Diffraction/Scattering

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A compact UHV X-ray diffractometer for studying surfaces and interfaces

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A compact ultra-high-vacuum (UHV) X-ray diffractometer for surface glancing X-ray scattering has been constructed. All the equipment, including a rotating-anode source of 18 kW and a UHV specimen chamber, is arranged on one optical table of dimensions 70×90 cm. The specimen chamber is 14 cm in diameter and 20 cm high and can be evacuated up to 3×10^{-8} Pa. It is equipped with two Be windows of thicknesses 0.2 and 0.4 mm. Specimen orientation in the vacuum is controlled from the outside. The specimen can be heated up to 773 K. The chamber has two evaporation cells and can be used for *in situ* observations of growing crystal surfaces. Using this instrument, we observed a mechanically polished Ag surface and successfully made an *in situ* observation of the layer-by-layer growth of a PbSe(111) surface. The instrument will be useful for preliminary experiments using laboratory sources, prior to final measurements at synchrotron radiation facilities.

Keywords: X-ray glancing-angle scattering; surface roughness; crystal growth.

1. Introduction

In recent years, many studies of surfaces and interfaces have been made using glancing-angle X-ray scattering techniques. Very intense X-ray sources and ultra-high-vacuum (UHV) specimen chambers are necessary to perform experiments of this kind. Marra *et al.* (1979) incorporated a 60 kW rotating-anode X-ray source into their molecular beam epitaxy (MBE) system and first showed that glancing-angle X-ray scattering is effective for investigating semiconductor surfaces. Eisenberger & Marra (1981) reported the use of a baby chamber in their study of a Ge(001) reconstructed surface. They used a 60 kW rotating-anode X-ray source together with the baby chamber and showed that glancing-incidence X-ray diffraction experiments have an adequate sensitivity for determining the structure of monolayers.

After the emergence of synchrotron radiation sources, several instruments for glancing-angle X-ray scattering have been designed and constructed at beamlines at synchrotron radiation facilities. Brennan & Eisenberger (1984) described a novel X-ray scattering diffractometer for studying surface structures under UHV conditions. Fuoss & Robinson (1984) constructed an instrument for surface X-ray diffraction in UHV on beamline X16A at the NSLS. Vlieg *et al.* (1987) built a UHV chamber combined with an MBE system, which is part of a surface X-ray diffraction set-up at the wiggler beamline of the synchrotron radiation source at Daresbury. Ferrer & Comin (1995) built a surface diffraction station, which consists of a six-circle diffractometer and a UHV system with a welded Be window, on beamline BL7 at the ESRF.

All the above instruments used very intense X-ray beams from synchrotron radiation sources or from rotating-anode sources. Although the baby chambers may be small, they have no functionalities for specimen preparation.

For surface X-ray glancing scattering experiments to be performed in a small laboratory room, the present authors constructed a compact UHV X-ray diffractometer (Fujii *et al.*, 1997). Significant improvements were made in its simplicity, economy and ease of its construction and usage. An 18 kW rotating-anode source, a UHV specimen chamber and a two-circle diffractometer are all arranged on just one optical table of dimensions 70×90 cm. The UHV chamber is as small as a baby chamber, but has two evaporation cells and can be used for *in situ* observations of growing crystal surfaces. Its performance and a few results of experiments using this instrument will be described.

2. Description of the instruments

Figs. 1(*a*) and 1(*b*) show schematically a front and a top view of the whole set-up of the present UHV diffractometer, respectively. On the left-hand side of both figures, an 18 kW rotating-anode X-ray source is shown. The experiments were performed with a Cu $K\alpha_1$ X-ray beam from the rotating-anode source (wavelength 1.54 Å, energy 8 keV). The beam source is 0.7 mm wide and 0.5 mm high, and has a maximum brilliance of 51.4 kW mm^{-2} when operated at 18 kW (60 kV, 300 mA).

The beam from the source is introduced into an incident monochromator through the collimating slits-1, whose opening is 0.5 mm wide and 0.2 mm high. The incident monochromator is made of a usual Ge(111) channel-cut crystal. The first incident surface of Ge has an offset of 11.2° from the (111) surface and increases the X-ray brilliance from 1 to 10. The cross section of the X-ray beam coming out of the monochromator is 0.05 mm wide and 0.2 mm high. The beam is then introduced into the UHV chamber through the collimating slits-2, whose opening is 0.05 mm wide and 0.1 mm high. The slits-2 determines the divergence of the incident beam. The divergence is 0.3 mrad measured from the specimen surface in the horizontal direction and 1.5 mrad in the vertical direction in Fig. 1(*a*).

Thus, a well collimated X-ray beam enters the UHV specimen chamber through a Be window, 0.2 mm thick and 30 mm in diameter. The beam is then incident upon the specimen surface at a glancing angle θ_i. The specimen is set, at present, with its surface normal lying in the horizontal direction.

Scattered X-ray beams from the specimen surface in the UHV chamber are taken out through another Be window, 0.4 mm thick and 60 mm in diameter, shown on the right-hand side of the UHV chamber, limited by slits-3, and received by a scintillation counter. The acceptance angle of the scattered X-ray beams is 0.3 mrad in the horizontal direction and 0.6 mrad in the vertical direction in Fig. 1(*a*).

The UHV chamber and a scintillation counter are mounted on a two-circle θ_i–θ_s diffractometer. The position of the diffractometer can be adjusted horizontally and vertically with the use of *YZ* crossed roller motions. The UHV chamber can be rotated by θ_i around the vertical axis up to 13°. The scintillation counter is also rotated by θ_s around the same vertical axis up to 145°. The

scintillation counter is mounted on a φ_s rotation mechanism which is superposed upon the θ_s rotation mechanism of the θ_i–θ_s diffractometer. The φ_s rotation is around the horizontal y axis, which is normal to the plane of Fig. 1(*a*) and is schematically shown at the top of Fig. 1(*b*).

The UHV chamber is equipped with two evaporation cell ports for crystal growth study and we can make *in situ* observations of the growing surfaces by X-ray glancing scattering technique. The chamber has an inner diameter of 14 cm and is 20 cm high. After baking at 423 K for 24 h, the chamber can be evacuated down to 3×10^{-8} Pa.

A specimen in the UHV chamber is mounted on a holder which is connected to the rotary-motion feedthrough, as shown in the upper part of Fig. 1(*b*). This rotary feedthrough, supported on crossed swivels *via* flexible bellows, allows us to control the azimuthal φ rotation around the y axis in the vacuum. This feedthrough also transmits the translations of the four-axis goniometer to the specimen in the UHV chamber. The specimen can be moved in the Y and Z directions, which lie in a plane perpendicular to the incident X-ray beam. It can also be tilted by χ around a horizontal axis which is perpendicular to the y axis. Thus, the specimen may move freely in five ways, *i.e.* translations in Y and Z directions, and φ, χ and θ_i rotations.

The specimen holder has a heater to anneal the specimen in UHV, which can heat the specimen to 773 K. This arrangement allows analyses of the growing surfaces at different temperatures.

The angular resolutions of the θ_i and θ_s rotation mechanism of the two-circle diffractometer are both 0.001°, and their angular accuracy in a coupled rotation is claimed to be less than 0.02°. The deviation between the centre of the θ_i rotation and that of the θ_s rotation is claimed to be less than 20 µm. The diameter of the confusion sphere of the whole of the present instrument, which consists of the above 20 µm coupled with the deviations of the four-axis specimen goniometer, is expected to be much larger than 20 µm and will be measured in the future.

Fig. 2 is a photograph showing the front view of the complete set-up. On the left-hand side, the rotating-anode X-ray source and the incident monochromator are seen. The UHV chamber and the scintillation counter are set on the two-circle θ_i–θ_s diffractometer. A sputter-ion pump is on top of the UHV chamber. The weight of all the equipment on the diffractometer is about 100 kg. Because the diffractometer has a weight limitation, the sputter-ion pump is hung on a counter balance, which is at the back and not visible in the figure. The evaporation cells have been removed in Fig. 2.

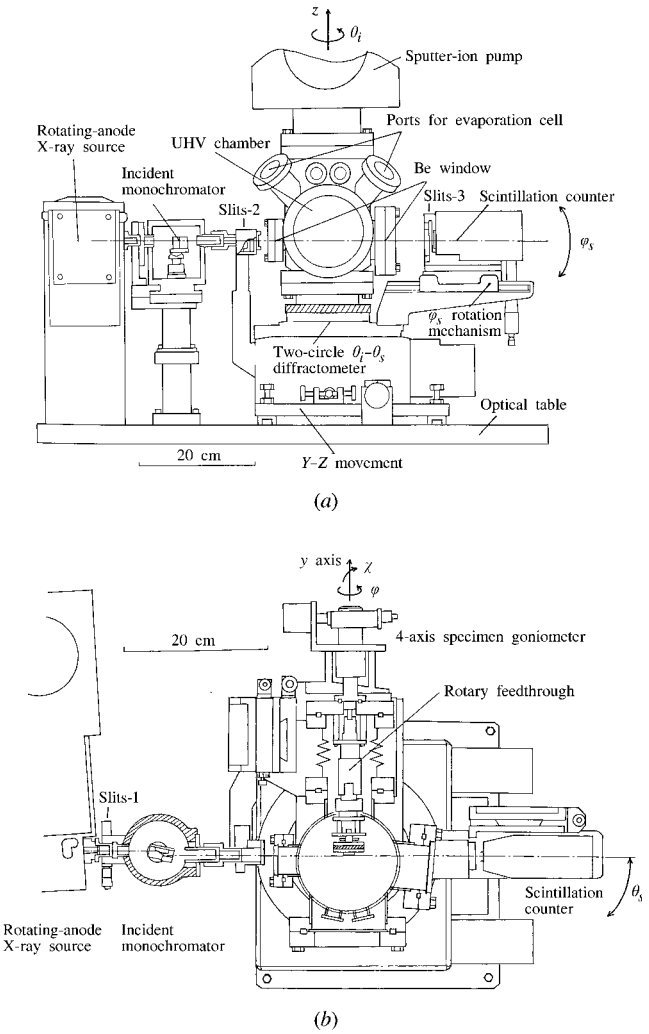

Figure 1
(*a*) A front view of the whole set-up of the present apparatus. The upper part above the hatched plate is mounted on the θ_i rotation mechanism. (*b*) A top view of the whole set-up. The upper part of the figure shows the four-axis specimen goniometer.

3. A few results

The preliminary measurements were performed on a silver polycrystal surface, 10 × 10 mm square, which was mechanically polished by alumina emery papers of powder diameter 0.3 µm. The results are shown in Fig. 3.

The sharp peak on the left in Fig. 3 is an angular distribution of an incident X-ray beam in the horizontal direction when the power of the Cu rotating-anode source was 60 kV, 300 mA. The

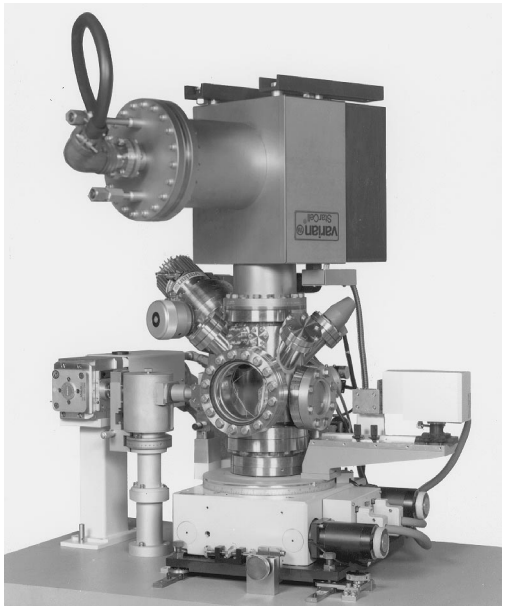

Figure 2
Photograph of the whole set-up of the UHV diffractometer. A sputter-ion pump seen at the top is hung on a counter balance not seen in the figure. The two evaporation cells had been removed when the photograph was taken.

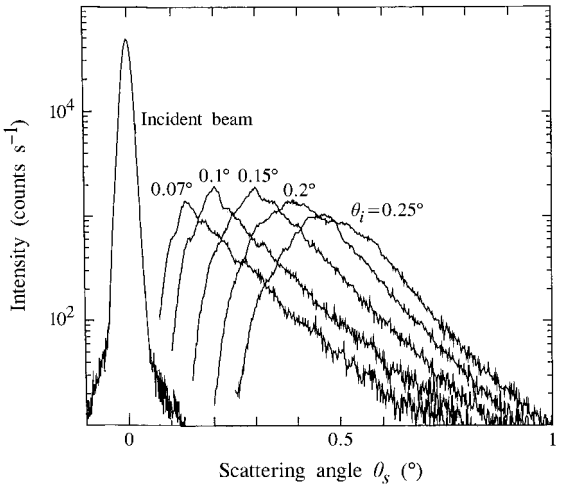

Figure 3
An angular θ_s distribution of an incident 8 keV X-ray beam, and angular θ_s distributions of the scattered X-ray intensity from a polished polycrystalline silver surface at several glancing incident angles, θ_i.

beam divergence is about 0.3 mrad full width at half-maximum. The maximum incident beam intensity is 45000 counts s^{-1}.

At the five fixed glancing incident angles θ_i, angular θ_s distributions of the scattered X-ray intensity were measured. The counting rates of specularly reflected X-ray beams are of the order of 10^3 counts s^{-1} for these standard operating conditions, as shown in Fig. 3. These intensity profiles will be enough to interpret the surface structures. We consider that these angular distributions include information about the surface roughness. When the surface is rough, the 00 truncation rod in the surface reciprocal space will be smeared out and the curves of the distribution become low and broad. The results of Fig. 3 can provide an estimate of the roughness of the polycrystalline silver surfaces.

It should be pointed out that the whole arrangement is compact. All of the instruments are set on only one optical table. In some cases the measurements by this instrument in a small laboratory room will serve as preliminary results to larger-scale expensive measurements using more intense X-ray beams from synchrotron radiation facilities. This instrument may also be used on a synchrotron radiation source itself instead of the rotating-anode source.

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