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DIFFRACTOMETER FOR SYNCHROTRON RADIATION EXPERIMENTS**

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**A NEW BEAUTY FOR "ADONE": A HIGH RESOLUTION POWDER
DIFFRACTOMETER FOR SYNCHROTRON RADIATION EXPERIMENTS**

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ABSTRACT

A high resolution powder diffractometer, connected to the wiggler magnet line BX1, is now operative at the Adone storage ring in Frascati. A Si channel-cut monochromator on the line allows operation in the range 1- 3 Å. To achieve the desired high resolution in the diffraction spectra, a "triple-axis configuration" has been chosen: a vertical standing $\theta/2\theta$ goniometer supports a flat Ge(111) crystal analyzer on the 2θ arm. With this configuration, a value of less than 0.02° for the FWHM of the diffraction peaks has been reached. The special design solutions adopted for a Seifert MZ VI goniometer and the microstep technology used in the stepper motor actuation assure a mechanical resolution better than 0.001° . A special supporting table, with six degrees of freedom, has been made for the diffractometer orientation in front of the X-ray beam. An IBM-PC is dedicated to the diffractometer positioning control and preliminary data collection. As a Macintosh IICX provides for the data processing, a special software package, named "Mac Dust", has been developed and is continuously updated. The first experimental results collected on-line during the instrument check-up are presented.

INTRODUCTION

At the end of 1988, a scientific cooperation project between various Italian, French, and Spanish scientific institutions became operative in the field of X-ray diffraction. The research project, financially supported by the European Economic Community is titled: "Realization and Setting up of Instrumentation in the X-ray Diffraction Field (Powders and Microcrystals) Near the Synchrotron Radiation Center of Frascati and Orsay".

For the purposes of the project, a high resolution powder diffraction station has been set up by the P.W.A. Group in Frascati, and a single crystal diffractometer at LURE by J.P. Lauriat and co-workers.

The diffractometer was connected to the Adone-Wiggler beam line BX1 at the end of February 1991, and a first test experiment was successfully carried out at the beginning of March 1991.

THE HIGH RESOLUTION POWDER DIFFRACTOMETER

Usually, a high resolution powder diffraction spectrum by means of synchrotron radiation gives a value in the full width half maximum of the diffraction peaks one order of magnitude less than that obtained with X-ray tubes. To achieve these values in powder spectra, the so-called "triple axis configuration" should be adopted for the diffractometer [1]. In this arrangement, the input monochromator (1st axis) and the sample (2nd axis) are followed by a crystal analyzer (3rd axis) which acts as an extremely narrow receiving slit. The use of synchrotron radiation instead of common X-ray sources enhances resolution due to the natural high collimation and intensity of the former. Thus, it is possible to reach FWHM values in the range of 0.02° . Our station, which must be characterized by high resolution, adopts the above-mentioned configuration, which is similar to those developed at CHESS [2] or at the SSRL [3].

In collaboration with the firm of Seifert [4], a special version of their MZ VI goniometer has been constructed according to our specifications. The diffractometer consists of three parts: a) the primary slit system; b) the $\theta/2\theta$ goniometer; c) the secondary slit system (see Figure 1).

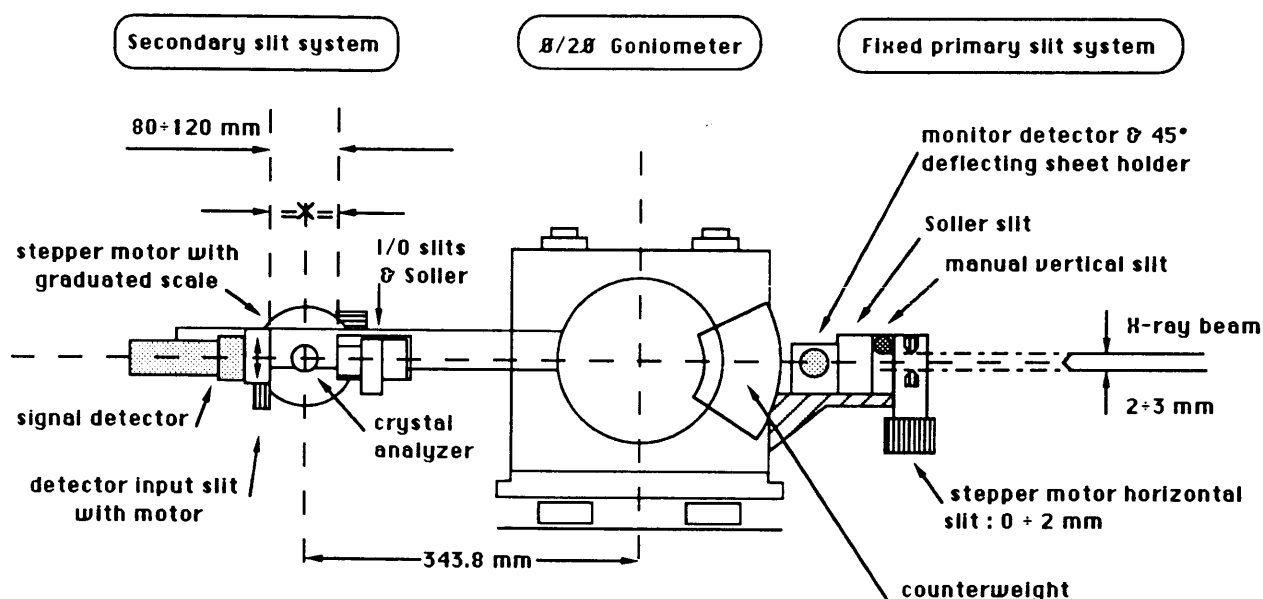


Figure 1. High resolution powder diffractometer - Schematic drawing

The primary slit system consists of a horizontal slit driven by a stepper motor, a manually controlled vertical slit, and a removable Soller slit. Behind the slits, a thin capton sheet at 45° and a scintillation detector allow the incoming X-ray radiation to be monitored.

The $\theta/2\theta$ goniometer, vertically placed, supports the secondary slit system on its 2θ arm. The arm is 343.8-mm long, thus permitting a recording scale of $1^\circ = 6$ mm. A set of input/output slits plus a second Soller are also located along the arm. All the parts are easily removable.

The 2θ arm also supports a small one-axis goniometer that consists of a stepper motor plus $1/20$ gear reduction system. This configuration allows an angular resolution of 0.0014° in the crystal positioning. A goniometric head holds a flat crystal analyzer in the (n,-n) setting mode with respect to the sample.

We have chosen a flat Ge [111] crystal analyzer. The choice is a compromise between the higher resolution achievable using a Si [111] crystal and the higher integrated intensity using a LiF [220] crystal.[2] The rocking curve of the crystal has a FWHM = 0.006° , according to our measurements with synchrotron radiation, so it acts as an angular slit of about 21 arc sec at 1.54 \AA .

A scintillation signal detector with its input slit, supported by a small graduated arm, completes the apparatus. A special counterweight, placed symmetrically with respect to the 2θ arm, is also present to assure good mechanical balance and to increase the mechanical reliability of the system.

All the stepper motors are driven using "microstep technology": a special electronic unit under a PC control allows one to easily reach a resolution better than 0.001° in mechanical positioning (see Figure 2).

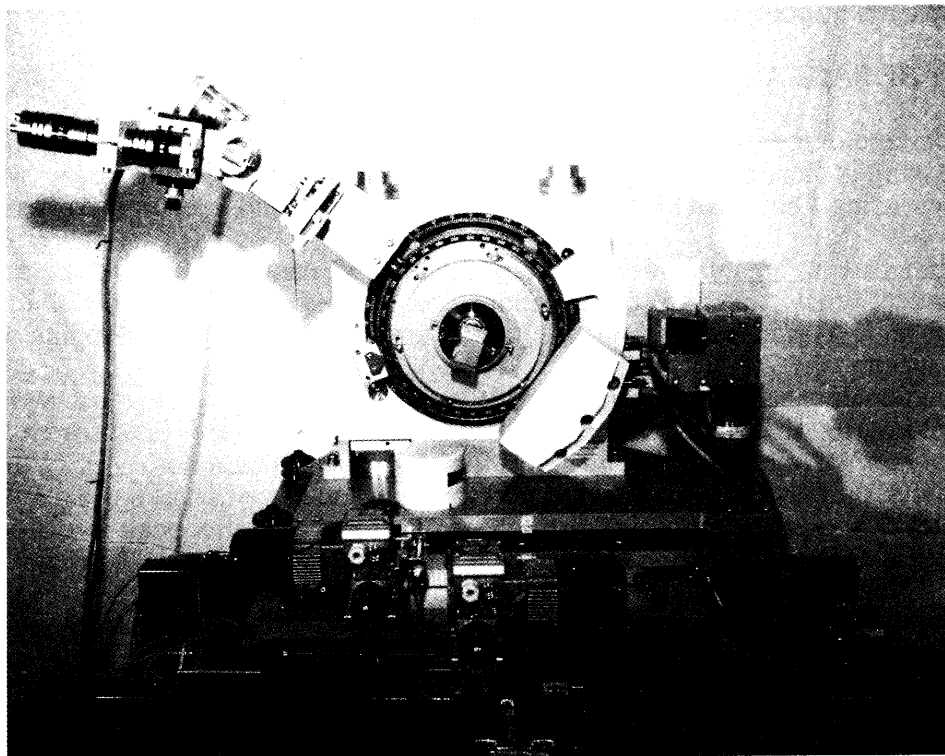


Figure 2. High resolution powder diffraction station on the supporting table

The following table reports the main parameters of the station.

Tab.1 - High Resolution Powder Diffraction Station - Main Parameters.

Goniometer Seifert MZ VI type	$\theta/2\theta$
Standing position	vertical
Axial deflection	< 0.001 mm
Axial speed	$0.01 - 20$ $^\circ/s$
Repeatability	~ 2 arc sec
Precision	~ 8 arc sec
Length of 2θ arm	343.8 mm
Diffractometer recording scale	$1^\circ = 6$ mm
Angular range	$-2^\circ \div 160^\circ$
Crystal analyzer	Ge(111)
FWHM of the Ge[111] rocking curve	0.006°
Crystal analyzer positioning resolution	0.0014°
Detector and Monitor, scintillation counter	NaI(Tl)
FWHM of the Si[111] peak, NBS sample	0.014°
Goniometer weight	~ 120 Kg

To increase the flexibility of the diffraction station, it is easy to place a X-ray tube vertically on the left of the goniometer using a special stand. By using a curved crystal analyzer instead of a flat one, and by moving the 2θ arm to the right it is also possible to use the diffractometer when synchrotron radiation is not available for both preliminary alignment and data collection.

A special supporting table has also been built that guarantees six degrees of freedom in positioning the diffractometer with respect to the X-ray incident beam [5]. The table platforms are moved by AC motors with encoders which are controlled by a Programmable Logic Control unit that is separate from both the goniometer control and the data acquisition electronics [6]. The encoder position is displayed by counters with nonvolatile memory [7].

THE SOFTWARE PACKAGE

All the goniometer movements and the data collection are under the control of an IBM-PC, while the data processing is managed by a Macintosh IICX. A special software package, "Mac Dust", has been developed using Quick BASIC compiler, for Apple Macintosh computers, as programming language [8].

The programs are all organized as selectable options from menus and submenus, according to the Macintosh standard graphic interface specifications. The graphic commands allow all possible graphic operation, such as X-Y mouse dragging and manual zooming facilities, overlapping and comparing of up to eight spectra.

Diffraction powder analysis programs provide instantaneous least square third degree polynomial smoothing; fast Fourier smoothing with Hanning filtering; first derivative peak search algorithm; and least square polynomial or cubic spline functions for background subtraction.

Profile analysis programs allow centroid and variance determination of a peak; FWHM and area calculation; least square polynomial fitting, useful for parabolic peak position and height determination; Gaussian, Lorentzian and Pearson VII nonlinear least square fitting; Pseudo-Voigt simplex fit; and Kalfa2 component subtraction for conventional X-ray sources.

The package also includes complete phase identification facilities: card managers, phase card to spectra graphic overlapping routines, and a Hanawalt algorithm based search-match program. It will also provide structure refinement programs, such as DBWS 9006 [9] (a new Macintosh version) and other analysis programs, e.g., the Warren-Averbach [10,11] method for microstrain and crystallite size determination.

FIRST EXPERIMENTAL TEST

At the end of February 1991, the high resolution powder diffraction station was connected to the wiggler magnet beam line BX1, where a channel-cut monochromator with Si [111] or Si [220]

crystals allows one to work in the range from 0.5 to 3 Å, with a resolution $\Delta E/E$ of $1.4 \cdot 10^{-4}$ and $6 \cdot 10^{-4}$, respectively.

As the instrumental resolution depends on the quality of the crystal analyzer, during the first instrumental test made with synchrotron radiation at the beginning of March 1991, we evaluated the FWHM value of the rocking curve of the Ge [111] crystal analyzer. The measurement was performed with the 2θ arm located at 0° position and with the detector placed at the correct angle for the Cu radiation (8.05 keV) that is selected by the channel-cut monochromator. An omega scan then gave a FWHM value of 0.006° for the rocking curve, as shown in Figure 3.

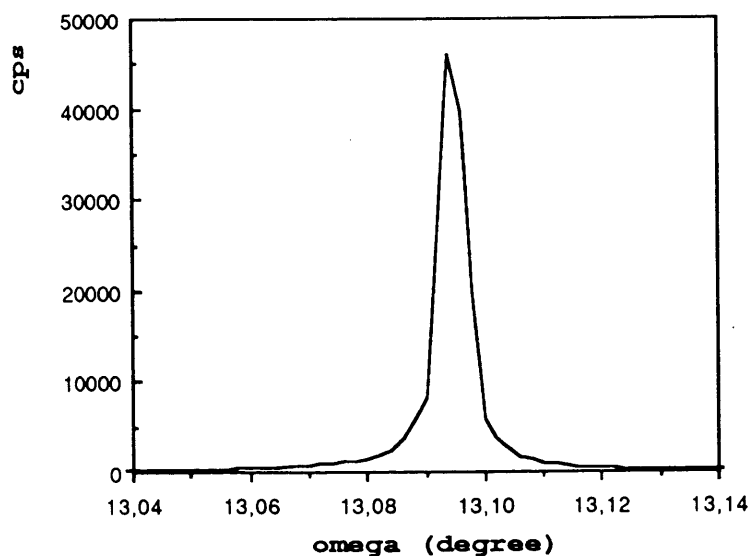


Figure 3. Rocking curve of the Ge[111] crystal, Synchrotron radiation 8.05 keV, FWHM = 0.006° , $\omega = 13.095^\circ$

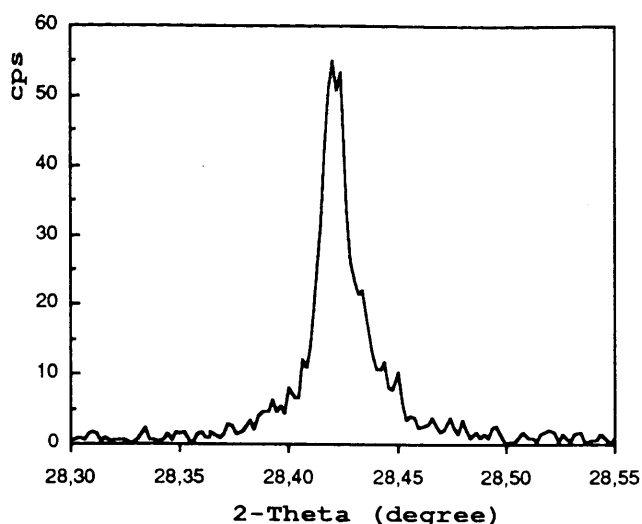


Figure 4. Si powder sample measured with synchrotron radiation 8.05 keV, peak [111], $2\theta = 28.420^\circ$, FWHM = 0.014°

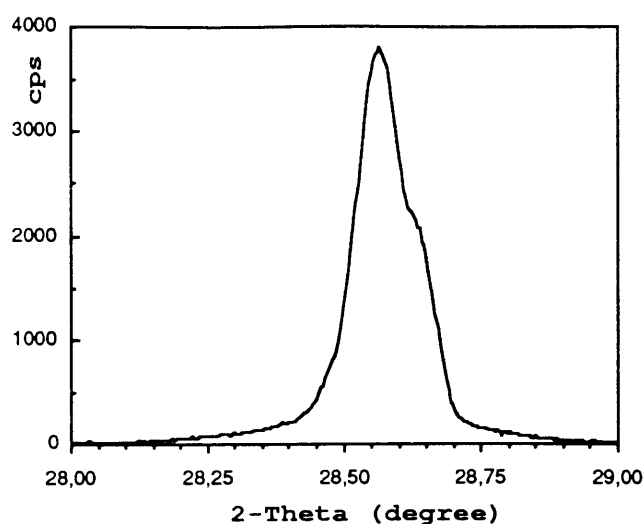


Figure 5. Si powder measured with Cu tube, peak [111], FWHM = 0.127°

Afterwards, a set of measurements were made on a Si powder sample (NBS standard 640a) by collecting the [111], [220], [311] and [331] peaks. As an example, Figure 4 shows the measured [111] diffraction peak without any smoothing and a FWHM of 0.014° . The same peak, collected with a standard Seifert two-circle powder diffractometer, is given for comparison in Figure 5. Here the FWHM is about 0.127° , just one order of magnitude larger than the previous value.

The next runs with synchrotron radiation will be dedicated to collecting first diffraction data on different powder samples at room temperature.

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