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An out of plane detector for surface X-ray diffraction.

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Abstract:

An out of plane detector movement has been built for the detector arm of the surface X-ray diffractometer on the wiggler beam line 9.4 on the synchrotron radiation source at Daresbury Lab. For a relative small cost,significantly increases the volume of reciprocal space that can be accessed with a corresponding improvement in the accuracy of the determination of the interatomic spacings.

We describe a simple modification to the detector arm of a diffractometer designed for surface X-ray diffraction (SXRD). For relatively little cost, it significantly increases the volume of reciprocal space that can be accessed.

With the introduction of high intensity synchrotron radiation sources, SXRD is now established as a major tool for the study of atomic structure at surfaces and interfaces. Many systems have been studied [1] and the technique has been extended to the monitoring of epitaxial growth in real time [2]. The power of the technique stems from the weakness of the interaction of short wavelength photons with matter which allows the scattered intensities to be interpreted within the kinematic scattering approximation, thus avoiding the complications of multiple scattering which characterise some other surface sensitive technique.

The diffractometer (fig 1) on the wiggler beamline 9.4 on the synchrotron radiation source at Daresbury Lab, is based on a modified four circle arrangement [3]. To exploit the horizontal polarisation and anisotopic resolution function, the axis containing the concentric delta (δ) and omega (ω) axes is fixed in the horizontal plane and the scattering plane containing the momentum transfer vector is close to the vertical The position of the environmental chamber precludes the use of a standard chi-circle (χ) and is replaced by two arcs of limited ($\pm$ 20°) movement. A fifth circle alpha (α) allows the grazing angle to be set independantly and increases the volume of reciprocal space that can be scanned. In practice, this is restricted by the dimensions of the beryllium window on the chamber and the beam line.

The volume in reciprocal space that can be reached with the detector in the normal position, that is, with its axis perpendicular to the ω–δ axis of the diffractometer is shown by the lighter shading in fig 2. It corresponds to an

X-ray wavelength of 0.8 Å with a beryllium window that subtends an angle of 20° to the sample and a maximum movement of the detector arm of 140°. For comparison, the accessible region of reciprocal space that can be reached for a low energy electron diffraction (LEED) experiment operating at normal incidence and with electron energy up to 150 eV, is also shown. For the determination of interatomic distances it is clear that SXRD in this arrangement gives better in -plane accuracy (typically ±0.02Å) than out-of-plane (typically ±0.10Å). The converse is true for LEED.

The accessible region of reciprocal space along the surface normal can be increased by adding a sixth (γ) table on the detector arm, with its axis orthogonal to the δ axis. This is, however, expensive; it limits the weight of the detector and introduces some redundancy in the control system since the χ arcs already provide the necessary fine movement. We have provided a simple solution to this problem which involves a mounting with two positions for the detector at γ=0 (the normal position) and γ=15°. As shown in fig.2, these positions define two overlapping regions of reciprocal space. The simplicity of the arrangement allows the mounting to be sturdy enough to accept a large liquid nitrogen cooled germanium detector.

The out of plane detector has two major elements, the detector mount and the detector beam pipes and associated components.The detector is clamped into two split synthetic resin bonded fabric cradles. This unit, in turn, slides between two pairs of curved aluminium beams, the cradles are held in place by bolts which pass through the slots in the top and bottom beams. The bolts position the detector and cradles in the beams and ensure that the detector points at the sample and also lock the detector and cradles in position after moving.

The two pairs of curved beams are machined from D79 Aluminium Tooling Plate which is inherently very stable; they were machined from the solid on a C.N.C. milling machine. The radii of the beams, 1134 mm and

934mm, are centred on the centre of rotation of the diffractometer. The beams are rigidly mounted to a table which is mounted onto a the dovetail slide on the detector arm. The stainless steel beam pipes are mounted from an aluminium plate, which is rigidly mounted to the Huber dovetail on the detector arm. The beam pipes have Kapton windows on each end and are continuously pumped to ≤ 1 mbar by a rotary pump .

Each beam pipe has its own set of post sample slits and detector slits . The post sample slits which are Huber 3011 4 jaw self centering are mounted on 2 MF 04 CC Microcontrolle translation stages at 90° which allow ± 4 mm of horizontal and vertical movement. The translation stages, which are fitted with encoders, are mounted off the aluminium plate and move independently of the beam pipes.The detector slits which are Huber 3010 independant 4 jaw slits, are mounted onto the end of the beam pipes. The translation stages and the D. C. motors that drive the detector slits can be operated remotely from outside the X-ray hutch. All the aluminium components except the curved beams, are machined from 6082 T651.

Once set up, changing the region of reciprocal space to be scanned involves a simple manual movement of the detector between the two positions without the need for realignment and a single instruction to the computer control, to run the six circle angle calculation code [4].

The arm is in routine use. An illustration of the increase in data that is available is shown in fig.3 for a scan along the (2 0) rod of the Si(111) surface after deposition of 0.5 monolayers of Er. [5]. Three Bragg peaks are reached rather than one. The asymmetric profile either side of the outer peaks is due to the in-phase and out-of-phase interference of the scattered waves from the surface and the bulk regions. Its measurements, is crucial for the accurate determination of the vertical position of the adsorbate structure.

References:

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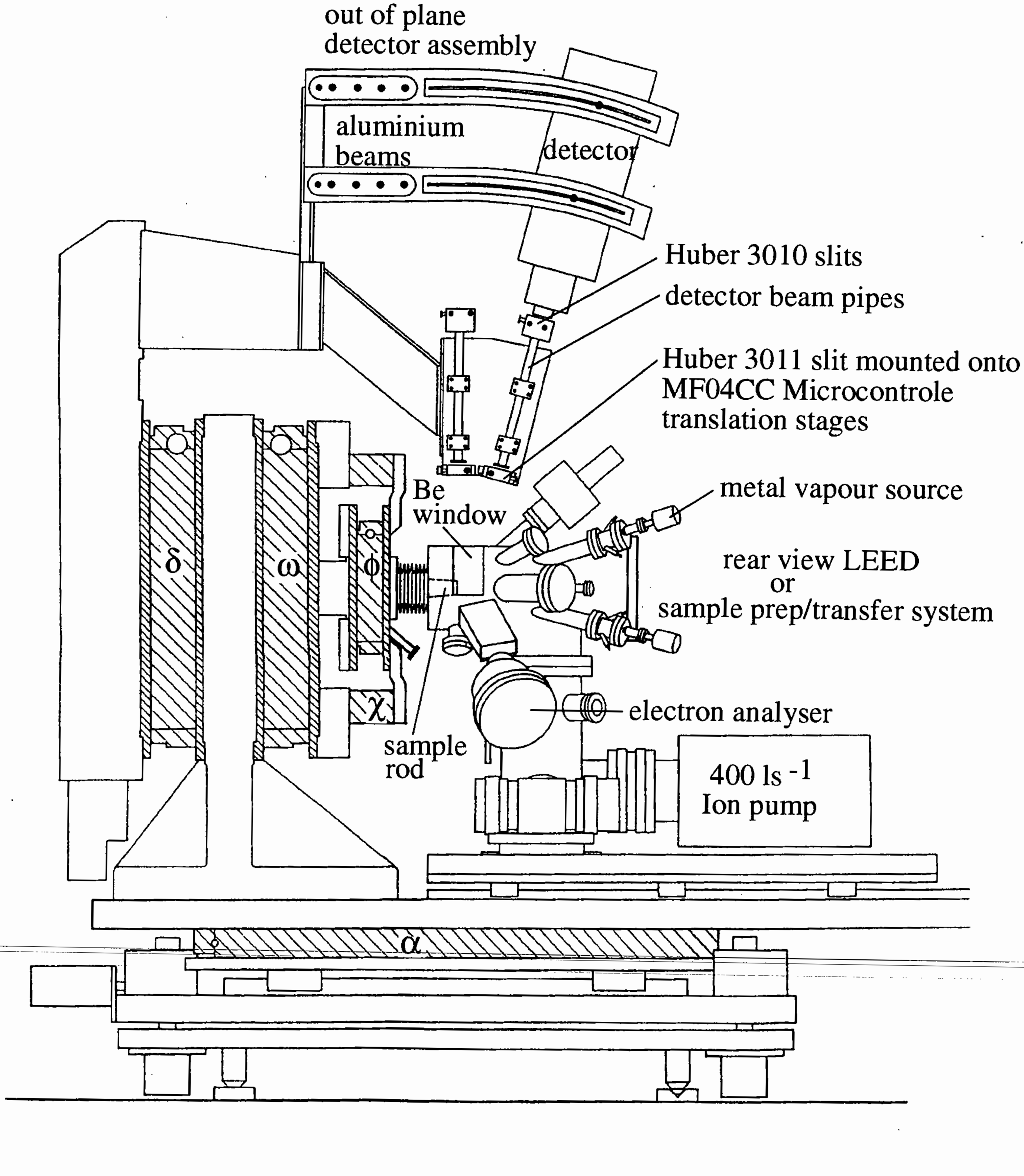
Figure Captions:

Fig. 1. Schematic diagram of the out of plane assembly mounted on to the surface X-ray diffractometer. The diffractometer is coupled to an environmental chamber designed for the study of epitaxial growth.

Fig. 2. The region of reciprocal space for Si(111) accessed by the diffractometer is approximately a flat disc oriented parallel to the surface plane. The discs defined by the two positions $\gamma=0$ and $\gamma=15$ are shown in profile.

Fig. 3. A scan along the (2,0) rod for the 0.5ML of Er on Si(111) [4],The points obtained with $\gamma=0°$ are shown by the filled in circles and those for $\gamma=15°$ by the open circles.



out of plane
detector assembly
aluminium
beams
detector
Huber 3010 slits
detector beam pipes
Huber 3011 slit mounted onto
MF04CC Microcontrole
translation stages
Be
window
metal vapour source
rear view LEED
or
sample prep/transfer system
δ
ω
φ
χ
sample
rod
electron analyser
400 ls -1
Ion pump
α

Fig.1

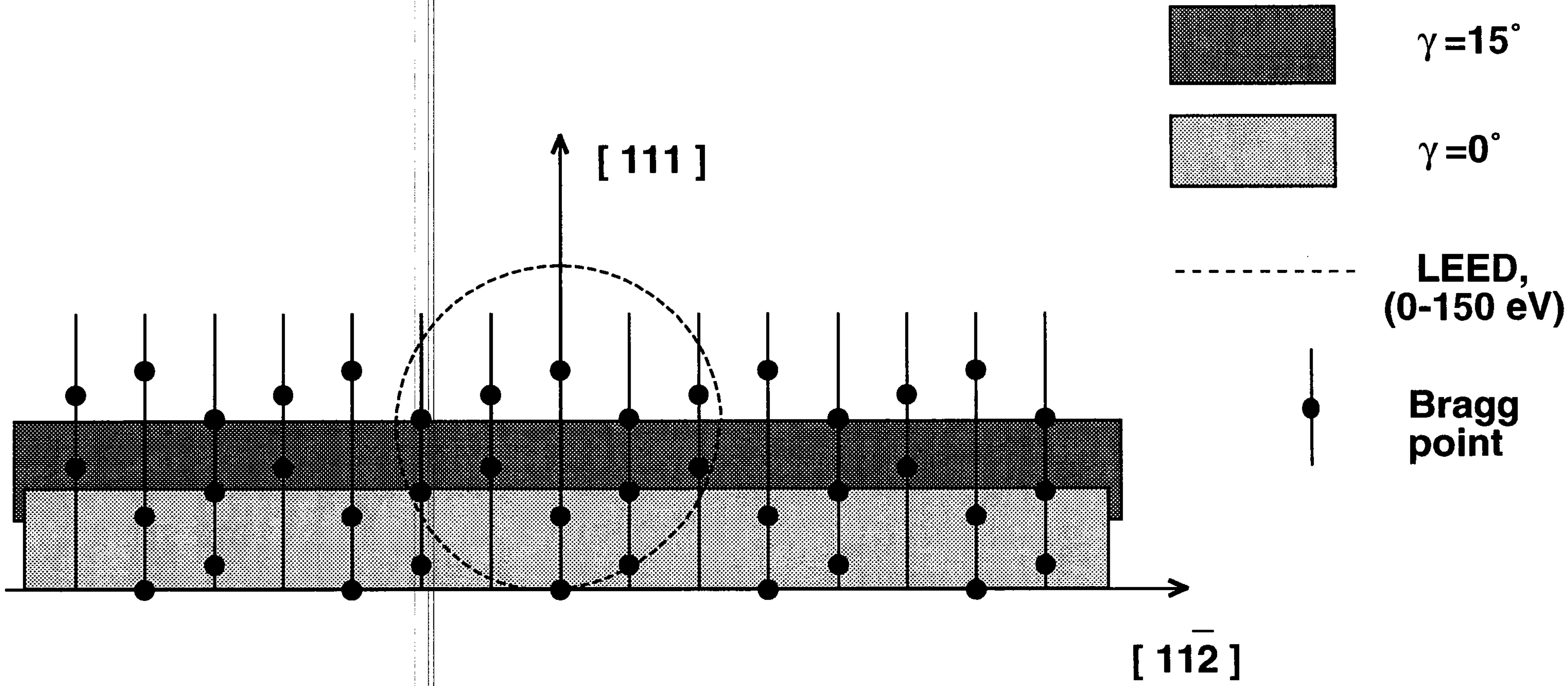
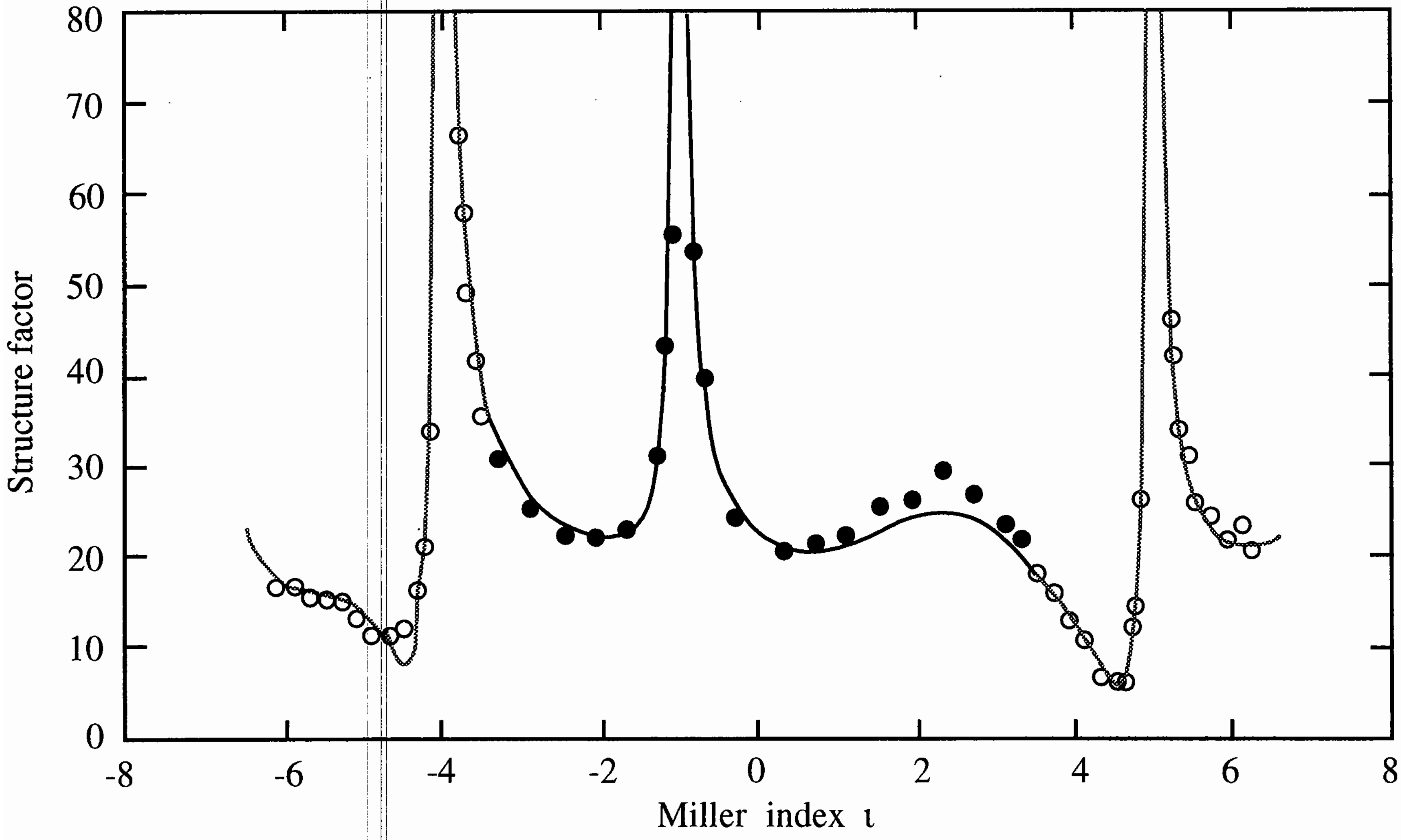


Fig.2



80
70
60
50
40
30
20
10
0
Structure factor
-8
-6
-4
-2
0
2
4
6
8
Miller index ι

Fig.3