The use of X-Ray Reflectometry for single film thickness analysis in GMR multilayer stacks

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ABSTRACT

The performance of giant magnetoresistive (GMR) sensor heads critically depends on the physical thicknesses of all functional layers involved in the so-called Spin-Valve stack, and consequently has to be controlled via very accurate thickness analysis methods. In this paper we demonstrate that X-Ray Reflectometry (XRR) analysis of the full GMR multilayer stack provides an absolute means to extract selected single film thicknesses with Angstrom accuracy. Secondly, we present XRR data recorded from special reduced layer sequences which allow to determine the deposition rates of Co, NiFe, Cu and Ru with an error of better than 0.04 Å/sec. We further show how for the analysis of GMR full stacks the XRR technique can be usefully combined with X-Ray Fluorescence (XRF) to measure the individual thicknesses of all layers.

INTRODUCTION

The rapid annual increase of the areal recording density of magnetic hard disk drives has led to the development of a new generation of sensor heads, called GMR or Spin-Valve sensors [1]. The GMR effect is based on the spin-dependent scattering and reflection of the conduction electrons. Consequently for the design of Spin-Valve devices, the choice of the appropriate crystallite size and orientation plays an important role as well as individual layer thicknesses and interfacial roughnesses.

A typical Spin-Valve as shown in Fig. 1a consists of two ferromagnetic layers separated by a nonferromagnetic metal layer. The magnetization of the pinned layer (PL) is firmly held in place by a strong antiferromagnet (e.g. NiO, FeMn), whereas the magnetic moment of the free-layer (FL) may rotate freely.

![Diagram](image)

Fig. 1: a) functional layers of a simple Spin-Valve stack. b) AP-pinned layer sequence.
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The GMR sensor exhibits a resistivity change $\Delta R/R$ when the FL magnetization follows the magnetic field of the thin film disk and thus changes its relative orientation to the PL magnetization. On a lab scale $\Delta R/R$ of more than 20% have been achieved with multilayer systems [2], whereas current GMR products using the ‘simple’ Spin-Valve trilayer structure typically show a $\Delta R/R$ of the order of 5%-9%.

The choice of the right thickness for the conducting spacer layer (typically Cu) is a trade-off between two effects: a thinner Cu layer results in a higher $\Delta R/R$, but also increases the magnetic interaction between free and pinned layer which is unfavorable for an easy rotation of the FL magnetization. To stabilize pinning and to minimize the net external field caused by the pinned layer the anti-parallel pinned (AP pinned) structure, which is illustrated in Fig. 1b, was introduced. The thickness of the Ru spacer layer critically determines the relative magnetic orientation of the two pinned layers [3]. All in all, for an optimum performance of the Spin Valve sensor stack a highly accurate thickness control for all sensor layers is required.

Previous studies of FeMn-based Spin-Valves without AP-pinning showed that XRR is a powerful method to control the individual thicknesses of all films contained in the full stack as well as to monitor the evolution of interfacial widths during successive annealing processes [4], [5]. In this paper, XRR data recorded from AP-pinned IBM GMR prototype sensors deposited on monitor coupons are shown. Capabilities and limitations of the XRR analysis of the Spin-Valve full stack are discussed. We report XRR studies of special reduced layer sequences designed to determine the deposition rates of all functional layers with a sub-Å accuracy. XRR is a thickness probe that serves as a calibration tool of indirect monitoring methods, e.g. XRF. The last section illustrates the complementarity of the thickness information obtained from a combined XRF and XRR analysis of GMR full stacks.

**EXPERIMENTAL**

The multilayer systems were prepared via ion beam deposition on 3.8 mm thick glass substrates (substrate rms roughness: ca. 5 Å). Earlier attempts with deposition on pure Si wafers led to the formation of Ni-Silicide and Ni-deficient Ni-Oxide interfacial layers which made an interpretation of the experimental XRR patterns difficult.

XRR data were collected with a Siemens D5000 XRD system equipped with a 2.2 kW long fine focus sealed tube source. To obtain a parallel and monochromatic beam (Cu Kα radiation, $\lambda = 1.5406$ Å), a collimating parabolic multilayer optic (Goebel mirror) is used. With a 0.05 mm exit slit on the primary side and a 0.2 mm anti-scatter plus a 0.1 mm detector slit on the secondary side, a dynamic intensity range of ca. 7 orders of magnitude is accessible. In the region of total external reflection a 0.1 mm Cu absorber is inserted to avoid dead time effects of the scintillation counter. For the analysis of all systems described in the following sections a step width of $\Delta 2\theta = 0.01^\circ$ was used.

Data evaluation was carried out with the commercial Bede REFS Mercury software package [6]. It employs the optical matrix method, based on the expressions of Parratt [7] and Névot and Croce [8], for the analytical calculation of XRR curves of layered systems. From a best fit of the experimental data - i.e. from the minimization of a cost function $\chi^2$ - individual film thicknesses, roughnesses and densities of a multilayer stack can be extracted.
To appropriately take into account the high angular part of the XRR patterns, an absolute log weighting of the difference between simulated and experimental data was chosen. For a better consistency of the fit results and a minimization of the number of fit parameters, the individual film densities were kept fixed at nominal values during the fit, i.e. only thickness and roughness values were optimized.

**XRR ANALYSIS OF GMR FULL STACK SAMPLES**

Fig. 2 shows the complete Spin-Valve layer sequence studied here and its corresponding electron density profile. The antiferromagnetic NiO film exhibits a strong coupling with the neighboring Co, pinned layer film and thus pins the direction of the PL (and consequently the Co AP-pinned layer) magnetization. The insertion of a nano-layer (Co) between the Cu spacer and the free layer has proved to enhance the GMR effect [2].

![Diagram of Spin-Valve stack and corresponding electron density profile](image)

*Fig. 2: Spin-Valve stack and corresponding electron density profile (unscaled).*

X-rays are reflected only at interfaces between materials with a sufficient electron density contrast. Consequently for the Spin-Valves presented in Fig. 2 interfaces separating adjacent Co, NiFe, and Cu films do not contribute to the XRR pattern, and only a total (Co+Cu+Co+NiFe) thickness under the reflective Ta cap layer is accessible via XRR. Thus, taking into account a natural Oxide layer Ta$_2$O$_5$ on top of the Spin-Valve stack, a 6-layer structure on glass is investigated.

A typical XRR pattern recorded from a GMR stack and its best fit are shown in Fig. 3. An excellent agreement between experimental data and theoretical simulation was obtained with the fit parameters listed in the Table 1.
The error for the measured individual thickness values is of the order of $\pm 1$ Å. For the error estimate, in the Bede REFS Mercury software each fit parameter is varied around its best fit value while keeping the rest of the parameter set fixed. Low and high end error bounds are identified as those values where the cost function $\chi^2$ has increased by 5%.

<table>
<thead>
<tr>
<th>layer material</th>
<th>thickness [Å]</th>
<th>roughness [Å]</th>
<th>density [g/cm³]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Glass</td>
<td>-</td>
<td>5.3</td>
<td>2.2</td>
</tr>
<tr>
<td>NiO</td>
<td>464</td>
<td>3.1</td>
<td>6.7</td>
</tr>
<tr>
<td>Co</td>
<td>35</td>
<td>3.8</td>
<td>8.8</td>
</tr>
<tr>
<td>Ru</td>
<td>9.6</td>
<td>2.7</td>
<td>12.3</td>
</tr>
<tr>
<td>Co+Cu+Co+NiFe</td>
<td>128</td>
<td>8.6</td>
<td>8.8</td>
</tr>
<tr>
<td>Ta</td>
<td>32</td>
<td>4.5</td>
<td>16.6</td>
</tr>
<tr>
<td>Ta$_2$O$_5$</td>
<td>31</td>
<td>5.9</td>
<td>8.5</td>
</tr>
</tbody>
</table>

Tab. 1: Best fit parameters for XRR curve from IBM GMR stack (cf. Fig. 3)

The weekly XRR analysis of similar Spin-Valve monitor specimens has been established as an important control of the IBM sensor production as well as of in-line thickness probe methods. However, as was discussed in this section, not each individual film thickness can be extracted from the XRR characterization of the full Spin-Valve stack.

**XRR ANALYSIS OF REDUCED LAYER SEQUENCES**

To determine the individual deposition rates of all functional layers of the GMR structure via XRR, it is necessary to produce special experiment samples with reduced layer sequences, i.e. only selected parts of the Spin-Valve full stack. The following tables 2a and 2b summarize the
XRR analysis results of a Ru and NiFe thickness matrix experiment designed by IBM location Mainz head manufacturing.

<table>
<thead>
<tr>
<th>Ru deposition time [s]</th>
<th>total Co+Ru+Co thickness [Å]</th>
<th>Ta thickness [Å]</th>
<th>Ta₂O₅ Thickness [Å]</th>
</tr>
</thead>
<tbody>
<tr>
<td>16</td>
<td>55.2</td>
<td>34.2</td>
<td>29.9</td>
</tr>
<tr>
<td>18</td>
<td>55.9</td>
<td>34.5</td>
<td>30.3</td>
</tr>
<tr>
<td>20</td>
<td>56.6</td>
<td>34.5</td>
<td>30.4</td>
</tr>
<tr>
<td>40</td>
<td>64.7</td>
<td>34.7</td>
<td>29.5</td>
</tr>
</tbody>
</table>

Tab. 2a) Ru sweep: 57s Co + var. Ru + 57s Co + 167s Ta on glass substrate

<table>
<thead>
<tr>
<th>NiFe deposition time [s]</th>
<th>total Cu+NiFe thickness [Å]</th>
<th>Ta thickness [Å]</th>
<th>Ta₂O₅ Thickness [Å]</th>
</tr>
</thead>
<tbody>
<tr>
<td>22</td>
<td>36.2</td>
<td>29.2</td>
<td>31.5</td>
</tr>
<tr>
<td>96</td>
<td>70.6</td>
<td>33.9</td>
<td>30.1</td>
</tr>
<tr>
<td>131</td>
<td>89.5</td>
<td>33.6</td>
<td>30.0</td>
</tr>
</tbody>
</table>

Tab. 2b) NiFe sweep: 40s Cu + var. NiFe + 167s Ta on glass substrate

XRR showed the best sensitivity to the total thickness values given in the two tables. Plots of these highly accurate thicknesses vs. deposition times (cf. Fig. 4b) show a well-defined linear behavior, indicating constant deposition rates for all film materials.

Fig. 4: a) XRR curve (×) and best fit (—) of a Co+Ru+Co+Ta layered system, Ru deposition time = 40 s. b) Ru thickness sweep: plot of total (Co+Ru+Co) thickness vs. Ru deposition time.
Consequently from linear fits of the curves deposition rates of Ru, Co, NiFe and Cu were calculated, using the slopes and the y-intercepts divided by the respective deposition times:

- Ru: \(0.40 \pm 0.01\) Å/s
- Co: \(0.43 \pm 0.01\) Å/s
- NiFe: \(0.49 \pm 0.02\) Å/s
- Cu: \(0.63 \pm 0.04\) Å/s

It is noted that for the study of GMR full stacks, XRF was successfully calibrated via XRR. For the calibration, similar individual film thickness sweep experiment specimens as described in this chapter were analyzed by XRR and XRF.

**COMBINATION OF XRR AND XRF FOR GMR FULL STACK ANALYSIS**

The results of an XRF analysis of the Spin-Valve stack displayed in Fig. 2 are complementary to the thickness information obtained via XRR.

First of all, the following effective thickness values can be determined via XRR and XRF, respectively:

\[
\begin{align*}
\text{XRF: } t_{\text{eff}} & = Co_1 + Co_2 + Co_3 \\
\text{XRR: } t_{\text{eff}} & = Co_2 + Cu + Co_3 + \text{NiFe}
\end{align*}
\]

In addition, XRF allows to directly measure the Cu spacer thickness, whereas the Ru thickness can be independently determined either via XRR or XRF. A combination of the two techniques yields the free layer thickness \(\text{NiFe} - t_{\text{NiFe}}\), which can also be checked with XRF alone by measuring the Fe intensity and by using the result of a previous analysis of the NiFe composition. It is justified to assume the measured Co rate to be constant throughout one deposition run which allows to calculate the \(Co_1\), \(Co_2\) and \(Co_3\) thicknesses from their respective deposition times.

Thus, from a combined application of XRR and XRF for GMR full stack analysis the deposition rates and hence individual thicknesses of all functional layers can be extracted.

**SUMMARY**

The XRR analysis of prototype AP-pinned GMR sensor stacks deposited on glass substrates has proved to be an accurate control tool of the individual film thicknesses of Ru and NiO, Ta and Ta₂O₅. For the XRR data evaluation of the complicated 6-layer sequences the Bede REFS Mercury analysis software was successfully applied. XRR studies of thickness sweep samples using functional parts of the Spin-Valve multilayer sequence helped to separate the deposition rates of Co, NiFe, Cu and Ru with an error of better than 0.04 Å/s.

XRR is a highly versatile absolute film thickness probe, which in the magnetic storage production is routinely used for the calibration of quicker in-line monitoring methods as e.g. XRF. Furthermore, the thickness information gathered by XRR and XRF studies of GMR full stacks is complementary, and a combination of the two methods allows to determine all individual film deposition rates.
REFERENCES