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# Accessible length scale of the in-plane structure in polarized neutron off-specular and grazing-incidence small-angle scattering measurements

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**Abstract.** Polarized neutron off-specular and grazing-incidence small-angle scattering measurements are useful methods to investigate the in-plane structure and its correlation of layered systems. Although these measurements give information on complementary and overlapping length scale, the different characteristics between them need to be taken into account when performed. In this study, the difference in the accessible length scale of the in-plane structure, which is one of the most important characteristics, was discussed using an Fe/Si multilayer together with simulations based on the distorted wave Born approximation.

## 1. Introduction

Layered magnetic structures exhibit interesting and important magnetic properties which are not present in the bulk, such as exchange coupling between layers, giant magnetoresistance, and tunnel magnetoresistance [1, 2, 3]. These anomalous magnetic properties arise from the structure in the out-of-plane and/or in-plane directions reduced to the nm range. To elucidate those mechanisms and to open the possibility of new applications, it is important to obtain information on the in-plane magnetic structure.

Off-specular scattering (OSS) and grazing-incidence small-angle scattering (GISAS) using polarized neutrons are unique and powerful techniques to observe correlations of small magnetic objects in layered systems. Since there is a difference between the OSS and GISAS in the accessible range and resolution of the lateral component of the momentum transfer  $\mathbf{q}$ , one has to verify if the length scale of the in-plane structure of the sample matches the in-plane  $\mathbf{q}$ -range and resolution [4]. The in-plane component of the coherence volume can be estimated by the in-plane  $\mathbf{q}$ -resolution. The interpretation of the data can be different if the lateral dimension of the in-plane structure of the sample is sufficiently small or large compared with the in-plane component of the coherence volume in that dimension. OSS only measures a limited range of the in-plane component of  $\mathbf{q}$  due to the restriction in the scattering geometry, making difficult the access to the lateral correlation length smaller than one  $\mu\text{m}$ . This can be overcome by GISAS, which allows measurements to a larger in-plane component of  $\mathbf{q}$  and hence to a smaller lateral correlation length [5]. This study has mainly discussed the difference in the accessible in-plane



$q$ -range between the OSS and GISAS using polarized neutrons to illustrate the limitation of the accessible lateral correlation length for both measurements.

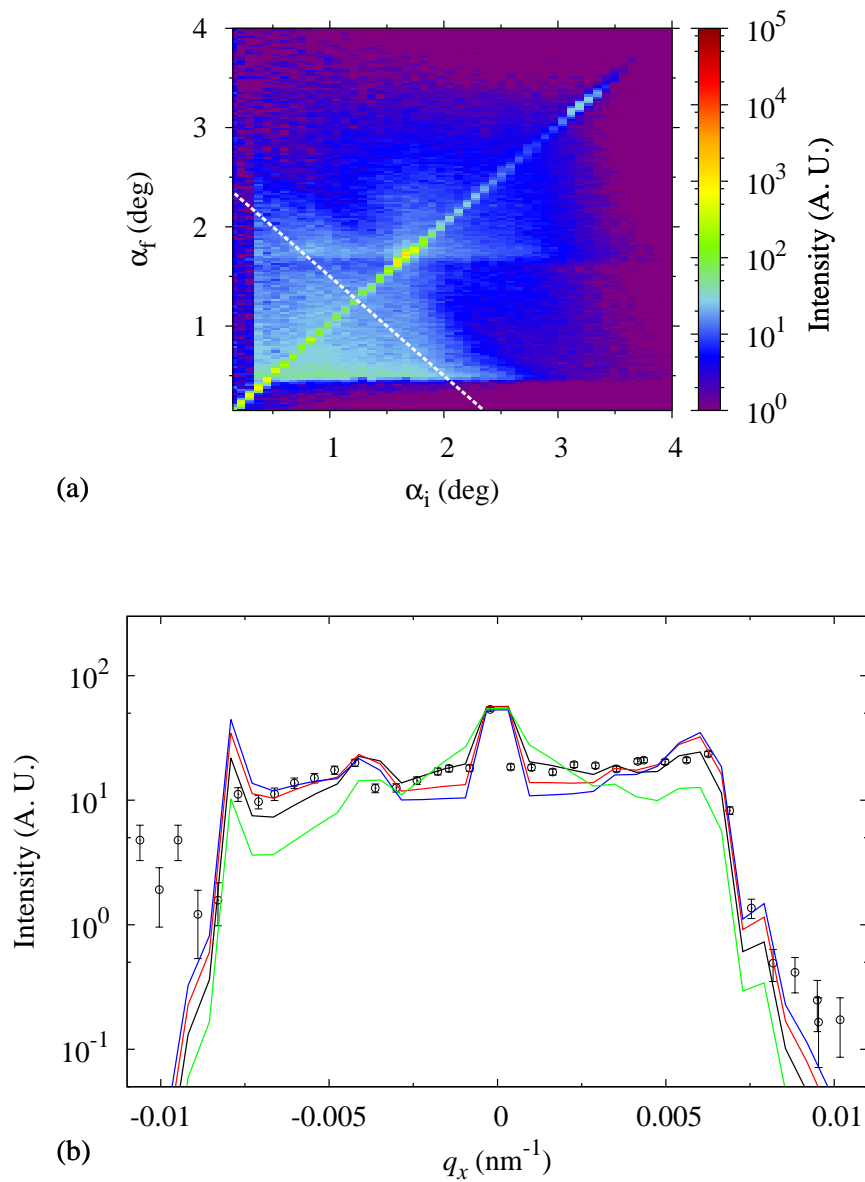
## 2. Results

Let us assume a scattering geometry where the sample surface is parallel to the  $(x, y)$  plane and neutrons incident on the sample at incident angle  $\alpha_i$  are scattered at exit angles of  $\alpha_f$  and  $2\theta_f$  in the planes within and perpendicular to the  $(x, z)$  plane [6]. The scattering intensity dependence on  $q_x$  is measured in OSS, whereas that on  $q_y$  observed in the GISAS. In this study, the OSS and GISAS data were analyzed in the framework of the distorted wave Born approximation (DWBA) given by Toperverg *et al.* [4, 7, 8].

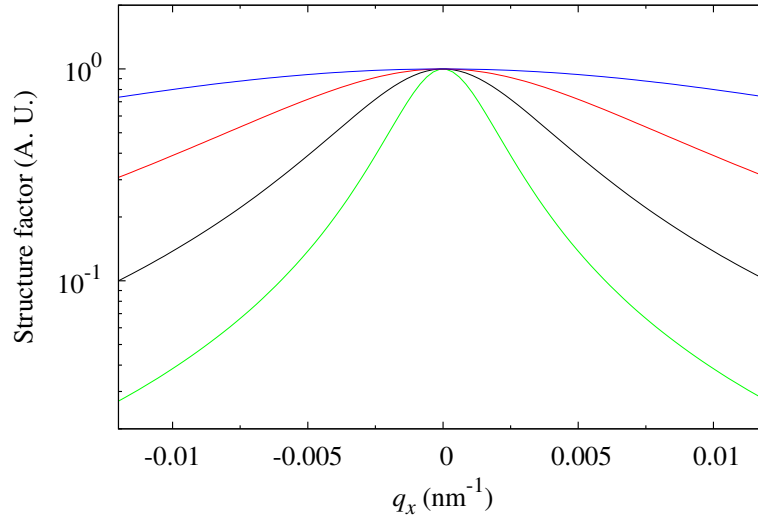
Figure 1(a) shows the measured polarized neutron OSS image in the  $I_{-+}$  spin-flip channel for an Fe/Si multilayer of 30 bilayers with a thickness of 10 nm, which was fabricated using the DC-magnetron sputtering [9]<sup>1</sup>. The measurement was performed on the D17 polarized neutron reflectometer installed at the high-flux reactor at the Institut Laue Langevin (ILL) [10, 11]. The monochromatic mode of operation with an average wavelength of 0.555 nm and wavelength spread of 4.0% in the full width at half maximum (FWHM) was used. The incident beam was collimated to keep the angular resolution less than 3.6%. Further detail of the measurement can be found in Ref. [6]. The spins in the sample are assumed to be aligned within the sample plane and to be perpendicular to  $\mathbf{q}$  because of  $q_x \ll q_z$  in the OSS. Since the sample was magnetized to 70% of saturation under an external field of  $4.6 \times 10^3$  A/m, the magnetic scattering is seen in both of the spin-flip and non-spin-flip channels. The lateral correlation length for the magnetic scattering,  $\xi_{\parallel}$ , corresponding to the length of an area in which the spins are aligned to the same direction, can be determined by the scattering intensity dependence on  $q_x$ . The intensity profile under the condition of  $\alpha_i + \alpha_f = \text{const.}$ , which is approximately considered as  $q_z = \text{const.}$ , is useful because it does not depend on the  $z$ -correlation length [12]. The measured and simulated OSS profiles along the broken line on Fig. 1(a) are shown in Fig. 1(b), where  $\alpha_i + \alpha_f = 2.5^\circ$  ( $q_z \approx 0.49 \text{ nm}^{-1}$ ) is satisfied. The simulated profile does not vary for small values of  $\xi_{\parallel}$  less than 250 nm, which makes it difficult to determine  $\xi_{\parallel}$  and the error of it. Since  $\mathbf{q}$  is oriented close to parallel to  $z$ -axis in the OSS,  $q_x$  is smaller than a few percent of  $|\mathbf{q}|$  for most cases. Although the  $q_x$ -range can be increased by increasing  $q_z$ , this causes another problem of the reduction in the scattering intensity. In general, it is difficult to obtain sufficient scattering intensity over full  $q_x$ -range for  $q_z > 0.5 \text{ nm}^{-1}$ . This limits the accessible  $q_x$ -range to  $|q_x| < 0.01 \text{ nm}^{-1}$  in the OSS. Let us consider the  $q_x$ -dependence of the structure factor in the simulation to understand this. Since the coupled areas with uniform orientation of the spins are modeled as rectangular boxes with lengths of  $2\xi_{\parallel}$  within the plane and a height corresponding to the layer thickness, the structure factor is approximately given by the Lorentzian function,  $1/\{1 + (q_x \cdot \xi_{\parallel})^2\}$  [13, 8, 6]. Figure 2 shows the Lorentzian profiles for different values of  $\xi_{\parallel}$ . The FWHM of the Lorentzian function becomes comparable to the accessible  $q_x$ -range with decreasing  $\xi_{\parallel}$ . This means that only a small part of the full peak profile is measured, making the determination of  $\xi_{\parallel}$  more ambiguous. The condition of  $q_{x,\text{max}} \cdot \xi_{\parallel} \gg 1$  needs to be taken into account when performing the OSS measurement, where  $q_{x,\text{max}}$  is the maximum value of  $|q_x|$  in the measurement. For this case with  $q_{x,\text{max}} = 0.01 \text{ nm}^{-1}$ , the GISAS measurement is needed to obtain quantitative information on the in-plane structure with a length scale smaller than 500 nm. The in-plane component of the coherence volume,  $l_x \approx 2\pi/\delta q_x$ , was estimated as  $l_x \gtrsim 10 \mu\text{m}$  because  $\delta q_x$  was roughly given by  $\delta q_x \lesssim 5 \times 10^{-4} \text{ nm}^{-1}$  (5% of  $q_x$ ) for this OSS measurement. Since  $l_x$  is much larger than the length of the coupled area, the magnetic scattering due to the fluctuation of the spins can be expected.

Figure 3(a) shows the measured polarized neutron GISAS image in the  $I_{-}$  channel for the

<sup>1</sup> Figure 1(a) is reprinted from [6], with permission from Elsevier.



**Figure 1.** (a) Measured polarized neutron OSS contour plot of an Fe/Si multilayer. (b) Measured (symbols) and simulated (lines) OSS profiles along the broken line in (a), where  $\alpha_i + \alpha_f = 2.5^\circ$  ( $q_z \approx 0.49 \text{ nm}^{-1}$ ) is satisfied. Green, black, red, and blue line represents the simulated profile with  $\xi_{\parallel} = 500, 250, 125,$  and  $50 \text{ nm}$ , respectively.



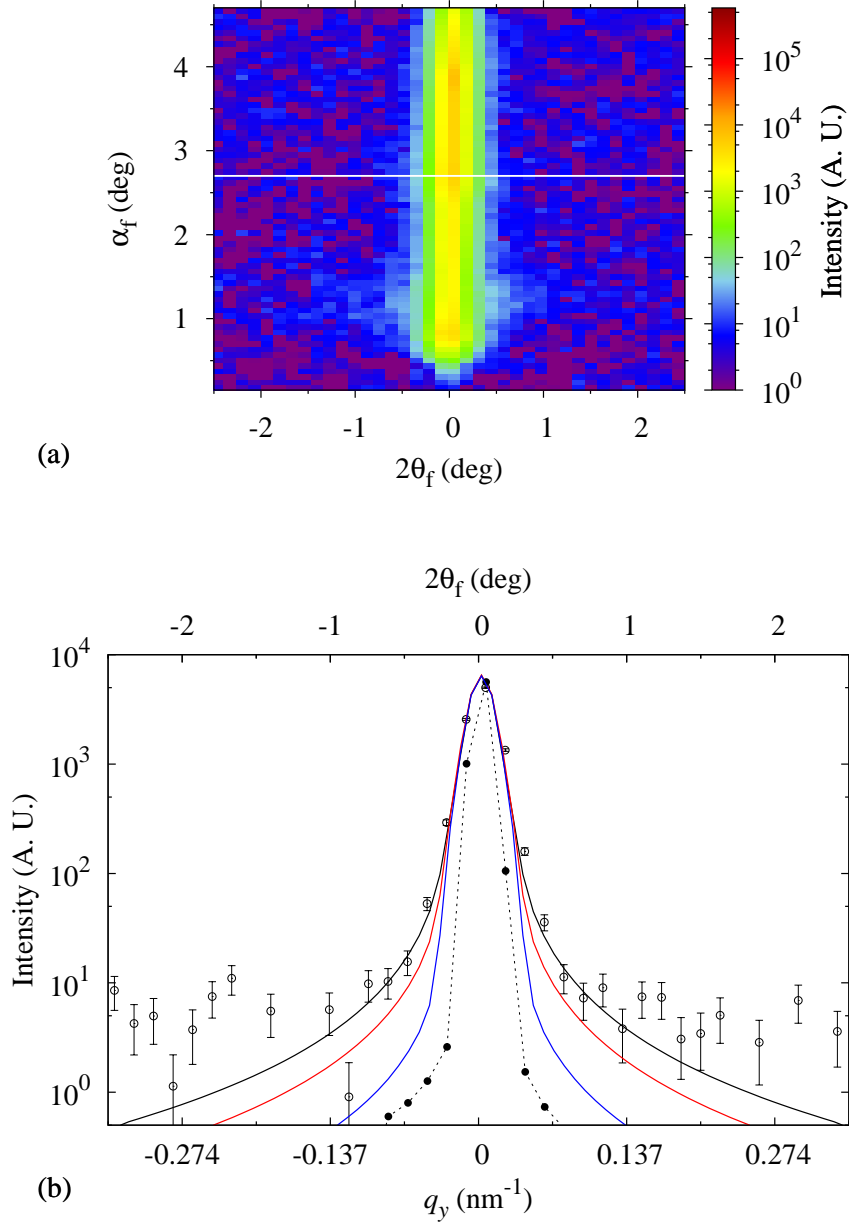
**Figure 2.** Structure factors proportional to the Lorentzian function,  $1/\{1 + (q_x \cdot \xi_{\parallel})^2\}$ . Green, black, red, and blue line indicates the profile with  $\xi_{\parallel} = 500, 250, 125,$  and  $50$  nm, respectively.

same sample<sup>2</sup>. The measurement without polarization analysis was performed on the D33 small angle neutron scattering (SANS) instrument at the ILL [14, 15]. The monochromatic mode of operation with an average wavelength of  $0.80$  nm and wavelength spread of  $10\%$  in FWHM was used. The beam divergence was less than  $1.95$  mrad. Further detail of the measurement can be found in Ref. [6]. In the GISAS,  $\alpha_i$  was chosen to  $3.8^\circ$  to include the intense magnetic scattering observed in the OSS image Fig. 1(a) at  $\alpha_i = 2.6^\circ$  and  $\alpha_f = 1.9^\circ$ , taking into account the difference in wavelength between the two techniques. The values of the angle given in the parentheses are the corrected angles for a wavelength of  $0.80$  nm. The spin-flip scattering is considered as dominant at  $\alpha_f = 2.7^\circ$  in Fig. 3(a). The measured and simulated profiles at  $\alpha_f = 2.7^\circ$  in Fig. 3(a) are shown in Fig. 3(b). The determination of  $\xi_{\parallel}$  in the GISAS is simpler and more reliable than that in the OSS since the intensity profile along  $q_y(2\theta_f)$  does not depend on the Fresnel coefficients in the layers. The simulated peak profile becomes sharp with increasing  $\xi_{\parallel}$  and the peak broadening due to the scattering is very small for  $\xi_{\parallel} = 1000$  nm, which would be close to the upper limit of the accessible range of  $\xi_{\parallel}$  for the GISAS measurement using a pin-hole-type SANS instrument because a well-collimated beam with a relatively long wavelength was used in this measurement. In addition, the in-plane component of the coherence volume,  $l_y \approx 2\pi/\delta q_y$ , is estimated to be  $l_y \lesssim 1000$  nm because  $\delta q_y$  is in the order of  $10\%$  of  $q_y$ . It is no longer possible to discuss the length scale of the in-plane structure with  $\xi_{\parallel} \geq 1000$  nm using this GISAS setup. For an in-plane structure with larger length scale, more reliable parameter would be obtained by the OSS.

### 3. Conclusions

The accessible range of the in-plane component of  $\mathbf{q}$  and the relation between the in-plane component of the coherence volume and length scale of the in-plane structure of the sample for the OSS and GISAS measurements with polarized neutrons were discussed using the measured data for the Fe/Si multilayer with the simulation based on the DWBA.  $\xi_{\parallel}$  for the magnetic scattering of this sample was determined to be  $325 \pm 80$  nm, where the result of the GISAS was more reliable [6]. To precisely determine  $\xi_{\parallel}$ , the condition of  $q_{x,\max} \cdot \xi_{\parallel} \gg 1$  has to be satisfied

<sup>2</sup> Figure 3(a) is reprinted from [6], with permission from Elsevier.



**Figure 3.** (a) Measured polarized neutron GISAS image of an Fe/Si multilayer. (b) Measured ( $\circ$ ) and simulated (lines) GISAS profiles along the solid line in (a). Black, red, and blue line represents the simulated profile with  $\xi_{\parallel} = 325, 500$ , and  $1000$  nm, respectively. Direct beam profile ( $\bullet$ ) is plotted with scaling. Statistical errors for the direct beam profile are smaller than the size of the symbols.

for the OSS (usually  $\xi_{\parallel} \gtrsim 100$  nm), whereas  $\xi_{\parallel} \lesssim 1000$  nm is needed for the GISAS. For the overlapping length scale of  $100 \lesssim \xi_{\parallel} \lesssim 1000$  nm, the accuracy of the obtained parameter can be improved by performing both OSS and GISAS measurements.

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## References

- [1] Grünberg P, Schreiber R, Pang Y, Brodsky M B and Sowers H 1986 *Phys. Rev. Lett.* **57** 2442
- [2] Baibich M N, Broto J M, Fert A, Nguyen Van Dau F, Pertroff F, Etienne P, Creuzet G, Friederich A and Chazelas J 1988 *Phys. Rev. Lett.* **61** 2472
- [3] Binasch G, Grünberg, Saurenbach F and Zinn W 1989 *Phys. Rev. B* **39** 4828
- [4] Zabel H, Theris-Bröhl K and Toperverg B P 2007 *Polarized neutron reflectivity and scattering from magnetic nanostructures and spintronic materials Handbook of Magnetism and advanced magnetic materials* ed Kronmüller H and Parkin S (Chichester: John Wiley & Sons Ltd.) pp 1237
- [5] Salditt T, Metzger T H and Peisl J 1994 *Phys. Rev. Lett.* **73** 2228
- [6] Maruyama R, Bigault T, Wildes A R, Dewhurst C D, Soyama K and Courtois P 2016 *Nucl. Instrum. Methods Phys. Res. A* **819** 37
- [7] Toperverg B P 2002 *Appl. Phys. A* **74** (Suppl.) S1560
- [8] Kentzinger E, Rücker U, Toperverg B, Ott F and Brückel T 2008 *Phys. Rev. B* **77** 104435
- [9] Høghøj P, Anderson I, Siebrecht R, Graff W and Ben-Saidane K 1999 *Physica B* **267-268** 355
- [10] Cubitt R and Fragneto G 2002 *Appl. Phys. A* **74** (Suppl.) S329
- [11] Andersen A H, Cubitt R, Humblot H, Jullien D, Petoukhov A, Tasset F, Schanzer C, Shah V R and Wildes A R 2006 *Physica B* **385-386** 1134
- [12] Maruyama R, Yamazaki D, Ebisawa T and Soyama K 2009 *J. Appl. Phys.* **105** 083527
- [13] Kentzinger E, Rücker U, Toperverg B, Brückel T 2003 *Physica B* **335** 89.
- [14] Dewhurst C D 2008 *Meas. Sci. Technol.* **19** 034007
- [15] Dewhurst C D 2012 *Nucl. Instrum. Methods Phys. Res. A* **683** 16