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Magnetic domain observation of hydrogenation disproportionation desorption recombination processed Nd–Fe–B powder with a high-resolution Kerr microscope using ultraviolet light

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A Kerr microscope that uses ultraviolet (UV) light for high-resolution domain observation was built, and the domain structure and magnetization process of hydrogenation disproportionation desorption recombination (HDDR) powder were examined. The UV Kerr microscope could observe nanometer-sized domain patterns. Applying a dc field of 1.0 kOe to HDDR powder at a desorption recombination (DR) time of 12 min produced abrupt wall motion. The pinning force exerted by the grain boundaries is inadequate for producing high coercivity because the Nd-rich phase layers along these boundaries are absent at a DR time of 12 min. For HDDR powder at a DR time greater than 14 min, changing the magnetic field by up to 1.0 kOe produced no observable wall motion. It follows that the high coercivity of HDDR powder is due to domain wall pinning at the grain boundaries. © *2010 American Institute of Physics*. [doi:10.1063/1.3339816]

I. INTRODUCTION

The hydrogenation disproportionation desorption recombination (HDDR) process was developed as a method for producing high-coercivity Nd–Fe–B powder.[1] The average grain size of the $Nd_2Fe_{14}B$ phase is about 300 nm, almost identical to the critical size of a single magnetic domain of this hard magnet.[2,3] Despite the fine grain size, the coercivity reported for HDDR powder is much lower than expected from magnetically isolated single-domain particles. It is therefore necessary to establish the relationship between the microstructure and domain configurations through domain observation. Domain observation of Nd–Fe–B magnets has been reported by using magnetic force microscopy and transmission electron microscopy in the Lorentz mode among others.[4,5] However, it is not easy to study the evolution of magnetic domains in external magnetic fields and at high temperatures. Kerr microscope is useful for *in situ* domain observation in a high magnetic field at high temperatures;[6,7] however, its resolution is insufficient for observing fine-grain HDDR powder. In this study, we developed a Kerr microscope that uses ultraviolet (UV) light for high-resolution domain observation and examined the domain structure and magnetization process of HDDR powder. This study uses wide-field illumination and video detection with a rapid raster, instead of spot focused illumination with a slower mechanical raster, as reported regarding high resolution Kerr microscopy in Refs. 8 and 9.

II. EXPERIMENTAL

A Kerr microscope was modified by incorporating optical elements for UV light, as shown in Fig. 1. The theoretical image resolution of an optical microscope is given by the Rayleigh criterion

$$R = 0.61\frac{\lambda}{\mathrm{NA}}, \tag{1}$$

where λ is the wavelength and NA is the numerical aperture of the objective lens.[10] Because a wavelength of 365 nm and an NA of 1.45 were used in the UV Kerr microscope, its theoretical image resolution was about 150 nm. We used a Xe–Hg lamp as a high-intensity UV light source, Glan–Taylor prisms as a polarizer and an analyzer for UV light, and a dielectric multilayer mirror as a 365 nm wavelength filter. UV Kerr images were captured using an intensified

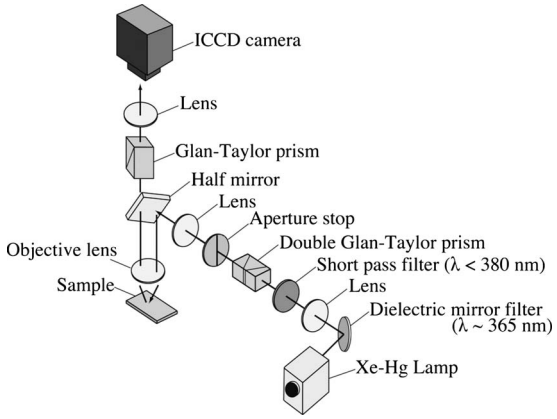

FIG. 1. Schematic diagram of the UV Kerr microscope.

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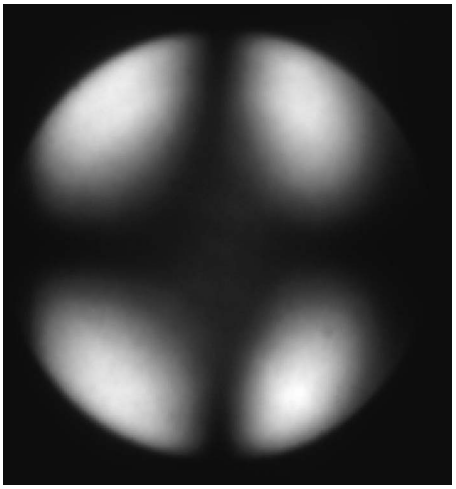

FIG. 2. Conoscopic image of the UV Kerr microscope.

charge-coupled device camera from Hamamatsu Photonics.

Magnetic domains of Nd–Fe–B magnets obtained by the HDDR process were observed using the UV Kerr microscope. The average grain size of the HDDR powder was 300 nm, and the size of a powder was about 100 μm. A commercially anisotropic bonded magnet (high-B type, produced by Hitachi Metals, Ltd., coercivity H_{cJ}=13 kOe) was cut into a 1.5-mm-thick, 3.5-mm-long, and 3.0-mm-wide sample. The domain configuration at the c-plane of the bonded magnet was observed by polar Kerr microscopy. Further, to explore the domain configuration and magnetization process by longitudinal Kerr microscopy, a dc field was applied at the polished surface of $Nd_{12.5}Fe_{72.8}Co_8B_{6.5}Ga_{0.2}$ HDDR powder embedded in a polyester resin. The powder has a low coercivity of 0.60 kOe at a DR time of 12 min. Increasing the DR time from 14 to 18 min abruptly increased the coercivity of the powder to about 16.8 kOe.

III. RESULTS AND DISCUSSION

Polarization-dependent reflection and transmission effects on curved lens surfaces and other optical elements within the microscope add to the depolarization of the sample, reducing the Kerr contrast. The influence of the optical elements for UV light on depolarization can be explored by observing the conoscopic image visible in the back focal plane of the objective lens of the UV Kerr microscope.[11] Figure 2 shows the conoscopic image obtained with the polarizer and analyzer adjusted to maximum extinction. The maximum extinction zone is cross shaped in the conoscopic image. This indicates that the zone receives sufficient polarization light for observing the magnetic domain image.

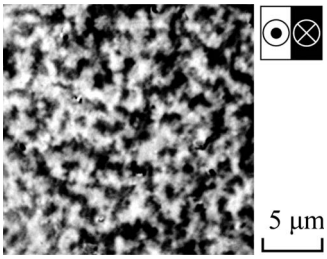

FIG. 4. Domain images of a Nd–Fe–B bonded magnet.

Figure 3 shows line patterns (200 nm width; 2 μm spacing) observed using visible light (546 nm) and UV light. The figure demonstrates that UV light observation shows the lines more clearly than visible light observation.

Figure 4 shows the domain patterns of the bonded magnet in the demagnetized state. In the images, bright and dark domains have magnetizations directed perpendicular to the observation surface. A maze domain pattern was observed at the c-plane. Note that the UV Kerr microscope allows observation of small domain patterns, approximately a few hundred nanometers in size. The maze pattern size is comparable to the grain size of HDDR powder.

Figure 5 shows the motion of domain configurations of an HDDR particle at a DR time of 12 min with a dc field changing from −0.5 to +1.0 kOe. In the images, bright and dark domains have magnetizations in the downward and upward directions, respectively. Domains with an upward component of magnetization are observed at a field of −0.5 kOe; they have a lateral size of about a few micrometers, as marked by the white lines in Fig. 5(a). The field was then changed gradually; at +1.0 kOe, the area of dark domains increased abruptly, as marked by the arrows in Fig. 5(b). In Fig. 5(b), the expanded domains appear to be marked by white lines and the original domains by dotted lines. The wall displacement is greater than the grain size of the $Nd_2Fe_{14}B$ phase which is about 300 nm in diameter. These data indicate that, with an increasing magnetic field, domain walls easily moved beyond several grain boundaries which function as the pinning sites of the domain walls.[5] Therefore, it can be concluded that the pinning force exerted by the grain boundaries is inadequate for producing high coercivity because the Nd-rich phase layers along these boundaries are absent at a DR time of 12 min.[12] Figure 6 shows the domain configurations of HDDR particle at a DR time of 18 min. For

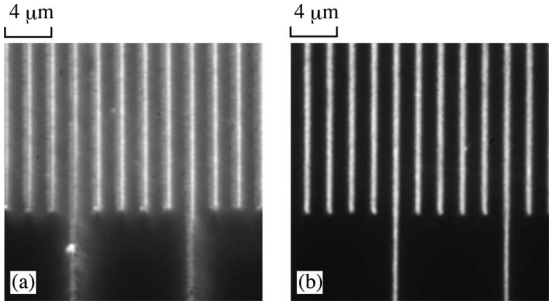

FIG. 3. Image of 200-nm-wide line patterns: (a) visible light and (b) UV light.

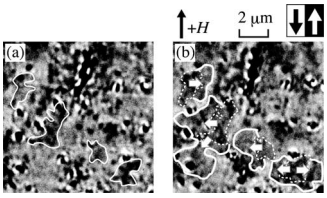

FIG. 5. Magnetization process of HDDR particle at a DR time of 12 min: (a) −0.5 kOe and (b) +1.0 kOe.

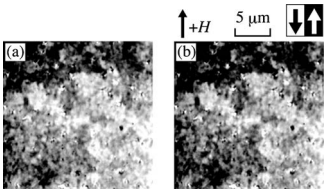

FIG. 6. Magnetization process of HDDR particle at a DR time of 18 min: (a) 0 kOe and (b) +1.0 kOe.

an HDDR particle at a DR time greater than 14 min, changing the magnetic field by up to 1.0 kOe did not produce observable wall motion. It follows that the high coercivity of HDDR powder is due to domain wall pinning at the grain boundaries.

IV. CONCLUSION

In this study, we developed a Kerr microscope that uses UV light for high-resolution domain observation and examined the domain structure and magnetization process of HDDR powder. When a dc field of 1.0 kOe was applied to HDDR powder at a DR time of 12 min, abrupt wall motion occurred. The wall displacement was greater than the grain size of the $Nd_2Fe_{14}B$ phase. It is concluded that the pinning force exerted by the grain boundaries is inadequate to produce high coercivity because the Nd-rich phase layers along these boundaries are absent at a DR time of 12 min. For HDDR powder at a DR time greater than 14 min, changing the magnetic field within 1.0 kOe produced no observable wall motion. Hence, domain wall pinning is the coercivity mechanism of HDDR powder. Moreover, it was shown that nanometer-sized domain patterns could be observed with the UV Kerr microscope. The high-resolution UV Kerr microscope will allow clarification of the coercivity mechanism of Nd–Fe–B magnets.

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