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Direct measurements of magnetostrictive process in amorphous wires using scanning tunneling microscopy

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We demonstrate a versatile capability to measure directly the magnetostrictive properties through the magnetization process on a nanometric scale using a modified scanning tunneling microscope. Single 10 mm long, 125 μm diam amorphous wires of both positive and negative magnetostriction have been studied and the data are compared with the hysteretic loops determined by both ac and SQUID magnetic measurements. This improved technique promises interesting possibilities, from both fundamental and applications points of view, in a number of scientific disciplines especially of interest in life and environmental sciences. © *1995 American Institute of Physics.*

Magnetostriction, change in dimensions due to an external magnetic field, is an important physical property for a wide variety of applications of magnetic materials. In designing and building magnetic field sensors, flux multipliers, magnetic recording heads, or even high performance transformers, the main requirement is to obtain a material with zero magnetostriction over a wide temperature range, whereas to develop stress sensors or actuators a high, positive/negative, magnetostrictive material is required. In recent years, by rapid quenching in a liquid media amorphous wires of diameter 100 μm or less have become available. These wires have a unique magnetic structure which can be exploited to produce novel responses to torsion, tension, and induced magnetic anisotropies in them. Furthermore, these amorphous wires can be cold-drawn to diameters as small as 20 μm without loosing their unique magnetic properties even at lengths of the order of 10 to 15 mm, thus providing a new convenient material for applications as magnetic sensors. Direct determination of the magnetostrictive properties of such wires is thus of considerable interest. Among the prevalent methods to determine the magnetostrictive constants are (1) strain gauge technique, which is useful only to study massive samples; (2) capacitance or inductance gauges; and (3) indirect methods that exploit the magnetoelastic properties like ferromagnetic resonance (FMR), and the small angle magnetization rotation method, which gives only the saturation magnetostriction values.[1] A cantilever method has been devised for determining the magnetostriction of thin films.[1] A method to determine the magnetostriction of long wires in a liquid media using the STM tip as a strain detector has been recently reported.[1,2] We have developed an STM-based technique to measure directly the magnetostrictive response through the magnetization process, *especially for small wires.* Thus, we have potentially a powerful local probe technique tomeasure the dynamic length changes at an Å scale.

In this letter we present the relation between STM-based direct measurements of the elongation (contraction) of two ferromagnetic as-quenched $Fe_{77.5}Si_{7.5}B_{15}$ and $Co_{72.5}Si_{12.5}B_{15}$ amorphous wires having positive (negative) magnetorestrictive properties, respectively, and the longitudinal magnetization process data on the *same samples* when subjected to dc magnetic fields. The changes in dimensions at an Å-length scale are measured in a fairly direct fashion. Although the sensitivities of early STM, capacitance, and interferometric methods are considered to be similar,[1] the versatility of the STM method lies in that both positive and negative magnetostriction can be measured on a nanoscale without any modification in the experimental setup. Moreover, with this method the samples of virtually any size can be measured (if they are short enough these can be almost free standing). The sensitivity of this method is limited only by the stability of the STM itself, which if properly isolated can reach well below the 0.1 Å level. Furthermore, STM is extremely sensitive to vertical displacements (fractions of Å) and thus provides an additional simultaneous information which is not available in the interferometric and capacitance methods. In the interferometric method an interference pattern is observed, while in the capacitance technique a resonant frequency is usually measured, both of which are indirect methods. To our knowledge, capabilities to carry out measurements on nearly free-standing single small samples of wires using STM have not been demonstrated before. It is useful to note that STM has also the additional capability of lateral scanning, which opens the virtually unexplored field of local magnetostriction studies in complex systems.

The scanning tunneling microscope (STM) used in this study is a compact instrument ($2\times2\times6$ cm^3 in size) consisting of two concentric piezotubes, one for scanning and the other for inertial sample translation as well as thermal compensation. This setup makes the microscope rather insensitive to thermal drifts. The described arrangement, together with the small size of the microscope, makes the STM stable with hardly any need for damping.[3]

The STM was placed in the center of a coil capable of producing over 200 Oe. Although the measurements presented in this article apply to longitudinal applied fields, a further modification of the facility to apply transverse fields is feasible and in fact under development in our experimental setup. One single amorphous wire of about 125 μm in diameter and 10–15 mm in length is placed inside a special cop-

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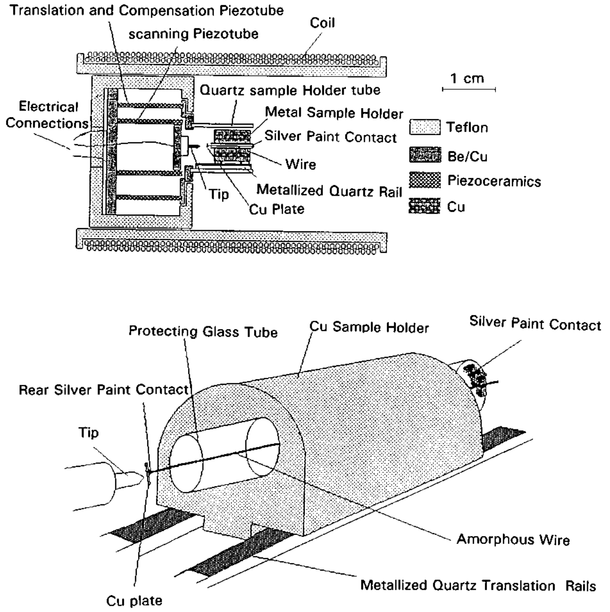

FIG. 1. Experimental setup for magnetostriction measurements in amorphous wires using a scanning tunneling microscope.

per sample holder. One end of the wire was glued using conductive silver paint to the center of the rear end of the sample holder. The wire is coaxial to a small glass tube of 1.5 mm i.d. to avoid possible lateral contacts. However, such a short wire is quite rigid and remains centered throughout the experiments, i.e., no lateral contact of the wire was present at any stage. At the other end of the wire a small ($0.03\times1\times1$ mm^3) copper plate was glued using conductive silver paint in order to simplify the approach to the sample and to the electron tunneling procedure. A schematic of the complete setup is shown in Fig. 1. When tunneling was achieved (typically with $V_t = 0.5$ V and $I_t = 2$ nA), the dc field was slowly increased in steps while monitoring the displacement of the *z*-piezo without altering the tip position with respect to the copper plate.

The hysteresis loops *of the same wires* used in the STM experiments were independently measured in a home-built loop tracer consisting of a primary coil that provides 22.9 Oe/A, a 1000 turns secondary, a compensation coil, and an integrator. The primary was fed with a 20 Hz alternating current and the loops were visually monitored and quantitatively studied in a digital oscilloscope. The longitudinal magnetization process curves were obtained measuring the amplitude of the peak-to-peak integrated voltage as a function of the amplitude of the current. The dc hysteresis loops were also measured by SQUID magnetometry to find that there was no appreciable difference with the switching curves obtained by the ac technique. It is worth mentioning that in all cases the initial state of the magnetization in these wires is determined and found to be similar to our earlier magnetization and magnetoresistance measurements.[4,5]

The elongation as a function of the applied dc longitudinal magnetic field for a 15 mm long $Fe_{77.5}Si_{7.5}B_{15}$ positive magnetostrictive amorphous as-quenched wire is shown in Fig. 2, where the elongation is presented directly in Å units in order to stress the sensitivity of the measurements. The inset shows data at low applied fields on an expanded scale. The overall magnetic response is consistent with the behavior of a positive magnetostrictive material. The wire elongates in the field direction, reaching a value of about 5000 Å for field values over 60 Oe. We can distinguish several marked regions with different dependencies of the elongation on the applied field. Up to ≈1 Oe there is no elongation within our experimental sensitivity. A linear dependence up to ≈18 Oe with a change of slope at ≈12 Oe follows. Then, we observe an abrupt change of the elongation followed by an approach to saturation behavior. The obtained saturation value corresponds to a value of $\delta l/l|_{\mathrm{sat}} \approx 3.3\times10^{-5}$. To estimate the saturation magnetostriction constant [$\lambda_s = \delta l/l|_{\mathrm{sat}} - \delta l/l|_{\mathrm{demag}}$ (Ref. 6)], there are two options; either we measure the longitudinal elongation when we saturate the wire perpendicularly, or we make an estimation of the $\delta l/l|_{\mathrm{demag}}$ (this in turn implies a determination of the spatial distribution of the magnetization in the demagnetized state). The ferromagnetic anisotropy of the resistance (FAR) could also be used for this purpose,[3] but it is not very clear yet[5,7] as to what kind of phenomena is affecting the magnetoresistance in these materials. The obtained $\delta l/l|_{\mathrm{sat}} \approx 3.3\times10^{-5}$ compares reasonably well with the value 2.8×10^{-5} of the saturation magnetostriction constant reported by us previously.[8]

The longitudinal magnetization process of the same sample is shown in Fig. 3, the inset showing the low field behavior. In principle, magnetization has a spatially averaged $M_s \cos\theta$ distribution, while magnetostriction has a spatially averaged $M_s \cos^2\theta$ behavior and are thus mutually related. The magnetostrictive behavior of the sample is more or less saturated at field values over 60 Oe (real saturation, 14 000 G, occurs for field values over 200 Oe). We can observe a marked change of slope at a field of about 1 Oe, which should be related to the above-mentioned onset of elongation. The fact that up to 1 Oe the longitudinal magnetization process is linear and it does not produce any appreciable elongation of the sample points unambiguously to a 180° domain wall movement as the operative magnetization pro-

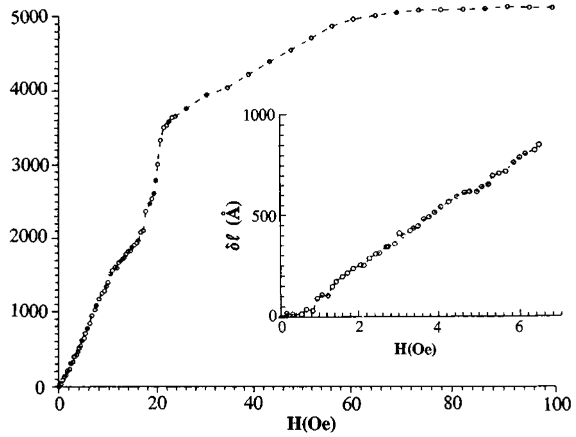

FIG. 2. Elongation measured as a function of the applied dc field for a 15 mm long as-quenched amorphous $Fe_{77.5}Si_{7.5}B_{15}$ wire. The inset shows the low field behavior.

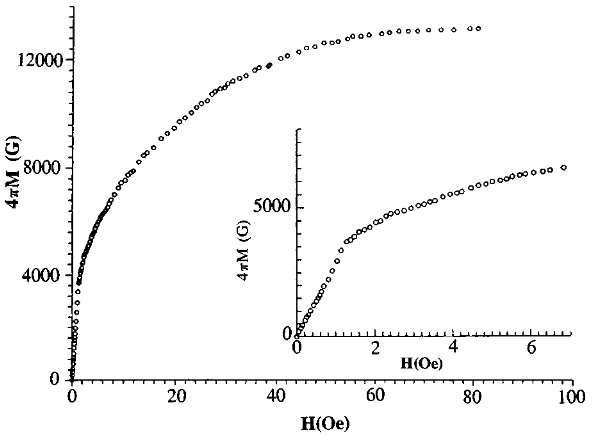

FIG. 3. Longitudinal magnetization as a function of the applied dc field for the same 15 mm long as-quenched amorphous $Fe_{77.5}Si_{7.5}B_{15}$ wire. The inset shows the low field behavior.

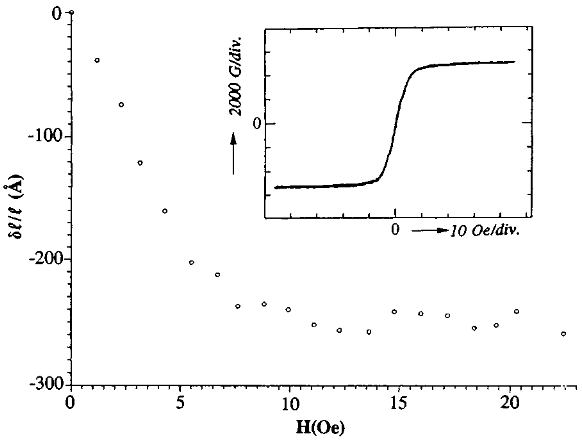

FIG. 4. Elongation measured as a function of the applied dc field for 12 mm long as-quenched amorphous $Co_{77.5}Si_{12.5}B_{15}$ wire. The inset shows a 20 Hz, ±49 Oe longitudinal hysteresis loop of the same sample.

cess. This conforms also with the direct magnetostriction measurements and can help to discern the active magnetization process. At field values over 1 Oe the magnetic susceptibility decreases coinciding with the onset of rotational processes as seen in the magnetostriction measurements, suggesting that the 180° domain wall movement is hindered as rotations start to dominate the magnetization process. For field values around 20 Oe, we notice that the magnetization process is basically linear. This coincides with the abrupt change in elongation and points to a rotation of the magnetization as the only effective magnetization process. A slow approach to saturation then follows.

In the case of a 12 mm long $Co_{72.5}Si_{12.5}B_{15}$ amorphous negative magnetostrictive wire, the magnetic field shrinks the sample, as seen in Fig. 4. The total effect is one order of magnitude lower than in the case of the Fe-based wire, saturating at $\delta l \approx -280$ Å. The initial behavior is linear with a negative slope that saturates at a field of about 10 Oe. The obtained value of $\delta l/l|_{\mathrm{sat}} \approx -2.3\times10^{-6}$ from the data in Fig. 4 compares well with values of the saturation magnetostriction constant obtained from the dependence of the initial susceptibility on the applied longitudinal stress (-2.3×10^{-6}) and using the SAMR method (-2.1×10^{-6}).[5,7]

A 20 Hz hysteresis loop of the same sample is shown in the inset of Fig. 4. The longitudinal magnetization process is linear for amplitudes of the field <10 Oe saturating at this value of the field. The observed behavior is totally consistent with the magnetostriction measurements shown above.

In summary, the versatility of using a STM to measure magnetostriction on a nanometric length scale in small samples has been demonstrated. The magnetostrictive process has been studied in both positive and negative magnetostrictive amorphous wires. The obtained magnetostriction values agree well with saturation magnetostriction constants determined from indirect methods. The field dependence of the magnetostriction, together with the longitudinal magnetization data, helps one to discern the operative magnetization processes in the wires. The Co-based exhibits a rotation of the magnetization from zero field to the saturation state. The low field magnetization process in the Fe-based wire is unambiguously determined to be 180° domain wall movement. The onset of the rotations coincide with the completion of this wall movement. The fact that the field evolution of the magnetostrictive process in the Fe-based model displays different slopes points to a more complicated domain structure than in the Co-based case.

STM is an extremely versatile instrument, which in our modified form can be used to study dimensional changes on an Å scale in different environments as well. As an example, we can mention an unexpected finding during our experiments: we silver pasted a copper plate onto our wires to ease the tunneling approach. We waited until the silver paste dried and we performed the tunneling approach; the whole process took over 1 h. Once tunneling was achieved we still observed drifts in the copper place displacement for hours. We attribute this to the silver paste solidification process. Even though the silver paste ‘‘looked’’ dry, the STM was telling us that our paste was not as stiff as it looked.

This versatile technique opens interesting new possibilities, from both applications and fundamental studies points of view, in various other scientific disciplines, especially on topics of interest in life and environmental sciences.

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