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CONTENTS

Thin Films, Fine Particles, Multilayers, Superlattices

- Dependence of Thickness of Fe Buffer Layer on Magnetic Properties for Mn₂6Ga Thin Films
..... K. Sato, Y. Takahashi, H. Makuta, T. Shima and M. Doi 48

Measurement Technique, High-Frequency Devices

- ウェハレベルの磁性薄膜における高周波磁気透磁率・磁歪定数評価法の開発
..... 森修・遠藤恭・島田寛・藪上信・内海良一 52

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Dependence of thickness of Fe buffer layer on magnetic properties for Mn_{2.6}Ga thin films

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*DO*₂₂-Mn_{2.6}Ga thin films were fabricated by an ultra-high vacuum electron beam vapor deposition system on a MgO (100) single crystalline substrate with different buffer layer thicknesses (0.7 - 5 nm). As the annealing temperature T_a increased to 400°C, the squareness of the *M-H* curves improved, and the saturation magnetization M_s and magnetic anisotropy K_u increased. At that time, from XRD patterns, *DO*₂₂-Mn₃Ga (002) superlattice and (004) fundamental peaks were clearly observed from all samples. Our results suggest the existence of a magnetic dead layer of about 3.5 nm in the MnGa layer. Because of this magnetic dead layer, a relatively high K_u of 9.3 Merg/cm³ and low surface roughness R_a of 0.93 nm were obtained from Mn_{2.6}Ga thin film on a 2-nm Fe buffer at $T_a = 400^\circ\text{C}$. The results in this work suggest that Fe is a suitable buffer layer for perpendicular magnetized *DO*₂₂-MnGa thin film.

Key words: *DO*₂₂-MnGa thin film, saturation magnetization, magnetic anisotropy, electron beam vapor deposition system, surface roughness, buffer layer

1. Introduction

MnGa alloys show *DO*₂₂ and *L1*₀ structure in the composition range at approximately 69 - 76 (η phase) and, 64 - 67 (γ₁ phase) and 58 - 61 (γ₂ phase) at.% Mn in the binary phase diagram¹⁾. Owing to outstanding magnetic properties such as a high magnetic anisotropy $K_u \sim 10 - 23.5$ Merg/cm³²⁻⁴⁾, a low Gilbert damping constant $\alpha \sim 0.008 - 0.015$ ⁵⁾, a relatively low saturation magnetization $M_s \sim 600$ emu/cm³²⁻⁴⁾ and a high spin polarization $P \sim 58\%$ (experimentally)⁵⁾, the MnGa thin films have been studied for applications of spintronic devices, including a magnetic random access memory operated by spin transfer torque (STT-MRAM). This device requires a guarantee of thermal stability and high speed writing and reading. That is to say, STT-MRAM needs perpendicularly magnetized magnetic tunnel junctions (p-MTJs) that have high K_u and P . Nowadays, MTJ is demanded to achieve a high $K_u \geq 10$ Merg/cm³, a high $P \geq 70\%$, a low $\alpha \leq 0.01$ and a low $M_s \sim 100$ emu/cm³^{6,7)}. From the point of view, a great potentiality for magnetic layers of MTJ is derived from MnGa thin films which shows high K_u , high P , low α and relatively small M_s ⁸⁻¹³⁾. However, there have been few reports about buffer layers for growth of MnGa thin films. Therefore, it is necessary to obtain the knowledge of buffer layers' dependence on magnetic properties. The lattice mismatch between *DO*₂₂-Mn₃Ga and Fe is smaller than that of *DO*₂₂-Mn₃Ga and Cr. However, it is well known that Fe behaves an in-plane easy magnetization axis. On the other hand, a large perpendicular magnetic anisotropy (PMA) of 14 Merg/cm³ from ultrathin Fe/MgO interface has been presented by J. W. Koo et al.¹⁴⁾. The, in-plane easy magnetization axis and the relatively high average roughness R_a of 2.63 nm were observed from MnGa thin films on 5-nm Fe buffer at our previous

work¹⁵⁾. Improvements of these properties are indispensable for application of MTJ films.

In this work, dependence of thickness of Fe buffer layers on magnetic properties for Mn_{2.6}Ga thin films have been investigated by using an ultra-high vacuum electron beam (UHV-EB) vapor deposition system.

2. Experimental procedure

All the samples prepared in this work were grown by an ultra-high vacuum electron beam vapor deposition system with base pressure less than 8.9×10^{-7} Pa. MnGa target was fabricated from raw materials of Mn (5N) and Ga (6N), through the arc melting method in argon atmosphere with base pressure less than 10^{-3} Pa. The stacking structure is the MgO (100) single crystalline substrate / the Fe buffer layers (0.7 - 5 nm) / Mn_{2.6}Ga (20 nm) / the Cr capping layer (10 nm). The MgO (100) single crystal substrate was thermally flushed up to 700°C in the UHV chamber half an hour. The Fe buffer layers and the MnGa layer were deposited at room temperature and 300°C, respectively. Finally, the Cr capping layer was deposited at room temperature. Annealing process was carried out at 300 - 500°C 3 hours to improve the crystal quality. All growths are monitored in real time by using reflection high-energy electron diffraction (RHEED). The compositions of the films and the crystal structure of the samples were determined by an energy dispersive X-ray spectroscopy (EDX) and X-ray diffraction (XRD) with Cu-*K* α radiation, respectively. The surface roughness of film was investigated by atomic force microscopy (AFM). The magnetic properties of thin films were measured by using superconducting quantum interference device (SQUID).

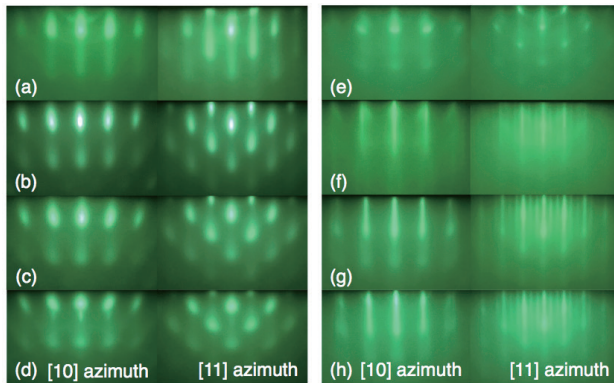


Fig. 1 RHEED patterns for different Fe buffer layer thicknesses and $\text{Mn}_{2.6}\text{Ga}$ thin films. Fe buffer: (a) 5 nm, (b) 2 nm, (c) 1 nm and (d) 0.7 nm. $\text{Mn}_{2.6}\text{Ga}$ thin film on Fe buffer: (e) 5 nm, (f) 2 nm, (g) 1 nm and (h) 0.7 nm.

3. Results and discussion

Figure 1 shows RHEED patterns for different Fe buffer layer thickness ((a)-(d)) and $\text{Mn}_{2.6}\text{Ga}$ thin films on Fe buffer: (e) 5 nm, (f) 2 nm, (g) 1 nm and (h) 0.7 nm, respectively. As the thickness of Fe decreases, RHEED patterns of Fe (From (a) to (d)) became spotty. The result suggested that Fe do not grow layer by layer (Frank-van der Merwe) but island growth (Vol-mer-Weber). Therefore, as Fe becomes thinner, surface roughness of Fe becomes worse. The epitaxial growth of $\text{Mn}_{2.6}\text{Ga}$ and surface reconstruction structure were observed from (e).

Figure 2 shows XRD patterns for $\text{Mn}_{2.6}\text{Ga}$ thin films on Fe buffer: (a) 5 nm, (b) 2 nm, (c) 1 nm and (d) 0.7 nm. $\text{DO}_{22}\text{-Mn}_3\text{Ga}$ (002) superlattice and (004) fundamental peaks were observed from all samples. Although the broad peaks are shown in (a) and (b), this might be because of the diffusion of Fe. The (004) peak sifted to low angle side, which showed the c-axis became long. The highest ordering parameter $S_{002} = 0.69$ was obtained from (b) 2-nm Fe buffer.

Figure 3 shows magnetization curves for $\text{Mn}_{2.6}\text{Ga}$ on Fe buffer: (a) 5 nm, (b) 2 nm, (c) 1 nm and (d) 0.7 nm. The black and red lines indicate the out-of-plane and in-plane magnetization curves. The volume of magnetic layer was calculated by combining the thickness of Fe and MnGa. K_u is given by the equation $K_u^{\text{eff}} + 2\pi M_s^2$ where K_u^{eff} is the effective perpendicular magnetic anisotropy (PMA) constant. The computation for the determination of K_u^{eff} was carried out in subtracting the area from H_k^{eff} to 0 in the in-plane magnetization curves from the out-of-plane magnetization curves. Here, the effective anisotropy field (H_k^{eff}) was defined as the extrapolated intersection of in-plane magnetization curves with the value of saturation magnetization of out-of-plane magnetization curves. Fig. 3 shows that in the case of (a), the easy magnetization axis is the in-plane direction and (b)-(d) are out-of-plane directions. The result of (a) 5-nm Fe buffer, which shows the easy axis to the in-plane direction, was considered due to

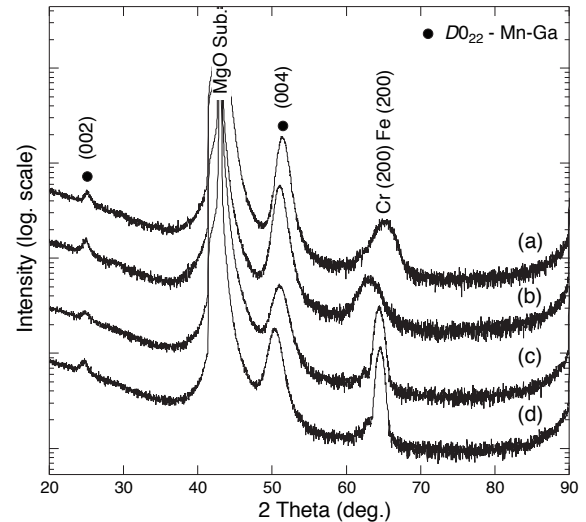


Fig. 2 XRD patterns for $\text{Mn}_{2.6}\text{Ga}$ thin film on Fe buffer: (a) 5 nm, (b) 2 nm, (c) 1 nm and (d) 0.7 nm.

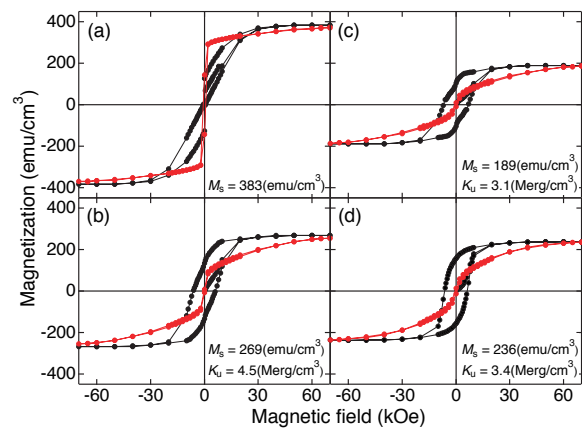


Fig. 3 Magnetization curves for $\text{Mn}_{2.6}\text{Ga}$ thin film on Fe buffer: (a) 5 nm, (b) 2 nm, (c) 1 nm and (d) 0.7 nm. Black and red lines indicate out-of-plane and in-plane magnetization curves.

interface roughness for Fe layer (Neel “orange-peel” coupling though the magnetic dead layer^{16,17}). The highest K_u of 4.5 Merg/cm³ was observed for (b) 2-nm Fe buffer.

Figure 4 shows the product of M_s and t as a function of t . Here, t is a thickness of MnGa layers and M_s is estimated by subtracting magnetization of Fe from total magnetization. The black circles and squares indicate the reference values of $\text{Mn}_{1.5}\text{Ga}$ thin films on 5-nm Cr buffer presented by Y. Takahashi et al³), and $\text{Mn}_{2.6}\text{Ga}$ thin films on 2-nm Fe buffer in this work, respectively. A magnetic dead layer thickness ~ 2 nm in the Cr buffer case and ~ 3.5 nm in the Fe buffer case were shown in Fig. 4. Here, the magnetic dead layer means a layer of non-magnetic ones. It was shown that when Fe used for the buffer layer, the magnetic dead layer becomes larger than in the case of

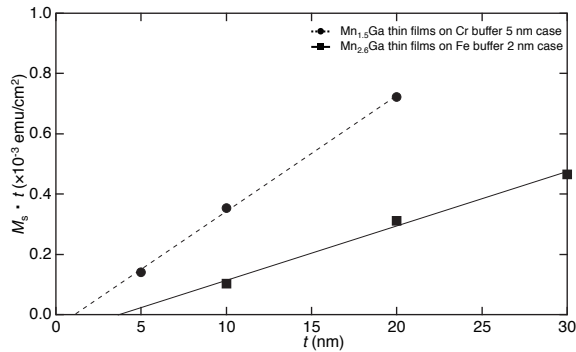


Fig. 4 Product of M_s and t as a function of t . Black circle and square indicate reference value of $Mn_{1.5}Ga$ thin films on 5-nm Cr buffer³⁾ and $Mn_{2.6}Ga$ thin films on 2-nm Fe buffer in this study, respectively.

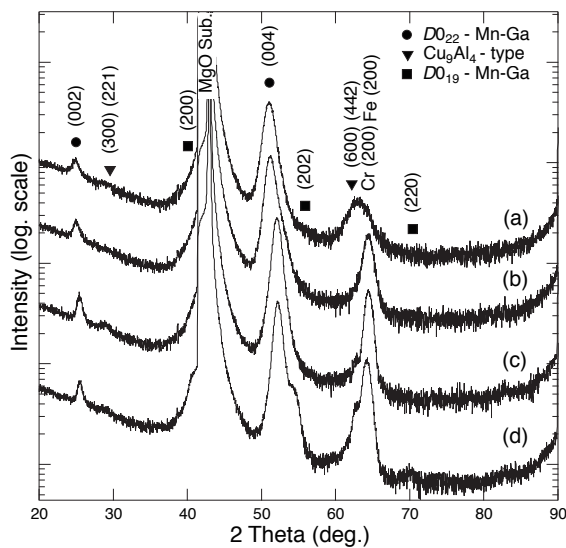


Fig. 5 XRD patterns for $Mn_{2.6}Ga$ on 2-nm Fe buffer (a) without annealing and at (b) $T_a = 300^\circ C$, (c) $400^\circ C$ and (d) $500^\circ C$.

Cr buffer layers. This result suggest that mixing of MnGa with Fe between their interfaces gives rise to the non-magnetic dead layer. The magnetization is decreased at the mixed layer, which agrees with our previous research that shows the drastic decrease of magnetization by adding Fe into MnGa alloys¹⁸⁾.

Figure 5 shows XRD patterns for $Mn_{2.6}Ga$ on the 2-nm Fe buffer (a) without annealing and at (b) $T_a = 300^\circ C$, (c) $400^\circ C$ and (d) $500^\circ C$. DO_{22} - Mn_3Ga (002) superlattice and (004) fundamental peaks were clearly observed from the all samples. As the T_a increased to (c) from (a), the (004) peak was shifted to a high angle side, which suggest that the c-axis turns to short. In addition, the ordering parameter (S_{002}) increased from 0.69 (a) to 0.78 (c). These results lead to a conclusion that annealing process was effective for the improvement of the crystal quality under $T_a = 400^\circ C$.

Figure 6 shows magnetization curves for $Mn_{2.6}Ga$ on the 2-nm Fe buffer (a) without annealing and at (b) $T_a = 300^\circ C$, (c) $400^\circ C$ and (d) $500^\circ C$. As the T_a increased from

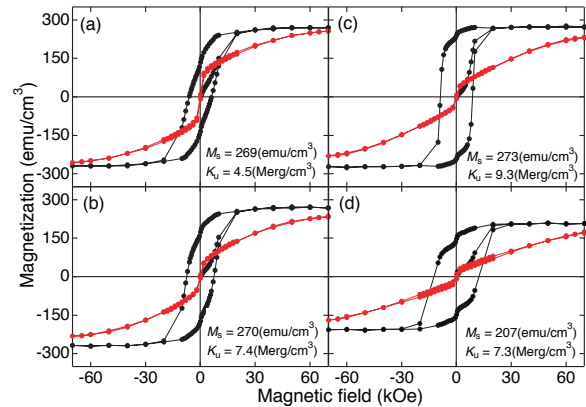


Fig. 6 Magnetization curves for $Mn_{2.6}Ga$ on 2-nm Fe buffer (a) without annealing and at (b) $T_a = 300^\circ C$, (c) $400^\circ C$ and (d) $500^\circ C$. Black and red lines indicate the out-of-plane and in-plane magnetization curves.

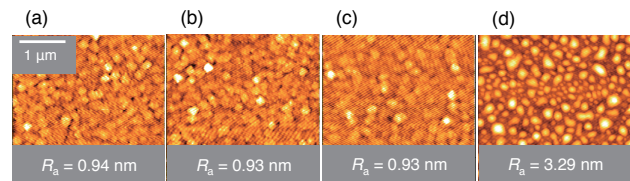


Fig. 7 AFM images for $Mn_{2.6}Ga$ on 2-nm Fe buffer (a) without annealing and at (b) $T_a = 300^\circ C$, (c) $400^\circ C$ and (d) $500^\circ C$.

(a) to (c), the squareness of the $M-H$ curves improved, and value of both M_s and K_u increased. These values, however, decreased at (d). This result is in good agreement with the result of Fig. 5. The highest M_s of 273 emu/cm^3 and K_u of 9.3 $Merg/cm^3$ were obtained at (c).

Figure 7 shows AFM images for $Mn_{2.6}Ga$ on the 2-nm Fe buffer (a) without annealing and at (b) $T_a = 300^\circ C$, (c) $400^\circ C$ and (d) $500^\circ C$. The relatively low surface roughness R_a of 0.93 nm was observed from (b) and (c). In the case of $T_a = 500^\circ C$, R_a became worse.

4. Summary

In this work, dependence of thickness of Fe buffer layers on magnetic properties for $Mn_{2.6}Ga$ thin films have been investigated by using an ultra-high vacuum electron beam (UHV-EB) vapor deposition system. The highest K_u was obtained from the 2-nm Fe buffer. When T_a is increased to $400^\circ C$, improvement of squareness of the $M-H$ curve and increase of M_s and K_u were observed. From the XRD patterns, DO_{22} - Mn_3Ga the (002) superlattice and the (004) fundamental peaks were clearly observed from the all samples. Our result indicates that Fe buffer layers made about 3.5 nm of the magnetic dead layers in MnGa. Because of the existence of about 3.5 nm of magnetic dead layer, the relatively high K_u of 9.3 $Merg/cm^3$ and the low surface roughness R_a of 0.93 nm might be obtained from $Mn_{2.6}Ga$ thin film

on 2-nm Fe buffer at $T_a = 400^\circ\text{C}$. The results in this work suggest that Fe is a suitable buffer layer for application of high K_u D_{022} -MnGa.

Acknowledgements This work was supported in part by the Ministry of Education, Culture, Sports, Science and Technology project for the “Hi-tech Research Center of Tohoku Gakuin University and Grants-in-aid for Scientific Research. Part of this work was supported by “Collaborative Research Based on Industrial Demand” program from Japan Science and technology Agency.

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ウェハレベルの磁性薄膜における高周波磁気透磁率・磁歪定数評価法の開発

In-site measurement of permeability and magnetostriction constant of magnetic films deposited on Si wafers

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A small microstrip-line probe developed to measure the RF properties of magnetic materials for a wide frequency range has been successfully applied to the on-site permeability measurement of thin films on Si wafers. In this study it is applied to determine the saturation magnetostriction constants of films on Si wafers. The results suggest that on-site and simultaneous measurements of permeability and magnetostriction for films on Si wafers are possible.

Key words: magnetostriction, permeability, thin film, Si wafer, tensile stress, magnetostrictive effect

1. はじめに

スマートフォンや PC などの電子デバイスには、多くの薄膜磁性材料が利用されている。これらの薄膜は一般的にウェハ上に製膜され、透磁率や磁歪定数などの磁気特性の計測が必要となる。しかし、磁歪については、光てこ法等による従来の評価方法は、評価用にサンプルを切り出す必要があり、煩雑かつ破壊検査となっている。これに対し、藪上¹⁾らは、非破壊で高周波計測が可能なマイクロストリップライン (Microstripe Line : MSL) 構造のプロープを用いた計測システムを開発してきた。さらに、この MSL プロープを応用して、Si ウェハ基板上的薄膜試料の局所的な透磁率測定を可能にした高周波磁性薄膜評価装置を開発した。本装置ではウェハや角型基板上的磁性薄膜を 40GHz までの透磁率測定が可能であることが報告されている²⁾。

本稿では、このプロープを応用して、透磁率評価と同時に飽和磁歪を計測する方法について報告する。

この計測方法により、ウェハ上の磁性膜の局所的な透磁率と磁歪を非接触、非破壊で同時計測することが可能となり、従来困難であった生産工程における磁性膜評価に有用な方法と考えられる。

2. 磁歪測定原理および計測システム

2-1 磁歪測定原理

本研究における磁歪の計測原理について説明する。

Fig. 1(a)のように、基板上に製膜された磁性薄膜の共鳴周波数 f_{r0} と磁気異方性 H_{kf} の関係は(1)式で表される³⁾。

$$f_{r0} = \frac{\gamma}{2\pi} \sqrt{H_{kf} \cdot 4\pi M_s} \quad (1)$$

これに対して、Fig. 1(b)のように一定の曲率半径 r で基板を湾曲させて磁性膜に張力を印加すると、磁気弾性効果によって共鳴周波数が高周波または低周波にシフトする。この時の共鳴周波数を f_{r+} とすると、(2)式ようになる。

$$f_{r+} = \frac{\gamma}{2\pi} \sqrt{4\pi M_s (H_{kf} + H_{k\sigma})} = \frac{\gamma}{2\pi} \sqrt{4\pi M_s \left(H_{kf} + \frac{3\lambda_s h_s E_f}{2r M_s} \right)} \quad (2)$$

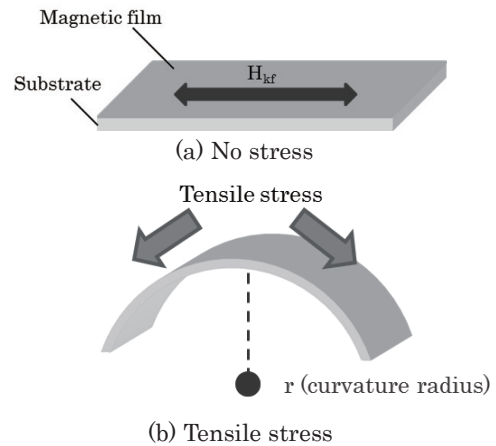


Fig. 1 Stress applied to magnetic thin film.

この時、 M_s は磁性膜の飽和磁化、 E_f は磁性膜のヤング率、 h_s は基板厚、 H_{ko} は磁気弾性効果による異方性磁界、 λ_s が磁性膜の飽和磁歪定数である。外部磁場 H_{ex} を印加した場合、(1)式、(2)式より f_{r0} と f_{r+} は(3)式の関係で表され、(4)式が成り立つ。

$$f_{r+}^2 = f_{r0}^2 + \left(\frac{\gamma}{2\pi}\right)^2 (4\pi M_s + H_{ex}) \left(\frac{3\lambda_s h_s E_f}{2r_+ M_s}\right) \quad (3)$$

$$\frac{f_{r+}^2 - f_{r0}^2}{f_{r0}^2} = \frac{1}{H_{kf} + H_{ex}} \left(\frac{3\lambda_s h_s E_f}{2r_+ M_s}\right) \quad (4)$$

(4)式により、 H_{ex} を掃引して f_{r0} と f_{r+} を測定すると、(4)式の比例係数から λ_s を求めることができる。

2-2 測定サンプル

本実験では 0.2 mm 厚の 4 インチ Si ウェハ基板上面に 500 nm 厚の Co-Zr-Nb 膜を RF スパッタにより製膜した。Fig. 2 では、円弧状の治具に沿わせてウェハを固定し、Co-Zr-Nb 膜に張力を印加している。

2-3 計測システム

Fig. 3 は磁歪計測システムの構成を示したものであり、MSL プロブ、電磁石、レーザー変位計、ネットワークアナライザ(アジレントテクノロジー製 N9928A)および PC から構成される。測定は MSL プロブを厚さ 20 μ m のポリスチレンフィルムを介して測定サンプルに近接配置する。MSL からの高周波磁界は薄膜の張力方向に直交する方向 (Fig.2 の紙面垂直方向) に印加され、高周波磁界方向の磁気共鳴周波数を検出する。この状態で Fig.4 のように電磁石から張力方向に 1100 Oe の直流磁界 (H_{ex}) を印加し、試料の磁化を飽和させた状態でネットワークアナライザをキャリブレーションする。次に H_{ex} を変化させつつ透過係数 (S_{21}) を測定する。この測定を無負荷状態および張力を印加した状態で繰り返し、 S_{21} から共鳴周波数を決定し、(4)式から磁歪 λ_s を算出した。レーザー変位計で測定した応力印加時のウェハの曲率半径 r は、278 mm であった。

なお、本計測の際には、Co-Zr-Nb 膜の作製時に形成された磁気異方性の磁化困難軸方向を MSL の幅方向と平行にした。

3. 測定結果

Fig. 5 は、Co-Zr-Nb 膜に H_{ex} (130~975 Oe) を印加した際の共鳴損失に相当する $(1-S_{21})/S_{21}$ を測定した結果である。破線は無負荷時、実線は張力負荷時を示している。共鳴周波数 f_r は、張力負荷によって、 H_{ex} の増加にともない 30 MHz から 4 MHz 程度、低周波数帯域へシフトしている。Fig. 6 は、Fig.5 のピーク値より求めた、 $(f_{r+}^2 - f_{r0}^2)/f_{r0}^2$ と $1/(H_{kf} + H_{ex})$ の関係をプロットしたグラフである。これらのパラメータは線形関係となっていて、(1)式によるフィッティングが可能である。このフィッティングにより得られた傾きが $(3\lambda_s h_s E_f / 2r M_s)$ にあたるので、 λ_s を算出したところ、 -7.2×10^{-7} であった。

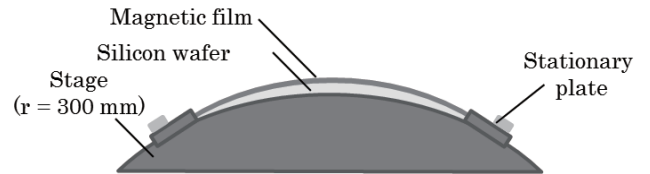
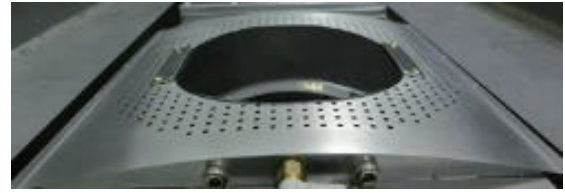


Fig. 2 Measurement sample.

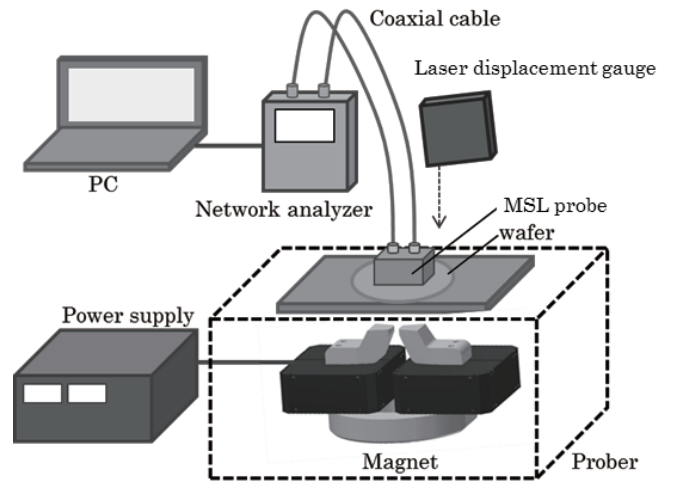


Fig. 3 Magnetostriction measurement system.

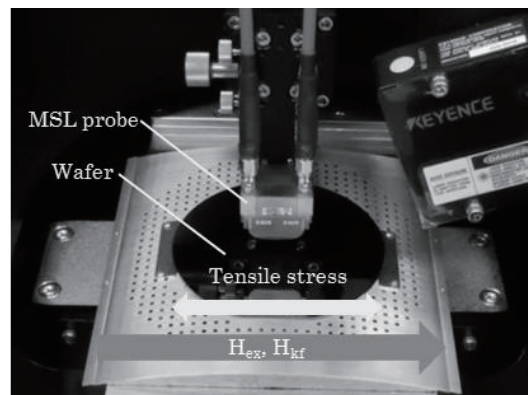


Fig. 4 The state of the measurement.

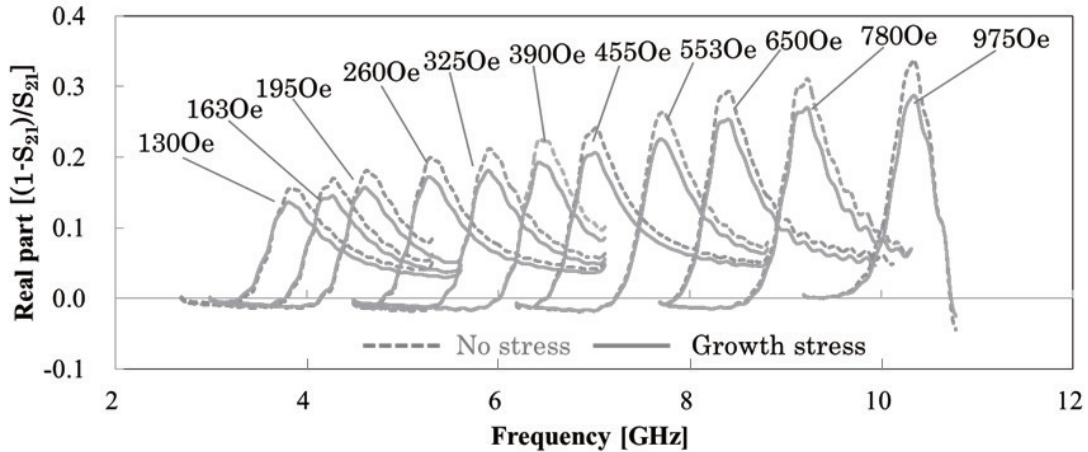


Fig. 5 FMR profiles of 500nm CoZrNb film with and without stress

Table 1 は膜厚 500 nm, 250 nm, 50 nm, の Co-Zr-Nb 薄膜を本測定法および光てこ法(東栄科学製薄膜磁歪測定装置)にて磁歪 λ_s を測定した結果を比較したものである。光てこ法による測定 ⁴⁾は、短冊形ガラス基板の上に Si ウェハ基板と同時作製した Co-Zr-Nb 薄膜を用いて行った。Fig. 7 は Table 1 の本測定法および光てこ法による各膜厚の磁歪測定値を比較したもので、測定値は直線上にあるが、本測定法の値が光てこ法の値に比べて若干小さくなる傾向がある。

4. 考察

本研究の結果から、本測定法について以下の利点が考えられる。

1. 本実験の薄膜試料に制御された応力を印加する方法では、 10^{-9} の測定感度を持ち、従来の光てこ法では困難であった 5nm 以下の極薄の試料も測定対象になる。
2. MSL プローブのサイズ程度の局所的な計測が可能であり、プローブを小型化する事で、MSL の線路長に応じてウェハ上の薄膜の透磁率、磁歪定数の分布が同時測定できる。
3. 本実験で示された、光てこ法による静的な磁歪と磁気共鳴による動的な磁歪に差が生じていることは、実験システムによる誤差、あるいは何等かの物理的理由がある可能性を考察中である。

5. 結言

薄膜試料の応力と強磁性共鳴周波数の関係を利用して磁歪定数を求める方法を提案し、その有用性を検証した。今後の課題として、プローブの小型化と高感度化、測定対象の拡大、実用システムの製作などを準備中である。

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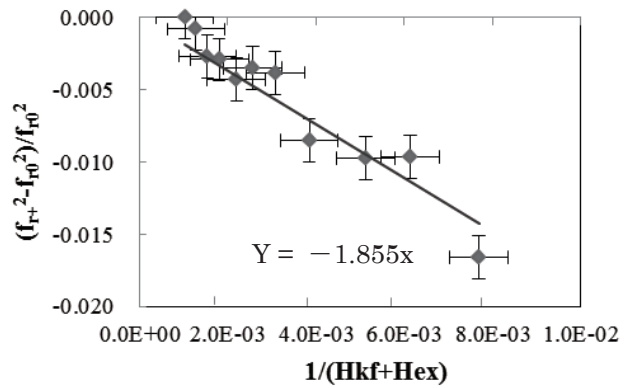


Fig. 6 Magnetic field dependence of resonance frequency.

Table 1 Results for different film thicknesses.

Magnetic film	Film thickness [nm]	λ_s (this measurement)	λ_s (cantilever method)
Co-Zr-Nb	500	-7.19×10^{-7}	-1.36×10^{-6}
Co-Zr-Nb	250	-2.31×10^{-6}	-3.07×10^{-6}
Co-Zr-Nb	50	-4.29×10^{-6}	-6.44×10^{-6}

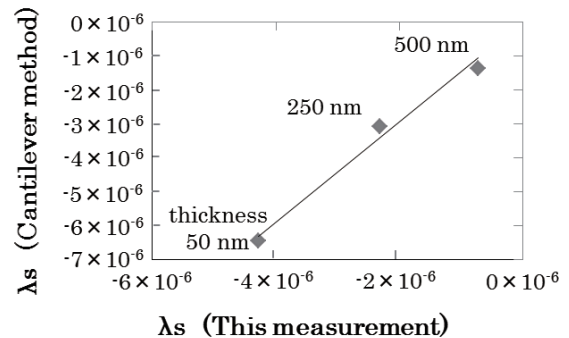


Fig. 7 Comparison between present and cantilever methods.

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