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B. Dobson, S. Hasnain, M. Hart, M.J. van Der Hoek, P. van Zuylen. AN INVESTIGATION OF THE PERFORMANCE OF A NOVEL DOUBLE CRYSTAL X-RAY MONOCHROMATOR FOR EXAFS AND XANES MEASUREMENTS. *Journal de Physique Colloques*, 1986, 47 (C8), pp.C8-121-C8-125. 10.1051/jphyscol:1986822 . jpa-00226132

HAL Id: jpa-00226132

<https://hal.science/jpa-00226132>

Submitted on 1 Jan 1986

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AN INVESTIGATION OF THE PERFORMANCE OF A NOVEL DOUBLE CRYSTAL X-RAY MONOCHROMATOR FOR EXAFS AND XANES MEASUREMENTS

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Abstract

A new double crystal x-ray monochromator for EXAFS and XANES measurements is described. The monochromator is designed to provide high intensity with good resolution by the use of bent crystals, when used on line 8 of the Daresbury Synchrotron Radiation Source. The details of the monochromator design have been reported before [1]. Examples of EXAFS spectra demonstrate the success of this design. Further aspects of the performance are considered. The stability of the monochromator, its ability to remove harmonics from the monochromatic beam and the use of doubly bent crystals to provide focussing are reported.

Finally, the appearance of anomalies in the output of the monochromator is shown to arise from multiple diffraction effects. These effects and efforts to minimise or remove them are discussed.

1. INTRODUCTION

The EXAFS station on line 8, station 8.1, has become operational this year. The monochromator is a novel design which has proved to be exceptional in its ability to produce a high intensity of monochromatic x-rays with accompanying good resolution. Some anomalies in the output of the monochromator (commonly known as 'glitches') have been attributed to multiple diffraction phenomena in the crystals. The effort expended in understanding and minimising these effects is of general interest for all crystal monochromators which are used for EXAFS measurements.

This report will summarise the design details of the monochromator and briefly discuss work that has been carried out. A discussion of 'glitches' is included.

2. THE DESIGN OF THE MONOCHROMATOR

The monochromator was designed and built in The Netherlands by the Institute of Applied Physics (TPD), Delft. It is installed on line 8 of the SRS. A fuller description of the monochromator is available elsewhere [1,2], but for completeness we briefly describe the design below.

The source of radiation for the beamline is from an upstream tangent point on dipole 8 (which gives the beamline its number) of the storage ring, where the source size is 4.5×1.1 mm, and the maximum horizontal aperture of the EXAFS station is 5 mrad. The monochromator is located 16 m from the tangent point, enclosed in a high vacuum vessel. There are beryllium window valves either side of the monochromator vessel for vacuum protection. It is hoped in the future to be able to

Fig.1. The absorption spectrum of a 10 μm copper foil at the K-edge. The inset shows in detail the edge feature demonstrating the high resolution achievable with this monochromator.

Fig.2. The absorption spectra of a 10 μm chromium foil at the K-edge, (a) was recorded with the monochromator crystals offset to achieve harmonic rejection, (b) was recorded with no harmonic rejection. Note the decrease in the effect of the glitch with increased rejection of harmonics.

open these to reduce absorption losses. A post-focussing mirror is located 2 m after the monochromator, again enclosed in a high vacuum vessel. Finally, there is a beryllium window valve immediately before the sample and detector stage. Immediately following the monochromator vessel, and just before the final beryllium window, are two sets of motorised slits, which allow control of the horizontal and vertical sizes of the monochromatic beam.

The monochromator is a double crystal design which uses three separate pairs of crystals to cover the wavelength range 0.9 - 4.0 Å. Two of the pairs consist of Si220 crystals which cover the ranges 0.9 - 1.4 Å and 1.1 - 2.5 Å. The third crystal pair uses Si111 crystals to cover the 2.0-4.0 Å range. As the full radiation load falls on the first crystal it is water cooled. All the crystals are pre-bent in the plane of diffraction to correct for the vertical divergence of the x-ray radiation from the storage ring. The second crystal is moved by a stepper

motor along an angled track as the monochromator wavelength is varied, so that the diffracted beam from the first crystal always falls centrally on the second. This also helps maintain the position of the beam on the sample. The resulting movement of the beam during scans is very small, e.g. over a 1 keV scan at 10 keV beam movement is 150 microns. A small DC motor-driven micrometer allows the second crystal to be tilted relative to the first. This permits the transmission of the monochromator to be fine-tuned to maximise output or to reject higher order harmonics. This motor is controlled from a servo-driver unit which stabilises the output of the monochromator.

The crystal pair for the shortest wavelength range (0.9 - 1.4 Å) relies on the second crystal to focus the monochromatic beam. As well as being bent on the plane of the diffraction, this crystal is also bent to a radius of 2.5 m perpendicularly to the plane of diffraction. The focussing action of this crystal is of course dependent on the wavelength of the beam, but produces a spot size of less than 3×2 mm at the sample for a beam energy of 11 keV.

3. TYPICAL RESULTS OBTAINED WITH THE MONOCHROMATOR

The detailed discussion of the behaviour of the monochromator has been documented elsewhere [1,3,4]. Figure 1 shows an example of an absorption spectrum of a 10 micron copper foil obtained with the focussing Si220 crystal set, the monochromator accepts the full vertical height of the beam. The resolution is better than 2 eV, which can be judged by the width of the feature on the absorption edge. The intensity of the monochromatic beam is over ten times that of a conventional EXAFS experiment where a toroidal mirror prefocusses the beam on to a double flat crystal monochromator and where intensity has to be sacrificed by reduction of the vertical aperture to obtain similar resolution.

Since the beginning of this year groups from the UK, the USA and The Netherlands have been using the station. The various crystal sets have been utilised to take data ranging in energy from the calcium K-edge at 4 keV up to the gold L_{III} edge at 11.9 keV, both in transmission and fluorescence modes. Data has been taken over a diverse selection of samples ranging from catalytic materials containing various transition metal compounds to biologically important materials, especially iron containing complexes such as the iron transport protein transferrin.

4. MONOCHROMATOR OUTPUT ANOMALIES, OR 'GLITCHES'

In the course of testing the monochromator it became apparent that at some positions the output of the monochromator undergoes significant changes in output intensity over small (typically 10 millidegrees) changes in Bragg angle. These fluctuations do not fully ratio out when the absorption spectrum is calculated from the signal (I) and reference (I_0) channels. These effects are known in other monochromators [5,6,7] and are generally referred to as glitches. Figure 2 shows an absorption spectrum of chromium foil at the K-edge. The dips in the spectrum at 6.04 keV are examples of typical glitches. The two spectra are recorded with different amounts of harmonic rejection ($I_0/I_{0,max} = 10\%$ and 50%) and show the reduction in the effect of the glitch at higher harmonic rejections, an effect which is explained below.

The origin of the monochromator glitches has been discussed before [see, for example ref.8]. They arise from the fact that the diffracting crystals are three-dimensional arrays of scattering centres. As a result, as well as the primary plane of diffraction, there are always other planes within the crystal which can diffract the incoming radiation. At certain Bragg angles the Bragg condition can be satisfied by planes $\{hkl\}$ other than the primary plane of diffraction. In general these $\{hkl\}$ planes will have different d -spacing compared to the primary plane, and thus their reflection curves will be different from the primary plane reflection curves for the selected energy and from harmonics of that energy. It is thought that the resultant change in energy content across the glitch is the primary effect that prevents normalisation of the glitches in EXAFS spectra.

In a crystal monochromator with both crystals in the same orientation it has been suggested that the glitches arising from the first crystal would be transmitted by the second crystal. If the second crystal were to be rotated about a normal to the primary plane of diffraction, this should reduce the effect of the glitches [5,6]. However other sources suggest that this is not so and that such an arrangement would lead to an increase in the number of glitches [7]. As a means of testing the effect of crystal rotation on the monochromator performance, a flat silicon wafer was installed on the medium energy Si220 crystal set. This wafer was cut in such a way that the primary plane of diffraction was the [220] plane, which was oriented parallel to the crystal surface, and the direction along the projection of the incident diffracted beams was the [1,-1,0] direction. Comparison of the glitches recorded with the new arrangement in comparison to those of the original arrangement (with both crystals in the [1,0,0] orientation), showed clearly that the original glitches could still be seen in the new arrangement. In addition there were extra glitches arising from the new orientation of the second crystal. This was confirmed by calculations of the glitch positions for the two crystal orientations carried out by Dr. C.S.G. Cousins [8]. The measured angles correspond to the calculated values to within 5 mdeg, the step resolution when the data were obtained. Only one feature is calculated and not found, and the only features found but not calculated are very small and/or broad. Thus the entire glitch pattern can be explained by a simple superposition of the glitch pattern of the separate crystal orientations.

5. CONCLUSION

The EXAFS station on line 8 is now providing useful beam time for a wide variety of studies. Use of the station by an increasing number of users has encouraged a continuing refinement of the facilities available. The glitches which have been found to be present are becoming better understood and their effects minimised. Such a reduction in the effect of glitches on the spectra can be minimised by increasing harmonic rejection. Efforts are also being made to reduce their effect by better matching of signal and reference channels (especially in fluorescence), by sample geometry and by monochromator output servo systems. However, full eradication of these effects seems unlikely, and much more work on understanding the underlying mechanisms is required to improve the performance of all such crystal monochromators in future.

6. ACKNOWLEDGEMENTS

We would like to thank the SERC and ZWO who provided funds for this work under the terms of the Anglo-Dutch collaboration. The calculations of the glitch patterns were kindly provided by Dr. C.S.G. Cousins of the University of Exeter. Finally we wish to thank the numerous research associates and Daresbury staff who have been involved in this work, especially Drs. J. Harries, J. Arbor, R. Garratt, C. Morrell and J. Baines.

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