}d\lambda_s/d\sigma$ versus J_s and fitting the points to straight lines. This procedure has been repeated for samples annealed at different temperatures. The values of $d\alpha/d\sigma$ are negative with high absolute values, ranging from -4.7×10^{-6} to -2.2×10^{-6} $GPa^{-1}T^{-3}$ while those of $d\beta/d\sigma$ are positive going from 3.5×10^{-6} to 1.5×10^{-6} $GPa^{-1}T^{-2}$. Thereby, the final value of $d\lambda_s/d\sigma$ is low because of a compensation of two opposite contributions, just as occurs with the value of λ_s in this alloy.

In $(Co_{1-x}Fe_x)_{75}Si_{15}B_{10}$ alloys, the single-ion coefficient is of the form[4] $\alpha=a_0+a_1x$ for low iron concentration $(x<0.12)$. Here the term a_0 corresponds to the Co ions

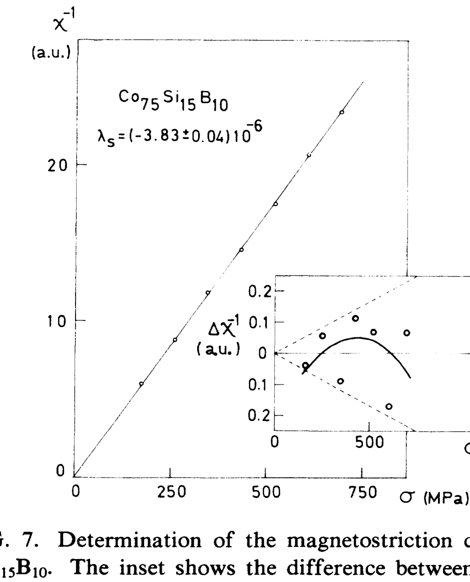

FIG. 7. Determination of the magnetostriction constant in $Co_{75}Si_{15}B_{10}$. The inset shows the difference between the least-squares fitting and the experimental values. Dashed lines represent a 1% deviation and the solid curve the expected behavior if $d\lambda_s/d\sigma=-2\times10^{-7}$ GPa^{-1}. $\Delta\chi^{-1}=\chi_f^{-1}-\chi_e^{-1}$, where f denotes the fitting value and e the experimental (points) or expected (line) value.

and has the value 7×10^{-6} T^{-3}. $Co_{75}Si_{15}B_{10}$ glasses exhibit only this single-ion contribution to the magnetostriction $\lambda_s = a_0 J_s^3$, therefore $da_0/d\sigma$ can be determined in this alloy and a separation of the Co and Fe contribution to the observed stress dependence of the magnetostriction should be possible.

Figure 7 shows the measurements of λ_s performed in $Co_{75}Si_{15}B_{10}$ by the χ^{-1} method. In order to improve the accuracy of the method and the sensitivity to detect small changes in λ_s as a function of the stress, each value of χ^{-1} was determined by a linear least-squares fitting of 8—10 points in the initial part of the magnetization curve.

The inset of Fig. 7 shows no significant deviations from a linear behavior of the points obtained in this way. This result indicates an upper limit of $\pm2\times10^{-7}$ GPa^{-1} for $d\lambda_s/d\sigma$ or $\pm3.4\times10^{-7}$ GPa^{-1-} T^{-3} for $da_0/d\sigma$, i.e., 1 order of magnitude below the values observed in $(Co_{0.94}Fe_{0.06})_{75}Si_{15}B_{10}$. Therefore the whole effect is to be attributed only to the iron atoms, present in very low concentration, so that a really strong contribution to the stress dependence of λ_s from each iron atom has to be postulated. A more likely interpretation can be envisaged by taking into account the stress dependence of the band configuration in these alloys, this dependence being noticeable because of the low value of λ_s itself. On the other hand, the possible existence of Fe-rich clusters in the Co matrix and the effect of stress on such a configuration could also explain the observed behavior. In fact, very recent results obtained by varying the quenching rate in $Co_{80}Nb_8B_{12}$ glasses[24] suggest the existence in these materials of two different phases, having magnetostrition constants of opposite sign.

IV. CONCLUSIONS

We can conclude that high-sensitivity magnetostriction measurements are appropriate for monitoring structural relaxation in low-magnetostriction metallic glasses. The SAMR method is specially suitable for measurements at different temperatures because the measuring current can be used for heating the sample. From the measurements in $(Co_{0.94}Fe_{0.06})_{75}Si_{15}B_{10}$ we conclude that the ratio between the two-ion and single-ion contributions to the magnetostriction follows the same kinetics as the chemical short-range order in this alloy. Moreover, an intimate relationship between CSRO, magnetostriction and stress induced anisotropy has been found experimentally.

Finally, a strong dependence of the magnetostriction on the stress applied during the measurement has been found for the first time. Such dependence can even change the sign of the magnetostriction constant by increasing the stress. An analysis of the influence of the stress on the single-ion and two-ion contributions to the magnetostriction, together with high accuracy measurements on $Co_{75}Si_{15}B_{10}$, indicates that this effect is due to the presence of iron. The exact microscopic origin remains, however, unclear.

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