Whole wafer magnetostriction metrology for magnetic films and multilayers


**Published in:**
*Measurement Science and Technology*

**Document Version:**
Peer reviewed version

**Queen's University Belfast - Research Portal:**
Link to publication record in Queen's University Belfast Research Portal

**Publisher rights**
© 2013 IOP Publishing Ltd

**General rights**
Copyright for the publications made accessible via the Queen's University Belfast Research Portal is retained by the author(s) and / or other copyright owners and it is a condition of accessing these publications that users recognise and abide by the legal requirements associated with these rights.

**Take down policy**
The Research Portal is Queen's institutional repository that provides access to Queen's research output. Every effort has been made to ensure that content in the Research Portal does not infringe any person's rights, or applicable UK laws. If you discover content in the Research Portal that you believe breaches copyright or violates any law, please contact openaccess@qub.ac.uk.
Whole wafer magnetostriction metrology for magnetic films and multilayers

C B Hill\textsuperscript{1,*}, W R Hendren\textsuperscript{1}, R M Bowman\textsuperscript{1,\#}, P K McGeehin\textsuperscript{2}, M A Gubbins\textsuperscript{2} and V A Venugopal\textsuperscript{2}

\textsuperscript{1}Centre for Nanostructured Media, School of Mathematics and Physics, Queen's University Belfast, Belfast, BT7 1NN, UK

\textsuperscript{2}Seagate Technology, Springtown, Northern Ireland, BT48 0BF, UK

Corresponding authors: * chill12@qub.ac.uk \# r.m.bowman@qub.ac.uk
Abstract

The requirements for metrology of magnetostriction in complex multilayers and on whole wafers present challenges. An elegant technique based on radius of curvature deformation of whole wafers in a commercial metrology tool is described. The method is based on the Villari effect through application of strain to a film by introducing a radius of curvature. Strain can be applied tensilely and compressively depending on the material. The design, while implemented on 3” wafers, is scalable. The approach removes effects arising from any shape anisotropy that occurs with smaller samples, which can lead to a change in magnetic response. From the change in the magnetic anisotropy as a function of the radius, saturation magnetostriction $\lambda_s$ can be determined. Dependence on film composition and film thickness was studied to validate the radius of curvature approach with other techniques. $\lambda_s$ decreases from positive values to negative values through an increase in Ni concentration around the permalloy composition, and $\lambda_s$ also increases with a decrease in film thickness; in full agreement with previous reports. We extend the technique by demonstrating the technique applied to a multilayered structure. These results verify the validity of the method and are an important step to facilitate further work in understanding how manipulation of multilayered films can offer tailored magnetostriction.

Keywords: Magnetostriction, Magnetic Films, Whole Wafers, Multilayers
1. Introduction

Magnetostriction of magnetic thin films is an area of interest in the magnetic recording industry. Very specific magnetic properties are required in elements such as recording heads; typically films with high saturation magnetization $B_s$, low coercivity and low magnetostriction. The need for low magnetostriction arises because when areal densities become greater, anisotropy effects become significant within magnetic recording heads. It is essential that they must be controlled in order to ensure that stresses are minimised in the presence of magnetic fields, and/or that magnetic properties will not be altered due to external stresses. Failure to control these two effects degrades the many operational performance indicators in a recording head [1-3].

At the same time, there has also been recent interest in magnetostriction control for micro-actuator applications, through the need to develop rare earth free alloy films that will give large magnetostriction. Recently, a re-visitation of Co$_{1-x}$Fe$_x$ has demonstrated that manipulation of parameters such as composition and temperature allow the magnetostriction to be manipulated to a significant degree [4].

Magnetostriction is either measured directly or indirectly. Direct measurements involve a dimensional change, whereas indirect measurements will measure a change in the magnetic property of the material as a result of strain. Magnetostriction then has to be calculated by relating it to the change in the anisotropy field. These indirect methods will yield $\lambda_s$, the saturation magnetostriction, while direct methods will generally yield $\lambda_e$, which is the field dependent magnetostrictive strain [5].

Direct methods involve application of a magnetic field which will bend the film-substrate bilayer due to magnetostrictive stress. Generally a uniform rotating field is applied to the sample, magnetizing it in one direction, while a magnetic field is applied in short pulses in the perpendicular direction; the directions depending on anisotropy being in or out of plane [6]. This produces a change in length of the sample where this deflection directly reflects the total film magnetization, and magnetostriction can be calculated from substrate deflection [7]. Up to a certain point, the $\lambda$–H response of these measurements will show a larger magnetostriction for a larger applied field value [8, 9]. These deflections will also be time sensitive and so time and field are of great importance in direct measurements, as they will be dependent on these two factors.

Direct measurements, making use of the Joule effect, typically make use of mechanical or optical methods such as strain gauges [10], interferometers [11, 12] and the more sensitive capacitance technique [13-19]. Strain gauges and interferometers however, have the disadvantage where they require special sample preparation (e.g. ribbons, wires and thin film cantilevers) causing undue restrictions. It has been shown that the capacitance technique is able to measure samples of various lengths and even irregular shaped samples. For the shape of the beam used however, it is normally
assumed that the width is small compared with the length so any curvature arising from displacement in the axis of the width can be neglected [17]. However in the case of a disc shaped sample, this assumption is not valid and curvature across the width of the sample cannot be ignored. The sample will not be under uniaxial stress and so capacitive measurements have limited applications for thin film wafers [5, 16-19].

Indirect measurement techniques, where as mentioned previously, a change in the magnetic response is measured, are viewed as more feasible and straightforward. There are three main techniques where strain can be applied to a sample; the three point bend, four point bend and applied radius of curvature. The three point bend technique involves upper and lower knife edges that exert a downward force on the sample. This technique has been employed in commercial instruments such as the SHB Instruments MESA inductive B-H looper whereby it can be equipped with an integrated magnetostriction three point assembly [6, 20]. The sample is stressed by applying a downward force which is derived from a cantilever assembly at the back of the pickup, and can be varied with adjustable weights. This technique can be applied to whole wafers, however it has been noted that the use of knife edges in this manner do not strain the sample uniformly [21].

The four point bend consists of a sample with two load applicators and two supports. Upon bending, the two supports below the neutral axis of the sample create tension, and the two load applicators above this axis create compression [22]. The advantage of this method is that it offers a region of constant bending moment between the load applicators at the centre point, where in this region the stress would vary only along the direction of specimen thickness. Hence, for a magnetostriction measurement, if a pick up coil is around this region of the beam, the thickness-averaged B field measured here would be independent of the position and length of the coil [23]. This technique however, is based on the Euler-Bernoulli beam model and was developed for beam geometries; therefore could also be argued that it is not a suitable technique for whole wafers.

Alternatively, the radius of curvature technique will apply a uniform uniaxial strain to the whole sample. Unlike the three point bend where the strain can vary largely between the region above the knife edge and the two ends, the strain applied from the curved surface of a radius of curvature is uniform. This technique has been applied in conjunction with a vibrating sample magnetometer or the magneto-optic Kerr effect. A holder is generally made with a scheme to introduce a variable radius of curvature and a sample is fixed to this. Given the size of the stimulus/detection scheme (a laser spot in MOKE and pick-up coils in VSM) samples are usually diced into small strips. By straining a sample over a range of curvature radii allows the change in the anisotropy field to be obtained [21, 24-28].

In the indirect measurement of magnetostriction, the magneto optical Kerr effect is often used but the probing laser field will only penetrate a certain distance, approximately 24nm [29, 30] beyond the surface of the sample, thus it is only suitable for very thin films and not suitable for thicker or
composite multilayered films. Further, the routine implementation is to apply a tensile strain exploiting the reflective nature of the films surface and frequently wafers are diced to fit the measurement tool. This can lead to an issue whereby dicing of a sample changes the anisotropy of a material and results in loss of the hard axis.

Taking all of the above into consideration, there is a need for a simple solution that addresses these three requirements – applicability to varying thickness of the thin film/multilayer, capability of tensile and compressive strain application, and the implementation of these conditions on whole wafers. In this paper we describe a universal extension to existing metrology that meets these requirements and allows the accurate measurement of magnetostriction of single films and multilayers in whole wafer samples.

2. Experimental

All samples were deposited in a load-locked UHV co-sputtering system with a base pressure of around 1 x 10^{-9} mbar. In the process chamber both the Ni and Fe targets are positioned at an angle of 15^\circ to the substrate. Ni_{x}Fe_{100-x} films were deposited onto Si/SiO_{2} wafers at room temperature at a deposition rate of 3 Å/s and an Ar process gas pressure of 5 x 10^{-3} mbar. The compositions of the films were controlled by altering the power of the magnetrons and by having a fixed deposition rate for each atomic composition percentage. The substrate was rotated at a speed of 20 RPM in order to obtain uniformity. Samples were also capped in a 5nm layer of Ta to avoid oxidation. Samples were then ex-situ annealed for 2 hours in vacuum at 225^\circ C with an applied field of 0.3 T and left to cool down over 12 hours.

The compositions and microstructures of films were revealed through EDX equipped for TEM and SEM. X-ray Diffraction was used to confirm crystalline orientation and lattice parameter calculation. Magnetic hysteresis loops were measured on a commercial SHB-instruments Model 110 BH-looper. The instrument was calibrated through cross reference with other magnetic metrology instruments in the laboratory already calibrated with NIST reference samples.

While recognising that SHB instruments provide a specialised three point bend magnetostriction pick up assembly for the MESA Inductive BH-looper [6], our model is not so equipped. Taking into account the limited dimensions within the instrument sample aperture, we have made an ingenious adaptation of the regular sample paddles that incorporates the radius of curvature to the wafer, with each paddle curvature ranging from 300mm to 1500mm. This development allows us to ameliorate the challenges identified above, i.e. uniform sample stress for both tensile and compressive application, whole wafer capability obviating magnetic changes that can arise from sample dicing, and capability of handling layers of large thickness ranges as well as multilayer configurations.
The paddles were precision machined from sheets of Tufnel using CNC milling. In this implementation the paddle was designed specifically for full 3” wafers (Figure 1). The wafer was positioned across the centre of the curvature and then mechanically secured by clamping it down at either end to ensure it was stressed fully across the radius. The distance to which the clamps are placed over the sample was kept consistent for each paddle, thus the strain was uniformly applied to the axis that is perpendicular to the applied magnetic field during annealing. The centre line of the paddle is co-incident with the pick-up assembly within the instrument.

Due to the simple nature of the paddle sample holder and the inductive detection scheme employed in the BH-Looper, the stress applied could be either tensile or compressive depending on how the sample was loaded onto the fixture. This is in marked contrast to MOKE based approaches. This is an important aspect as materials with different magnetostrictive signs will act differently depending on how the stress is applied [24].

Strain can be applied at any point during the fabrication process; however it is more beneficial to apply a strain during the measurement, as opposed to during deposition or annealing, as it allows a range of independent strains from a single film. Like most inverse measurement techniques, our method involves measurement of the change in the anisotropy field as a result of the applied strain. Further a result from one single film averaged over several strains will give more accuracy [31]. The change in anisotropy field $H_k$ as a result of applied strain was measured from the change in the hard axis loops between stressed samples of different radii of curvature. It is important that the hard axis is measured so that there is a linear response of the magnetization with the field outside of saturation regions [32]. We also note that the simplicity of the holder facilitates minor angular motion to identify the optimum orientation for closure of hard axis where achievable in samples. Through doing so this gives a more accurate representation of the anisotropy energy.

### 3. Results and analysis

To demonstrate the effectiveness of the design, initial measurements were focused on a range of Nickel-Iron samples with composition around the zero magnetostriction point, which from literature is taken to be approximately Ni$_{80}$Fe$_{20}$ [33]; films with higher Ni content have a negative magnetostrictive value, and films with lower Ni content will have a positive magnetostrictive value. Further, we highlight that accurate measurement of small values of magnetostriction around zero are most demanding [34].

The three compositions (Ni$_{77}$Fe$_{23}$, Ni$_{80}$Fe$_{20}$ and Ni$_{83}$Fe$_{17}$) were prepared with expected magnetostrictions to be positive, close to zero, and negative, respectively. Figure 2 shows the effects of the application of strains to the films. By definition, for a positive magnetostrictive material the magnetization will be increased by tensile strain and for a negative magnetostrictive material the
magnetization is decreased by tensile strain. Application of tensile stress to Ni$_{77}$Fe$_{23}$ creates an easier axis and application of compressive stress creates a harder axis, which is in agreement with how a positive material is defined. Ni$_{83}$Fe$_{17}$ demonstrates the reversal of this, as expected, while Ni$_{80}$Fe$_{20}$ shows little difference between the two.

A hard axis loop is required to calculate the magnetostriction and the magnetization must decrease when applying strain, so the sign of the magnetostrictive response of the material will determine whether compressive or tensile strain will be applied to obtain the anisotropy field. This is useful, particularly for films with unknown magnetostrictive values, as it will be clear on the first measurement whether tensile or compressive stress must be applied.

Radii of curvature used in this work ranged from 300 to 1500 mm, where the smaller the radius the sample is bent over, the greater the applied stress. Three of these were chosen which demonstrated the largest change in anisotropy for our chosen material. This is shown clearly in Figure 3, where the field at which the magnetization reaches saturation is greater for a larger stress, which corresponds to a smaller radius. The strain applied remains within the elasticity limit of the material and so it is expected that the anisotropy field should depend linearly on applied stress.

The change in the anisotropy field between each of the stressed loops can be determined by fitting a polynomial of order of best fit to the normalised hysteresis loop [21]. Saturation magnetostriction can then be calculated by substituting the gradient value for each radius into the following equation:

$$\lambda_s = \frac{d(H_s)}{d\left(\frac{1}{R}\right)} \frac{2\mu_0 M_s(1 - \nu^2)}{3t_s E_s}$$

where $\nu$ is the Poisson’s ratio of the substrate, $M_s$ is the saturation magnetisation, $t_s$ is the thickness of the substrate and $E_s$ is they Young’s modulus of the substrate [21, 28].

The Ni$_x$ Fe$_{100-x}$ films were investigated as a function of composition as above, and in addition, thickness. Figure 4 illustrates the variation of magnetostriction with respect to the Ni composition. The lowest Ni content of 77% has the highest positive magnetostriction value. As the Ni content increases the magnetostriction decreases in a linear trend, crossing over the zero value between approximately 80 - 81% and continues into the negative region. It is evident that the trend in our results and that found in other studies completely agree [33, 35-37].

Thicknesses of the three compositions from Figure 4 were also compared to understand how magnetostriction varies with film thickness. A positive, close to zero and negative composition were chosen and film thickness was varied from 10 – 60 nm. Figure 5 shows this relation and there is a clear trend where magnetostriction decreases with an increase in thickness within this range,
regardless of the magnetostrictive sign. This trend in results is in agreement with other published work [15, 27, 38] confirming the effectiveness and efficacy of this new technique.

Finally, to clearly illustrate the suitability for more challenging samples that could not be measured by other indirect techniques such as MOKE, bilayered films of opposite magnetostrictive polarity were investigated. The films were deposited with the following structure A (x nm) / B (60-x nm), where A is Ni$_{83}$Fe$_{17}$ and B is Ni$_{77}$Fe$_{23}$. Figure 6 shows these results plotted against the thickness of layer A, giving an expected trend where the magnetostriction changes from positive to negative as x$_A$ increases in thickness from 0 to 60nm. It appears from Figure 6 that at x$_A$ = 30nm, $\lambda_s$ reaches zero which would be the structure: A (30nm) / B (30nm). This observed trend is somewhat similar to other published work [34] and is a good indication that multilayered systems can indeed be manipulated and measured with this technique.

4. Conclusion

The analysis concludes that the radius of curvature fixture to be used in conjunction with a BH-looper, and applied to full wafers, is a viable approach for magnetostriction measurement. Preliminary results agree with literature by illustrating that (a) there is a thickness dependence of NiFe magnetostriction, (b) there is a NiFe composition dependence, whereby depending on the magnetostrictive properties of the film, compressive or tensile stress must be applied, and (c) layering of positive and negative magnetostrictive materials can be altered to reach zero. The method presented has important advantages as it utilizes full wafers, is a simple, cost effective set up, and can be applied to both thicker films and multilayers. This sets a fundamental basis for further work, in particular with multilayered films, in an effort to prove that manipulation of these layers will allow control of magnetostriction.

Acknowledgements

We thank Seagate Technology (Ireland) for their financial support to establish ANSIN (www.ansin.eu). We thank Philip Orr for invaluable technical assistance.
Figure 1:

Images of Tufnol paddle showing (a) cross section and (b) full view to be inserted in a BH-looper for a specific radius. 3” wafers are stressed and held in position with clamps where the stress may be tensile or compressive depending on how the sample is fixed.
Figure 2:

Hysteresis loops in unstressed conditions and with equal applied tensile and compressive stress corresponding to a radius of curvature of 500mm for (a) Ni$_{77}$Fe$_{23}$ (b) Ni$_{80}$Fe$_{20}$ (c) Ni$_{83}$Fe$_{17}$. 
Figure 3:
Normalised magnetization of averaged hysteresis loops at different tensile stresses (or different radii of curvature) for Ni$_{83}$Fe$_{17}$ grown on SiO$_2$ as a function of applied field.

Figure 4:
Measured saturation magnetostriction $\lambda_s$ as a function of Ni concentration (atomic %).
Figure 5:

Measured saturation magnetostriction $\lambda_s$ as a function of film thickness for Ni$_{77}$Fe$_{23}$, Ni$_{80}$Fe$_{20}$ and Ni$_{83}$Fe$_{17}$.

Figure 6:

Measured saturation magnetostriction $\lambda_s$ of bi-layered samples of A (x nm) / B (60-x nm), where A is Ni$_{83}$Fe$_{17}$ and B is Ni$_{77}$Fe$_{23}$, as a function of $x_A$ nm.


[24] Kundys B, Bukhantsev Yu, Szymbczak H, Gibbs M R J and Zuberek R 2002 Temperature dependence of saturation magnetostriction measured for Fe\textsubscript{81}Si\textsubscript{3.5}B\textsubscript{13.5}C\textsubscript{2} J. Appl. Phys. 35 1095 – 1098


[38] Kawai T, Ouchi S, Ohtake M and Futamoto M 2012 Thickness Effect on Magnetostriction of Fe and Fe<sub>80</sub>B<sub>2</sub> Thin Films IEEE Trans. Magn. 48 1585-1558