Development of alternating magnetic force microscopy: Local magnetic domain analysis by advanced magnetic field imaging with high functionalities for high performance magnets

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For further development of high performance magnets, magnetic domain observation is important to study the relationship between the nanostructure and hard magnetic properties. To observe the magnetic domain structure of magnets, technique with a wide range of imaging area from nm scale to μm scale is required for domain boundary and inside parts magnetic grains. Recently, the magnetic domain observation of NdFeB sintered magnets by X-ray magnetic circular dichroism microscopy revealed that the coercivity of magnetic grains on the grain boundary fractured surface was higher than that on a polished surface and was similar to that of the bulk magnet [1]. Therefore, magnetic domain observation of rough fractured surface is highly desired.

Magnetic force microscopy (MFM) is a technique widely used to investigate the fine magnetic domain structure with relatively high spatial resolution. In order to improve the spatial resolution of MFM, decreasing tip-sample distance is quite important. However, conventional MFM has a difficulty to reduce the tip-sample distance because of topography artifacts near sample surface where short-range forces, such as van der Waals force are dominant. To solve the problem, we have developed alternating magnetic force microscopy (A-MFM). This enables near-surface imaging of DC and AC magnetic fields with high spatial resolution of less than 5 nm by using our developed sensitive ferromagnetic tips [2-3]. Here the definition of spatial resolution is the half of the minimum wavelength where MFM magnetic signal reaches white noise level for the A-MFM image. A-MFM utilizes frequency modulation of a cantilever oscillation generated by an off-resonance alternating magnetic force between a magnetic tip and a magnetic sample.

Table 1 shows the characteristics of conventional MFM and Alternating MFM. The A-MFM has more functionalities than conventional MFM. For detecting DC magnetic field, A-MFM uses AC magnetic field to drive the tip with periodically changing magnetic moment \( M^a(t) \cos(\omega_d t) \). For high performance magnets, it is noteworthy that the tip should not be magnetically saturated by magnetic field from the sample. Therefore, A-MFM utilizes frequency modulation of a cantilever oscillation generated by an off-resonance alternating magnetic force between a magnetic tip and a magnetic sample.

![Image of a topographic image, A-MFM signed image of DC magnetic field gradient, and A-MFM polarity image of DC magnetic field gradient for a fractured surface of demagnetized anisotropic Sr ferrite sintered magnet.](image)

**Table 1. Characteristics of conventional MFM and Alternating MFM.**

<table>
<thead>
<tr>
<th>Characteristic features</th>
<th>Conventional MFM</th>
<th>Alternating MFM</th>
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<tbody>
<tr>
<td>Magnetic field measurement near sample surface</td>
<td>×</td>
<td>○ [All magnetic tips]</td>
</tr>
<tr>
<td>Separated detection of magnetic field (including short range forces)</td>
<td>×</td>
<td>○ [All magnetic tips]</td>
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<tr>
<td>Polarity &amp; zero detection of magnetic field</td>
<td>×</td>
<td>○ [All magnetic tips]</td>
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<tr>
<td>Vector magnetic field measurement</td>
<td>×</td>
<td>○ [Soft magnetic tip]</td>
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<tr>
<td>Stroboscopic measurement of AC magnetic field</td>
<td>×</td>
<td>○ [Hard magnetic tip]</td>
</tr>
<tr>
<td>Fixed measuring direction of magnetic field</td>
<td>×</td>
<td>○ [Superparamagnetic tip]</td>
</tr>
<tr>
<td>Precise magnetic field measurement on rough surface</td>
<td>×</td>
<td>○ [Superparamagnetic tip]</td>
</tr>
<tr>
<td>Simultaneous imaging of DC &amp; AC magnetic field (Spectroscopic measurement of magnetic field)</td>
<td>×</td>
<td>○ [Soft magnetic tip] [Superparamagnetic tip]</td>
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<tr>
<td>Spatial resolution (half of the minimum detectable wavelength)</td>
<td>&gt; 10 nm (Necessary for vacuum atmosphere.)</td>
<td>&lt; 5 nm (Air atmosphere is OK.) [Soft magnetic tip] [Hard magnetic tip]</td>
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Fig. 1 (a) topographic image, (b) A-MFM signed image of DC magnetic field gradient, (c) A-MFM polarity image of DC magnetic field gradient for a fractured surface of demagnetized anisotropic Sr ferrite sintered magnet.
we have recently developed a sensitive FeCo-Gd$_2$O$_3$ superparamagnetic tip. A-MFM with a superparamagnetic tip enables the precise imaging of near-surface static magnetic field with a fixed direction parallel to the tip magnetic moment driven by AC magnetic field. Even rough fractured surface can be imaged in this way.

Fig. 1 shows A-MFM images on a fractured surface of demagnetized anisotropic Sr ferrite magnet. Fig. 1(a), (b) and (c) are the topographic image, the signed intensity image of DC magnetic field gradient (Lock-in X image) and polarity image of DC magnetic field gradient (Lock-in θ image), respectively. Lock-in amplifier signals of A-MFM as follows.

\[
X + iY = R \exp(i\theta) \propto M^x_m \cos(\omega_m t)(\partial^2 H^x_m / \partial z^2) + i M^y_m \sin(\omega_m t)(\partial^2 H^y_m / \partial z^2)
\]

\[
R = \sqrt{X^2 + Y^2} \propto M^z_m (\partial^2 H^z_m / \partial z^2) \propto (\partial^2 H^z_m / \partial z^2)
\]

\[
H^z_m \Rightarrow -H^z_m, \quad X = M^x_m (\partial^2 (-H^z_m) / \partial z^2) \cos(\omega_m t) = M^x_m (\partial^2 H^z_m / \partial z^2) \cos(\omega_m t \pm \pi)
\]

Here X, R and θ signals correspond to in-phase signed magnetic field gradient, intensity of unsigned magnetic field gradient (absolute value of magnetic field) and phase of magnetic field gradient (The θ change of π corresponds to the polarity change of magnetic field gradient and surface magnetic charge), respectively.

A-MFM can clearly observe DC magnetic field gradient and polarity change of surface magnetic charges in Fig.1(b) and (c) even on the fractured surface of which surface roughness is about 1 μm. On the other hand, the interpretation of conventional MFM image is not easy due to the topography artifact.

The superparamagnetic tip can also solve the problem of ferromagnetic tip that the strong magnetic force of the ferromagnetic tip in high magnetic field from the sample deteriorates the control of constant tip-sample distance near the sample surface. However, the moderate magnetization of superparamagnetic tip prevents its magnetic snapping to the sample surface.

Simultaneous imaging of DC and AC magnetic field by A-MFM is also valid to understand the magnetic homogeneity of magnets by changing the amplitude of AC magnetic field to sample space including a tip and a sample. The magnetic imaging of AC magnetic susceptibility at magnetically reversal area becomes possible.

Fig. 2 shows A-MFM unsigned intensity images of DC and AC magnetic field gradients (Lock-in R images of \(\omega_m\) and 2 \(\omega_m\) (\(\omega_m\) : AC magnetic field frequency) under external AC magnetic field with the amplitude of 0.2 and 1.0 kOe for the fracture surface of demagnetized anisotropic Sr ferrite sintered magnet. By using the lock-in R signal of 2 \(\omega_m\) (\(\propto M^z_m (\partial^2 H^z_m / \partial z^2)\)), the grains having reversible magnetization and generating AC magnetic field can be imaged. With the increase of external AC magnetic field amplitude from 0.2 kOe to 1.0 kOe, the number of magnetically reversible grain increases. These grains have a large scale distribution of magnetic field intensities, which source is unvaried magnetization in external AC magnetic field. Simultaneous imaging of DC and AC magnetic field is thought to be useful for analyzing the magnetic inhomogeneity analysis.

In conclusion, our developed A-MFM with the superparamagnetic tip can provide precise magnetic field imaging with a fixed magnetic direction. It is thought to be quite effective method to analyze local magnetic domain structure of various permanent magnets.

ACKNOWLEDGMENT

This work was supported by JST-SENTAN. We thank Hitachi Metals, Ltd. for providing the sample of anisotropic Sr ferrite sintered magnet.

References