

Effect of Oxidation Protection Layer on the Performance of Magnetic Force Microscope Tip

Keiichi Kato, Mitsuru Ohtake, Masaaki Futamoto, Fumiyoshi Kirino*, and Nobuyuki Inaba**

Faculty of Science and Engineering, Chuo University, 1-13-27 Kasuga, Bunkyo-ku, Tokyo 112-8551, Japan

*Graduate School of Fine Arts, Tokyo University of the Arts, 12-8 Ueno-koen, Taito-ku, Tokyo 110-8714, Japan

**Faculty of Engineering, Yamagata University, 4-3-16 Jyonan, Yonezawa, Yamagata 992-8510, Japan

Magnetic force microscope (MFM) tips are prepared by coating silicon tips of 4 nm radius with 20-nm-thick metallic magnetic films with and without 2-nm-thick oxidation protection layers. Iron (Fe) is used as the magnetic material, whereas carbon (C), silicon nitride (Si-N), or silicon carbide (Si-C) is employed as the protection layer. The MFM tips are exposed in an environment of high temperature of 70 °C and high relative humidity of nearly 100%. The effect of protection layer on the spatial resolution is investigated as a function of period of exposure to the environment. The resolution of MFM tip without protection layer deteriorates from 7.2 to 21.2 nm in 10 days. The deterioration is attributed to an increase in the tip radius and a loss of the detection sensitivity caused by oxidation of coated Fe material. In contrast, the resolutions of tips with C, Si-N, and Si-C layers are kept almost constant at 12.1 ± 0.5 , 12.1 ± 0.5 , and 14.8 ± 2.1 nm for a time span of 10 days, respectively. The coating of a very thin protection layer has been shown effective in keeping the MFM tip performance for a long period of time by preventing the oxidation of coated metallic magnetic material.

Key words: magnetic force microscope, tip, spatial resolution, oxidation protection layer

1. Introduction

Magnetic force microscopy (MFM) has been widely used to investigate the nano-scale magnetization structures of magnetic devices like hard disk drive (HDD) media. MFM tip is prepared by coating a non-magnetic sharp tip with a magnetic film and it is the key component which determines the spatial resolution. The areal density of HDD medium is now approaching 1 Tb/in², where the bit length is becoming narrower than 30 nm. MFM resolution of around 15 nm or better is thus necessary to observe the magnetization structures of high-end recording media. However, the resolution of commercially available MFM tip is limited at around 20–30 nm. In order to improve the resolution, the tip needs to be sharp so that a very small volume of magnetic material around the top interacts with a magnetic observation sample surface. Various methods such as tip fabrication by focused ion beam etching^{1–4}, magnetic material deposition on a sharp non-magnetic tip made of silicon (Si)⁵ or carbon nanotube^{6–8}, etc. have been tried. In our previous studies^{9–15}, MFM tips were prepared by coating Si tips with various magnetic materials like iron (Fe), iron-boron alloy, iron-cobalt alloy, cobalt-platinum ordered alloy, etc. The signal detection sensitivity of MFM tip was improved by coating a material with higher saturation magnetization (M_s). The resolution was influenced not only by the tip radius but also by the detection sensitivity.

MFM tips prepared by coating metallic magnetic materials are expected to oxidize when kept in a normal atmosphere due to chemical reaction with oxygen or water vapor. In this case, the resolution tends to deteriorate because the magnetic sensitivity of MFM tip will decrease depending on the oxidation of coated

metallic magnetic material. By applying a thin protection layer to MFM tip, it seems possible to keep a high resolution property for a long period of time.

In the present study, the effect of oxidation protection layer on the MFM performance is investigated by employing Fe-coated MFM tips. Fe-coated tips show high resolution characteristics when tested soon after preparation^{9,10,15}. However, it was noted that the resolution deteriorated when the tip was kept under an ambient atmosphere, possibly due to easy oxidation of Fe material. Therefore, it seems useful to employ Fe-coated tips to investigate the effect of thin protection layer on the variation of MFM tip performance as a function of exposure time. The protection layer must be very thin and continuous to keep the distance between the magnetic tip and a sample as small as possible and to prevent oxidation of magnetic material. Carbon (C), silicon nitride (Si-N), and silicon carbide (Si-C) are selected as the protection layer materials. These non-magnetic materials have been studied as the protection layers for magnetic recording media¹⁶ and they are also supposed to work as the protection layers for MFM tips. The variations of resolution as a function of time are compared between MFM tips with and without protection layers.

2. Experimental Procedure

A radio-frequency (RF) magnetron sputtering system with the base pressures lower than 4×10^{-7} Pa was employed for coating. Commercial Si tips with top radius of 4 nm were used as the base tips. Fe, C, Si₃N₄, and SiC targets of 3 inch diameter were used and the respective RF powers were fixed at 48, 300, 190, and 150 W. The distance between target and Si tip was 150

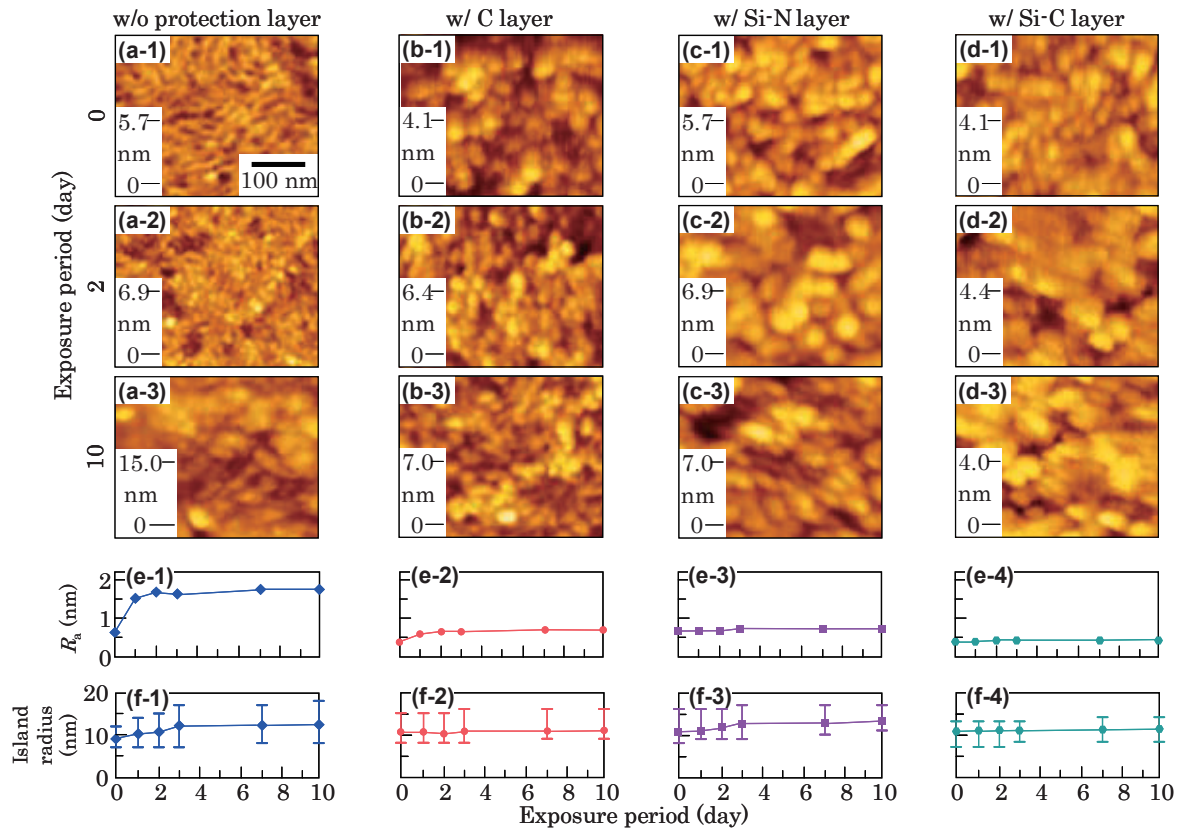


Fig. 1 (a)–(d) AFM images observed for Fe films (a) without and with (b) C, (c) Si-N, and (d) Si-C protection layers deposited on flat Si substrates, (a-1)–(d-1) before and (a-2)–(d-3) after exposure to an environment of 70 °C and nearly 100% relative humidity for (a-2)–(d-2) 2 and (a-3)–(d-3) 10 days. [(e), (f)] Dependences of exposure period on (e) R_a and (f) average island radius.

mm. The Ar gas pressure during sputtering was kept constant at 0.67 Pa. Under the conditions, the deposition rate was 0.020 nm/s for Fe, Si-N, and Si-C materials, while that was 0.017 nm/s for C material. 20-nm-thick Fe films and 2-nm-thick protection layers of C, Si-N, or Si-C were sequentially deposited on Si tips at room temperature (RT). MFM tips without protection layers were also prepared. The coating thicknesses were estimated for films deposited on flat Si substrates, which were located near the base tips in the sputter deposition system. The flat films were also used for structural and magnetic characterizations of coated film materials. The MFM tips and the flat magnetic films were exposed in an environment of 70 °C and nearly 100% relative humidity for acceleration of oxidation up to 10 days. Oxidation rate obeys the Arrhenius Law and the rate (v) is expressed as, $v \propto \exp(-E_a / k_B T)$, where E_a is activation energy, k_B is Boltzmann constant, and T is absolute temperature. The rate is considered to be proportional to the concentration of water vapor, H_2O , humidity. When $E_a = 28 \text{ kJ/mol}^{17}$ and $T(\text{RT}) = 293 \text{ K}$ (20 °C), 343 K (70 °C) are employed, the oxidation rate (v) at 70 °C is estimated to be 5.3 times of the rate at RT. When the humidity of ambient atmosphere, around 50%, is considered, the exposure test at 70 °C under 100% relative humidity corresponds to an acceleration of about 10 times. The tip shapes were observed by scanning electron microscopy (SEM). The surface

roughnesses and the magnetic properties of flat films were measured by atomic force microscopy (AFM) and by vibrating sample magnetometry, respectively.

MFM observation was carried out at RT under pressures lower than 0.1 Pa. A perpendicular medium recorded at linear densities ranging from 500 to 1800 kilo flux change per inch (kFCI) was used as an observation sample. MFM tips were magnetized along the tip axis so that the tip top possessed the south magnetic pole. In this case, the bright and the dark contrasts in an MFM image, respectively, correspond to the areas where repulsive and attractive forces are working between the tip and the observation sample. The quality factor value, the distance between tip and observation sample, and the scanning speed were respectively 3000–6000 (dimensionless), $4 \pm 1 \text{ nm}$, and 1.4 m/s. The resolutions of MFM tips were carefully determined by optimizing the observation conditions.

3. Results and Discussion

Figures 1(a)–(d) show the AFM images observed for Fe films without and with protection layers deposited on flat Si substrates before and after exposure to the environment of 70 °C and nearly 100% relative humidity. Figures 1(e) and (f) summarize the arithmetical mean surface roughness (R_a) values and the average island radiuses, respectively. Here, the island radius is estimated by using the relation, (radius)

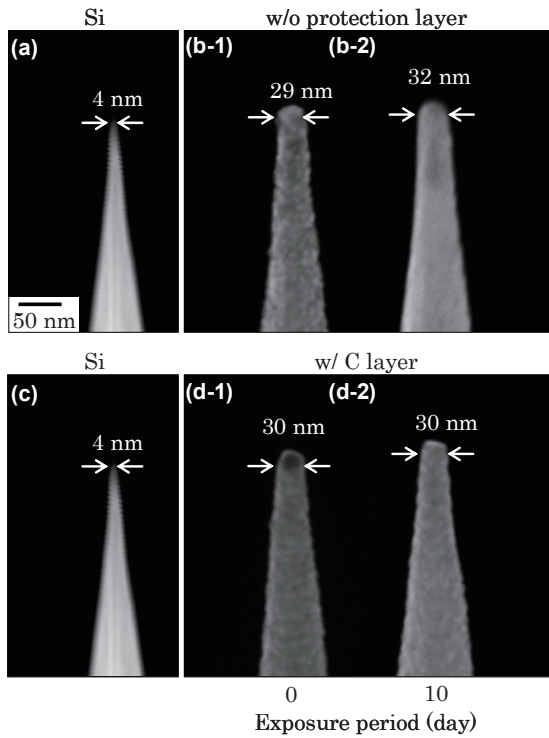


Fig. 2 (a) (c) SEM images of Si tips with 4 nm radius before film coating. (b-1) and (b-2) respectively show the profiles of tip coated with Fe-layer before and after exposure to an environment of 70 °C and nearly 100% relative humidity for 10 days. (d-1) and (d-2) respectively show the profiles of tip coated with Fe-layer and C-protection-layer before and after exposure to the environment for 10 days.

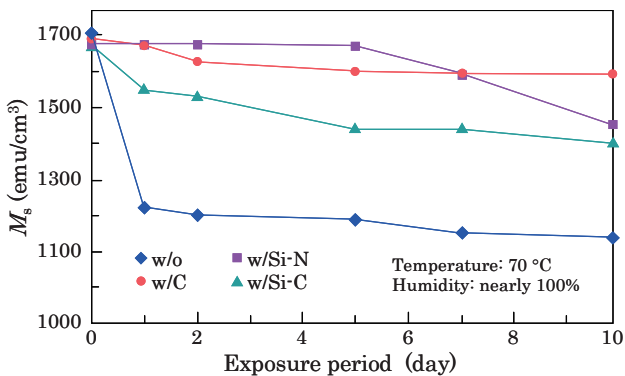


Fig. 3 Dependence of M_s on exposure period measured for Fe films without and with C, Si-N, and Si-C protection layers deposited on flat Si substrates.

$= [(area) / \pi]^{1/2}$. With increasing the exposure period from 0 to 10 days, the R_a value of magnetic film without protection layer increases from 0.6 to 1.8 nm. On the contrary, the R_a values of films with C, Si-N, and Si-C protection layers are kept constant around 0.5 nm during the oxidation test period. Furthermore, the average island radiuses of films with protection layers are smaller than those of films without protection layers. The results show that the film surface morphology is kept unchanged for the samples with protection layers, whereas the surface roughness changes with time for those without protection layers possibly depending on

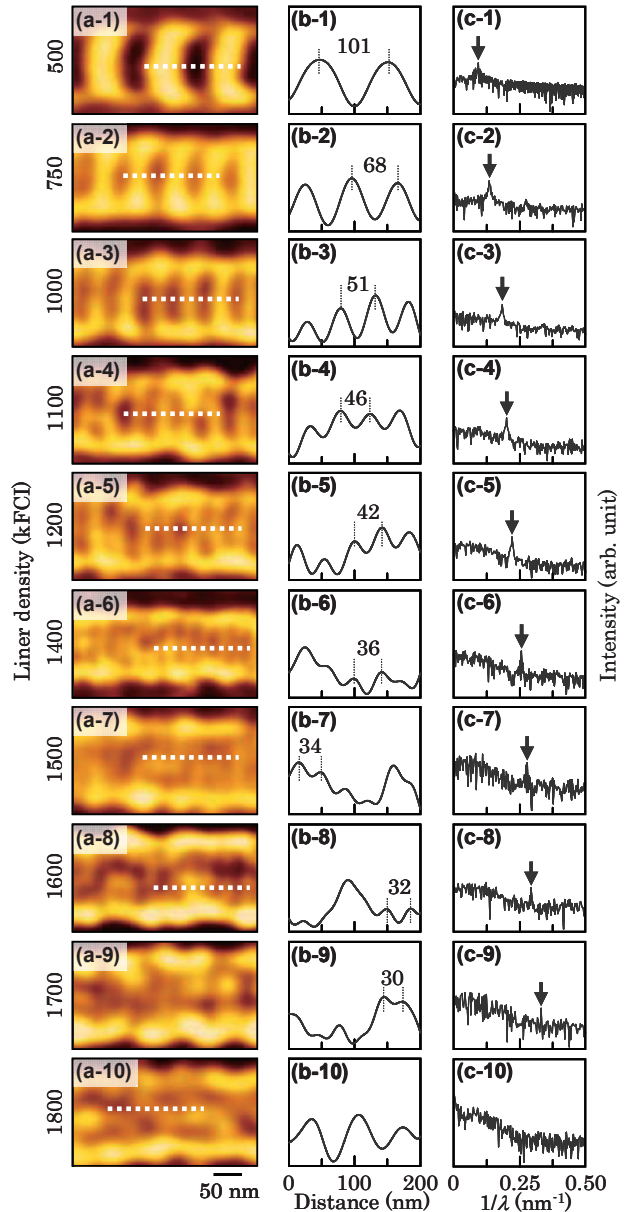


Fig. 4 (a) MFM images of a perpendicular medium recorded at (a-1) 500, (a-2) 750, (a-3) 1000, (a-4) 1100, (a-5) 1200, (a-6) 1400, (a-7) 1500, (a-8) 1600, (a-9) 1700, and (a-10) 1800 kFCI observed by using an MFM tip without protection layer before exposure to an environment of 70 °C and nearly 100% relative humidity. (b) Signal profiles along the white dotted lines in (a). (c) Power spectra analyzed for the magnetic bit images of (a).

the progress of iron material oxidation. As surface oxidation proceeds, Fe changes to oxides, FeO, Fe₂O₃, etc. which is related with the variations of crystal structure and density. The surface oxidation is enhancing the surface roughness estimated as R_a and island radius shown in Fig. 1.

Figures 2(a) and (c) show the SEM images observed for Si tips of 4 nm radius. Figures 2(b-1) and (b-2) show the SEM images observed for MFM tips without protection layers before and after exposure for 10 days, respectively. The Fe film thickness covering the base Si

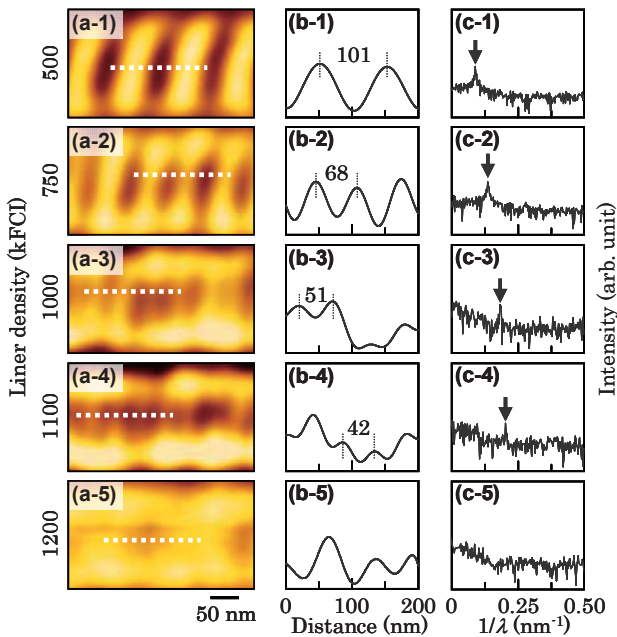


Fig. 5 (a) MFM images of a perpendicular medium recorded at (a-1) 500, (a-2) 750, (a-3) 1000, (a-4) 1100, and (a-5) 1200 kFCI observed by using an MFM tip with C protection layer before exposure to an environment of 70 °C and nearly 100% relative humidity. (b) Signal profiles along the white dotted lines in (a). (c) Power spectra analyzed for the magnetic bit images of (a).

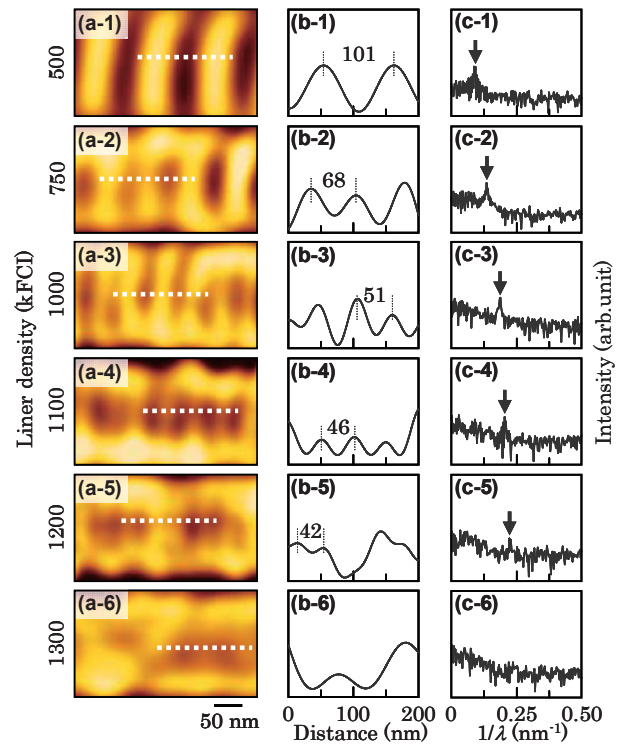


Fig. 7 (a) MFM images of a perpendicular medium recorded at (a-1) 500, (a-2) 750, (a-3) 1000, (a-4) 1100, (a-5) 1200, and (a-6) 1300 kFCI observed by using an MFM tip with Si-C protection layer before exposure to an environment of 70 °C and nearly 100% relative humidity. (b) Signal profiles along the white dotted lines in (a). (c) Power spectra analyzed for the magnetic bit images of (a).

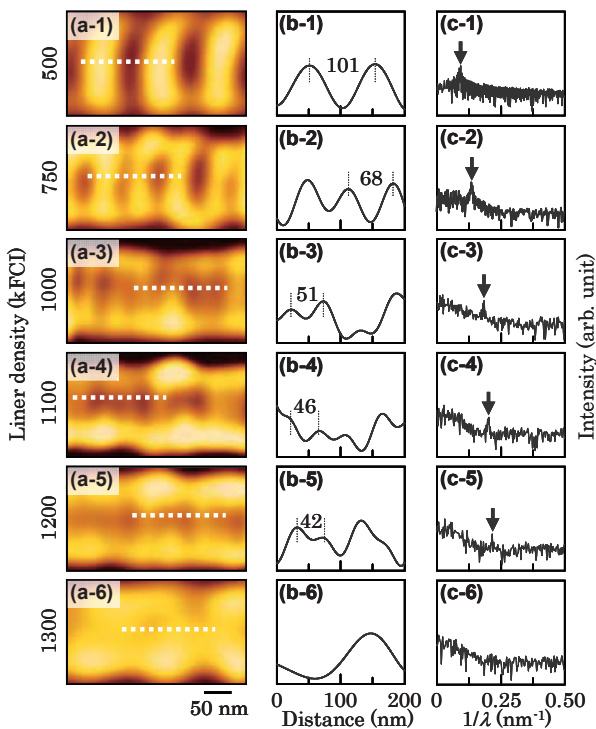


Fig. 6 (a) MFM images of a perpendicular medium recorded at (a-1) 500, (a-2) 750, (a-3) 1000, (a-4) 1100, (a-5) 1200, and (a-6) 1300 kFCI observed by using an MFM tip with Si-N protection layer before exposure to an environment of 70 °C and nearly 100% relative humidity. (b) Signal profiles along the white dotted lines in (a). (c) Power spectra analyzed for the magnetic bit images of (a).

tip can be estimated by comparing the tip profile before and after Fe deposition. The thickness around the top part of MFM tip is measured to be about 12 nm which is 60% of thickness measured for a flat sample (20 nm). However, the film thickness is expected to vary delicately depending on the incidence angle of material sputtered-out from the target. The relatively large thickness ratio of about 60% with respect to that measured for the flat sample is due to a large size (3-inch diameter) of sputter target, the small size of Si tip, and the target to sample distance (150 mm). The local thickness of top part, which gives a dominant influence on the MFM performance, is considered to be similar to that measured for the flat sample, since the incident angle is almost same between the two cases. Therefore, the thickness measured for flat sample is employed as the effective coating thickness of MFM tip. As the exposure period increases from 0 to 10 days, the tip radius increases from 29 to 32 nm, corresponding to the film surface morphology variation observed for the flat film samples. Furthermore, the tip surface profile is changing to be very smooth losing the small surface undulations which are observed for the tip before exposure. Such variation is interpreted to be caused by the surface oxidation of coated Fe film. Figures 2(d-1) and (d-2) show the SEM images observed for MFM tips with C protection layers before and after exposure for 10 days, respectively. The thickness of C protection layer on the side of Si tip is estimated to be about 1 nm. The radius and the surface roughness are almost similar between the two samples, indicating that the magnetic tip structure is maintained even after

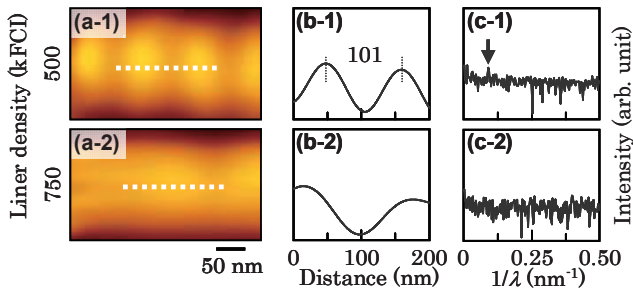


Fig. 8 (a) MFM images of a perpendicular medium recorded at (a-1) 500 and (a-2) 750 kFCI observed by using an MFM tip without protection layer after exposure to an environment of 70 °C and nearly 100% relative humidity for 10 days. (b) Signal profiles along the white dotted lines in (a). (c) Power spectra analyzed for the magnetic bit images of (a).

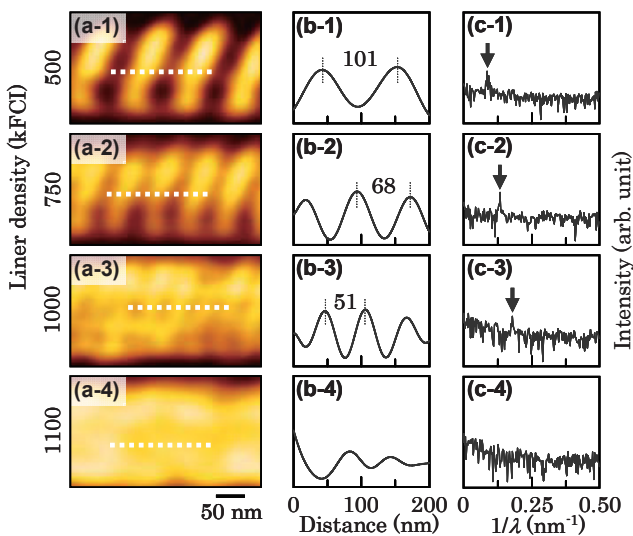


Fig. 9 (a) MFM images of a perpendicular medium recorded at (a-1) 500, (a-2) 750, (a-3) 1000, and (a-4) 1100 kFCI observed by using an MFM tip with C protection layer after exposure to an environment of 70 °C and nearly 100% relative humidity for 10 days. (b) Signal profiles along the white dotted lines in (a). (c) Power spectra analyzed for the magnetic bit images of (a).

exposure to the environment of high temperature of 70 °C and high humidity of nearly 100% for 10 days. The results indicate that the protection layer coating is effective in keeping the tip profile by preventing surface oxidation of magnetic material. Figure 3 shows the variations of M_s measured for flat film samples with and without protection layers. The M_s values are almost similar to that of bulk bcc-Fe material (1713 emu/cm³) for the samples with and without protection layers soon after preparation. The M_s value of film without protection layer decreases to 1150 emu/cm³ after exposure for 10 days, whereas those of films with C, Si-N, and Si-C protection layers show small variations to 1700–1600, 1700–1500, 1700–1400 emu/cm³, respectively. These results are suggesting that a high detection sensitivity is kept for a long time by introducing a thin protection layer on a magnetic tip.

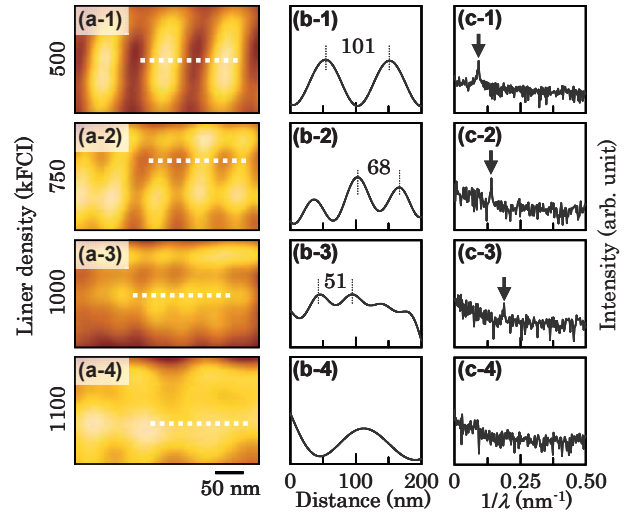


Fig. 10 (a) MFM images of a perpendicular medium recorded at (a-1) 500, (a-2) 750, (a-3) 1000, and (a-4) 1100 kFCI observed by using an MFM tip with Si-N protection layer after exposure to an environment of 70 °C and nearly 100% relative humidity for 10 days. (b) Signal profiles along the white dotted lines in (a). (c) Power spectra analyzed for the magnetic bit images of (a).

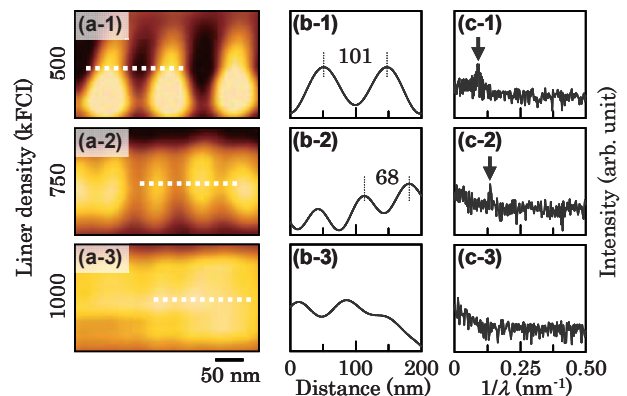


Fig. 11 (a) MFM images of a perpendicular medium recorded at (a-1) 500, (a-2) 750, and (a-3) 1000 kFCI observed by using an MFM tip with Si-C protection layer after exposure to an environment of 70 °C and nearly 100% relative humidity for 10 days. (b) Signal profiles along the white dotted lines in (a). (c) Power spectra analyzed for the magnetic bit images of (a).

Figure 4(a) shows the MFM images of a perpendicular medium recorded at linear densities ranging from 500 to 1800 kFCI observed by using an MFM tip without protection layer before oxidation. The sharpness of MFM image is degrading with increasing the linear density. In order to estimate the degradation quantitatively, MFM signal intensity profile measurement and fast Fourier transformation (FFT) analysis are carried out for the magnetic bit images of Fig. 4(a). Figures 4(b) and (c) show the intensity variation profiles measured along the white dotted lines in Fig. 4(a) and power spectra analyzed for the magnetic bits of Fig. 4(a), respectively. The bit lengths and the peaks corresponding to the recording

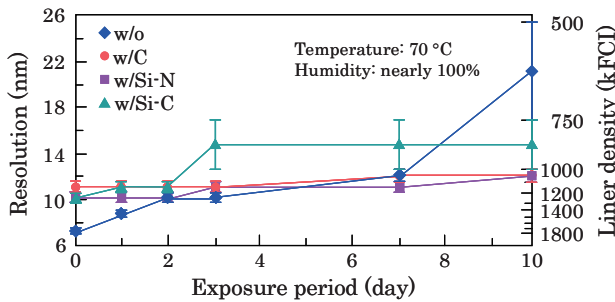


Fig. 12 Dependence of resolution on exposure period measured for MFM tips without and with C, Si-N, and Si-C protection layers.

densities ranging between 500 and 1700 kFCI are respectively recognized as shown in Figs. 4(b-1)–(b-9) and (c-1)–(c-9). Magnetic bits recorded at 1800 kFCI are not distinguishable as shown in Figs. 4(b-10) and (c-10). Therefore, the spatial resolution is between $14.9/2 = 7.5$ nm (1700 kFCI) and $14.1/2 = 7.1$ nm (1800 kFCI), that is, 7.3 ± 0.2 nm.

Figures 5, 6, and 7, respectively, show the MFM data obtained by employing tips with C, Si-N, and Si-C protection layers before exposure. When the tip with C protection layer is employed, the magnetic bits recorded below 1100 kFCI are observed, whereas those recorded at 1200 kFCI are not detected. The resolution is thus between $23.1/2 = 11.5$ nm (1100 kFCI) and $21.2/2 = 10.6$ nm (1200 kFCI), that is, 11.1 ± 0.4 nm. Similarly, the resolution is determined to be between $21.2/2 = 10.6$ nm (1200 kFCI) and $19.5/2 = 9.8$ nm (1300 kFCI), that is, 10.2 ± 0.4 nm for MFM tips with both Si-N and Si-C layers. The resolution slightly deteriorates by employing a protection layer. The total coating thickness is about 2 nm thinner for the tip without protection layer than that of a tip with protection layer. Therefore, the deterioration of resolution is considered mainly due to the increase of effective magnetic spacing between the top of magnetic tip and sample.

Figures 8–11 show the MFM data obtained by using MFM tips after exposure to the environment of 70 °C and nearly 100% humidity for 10 days. Figure 12 summarizes the variations of tip resolution as a function of exposure period. For tips without protection layer, the resolution apparently deteriorates with increasing the exposure period. The reason is partially due to an increase of tip radius because of Fe oxidation and partially due to a decrease in the MFM detection sensitivity. The data shown in Fig. 1(e), Fig. 3, and Fig. 12 are apparently supporting this interpretation. On the other hand, the resolutions of tips with C, Si-N, and Si-C layers are respectively almost constant at 12.1 ± 0.6 nm, 12.1 ± 0.6 nm, and 14.8 ± 2.1 nm. The result indicates that the introduction of C, Si-N, or Si-C protection layer is effective in keeping a high spatial resolution for a long period of time.

4. Conclusion

MFM tips are prepared by coating Fe films without and with C, Si-N, and Si-C protection layers on Si tips

and they are exposed to an environment of 70 °C and nearly 100% relative humidity, which is an acceleration test of about 10 times of room temperature environment (20 °C, 50%). The resolution of MFM tip without protection layer deteriorates from 7.2 ± 0.2 nm to 21.2 ± 4.3 nm with increasing the exposure period from 0 to 10 days, whereas those with C, Si-N, and Si-C protection layers stay almost constant at 12.1 ± 0.6 , 12.1 ± 0.6 , and 14.8 ± 2.1 nm for 10 days. The protection layers are shown effective in preventing oxidation of Fe-coated MFM tips and thus contribute in keeping high spatial resolutions for a long period of time.

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