Ultra-small-angle neutron scattering (USANS) with the use of perfect silicon crystals provides a resolution of the order of 10^{-5} \AA^{-1} in reciprocal space, which corresponds to \( \mu \)rad in scattering angles and \( \mu \)m structures in real space. From small-angle scattering by artificial lattices follows a unique test procedure for the related devices and techniques. Corresponding measurements were performed at the USANS facilities of the Atominstitut in Vienna and of the S18 instrument at the ILL. We observed diffraction patterns from samples being periodically structured in one and two dimensions. These measurements take advantage of the extended coherence function of the set-up and the high quality of the manufactured silicon sample lattices. Due to these characteristics up to 50 interference orders were obtained at the S18 instrument. Scattering from two-dimensional periodic structures was observed for different orientations of the sample which shows characteristic diffraction maps in reciprocal space.

1. Introduction

Ultra-small-angle neutron scattering (USANS), which has been developed decades ago (Bonse & Hart, 1965), is presently becoming an established technique for material characterization in the \( \mu \)m range, mainly because of the new tail-suppression method described by Agamalian et al. (1997). Several examples can be found in the survey of Hainbuchner et al. (2002). Further applications on various topics of materials science can be found in recent literature (Agamalian et al., 2000; Aizawa et al., 2000; Banhart, Bellmann & Clemens, 2001; Buckley et al., 2001; Matsuoka et al., 2000; Mozumder et al., 2001; Radlinski et al., 2000; Staron & Bellmann, 2002; Triolo et al., 2000). Model samples with known parameters may help to understand the basic features of this technique and clarify the performance of the instruments involved. In this work we will compare two instruments, the KWS facility of the Atominstitut in Vienna (Villa et al., 1999) and the USANS option of the neutron optics instrument S18 at the ILL (Hainbuchner et al., 2000). Artificial periodic structures have already become a topic of interest for the presentation of USANS performance possibilities (Hainbuchner et al., 2000; Treimer et al., 2001), and also for fundamental quantization aspects in neutron interferometry (Rauch et al., 2002). We present results from a series of experiments on different silicon lattices which we have performed over the last few years. Since USANS experiments may, in principle, be performed over a \( Q \)-range from 10^{-5} \AA^{-1} up to 10^{-2} \AA^{-1} it is of principal interest to verify that the USANS technique is valid and that the USANS instruments behave properly over this whole \( Q \)-range.

2. Theoretical basics

Interference effects are highlighting examples for the wave nature of neutrons. In principle, neutron interference phenomena may occur from diffraction on any periodic structure. If the distances which characterize the periodicity of the structure are of the order of several \( \mu \)m the corresponding scattering angles are of the order of several \( \mu \)rad for thermal and cold neutrons, and therefore accessible by USANS techniques only. In this section we want to present a summary of the basic quantities needed to describe the related effects. In any case, it is clear that the spatial width of the wave packet associated with the incoming neutron beam has to be sufficiently large to cover the spatial extension of the respective diffraction object. In other words, the coherence length of the neutrons has to be of the same order of magnitude as a certain size of the periodic sample. The theory for neutron diffraction by artificial lattices may essentially be taken from light optics (eg. Born & Wolf, 2002, pp. 446). In the case of USANS, however, the effect of the instrument on the scattering curve has to be taken into account additionally.

2.1. The scattering curve

Each USANS instrument is characterized by its own instrument resolution curve, often called the rocking curve, which arises from the rotation of the analyzer crystal with respect to the fixed monochromator crystal. The rocking curve of the empty instrument is basically obtained by the angular convolution of the reflectivities of both crystals plus an inevitable environmental background. The rocking curve depends on both the properties of the reflecting crystals and the set of lattice planes chosen for neutron reflection. Fig. 1 shows the instrument curves for the USANS facilities at the Atominstitut (KWS) and at the ILL (instrument S18). The bottom axis in Fig. 1 has been labelled by \( Q_y \). In reality, the corresponding values were derived from the rotation angle of the analyzer crystal \( \theta \) and the neutron wave number \( k \) by \( Q_y = k \theta \). The meaning of this notation is that the instrument curve, \( R(Q_y) = R(k \theta) \), is superimposed on any scattering signal in the shown \( Q_y \)-range which has to be properly taken into account for data analysis. The \( y \) subscript indicates that only scattering in one direction will be resolved by the USANS double crystal technique (in our cases the horizontal direction perpendicular to the neutron beam). The ATI instrument uses silicon 331 reflections at a wavelength of 1.76 \( \AA \) \( (\theta_B = 45^\circ) \) while the equivalent data for the S18 instrument are Si(220) and 1.9 \( \AA \) \( (\theta_B = 30^\circ) \). The dynamic range of S18 is about 3 orders of magnitude larger owing to its peak intensity to background ratio. The FWHM on the \( Q_x \)-scale amounts to \( 2 \times 10^{-5} \AA^{-1} \) for S18 and \( 10^{-5} \AA^{-1} \) for KWS with direct consequences for the resolvable structures.

The scattered intensity \( I_S \) by any homogeneous small-angle scattering object is essentially given by

\[
I_S(Q) = \alpha I_0 (|N_2| - |N_B|) |F(Q)|^2,
\]

where \( \alpha \) is a constant containing properties of the neutron beam and the sample, \( I_0 \) the incoming neutron flux, \( |N_B| \) the coherent neutron scattering length density of the sample, and \( |N_2| \) of the surrounding material. The scattering function \( |F(Q)|^2 \) is derived from the Fourier transform of the spatial region making up the scattering object.

Furthermore, the intensity \( I_m \) which is measured in the USANS double crystal diffractometer follows from the intensity \( I_S \) after slit height smearing (over the vertical direction \( x \)) and convolution with the instrument curve \( R(Q_x) \)

\[
I_m(Q_x) = \int dq_y \int dq_x I_S(Q_x,q_y) R(Q_x-q_y) + \int dq_y I_B(Q_y) + I_B,
\]

where \( I_B \) denotes the total scattering probability and \( I_B \) the inevitable background intensity. It should be emphasized that this relation is taken mainly for its practical purpose for data evaluation and that a more complex treatment may be found elsewhere (e.g. Carsuighi et al., 1997).