On the Design of a Novel High Resolution Xanes Spectrometer

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Abstract

The details of a novel, high resolution (ΔE at Cu K absorption edge region = 0.3eV), compact two crystal x-ray monochromator are presented. The design considerations include besides the resolution and compactness (30cm×30cm×30cm) an easy changeability of crystal monochromators, constancy of input and output x ray beam directions and the impingement of the central ray of the selected x ray beam at the same point on surfaces of both the crystals independent of the Bragg angle θ. The high resolution achievable using the Kohra formulation (Kazutake Kohra J. Phys. Soc. Japan **17**, 589 (1962)) is also discussed. The plan and the elevation of the monochromator are also given and elaborated.

1. Introduction

An x-ray absorption spectrum (XAS) is measured by varying the energy of the incident photon while monitoring the transmitted beam or fluorescent yield or non-radiative electron yield. The observed fine structure around or beyond the onset of an absorption edge in XAS of condensed matter is conventionally divided into two regions : XANES or NEXAFS - the x-ray absorption near edge structure or near edge x-ray absorption fine structure and EXAFS - the extended x ray absorption fine structure which pertains to the oscillations beyond 50eV or so from the absorption edge. While EXAFS has been proved to be a local-structure-sensitive tool, XANES contains a variety of wealthy information such as the density and the symmetry of the unoccupied electron states above E_F-the Fermi energy, bonding, oxidation state, coordination geometry, etc. However, a device for XANES data acquisition should have the energy resolution about 1eV or less besides the high flux source. This is so because sometimes the peaks in this region are separated by 0.5eV and sometimes they are very weak compared to the other peaks in XAS. Although the synchrotron radiation (SR) has been a major source for the XAFS community, the recent work by Sakurai[1-3] has rekindled the interest in the laboratory XAFS (lab-XAFS). Lab-XAFS facility has many advantages including a fast turn around time to modify and implement results.

In an XAS experiment the desire of an experimentalist is to get maximum flux for a given energy resolution as well as the ease and the speed of tunability of energy. The spectrometers designed earlier can be classified as either the focusing type (which give high flux at the sample stage but have very poor energy resolution e.g. 10eV at Cu K absorption edge E ≡ 9000eV) or the non-focusing type (which have good resolution such as 2eV at 9000eV but can produce very bleak output flux at the sample stage).

Besides the high resolution and an increase in the output flux, we also considered in the design of the spectrometer the following needs often sought for by an XAS worker: (a) constancy of input and the output beam direction, (b) easy alignment, (c) to make the central ray of a selected beam hit the same point of both crystals independent of θ or chosen energy, (d) the 2θ range from 10° to 140°, and (e) light weight, compactness and UHV compatibility.

A spectrometer based on the above considerations has been designed and developed recently by the author at the Department of Physics, Goa University, India. In the following sections we discuss the geometrical principle governing the design as well as its mechanism. Also presented are the physics of special crystals used to achieve high resolution and high flux simultaneously.

2. Geometrical Principle

Although the geometrical principle underlying the spectrometer shown in Fig 1, is similar to the one employed earlier by Golovchenko et

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al.[4] it differs with their design in many important aspects as shown below. As shown in Fig 1 the incoming and the outgoing x-ray beams which are parallel to each other are offset by a distance S. Thus if θ is the angle at which the x-ray beam is incident on the first crystal and $D(\theta)$ is the normal distance between the crystals AB and EF, then

$$D(\theta) = S/[2\cos(\theta)]. \qquad (1)$$

Thus one can tune the spectrometer by varying the distance D while keeping the distance S constant. The use of D to get the Bragg angle reduces the dimensions of the monochromator. In their design Golovchenko et al have made use of the linear distance between the two parallel crystals, viz., $S = \chi.\tan 2\theta$, where χ is the linear position readout of the second crystal (with zero position being the one when this crystal lies exactly below the first one). Thus the dimensions of the monochromator become quite large due to the use of a lead screw. Further the range of the Bragg angle gets reduced due to the requirement of high accuracy lead screw throughout its length. As the motion of such a bulky monochromator is necessarily achieved using an air cushion it makes the same to be useless at low energies. The present design overcomes all these difficulties due to its special features.

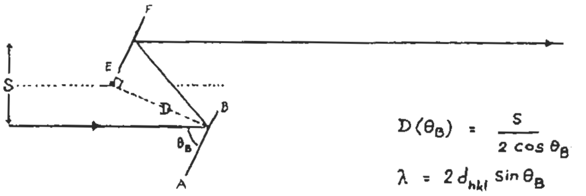

Fig. 1 Geometrical Principle

3. Design Mechanism

The plan and the side view of the construction of the spectrometer are shown in Figures 2 and 3 respectively. The spectrometer is designed to fit on a 20mm thick stainless steel (304 D) flange (12-inch diameter) ground to achieve lapping of surface parallelity of 5 micron and is thus suitable for UHV. As shown in Fig2. the first crystal is mounted on the base plate (which can be rotated on the flange) using a crystal holder which for fine adjustments can be translated back and forth as well as rotated about its axes with the help of micrometer screws. At the end of this crystal there is a high accuracy linear shaft (accuracy 5 micron) which can slide using a pair of linear bearings with their bases fixed to the base plate. The second crystal is mounted on the on the top of this linear bearing slide. Since the sliding direction of the latter shaft is normal to the surfaces of both the crystals, these crystals will always be arranged in a non-dispersive mode if the base plate is rotated, giving the outgoing beam direction parallel to the incoming one. The offset distance S between the two crystals is kept constant with the help of a linkage which adjusts the distance D between the two crystals automatically (eq (1)) when the base plate and hence the crystals are rotated. This is achieved by another linear bearing fixed to the flange with its sliding directions parallel to the incoming as well as outgoing beams. The linkage bar of which one end is connected to the top of the second crystal while the second one slides along this linear bearing enables the distance adjusted according to eq (1) giving the offset distance constant.

Thus as the base plate is rotated both the crystals as well as the linear bearing on it would rotate and simultaneously the second crystal translates along the bar. The spectrometer is

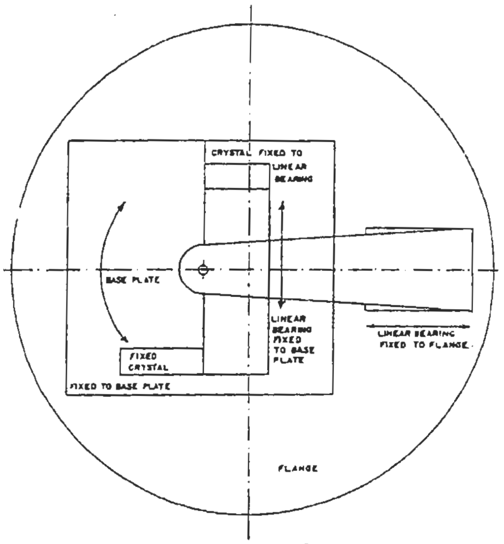

Fig. 2 Top View of the monochromator

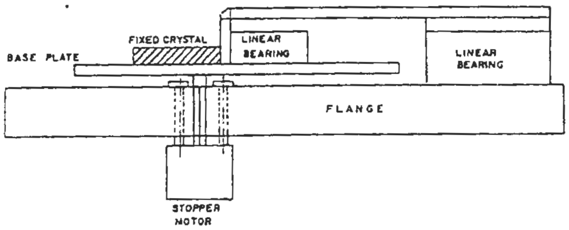

Fig. 3 Side View of the monochromator

driven by a stepper motor from the bottom side of the flange. The entire system is computer controlled. The details of the control electronics as well as the computer software are given elsewhere[5]

4. Achievement of High Resolution and Focusing

A spectral line is broadened due to many reasons such as the width of , slit assembly, choice of the crystal etc. However, keeping the first two parameters fixed the ultimate contribution to the broadening will be given by the choice of the crystal which is given below.

It is well known that in a 2-crystal spectrometer operated in a non-dispersive mode the angular spread of an x-ray beam reflected by a symmetric crystal cannot be minimized beyond a limit known as the Darwin width. Furthermore if the crystals are symmetric there also occurs the divergence of the beam resulting in the loss of intensity at the output stage. To achieve the gain in the intensity there should be a focusing of the beam after reflection (i.e. the crystal should act as both - a monochromator and a lens). While the focusing achieved normal to the diffracting planes is quite common by bending a crystal cylindrically with the position of the focus being a function of radius of the cylinder[6], the focusing in the diffracting planes is very different and is obtained by cutting the surface of a crystal asymmetrically. We discuss below the physics of such asymmetrically cut crystals (ACC) on the lines given by Kohra[7].

Referring to Fig 4 if the incoming ($\mathbf{k}_{in}$) and the outgoing ($\mathbf{k}_{out}$) wavevectors make angles θ_{in} and θ_{out} with the crystal surfaces then using the continuity condition for tangential components of these vectors and then differentiation yields

$$\Delta\theta_{out} = (m^{-1})\,\Delta\theta_{in} \qquad (2)$$

where

$$m = [\sin(\theta_{out})]/[\sin(\theta_{in})] \qquad (3)$$

is the asymmetry index or magnification factor. From the dynamical theory of diffraction if ω_s is the width of the rocking curve of the reflection for a symmetric crystal then for the ACC it is given by

$$\Delta\theta_{out} = [\omega_s/\sqrt{m}] \qquad (4)$$

The divergence of the incoming beam normally guarantees that the full range of the rocking curve width is utilized to produce the diffracted beam. Hence from the dynamical theory, $\Delta\theta_{in}$ is given by

$$\Delta\theta_{in} = \omega_s\sqrt{m} \qquad (5)$$

Thus if $m > 1$ the exit divergence will get reduced by a factor $\sqrt{m}$ while the acceptance is enlarged by the same factor and these will occur simultaneously (Figs. 5 and 6) . In our spectrometer we will be employing an ACC

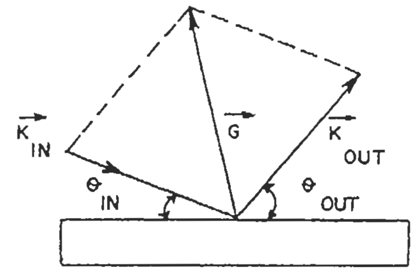

Fig. 4 Asymmetric Bragg Diffraction

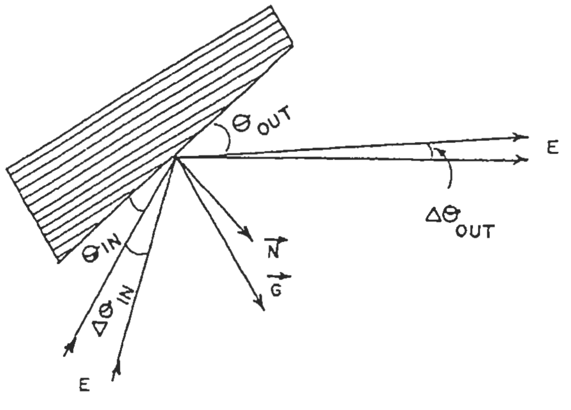

Fig. 5 The Effect of an Asymmetric Diffraction

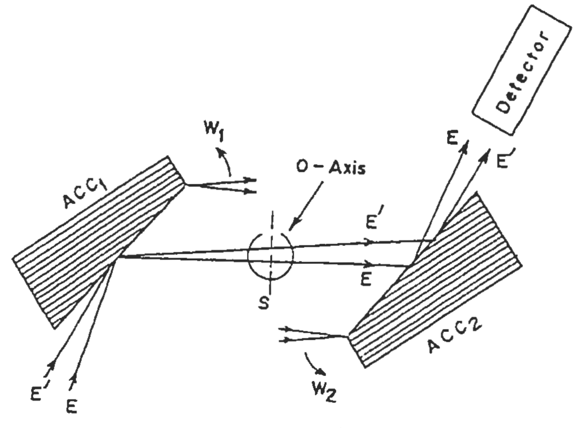

Fig. 6 Use of Two Asymmetrically Cut Crystals

Si(111) with m = 38 for which $\Delta\theta_{out}$ comes out to be 1.14 arc seconds giving $\Delta E = 0.3$eV at 9000eV (Cu K absorption edge (approx)).

5. Summary

We have presented a scheme for a novel 2-crystal Lab-XANES spectrometer to obtain a resolution of 0.3eV at Cu K absorption edge ($\equiv$ 9000eV (approx)) as well as focusing of x-rays resulting in the gain in intensity.

Acknowledgments

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