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Study of Nano-Granular Co-Zr-O Thin Films by Holography and HRTEM

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Developments in electric devices have led to an urgent request of higher frequency operation of magnetic devices. Thus higher magnetic anisotropy field (H_k), saturation magnetization (B_s) and electrical resistivity (ρ) are the most important parameters for distinguished high frequency soft magnetic materials[1] since the permeability (μ) is remarkably influenced by eddy current losses and the magnetic resonance at high frequency. Recently, such excellent magnetic properties have been obtained in the soft magnetic Co-Zr-O thin films[2] which have a nano-granular structure. In the present work the magnetic structure and microstructure of Co-Zr-O films were evaluated by holography and HRTEM, respectively.

Three kinds of Co-Zr-O film with different compositions were prepared on water-cooled glass substrates (Corning 7059, 0.5mm in thickness) by a RF reactive magnetron sputtering in an O_2+Ar atmosphere, using a Co-Zr alloy targets. The easy axes of the films were controlled by the magnetic field of permanent magnets. The details of the deposition condition were described elsewhere[3]. TEM samples were prepared by one-side grinding and ion-milling. JEM3000F (accelerating voltage 300kV) equipped with a biprism was used for electron holography. JEM4000EX (400kV), JEM1250 (1250kV) and JEM2010F (200kV) were used for the investigation of the microstructures, Lorentz observations and microanalyses, respectively.

Figures 1a, 1b and 1c are the Lorentz micrographs (under focus) of $Co_{71.5}Zr_{9.2}O_{19.3}$, $Co_{59.9}Zr_{10.3}O_{29.8}$ and $Co_{52.9}Zr_{12.0}O_{35.1}$ thin films, respectively. The bright and dark lines indicated by arrows illustrate the magnetic domain walls. It can be seen that the domains of $Co_{59.9}Zr_{10.3}O_{29.8}$, which has the highest H_k (=11kA/m), are smaller than that of $Co_{71.5}Zr_{9.2}O_{19.3}$, however domain walls can not be found in $Co_{52.9}Zr_{12.0}O_{35.1}$. In order to have an intuitive observation of the magnetic structure, holograms were taken from these samples. Figures 2a, 2b and 2c are the reconstructed phase images of $Co_{71.5}Zr_{9.2}O_{19.3}$, $Co_{59.9}Zr_{10.3}O_{29.8}$ and $Co_{52.9}Zr_{12.0}O_{35.1}$, respectively. The contour lines show the magnetic flux and the arrows indicate its direction. Between two adjacent contour lines, there is a constant flux of h/e (=4×10^{-15}Wb) flows. It can be easily seen that $Co_{59.9}Zr_{10.3}O_{29.8}$ shows clear magnetic domains, however $Co_{52.9}Zr_{12.0}O_{35.1}$ shows no distinct domains. The HRTEM images of these three kinds of Co-Zr-O films are shown in Figure 3. All of these films have a nano-granular structure. Through the diffraction patterns and the microanalysis results, it is shown that the metallic particles are mostly pure Co although it can not exclude the possibility that a little bit Zr would be contained in the Co grains. The amorphous matrix is zirconium oxide and it also seems that there are some Co atoms in the matrix which indicates to form Co-Zr amorphous structure. Although the particle sizes of Co are about a few nanometers for these three samples, $Co_{71.5}Zr_{9.2}O_{19.3}$ shows not only the biggest size but also the highest density of Co particles. This result exactly explains why $Co_{71.5}Zr_{9.2}O_{19.3}$ has the highest B_s (=12kG) among these three samples. On the other hand, $Co_{52.9}Zr_{12.0}O_{35.1}$, in which the Co particles are magnetically isolated, is in super-paramagnetic state.

References

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[2]. S. Ohnuma et al., *IEEE Trans. Mag.*, 37 (2001) 2251.
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[4]. The author thanks Dr. T. Ohsuna for his software on phase reconstruction and Prof. K. Hiraga and T. Oikawa for valuable discussions.

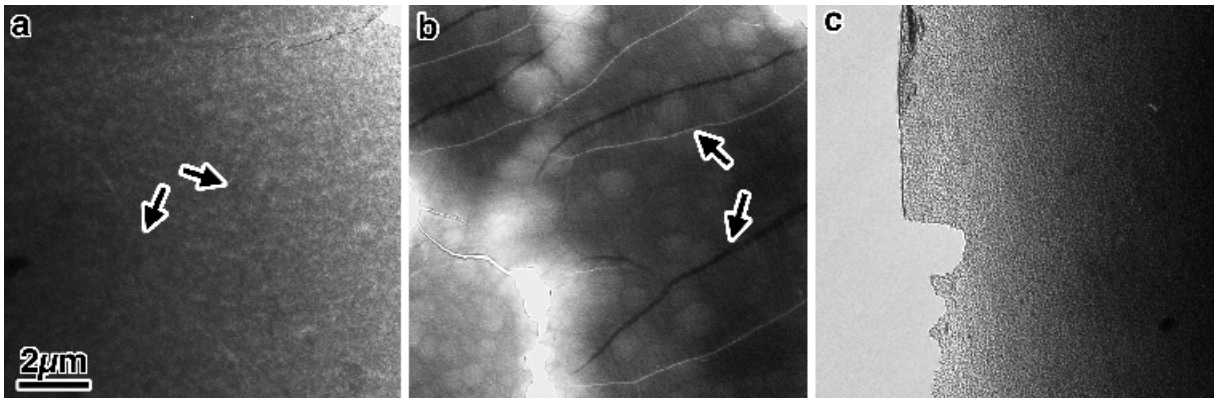

Figure 1: Lorentz micrographs (under focus) of $Co_{71.5}Zr_{9.2}O_{19.3}$ (a), $Co_{59.9}Zr_{10.3}O_{29.8}$ (b) and $Co_{52.9}Zr_{12.0}O_{35.1}$ (c) thin films.

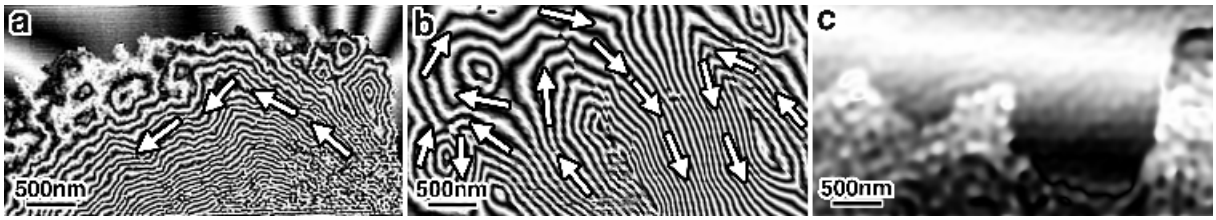

Figure 2: Reconstructed phase images of $Co_{71.5}Zr_{9.2}O_{19.3}$ (a), $Co_{59.9}Zr_{10.3}O_{29.8}$ (b) and $Co_{52.9}Zr_{12.0}O_{35.1}$ (c).

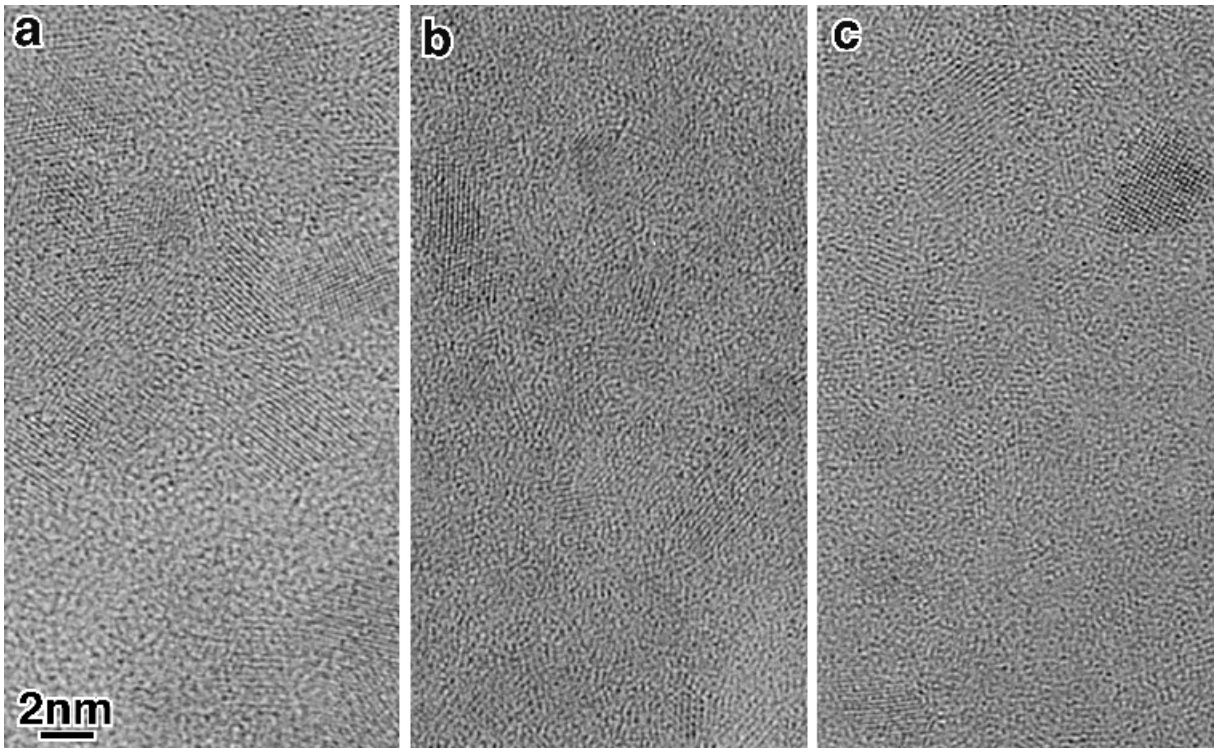

Figure 3: HRTEM images of $Co_{71.5}Zr_{9.2}O_{19.3}$ (a), $Co_{59.9}Zr_{10.3}O_{29.8}$ (b) and $Co_{52.9}Zr_{12.0}O_{35.1}$ (c).

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