

実験報告書様式(一般利用課題・成果公開利用)

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	提出日 Date of Report 2013/1/7
課題番号 Project No. 2012A0091 実験課題名 Title of experiment Combined assessment of polymer - magnetic nanoparticle films by diffuse scattering/GISANS and GISAXS 実験責任者名 Name of principal investigator 奥田浩司 Hiroshi Okuda 所属 Affiliation 京大工 Dept.Mater.Sci.Eng, Kyoto University	装置責任者 Name of responsible person 武田 全康 装置名 Name of Instrument/(BL No.) 写楽(17) 実施日 Date of Experiment 2012.11.6-8

試料、実験方法、利用の結果得られた主なデータ、考察、結論等を、記述して下さい。(適宜、図表添付のこと)
Please report your samples, experimental method and results, discussion and conclusions. Please add figures and tables for better explanation.

1. 試料 Name of sample(s) and chemical formula, or compositions including physical form. Fe ₃ O ₄ nanoparticle dispersed Polystyrene film cast on Si substrate

2. 実験方法及び結果 (実験がうまくいかなかった場合、その理由を記述してください。) Experimental method and results. If you failed to conduct experiment as planned, please describe reasons. Experimental: Sample was made from Fe ₃ O ₄ magnetic nanoparticle having 15 nm of diameter and toluene solution of polystyrene spin-cast on a 2inchi Si substrate. To measure the scattering intensity along qy directions around the Yoneda peak, the sample alignments was set with 90 degree rotation from standard reflectivity measurement setting, i.e., the sample normal is set parallel to the vertical direction. From the intensity assessment check made at the first beamtime in July, the slit in the qz direction was kept open for upper bound to gain as much neutrons available, since we are interested in the in-plane nanostructure in the present measurements. The second measurements were made for two days. Owing to the scan direction necessary for the present one, alignment of the sample took more time compared to the standard reflectivity. One major point is that X-ray does not penetrate through the substrate, but neutron easily does, so that the alignment procedure was found to be quite differenct from what we usually do at synchrotron facilities. The above two points made us use much time to fix the measurement condition
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2. 実験方法及び結果(つづき) Experimental method and results (continued)

than we first anticipated.

Preliminary measurement with X-rays

Figure 1 shows the preliminary GISAXS measurements made at Phoron Factory, BL06A station before the first beamtime of neutron. It shows a well defined interparticle interference peak at about $q_y=0.5 \text{ nm}^{-1}$ along the Yoneda peak and a strong diffuse scattering in the q_z direction at $q_y=0$. With a II-CCD, with high sensitivity and less dynamic range the exposure time was about a couple of seconds, and with Pilatus, it can be extended to 20 seconds for better statistics. To confirm that the film structure is uniform over the substrate, the X-ray was scanned across the sample with a beamsize of about 0.8 mm. The pattern was confirmed to agree well in the central area that we scanned in the present measurement, for about 1.5 inches. The magnetization curve obtained with SQUID showed that the magnetization almost saturated at the field of

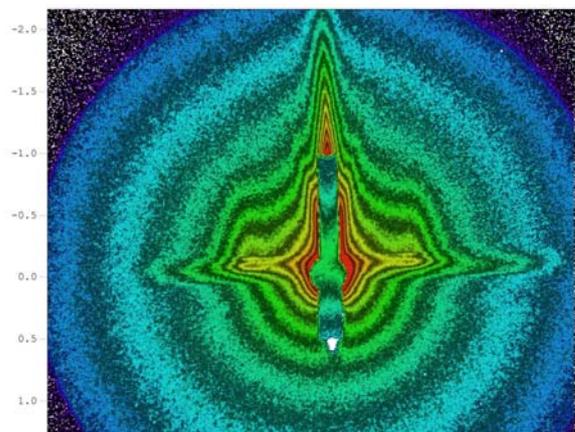


Fig.1 GISAXS profile with IICCD obtained at Photon factory for the same sample measured in the present beamtime.

about 1kOe(80%), although the signal is very weak and diamagnetic component from Si substrate was dominantly strong..

Figure 2 gives the present results with neutrons, averaged over the wavelength between 0.5 and 8 angstroms. A well-defined peak at the q_y corresponding to the interparticle interference in the GISAXS pattern was recognized in the present GISANS measurements. The intensity increase at lower q_y observed in the GISAXS, corresponding to the larger heterogeneity of the film, yet much smaller than the scale checked with scanning GISAXS, was not observed in the present condition. As seen in the figure, the present profile suggests that the scattering intensity is strongly enhanced at some of the wavelengths. The energy dependence of the enhancement depends on the films structure of the sample that gives dynamical effects, requiring reflectivity data. Further analysis is under way on this viewpoint.

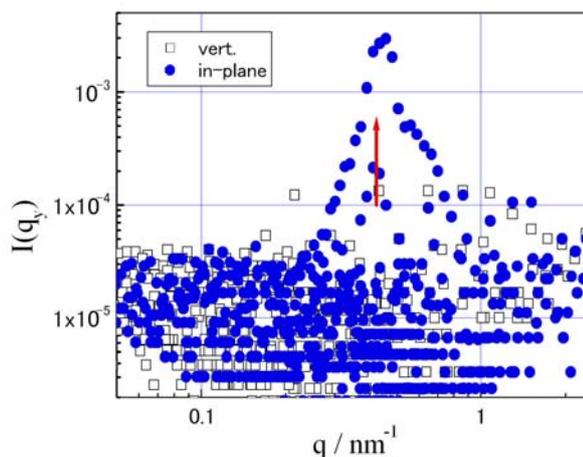


Fig.2 GISANS profile in the q_y direction using the wavelength between 0.5 and 8 Å. Vertical and in-plane denotes the direction of magnetic field applied to the sample. The intensity was summed with the wavelength resolution of about 10%