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Analysis of artificial silicon microstructures by ultra-small-angle and spin-echo small-angle neutron scattering

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Abstract

Ultra-Small-Angle Neutron Scattering (USANS) is currently becoming an effective technique for the analysis of structures in the micrometer range. The new Spin-Echo SANS (SESANS) method measures a signal in real space. In both cases microfabricated silicon gratings provide unique test procedures for the related devices and interpretations of the experimental data. A series of one-dimensional gratings was fabricated using a highly anisotropic ion etching technique (RIE) and measured at the USANS instrument S18 at ILL, Grenoble. Grating parameters derived from the experimental data are in agreement with the nominal values. Scattering length density correlation functions calculated from the USANS data are compared to SESANS correlation functions measured at the Delft University of Technology, demonstrating the reciprocity of the two scattering methods. Reconstruction techniques for one-dimensional scattering length density distributions are applied to the USANS data. The results are in good agreement with SEM micrographs of the samples.

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Keywords: USANS; SESANS; Neutron instrumentation; Neutron optics; Reconstruction

1. Introduction

Ultra-small-angle scattering with the use of perfect silicon crystals was developed decades ago for X-rays [1] and adapted for thermal neutrons [2]. This technique provides a resolution of the order of 10^{-5} Å^{-1} in reciprocal space corresponding to μrad in scattering angle and μm in structure size. Improvements of the experimental technique, mainly the tail-suppression method described by Agamalian et al. [3], are presently establishing ultra-small-angle neutron scattering (USANS) for material characterization. The typical structure size probed with the technique ranges from a few tenths of a micrometer up to a few tens of micrometers making this destruction-free method an interesting option for various branches in science and technology, including condensed matter physics, molecular biology and biophysics, polymer science and metallurgy [4].

The new spin-echo small angle scattering (SESANS) technique [5,6] developed at the Delft University of Technology provides structural information in real space. Probed structure sizes are in the range of 10 nm to about 15 μm. The scattering pattern is probed by scattering angle-dependent Larmor precession, which does not require collimation of the neutron beam. The applications are similar as to USANS.

With both methods the measured signal is analyzed in only one direction perpendicular to the incoming beam. Therefore, one-dimensional line gratings are suitable for calibration purposes and for a comparison of results obtained with the two techniques [7]. In the case of

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USANS, information about the spatial structure of the sample has to be calculated from scattering data, i.e. from information in reciprocal space. This usually involves more or less detailed model assumptions about the samples. An approach relying on only minimum assumptions are the so-called direct reconstruction methods. Again, one-dimensional line gratings are ideal samples to examine these methods for USANS data.

Testing devices and methods with model structures of well-known geometries will further pave the way of USANS and the novel SESANS technique towards standard methods. It also helps to test the validity of model calculations. Although the sizes of the scatterers in this case are large, they can be treated in the first Born approximation. However, to obtain direct results for the forward direction, methods like the phase object approximation will have to be applied [7].

2. Silicon line gratings

Artificial periodic structures have already become a topic of interest for fundamental quantization aspects in neutron optics [8–10] and for the demonstration of USANS performance possibilities [11]. For the studies presented here a series of silicon gratings with periods ranging from 12 to 28 μm was fabricated using an anisotropic dry etching process (reactive ion etching, RIE) [12,13]. This technique allows for high aspect ratios of the etched features with good profile control. Due to the nature of the process, a certain variation of the profile with etch depth was to be expected. Trench depths of 40 and 60 μm, in several cases also 80 μm for each grating type allow to test for this effect. Grating periods are not affected by this etch depth dependence of the profile. All fabricated gratings have a nominal trench width of 8 μm while the ridge widths were varied between 4 and 20 μm for different gratings. To match the neutron beam cross-section available at the instrument S18, located at the Institut Laue Langevin, Grenoble, the patterned area of these gratings was chosen exceptionally large at $22 \times 22\,\mathrm{mm}^2$. After optimization of the etching process a nearly ideally rectangular profile of the trenches was achieved [14], see Fig. 1.

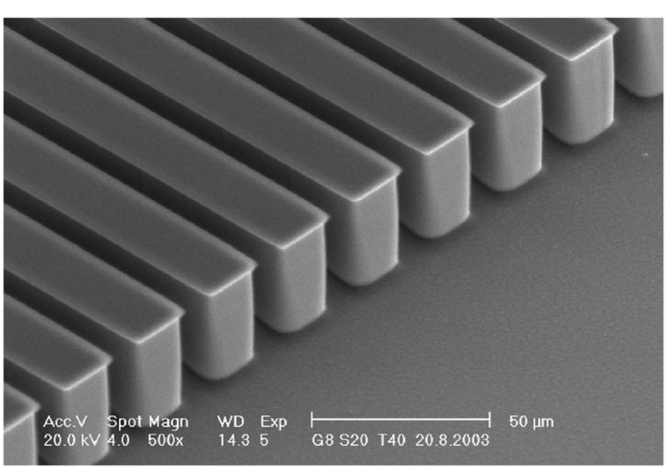

Fig. 1. Grating with period 28 μm, trench width 8 μm, ridge width 20 μm, trench depth 40 μm. The optimized etching process creates trenches with excellent control over geometry and depth of the etched features.

3. USANS measurements

The experiments were performed with the USANS option of the instrument S18 [15,16]. The set-up is a double perfect crystal diffractometer in Bonse–Hart configuration with two triple-bounce channel-cut perfect silicon crystals serving as monochromator and analyzer, which are mounted on a common optical bench, see Fig. 2. USANS patterns are recorded by rotating the analyzer crystal with typical sub-μrad step widths. The rocking curve $I_R(Q)$ (scattering vector Q in horizontal direction perpendicular to the rotation axis of the analyzer and to the incoming beam) of the empty instrument is described by a convolution of the reflection curves of the two perfect crystals as

$$I_R(Q) = I_0 \int R^3(Q')R^3(Q-Q')\,\mathrm{d}Q' \tag{1}$$

where $R(Q)$ is the reflection probability for a single reflection and I_0 is the incoming neutron intensity. Due to inevitable background the measured instrument curve is given by $I_{Rm}(Q) = I_R(Q) + I_B$ with the background intensity I_B which usually amounts to a few neutrons per minute.

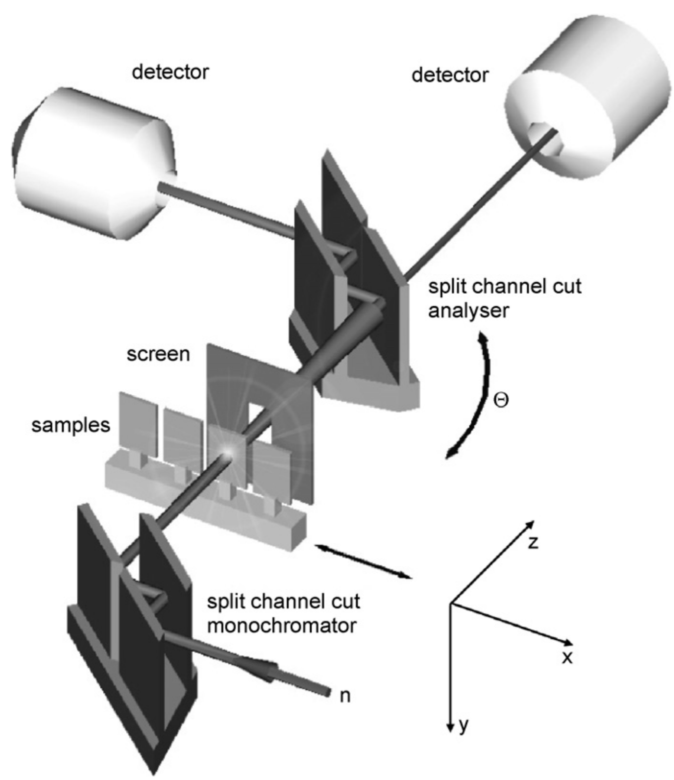

Fig. 2. Scheme of a double crystal diffractometer (Bonse–Hart camera) [15]. The scattered intensity is analyzed in the horizontal plane integrating over contributions in vertical direction.