Journal of Magnetism and Magnetic Materials 212 (2000) 355–360

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New optical technique for bulk magnetostriction measurement

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Received 29 September 1999; received in revised form 1 December 1999

Abstract

A new optical technique was applied to the measurement of magnetostriction in bulk samples. This technique utilizes an optical fiber bundle, AC-modulated light and lock-in detection. Deformation of the sample is determined from the ratio of the incident and reflected light intensities. Noise due to the instability of the light source is eliminated by obtaining the ratio of the incident and reflected light intensities, and the noise caused in the detector circuit can be reduced by lock-in detection. The performance of this method was characterized with a series of measurements using a gold film and crystal disks of pure iron and nickel. This technique offers a resolution of 0.5 nm and is sensitive enough to measure magnetostriction as small as 5×10^{-7} in 1 mm thick samples. © 2000 Elsevier Science B.V. All rights reserved.

Keywords: Optical method; Optical fiber; Magnetostriction measurement; High sensitivity; Bulk magnetostriction

1. Introduction

Magnetostriction is the fractional change in length ($\lambda = \Delta l/l$) that is associated with the magnetization process of magnetic substances and is an important parameter which determine the performance, such as permeability, of magnetic materials. It can be measured by various techniques employing optical, strain gauge, and capacitance methods. The traditional optical method is the simplest method [1]. Here, the deformation of magnetic substances is measured using an optical lever. The change in length can be measured as a change in the reflection angle of an optical beam that is reflected by a mirror which is attached to the optical lever [1]. When the sample deforms with an increase in the magnetic field, the deformation is transmitted to a rod and then the rod rotates the optical lever. This method, however, requires a relatively large sample for measurement.

The strain gauge method is the most facile technique for measuring magnetostrictive deformation of the sample [2,3]. Strain gauges in a Wheatstone bridge arrangement are used for these measurements. The deformation of the sample is measured as a change in the resistance of the strain gauge, which is glued onto the surface of the sample. The bridge arrangement enables highly sensitive measurements of deformations as small as $\lambda = 10^{-6}$. Metal wire strain gauges have been widely used for magnetostriction measurements.

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PII: S0304-8853(99)00832-X

However, the measurement is often disturbed by the magnetoresistance and the temperature dependence of the wire. Moreover, relatively large samples of more than 1 mm must be used for measurements because the minimum gauge size available is about 1 mm. Therefore, this method is not effective to measure magnetostriction for samples smaller than 1 mm.

As mentioned above, there are many difficulties in applying the optical lever method or the strain gauge method to the measurement of tiny samples with a size of less than 1 mm. The capacitance method is a highly sensitive magnetostriction measurement method for small samples [4–8]. In general, a three-terminal capacitance method is typically used. The deformation of the sample is measured as a change in the capacitance between two metal plates, one of which is fixed on the sample holder and the other of which is attached to the sample mounted on the holder. When the sample deforms under increasing magnetic field, the plate on the sample gets displaced, thus changing the capacitance between the two plates. The deformation of the sample can be determined from the capacitance change as measured by a capacitance bridge. Deformation as small as $\lambda = 10^{-7}$ can be measured with this method. The capacitance method is highly accurate and can measure relatively small samples. However, it requires very delicate electrical wiring because measurement is often disturbed by a floating capacitance induced in the wiring. Moreover, an electrode (metal plate fixed on an insulator) must be put on the surface of the sample mounted on the sample holder prior to the measurement. This setup causes serious problems, especially for tiny samples because the weight of the electrode often stresses the sample.

In the studies of ferromagnetic materials, it is essential to characterize basic ferromagnetic parameters using single-crystalline samples. However, it is often very hard for some novel materials to obtain large single-crystalline samples. We have sought after a new simple and convenient technique with a high accuracy for the measurement of magnetostriction for tiny single-crystalline samples. Recently, optical methods for the measurement of displacement have made remarkable progress [9–13]. Application of these optical methods can permit the measurement of magnetostriction in tiny samples with high accuracy and without any contact between the probe and the sample. Laser interferometry is one of the most promising methods and provides high measurement precision. However, resolution and performance depends on the wavelength of light that is used. Moreover, both thermal expansion and distortion of the fibers often cause drift when optical fiber is used in the measurement. This is problematic since it is sometimes necessary to perform magnetostriction measurement in a low-temperature cryostat with a superconducting magnet, in which case the thermal expansion and the bend of the light path are unavoidable. Thus, laser interferometry seems unsuitable for this type of measurement.

In this study, we have applied a simple optical fiber displacement measurement method that is based on the measurement of reflected light [13]. Although this method is not new, it has been improved by using an AC-modulated light source and lock-in detection. We have attained sub-nanometer sensitivity using this method.

2. Outline of the experimental setup

The experimental setup includes two important elements: a measurement probe and an optical fiber displacement detector (OFDD) [13]. A schematic illustration of the probe and the OFDD is shown in Fig. 1. The sample holder was made of duralumin

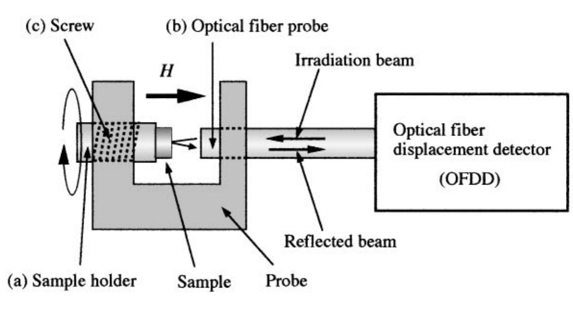

Fig. 1. Schematic illustration of the measurement probe. The probe consists of a duralumin sample holder and an optical fiber probe of an OFDD. The sample position can be adjusted by rotating a screw on the sample holder.

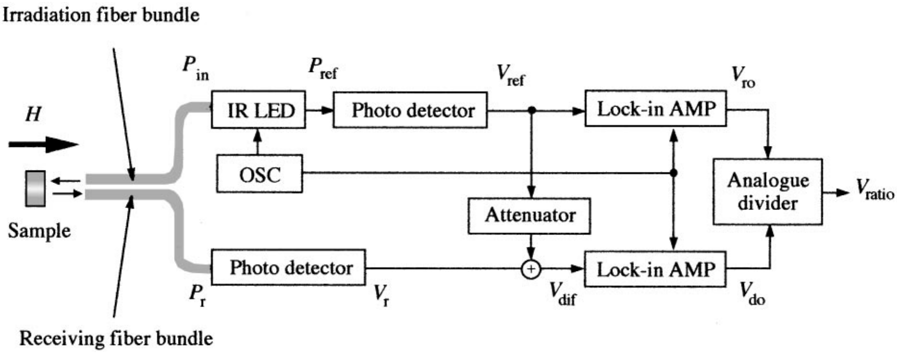

Fig. 2. Schematic diagram of an optical fiber displacement detecting system.

so as to avoid magnetic disturbances to the measurement, and has a sample position adjuster. The position of the sample in relation to the optical fiber probe of the OFDD can be adjusted by rotating a screw on the sample holder. The OFDD utilizes AC-modulation and lock-in detection. A schematic diagram of the OFDD is shown in Fig. 2. Hundreds of optical fibers are bundled and one end of the bundle is divided into irradiation and receiving fiber bundles (IFB and RFB). The other end of the fibers is set at a distance of about 50 μm from the surface of the sample. The AC-modulated light from an infrared light-emitting diode (IR-LED) irradiates the sample surface after being transmitted through the IFB. The light which is reflected by the surface enters the RFB and is detected by a PIN-diode photodetector after being transmitted through the RFB. The IR-LED and PIN-diode photodetectors have been mounted on an aluminum block to reduce thermal instability.

In this technique, optical power P_r, which is received by the RFB, depends on the distance d between the optical fiber and the surface of the sample. When d increases, P_r shows a significant increase and decreases monotonically after reaching maximum P_{max}. Here, P_r/P_{max} only depends on the geometrical parameters between the optical fiber and the light source. If each fiber bundle is calibrated for the light source, displacement Δd can be obtained from P_r/P_{max}. In the experiment the reflected light is detected by a PIN-diode photodetector, and output signal V_r of the detector is biased by the reference signal V_{ref} from the light source through an attenuator with gain G_a. After amplification of signals $V_{dif} = V_r - G_a V_{ref}$ and V_{ref} by lock-in amps with gains G_{dif} and G_{ref}, respectively, $V_{do} = G_{dif} V_{dif}$ is divided by $V_{ro} = G_{ref} V_{ref}$ through an analogue divider. When the gains are adjusted appropriately, the output $V_{ratio} = V_{do}/V_{ro}$ of the divider is proportional to P_r/P_{max} and shows the same behavior as that of P_r. The effect of instability in the light source can be eliminated by dividing $G_{dif} V_{dif}$ by $G_{ref} V_{ref}$ which is proportional to the power of the light source. In addition to the reduction of the effect of instability on the light source, the thermal drift due to the electronic circuit, which includes photodetectors, can be reduced effectively by using an AC-modulated light source and lock-in detection. A high sensitivity of this system has thereby been attained. Once the dependence of V_{ratio} on the distance d between the optical fiber and the surface of the sample is calibrated by using a piezo-actuator, measurement can be performed using a linear portion of a $V_{ratio}(d)$ curve. The deformation of the sample is calculated from V_{ratio} by using a conversion factor which was obtained by the calibration mentioned above. Since randomly bundled fibers and no optical interference are used in this method, the effect of bending the fibers is negligible on the measurement.

Magnetostriction measurement can be performed through the computer using software that is programmed to control the system. A single-crystalline sample is glued to the top of a sample holder using a small amount of adhesive (for example epoxy resin) after the crystallographic direction of the sample is determined using the X-ray

Laue back-reflection method. When the sample deforms under an increasing magnetic field, distance d between the sample and the optical fiber probe changes, and then V_{ratio} changes. V_{ratio} is measured by the DVM at a magnetic field which is discretely changed. The readouts from the DVM are transferred to the computer via GPIB and then transformed into magnetostriction using the value of the sample length and the calibrating parameters. Magnetostriction during measurement is displayed on a video-display as a function of the magnetic field intensity.

3. Characterization of the new technique

3.1. Performance of the system

The performance of the system was characterized by measuring a gold film which is formed on the top of the piezo-actuator by vacuum evaporation. The output voltage (V_{ratio}) of the OFDD is shown in Fig. 3 as a function of the displacement of the film against the optical fiber probe. An almost linear relationship between V_{ratio} and the distance can be observed at regions-I and II, which are shown in the figure. Although both regions can be used for measurement, highly sensitive measurements are attained by using region-I. Since V_{ratio} has been adjusted to give 10 V at the maximum, the operation point for measurement can be easily set to the center of region-I by adjusting the screw of the sample holder so as to give $V_{ratio} = 5$ V. The slope at region-I gives a displacement/voltage conversion factor of 9.3 μm/V for the system. In general, the deformation $\Delta l/l$ due to magnetostriction is very small, in the range 10^{-6} to 10^{-5}. Therefore, when a specimen with $l = 1$ mm is used for the measurement, the deformation Δl is in the range from 1 to 10 nm. Region-I has enough sensitivity to measure this range of deformation, and a deformation of 1 nm corresponds to an output change of 0.1 mV.

The noise level and the drift of the system were characterized and the results are shown in Fig. 4. As shown in Fig. 4a, the noise level is about 0.2 nm, peak-to-peak, corresponding to an rms value of less than 0.05 nm. Since this type of noise could not be reduced when the electronic circuit was shielded electrically, this seems to be caused in the electronic circuit. The drift during 7 h is shown in Fig. 4b. The drift level is within 2 nm for

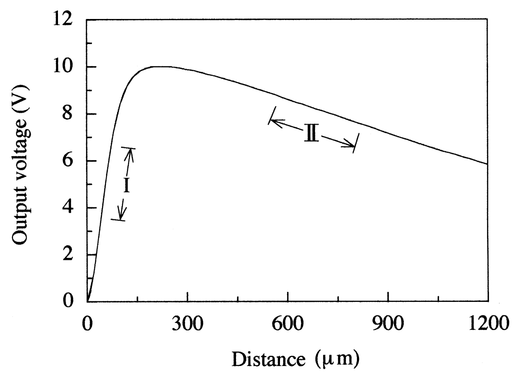

Fig. 3. A result of displacement measurement using a gold film mounted on a piezo-actuator. Vertical and horizontal axes respectively show output (V_{ratio}) of the OFDD and displacement.

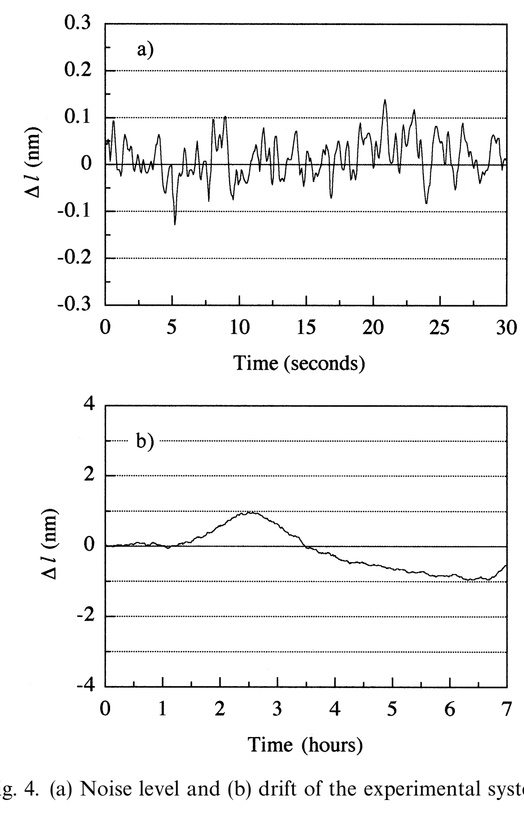

Fig. 4. (a) Noise level and (b) drift of the experimental system.

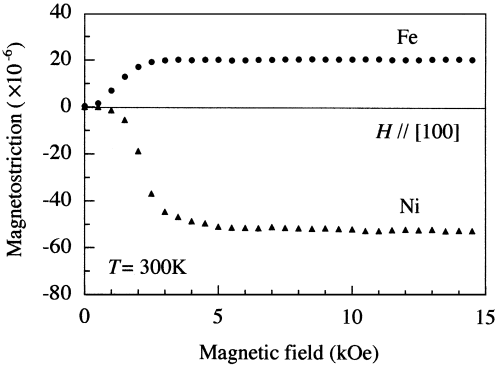

Fig. 5. Magnetic field dependence of magnetostriction as measured for pure iron and nickel single crystals in the [1 0 0] direction at 300 K.

temperature fluctuation of ± 2 K. The drift is the result of thermal expansion of the sample probe and holder due to a change in ambient temperature. If the system is operated at a well-controlled temperature within ± 0.5 K, the drift is reduced to 0.5 nm. Therefore, deformation as small as 5×10^{-7} in 1 mm thick samples can be measured by this system.

3.2. Examples of measurement

In order to characterize the performance of the measurement system, magnetostriction measurements were performed for small single crystals of pure iron and nickel. Fig. 5 shows the magnetic field dependence of magnetostriction as measured for single-crystalline samples of iron and nickel at 300 K. Disk-shaped crystals were used for the measurements. The disk plane of the sample was made parallel to the (1 0 0) plane of the cubic crystal. The diameter d and length l of the iron and nickel disks are, respectively, $d = 1.80$ mm and $l = 0.452$ mm and $d = 1.43$ mm and $l = 0.653$ mm. The disk sample was glued to the top of the sample holder using a small amount of epoxy resin. The epoxy resin was stable for the measurement at room temperature. A cyanoacrylate adhesive for paper strain gauge was also used to mount the sample and it has been confirmed as demonstrating a good performance. Magnetostriction was measured in the [1 0 0] direction by applying a magnetic field up to 14.5 kOe. In the case of iron, the magnetostriction increases as the applied magnetic field intensity increases and tends to become saturated at magnetic fields above 3 kOe. The magnetostriction constant in the [1 0 0] direction, which is the easy axis of magnetization, was determined to be $\lambda_{1\,0\,0} = 20.4 \times 10^{-6}$ from an analysis of the experimental curve. This is consistent with 20.3×10^{-6} (at 300 K), which was reported by Carr et al. [14].

Unlike iron, nickel has the easy axis of magnetization in the [1 1 1] direction at 300 K; and the [1 0 0] direction is the hard axis of magnetization. Since the disk plane of the sample was parallel to the (1 0 0) plane, magnetostriction was measured by applying a magnetic field parallel to the hard axis. Contrary to the results for iron, the magnetostriction decreases with increasing magnetic field and saturates for magnetic fields above 5 kOe. The magnetostriction constant in the [1 0 0] direction of nickel crystal was determined to be $\lambda_{1\,0\,0} = -52.4 \times 10^{-6}$ from an analysis of the experimental curve. This is consistent with the experimental values of -50.8×10^{-6} and -58.3×10^{-6} that were reported by Bozorth et al. and Carr et al., respectively [14,15].

4. Conclusion

A new optical technique, which utilizes an optical fiber bundle and lock-in detection, was applied to the magnetostriction measurement. High sensitivity of this method was shown through a series of measurements for a gold film and crystal disks of pure iron and nickel. Magnetostriction as small as 5×10^{-7} in 1 mm thick samples can be measured by this method.

Acknowledgements

This work was supported by The Science Research Fund of Japan Private School Promotion Foundation. A part of the work performed at Aoyama Gakuin University was supported by The Private School High-Tech Research Center Program of The Japanese Ministry of Education, Science, Sports and Culture.

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