A Linear Spectrometer Designed for EXAFS Spectroscopy

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A linear spectrometer, especially designed for EXAFS applications, has been built to maintain the Rowland geometry with the anode focal spot as fixed point on the Rowland circle. The performance of the mechanical construction of this spectrometer allows one to take incremental steps of 5 μm with a resetability of ± 5 μm.

INTRODUCTION

Several EXAFS spectrometers have been described in literature (1-5), which are based upon the Rowland circle configuration with the anode as fixed source point. One design uses 4 A.C. stepping motors to drive the Rowland circle mechanism, which seems to be more complicated than necessary for routine measurements. Other designs involve a much simpler drive-mechanism but lack adequate read-out or positioning control for sufficient reproducibility of the energy scale. Here we will describe a linear spectrometer suitable for EXAFS spectroscopy which has been especially designed to fulfil the following demands:

- Minimum incremental steps of 0.2 eV at 9000 eV with a resetability of ± 0.1 eV.
- Accurate and reproducible position read-out.
- Rapid and easy change of the radius of the Rowland circle, which makes the system adaptable to different, commercially available, monochromator crystals.
- Easy alignment procedure.
- Exchange of monochromator crystals without tedious alignment procedures.
- A possible load on the sample stage of at least 25 kg without any relevant influence on the positioning accuracy. To handle a wide variety of different samples and measuring conditions it must be possible to fit liquid- and in-situ cells and cryostats on the sample stage.

THE EXAFS LINEAR SPECTROMETER

The Rowland circle configuration as shown on the photograph (Fig.1) is based upon a mechanism consisting of the following elements:

1) The main slide which moves along the main guide in order to change the distance between the X-ray focal spot and the monochromator crystal.
2) The monochromator support which is attached to the main slide by means of a rotating axis.
3) The sample/detector slide which moves along a guide which is connected to the main slide by means of the same rotating axis as used for the

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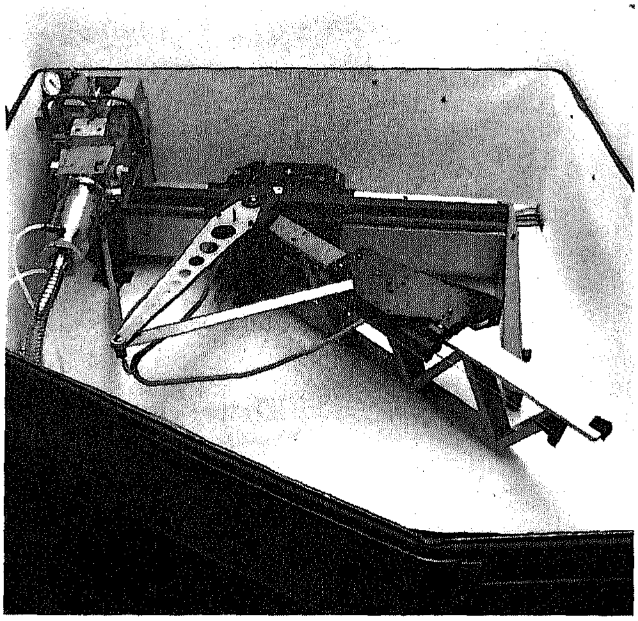

Figure 1 The linear spectrometer with Elliot type GX 21 rotating anode X-ray generator

monochromator support.

4) Three arms of equal length with one common central axis (center of the Rowland circle).
   - One arm from the anode fixed source point.
   - The monochromator arm rigidly attached to the monochromator support.
   - The sample/detector arm from the sample focal spot.

The elements described above maintain the Rowland circle configuration as long as the distances from the X-ray source point to the monochromator axis and from the monochromator axis to the sample focal spot are kept equal within the design specifications.

The angle between the monochromator crystal and the X-ray beam changes during the displacement of the main slide along the main guide. The wavelength (λ) of the photons reflected by the monochromator crystal is linearly dependent on the distance (x) between the crystal and the anode source point:

$$\lambda = \frac{d}{R} x$$

where R is the radius of the Rowland circle and d is the lattice spacing of the diffraction planes used for monochromatization.

The high positioning accuracy of the total mechanism has been achieved by paying full attention to the kinematical and statical design specifications of the total lay-out and its components. In addition all bearings and bearing points are preloaded in order to eliminate all virtual and actual clearance. Each slide is driven by statically and kinematically balanced friction wheels which are coupled to a D.C. motor. Using D.C. motors and ruler systems of high mechanical resolution, the positioning accuracy is realized by a computer electronic feedback system. With a radius R=500 mm of the Rowland circle and Si(400) crystal (d=1.3576 Å) "mechanical" incremental steps of 0.2 eV can be obtained with a resetability of ± 0.1 eV at the Cu-edge (9000 eV). The sample/detector slide can easily accomodate a weight of 25 kg without a change in relevant positioning accuracy.

PRELIMINARY RESULTS

A simple Si(400) Johann crystal as monochromator gives for a 3 μm Pt foil the results as presented in Fig.2.

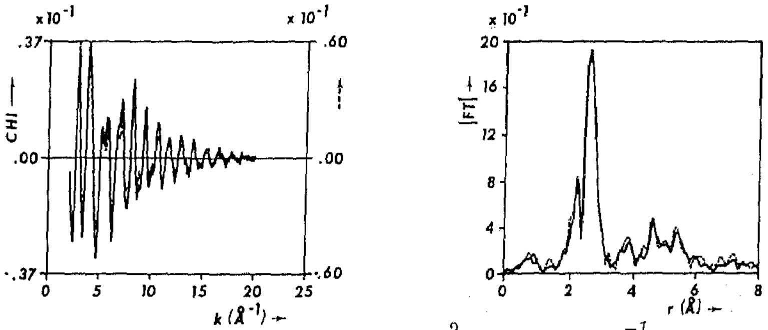

Fig.2 EXAFS and Fourier transform (k^2, Δk=2.6-19 Å^{-1}) of Pt-foil (RT). Solid line: Eindhoven EXAFS facility, with ΔE ~14 eV, 20 mm horizontal crystal spot: Mo anode: 22 kV, 200 mA, $3x10^6$ photons/sec. Dotted line: SSRL, 3 GeV, 40-80 mA.

With a Johansson Si(111) a resolution of 11 eV is obtained at the Cu-edge with a photonflux of $3x10^7$/sec.. Conditions: 70 mm horizontal crystal spot, Mo-anode 26 kV, 280 mA,. Test experiments with Johannson Si(311) and Ge (311) crystals will be carried out in due course.

LITERATURE

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