Electron Holography Study of Magnetic Flux Distribution in Fe-rich Nanocrystalline Soft Magnetic Material Fe$_{85}$Si$_2$B$_8$P$_4$Cu$_1$

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Fe-rich nanocrystalline material Fe$_{85}$Si$_2$B$_8$P$_4$Cu$_1$ [1] is a soft magnetic material which has high saturation magnetic flux density and low magnetic core loss because of high Fe concentration and small size of crystallite. This material has a homogeneous nanocrystalline structure composed of alpha-Fe grains with a size of about less than 20 nm which are realized by crystallizing the heterogeneous amorphous alloys with heat-treatment. In this study, we prepared specimens which were heat-treated at deferent temperatures, then we evaluated magnetic flux distributions by electron holography. Especially, the magnetic flux distribution of the specimen which has relatively large crystal grain and high amorphous volume-fraction, was analyzed in detail quantitatively.

In this experiment, we used the Fe$_{85}$Si$_2$B$_8$P$_4$Cu$_1$ sample that was heat-treated at 330°C. In order to make a thin film for TEM observations, we used JEM-9310FIB focused ion beam system (FIB). To record bright field images and holograms, we used HF3300S transmission electron microscope which is equipped with a 300 kV cold-type field-emission gun and a double biprism system. In order to extract the magnetic field information from holograms, we used a special specimen holder which enables to rotate a specimen by 180° without detaching the specimen from this holder.

Figure 1(a) shows a bright field image of the specimen. Figure 1(b) shows a bright field image which was recorded after reversing the specimen, and adjusting the diffraction contrast. Under these conditions, two holograms were recorded in order to extract the magnetic field information. Figure 1(c) shows a magnetic flux distribution which was obtained by subtraction of the phases reconstructed from the two holograms. The phase was amplified by a factor of ten. The effect of phase shift due to electron diffraction was reduced by adjusting the diffraction contrast. Figures 2(a) and 2(b) are enlarged bright field image and image of magnetic flux distribution, respectively. White circles show the positions of nanocrystals. We measured the ratio of the magnetic flux density of $\alpha$-Fe region to that of amorphous region from the line profiles which are taken from red and blue lines shown in Fig. 2(b). The ratio was 1.05. This ratio is found to be near to the value of 1.04 calculated with literature data [1,2].

References
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FIG. 1. (a) Bright field image of the Fe$_{85}$Si$_2$B$_8$P$_4$Cu$_1$ sample after 330°C heat-treatment. (b) Bright field image which was recorded after reversing the specimen, and adjusting the diffraction contrast. (c) Magnetic flux distribution (phase amplification is 10 times.)

FIG. 2. Enlarged (a) bright field image and (b) image of magnetic flux distribution corresponding to the rectangular regions in FIG. 1(a) and FIG. 1(c).