LASER-WRITTEN DOMAIN OBSERVATION USING HIGH RESOLUTION LORENTZ MICROSCOPY

S. YATSUYA and J. H. SEXTON

3M Co., Basic Materials Lab, St. Paul, MN 55144, USA

Abstract - The performance of future generations of MO (Magneto-Optical) recording media will depend more strongly on domain edge features because of pulse width modulation (PWM) encoding requirements and higher domain densities. Direct observation of these features would help greatly in correlating media performance to domain characteristics. This paper describes domain observation using Lorentz transmission electron microscopy (TEM) with a new sample preparation method; domains were dynamically written in thin films deposited on a large salt crystal substrate. Domains written both in a strongly on domain edge features because of pulse width modulation writing conditions were found. A resolution of near microscopy (TEM) with a new sample preparation method; domains were dynamically written in thin films SiN/TbFeCo/SiN/AI-Cr thin domain characteristics. This paper describes domain observation using Lorentz transmission electron microscopy for domain observation on media, the control of domain shapes and domain edge features is of paramount importance. The control of domain formation is dependent both on media properties and on record conditions. Direct observation of domain features would help greatly in correlating media properties and record conditions to domain characteristics. This paper describes a useful technique for making direct observation of domain features and discusses our current results.

There are several methods for detecting magnetic domains: the Bitter method [1], polarized light microscopy[2], Lorentz TEM [3], Lorentz scanning electron microscopy (SEM) [4], Lorentz STEM [5], spin-polarizing SEM [6], Interference Electron Micrography [7], and Magnetic Force Microscopy (MFM) [8]. The Bitter method and polarized light microscopy do not detect details of magnetic domains of less than 0.5 μm. The characterization of domains in M-O thin films, however, requires a 0.05 μm resolution.

In this report we chose Lorentz TEM for domain observation. Suits et al. [9] has reported on the use of Lorentz TEM for domain observation on thin films grown on carbon/mica substrates. A static tester was employed by Suits for writing domains. Other investigators have reported on Lorentz TEM observation of domains written by dynamic testers. The sample substrates used in those investigations included a silicon wafer with a grid of 80 μm x 80 μm thin film windows of SiN [10], and evaporated polycrystalline salt films on resinous disk substrates [11].

In this investigation a dynamic tester was employed for writing the magnetic domains. Sample preparation for Lorentz TEM observation of dynamically written domains can be very difficult depending on the approach. In this experiment we attempted to simplify this step by using a single NaCl crystal plate as a thin film substrate. The plate was large enough for use with a dynamic test-bed for writing the domains. Domains were written using a non-tracking (focus-only) optical head. Recorded domains were clearly seen with a resolution of 10 nm. One of the advantages of our method is the ease of sample preparation.

EXPERIMENTAL

Both Lorentz TEM and TEM were carried out to investigate magnetic domain features as well as physical microstructure. A JEOL200CX equipped with a Lorentz microscopy pole-piece (AMG2) for the objective lens was used. The maximum magnification reached around x 90,000. The "Out-of-Focus Method" was used in this experiment [3]. Contrasts of domains imaged using Lorentz microscopy for M-O thin films were strongly dependent on the angle between the normal to the film plane and the incident electron beam [9]. The tilt angle used was 30°. The defocus distance was 1 to 2 mm and the incident beam parallelism angle was 10^-5 radians [12]. The TEM was operated at 200 kV.

Two kinds of film stacks were deposited on a
single salt crystal (100) substrate (50 x 50 x 3 mm): One was a tri-layered thin film, SiN (15 nm) / TbFeCo (50 nm) / SiN (15 nm) and the other one was a quadri-layered one, SiN (70 nm) / TbFeCo (50 nm) / SiN (10 nm) / Al-Cr (50 nm). The thicknesses of the quadri-layered thin film were optimized using a thin film model [13]. The TbFeCo layers were dc magnetron sputtered at an Ar pressure of 2 mTorr and the SiN dielectric layers were RF sputtered at an Ar pressure of 2 mTorr. The TbFeCo film was made using iron, cobalt, and terbium sputtering targets.

The composition of the film was measured by X-ray fluorescence spectroscopy to be 77.3 at.% Fe, 4.7 at.% Co and 18.0 at.% Tb. Magnetic properties were measured by a vibrating sample magnetometer. The film had a perpendicular remanent moment with a saturation magnetization of 232 emu/cm$^3$ and a coercivity of 2.89 x 10$^3$ Oe at room temperature.

Magnetic domains were written on the film stacks with an 830 nm M-O disk recorder configured for air-incident recording (as opposed to conventional substrate-incident recording). Domains were written at a sample velocity of 2 m/s using laser pulse frequencies from 0.5 to 2.0 MHz. The write powers ranged from 8 mW to 12 mW with applied fields of 100 Oe and 200 Oe. Domain sizes ranged between 0.4 μm and 3 μm.

After writing domains with a dynamic tester, small pieces of the films were floated off the NaCl substrate in water and placed on TEM grids. These films were then used as samples for Lorentz TEM.

RESULTS AND DISCUSSION

![Lorentz micrograph](image)

Figure 1 shows Lorentz micrographs of domains which were under-focused. The domains were written on the thin film stack of SiN/TbFeCo/SiN using the conditions from (a) to (e) shown in Table 1. The written domain shapes ranged from oblong at long pulse lengths to round at shorter pulse lengths. The extraneous line-like domains are associated with radial positioning during erasure and the dot-like domains are likely due to film defects associated with the NaCl substrate.

![Lorentz micrograph](image)

Fig. 1 Lorentz micrographs of domains written on the thin film stacks of SiN/TbFeCo/SiN in recording conditions from (a) to (e) in Table 1.

Figure 1a and 1b show domains written at write fields of 100 Oe and 200 Oe, respectively. Both sets of written domains look similar, indicating that writing is occurring in the plateau region of the write field response. Reversed domains in the written domains were sometimes found (Fig. 1a and b).

The write power threshold for the tri-layer films was slightly less than 8 mW. With the write power set to 8 mW, the written

<table>
<thead>
<tr>
<th>Write power (mW)</th>
<th>Frequency (MHz)</th>
<th>Write field (Oe)</th>
<th>Duty cycle (%)</th>
<th>Domain length (μm)</th>
<th>Domain width (μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>a</td>
<td>12</td>
<td>0.5</td>
<td>100</td>
<td>40</td>
<td>1.9</td>
</tr>
<tr>
<td>b</td>
<td>12</td>
<td>0.5</td>
<td>200</td>
<td>40</td>
<td>1.9</td>
</tr>
<tr>
<td>c</td>
<td>8</td>
<td>0.5</td>
<td>200</td>
<td>40</td>
<td>Sub-domains</td>
</tr>
<tr>
<td>d</td>
<td>12</td>
<td>1.0</td>
<td>200</td>
<td>40</td>
<td>1.2</td>
</tr>
<tr>
<td>e</td>
<td>12</td>
<td>2.0</td>
<td>200</td>
<td>40</td>
<td>0.7</td>
</tr>
<tr>
<td>f</td>
<td>12</td>
<td>2.0</td>
<td>200</td>
<td>26</td>
<td>0.4</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Quadri-layered thin films: SiN/TbFeCo/SiN/Al-Cr/NaCl</th>
</tr>
</thead>
<tbody>
<tr>
<td>g</td>
</tr>
<tr>
<td>h</td>
</tr>
</tbody>
</table>
domains ((c) in Fig. 1 and Table 1) were composed of numerous sub-domains. A high resolution Lorentz micrograph of the domains written at 8 mW is shown in Fig. 2. The sizes of numerous sub-domains as indicated by arrows were from 100 nm to 500 nm in size. These sub-domains resemble those produced due to the demagnetization field effect [9].

![Fig. 2 Domains written at 8 mW of detail using high resolution Lorentz microscopy.](image)

For conditions (b), (d), and (e) the domain lengths and widths both decreased as the write pulses became shorter. The written domains shown in (e) in Fig. 1 sometimes showed smooth edges and sometimes irregular edges, depending on the positions. The irregular edge shapes appear to be associated with domain wall pinning at the dot-like domain sites (i.e. defect sites).

It was possible to produce very small domains by reducing the duty cycle to 26%. These domains were 0.4 μm across as shown in Fig. 3. The edges of the domains look smooth.

![Fig. 3 Domains of 0.4 μm across using the duty cycle of 26%, written at 12 mW and at 200 Oe.](image)

Figure 4 illustrates that the written domain shapes, and especially domain edge features, are clearly seen with the resolution of around 10 nm. Fig. 4 also shows magnetic domain walls marked by "A" and microstructures, probably columnar structures marked by "B". It was found that both magnetic contrasts and microstructures were imaged at the same defocus distance. This demonstrates the possible use of this technique for investigating the dependence of domain edge characteristics on physical microstructure.

![Fig. 4 Domain walls marked by A and microstructures marked by B.](image)

Domains were also recorded and observed on a quadri-layered film stack, SiN/TbFeCo/SiN/Al-Cr ((g) and (h) in Table 1), and are shown in Fig. 5. The shapes of the written domains here are much more uniform and are tear drop shaped in contrast to those of the tri-layered thin film stacks as shown in Fig. 1. The domains are also longer and wider than the

![Fig. 5 Recorded domains in the thin film stack of SiN/TbFeCo/SiN/Al-Cr: The five rows of domains marked by "A" were written at the write power of 12 mW and the following three rows marked by "B" of domains were recorded at 10 mW. The write field was 100 Oe.](image)
domains in the tri-layered thin film. The edges of domains looked smooth and the inside of the domains contained small reversed domains. The difference of domain shapes between quadri-layered and tri-layered film stacks results from the different thermal properties with the presence of the Al-Cr layer. Also the Al-Cr may act as a physical barrier preventing film interactions with NaCl — note the absence of the dot-like domains as seen in Fig.1.

SUMMARY

We have demonstrated a new technique with which dynamically laser-written domains were examined by Lorentz microscopy. The resolution of magnetic contrasts is around 10 nm which is good enough to characterize written domain shapes and the domain edges in M-O thin films. We have also shown that domain walls and microstructures in thin films were observed at the same time. It was demonstrated that domains written both on tri-layered thin films and quadri-layered ones looked very different.

ACKNOWLEDGMENTS

The authors wish to thank T. Arii of National Institute of Physiological Sciences, Japan for his useful discussions on Lorentz microscopy. The authors are grateful for M. B. Hintz for helping us by supplying many samples in this study, for W. A. Challener for optimizing the quadri-layered stack using his thin film model and for D. R. Callaby for preparing one of our samples. Many thanks go to M. Kersker and M. Kawasaki of JEOL-US for locating the AMG2 pole-piece.

REFERENCES