Versatile Powder X-Ray Diffractometer
( Ultima IV Type II )

Document No: AGB-S006-1

Rigaku Corporation
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The Ultima IV multipurpose diffraction system is a flexible and easy to use x-ray diffractometer. This versatile theta/theta design can incorporate both Cross Beam Optical technology (CBO) and an independent in plane scattering axis to provide the widest possible range of measurement geometries without the need for system re-configuration.

**Ultima IV optics**

[STANDARD]

BB\(^1\) (high-resolution \(\theta/2\theta\) scan): -

Thin film geometry: -

[OPTIONS]

BB (high-resolution \(\theta/2\theta\) scan, decreased UE\(^2\)): Soller Slit 2.5°

BB (high-resolution \(\theta/2\theta\) scan, decreased BG\(^3\)): PB/BB Diffracted Beam Monochromator

Thin film geometry (decreased BG): PB/BB Diffracted Beam Monochromator

Residual stress geometry: (Multipurpose Attachment)

Pole figure geometry: Slits for Pole Figure, Multipurpose Attachment

High intensity PB\(^4\) geometry (7 mm): CBO, (PB/BB Diffracted Beam Monochromator)

High resolution PB geometry (7 mm): CBO, PSA\(^6\) 0.114°

Small angle x-ray scattering geometry: CBO, Small Angle X-Ray Scattering Unit

Small area x-ray diffraction geometry: CBO, Small Area X-Ray Diffraction Unit

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\(^1\) BB: Bragg-Brentano focusing  
\(^2\) UE: Umbrella Effect  
\(^3\) BG: Background  
\(^4\) PB: Parallel Beam focusing  
\(^5\) CBO: Cross Beam Optics  
\(^6\) PSA: Parallel Slit Analyzer
1. Configurations

(1) X-Ray Generator
(2) X-Ray Radiation Enclosure
(3) Goniometer
(4) X-Ray Detector and Controller
(5) Standard Software
(6) Consumables
(7) Application Software
(8) Attachments

2. Specifications

2.1 X-ray generator system

2.1.1 X-ray generator

(1) Maximum power: 3 kW continuous rated maximum output power
(2) Voltage: 20 – 50 kV, 1 kV steps
(3) Current: 2 – 60 mA, 1 mA steps
(4) Stability: ± 0.01 % against ± 10 % mains fluctuation, voltage and current

2.1.2 X-ray tube

(1) X-ray tube: Cu target tube (normal, fine, or long fine focus)
(2) Focus size*: Normal focus 1.0 × 10 mm², from a target surface view [SELECT]
     Fine focus 0.4 × 8 mm², from a target surface view [SELECT]
     Long fine focus 0.4 × 12 mm², from a target surface view [SELECT]

*Fine or long fine focus is recommended for SAXS\(^7\) and/or thin film measurements.

2.1.3 X-ray tube shield

(1) Shutter: Electromagnetic shutter interlocked with radiation enclosure
(2) Focus: For line focus exclusive use

2.1.4 Safety features

Abnormal cooling water flow, pressure, and temperature detection
Abnormal generator overload detection
Leak current breaker
Shutter malfunction detection

\(^7\) SAXS: Small Angle X-ray Scattering
2.2 X-ray radiation enclosure

(1) Type: Two sliding doors with fail-safe control
(2) Window: 0.5 mm Pb equivalent acrylic panel
(3) Warning light: Located on the top of the radiation enclosure

2.3 Goniometer

2.3.1 Goniometer

(1) Geometry: Vertical $\theta/\theta$ (keeps sample horizontal and stationary)
(2) Scanning axis: $\theta_D$, $\theta_S$, $T_s$ (tube height), $Z_s$ (slit height)
(3) Scanning method: $\theta_D/\theta_S$ independent or coupled
(4) Range:
   - $\theta_D/\theta_S$ coupled  $-3 - +162^\circ$ ($2\theta$)
     * with the optional 20 mm receiving slit box
     - $\theta_S$ independent  $-1.5 - +77^\circ$
     - $\theta_D$ independent  $-5 - +154^\circ$
     - $T_s$  $-7 - +2.5$ mm
     - $Z_s$  $-3 - +1.5$ mm
(5) Min. step:
   - $\theta_D/\theta_S$ coupled  0.0002 $^\circ$ ($2\theta$)
   - $\theta_D/\theta_S$ independent 0.0001 $^\circ$
   - $T_s$  0.001 mm
   - $Z_s$  0.001 mm
(6) Speed:
   - $\theta_D/\theta_S$ coupled  0.02 – 100 $^\circ$/min. ($2\theta$)
   - $\theta_D/\theta_S$ independent 0.01 – 50 $^\circ$/min.
   *These values may differ depending on configuration.
(7) Radius: 285 mm

2.3.2 Slits [for powder sample analysis]

(1) Type: Computer-controlled, programmable automatic variable slit
(2) Slit width:
   - Divergence slit (DS)  0.05 – 7.00 mm, 0.01 mm step
   - Scattering slit (SS)  0.05 – 7.00 mm, 0.01 mm step
   - Receiving slit (RS)  0.05 – 7.00 mm, 0.01 mm step
   *The DS and SS can be controlled based on divergence angle when BB is selected.
(3) Height slits: 2.0 mm, 5.0 mm, Open
(4) K$\beta$ filter: Ni-filter for Cu radiation
2.3.2 Slits [OPTION]

(1) Type: Computer-controlled, programmable automatic variable slit

(2) Slit width:
- Divergence slit (DS) 0.05 – 7.00 mm, 0.01 mm step
- Scattering slit (SS) 0.05 – 20.00 mm, 0.01 mm step
- Receiving slit (RS) 0.05 – 20.00 mm, 0.01 mm step

*The DS and SS can be controlled based on divergence angle when BB is selected.

(3) Height slits: 2.0 mm, 5.0 mm, Open

(4) Kβ filter: Ni-filter for Cu radiation

2.3.3 CBO unit [OPTION]

(1) Optical device: Parabolic multilayer mirror

(2) X-ray wavelength: Cu Kα

(3) Divergence angle: < 0.06 °

(4) Selection slits: BB, PB, SAXS [OPTION], Small area x-ray diffraction [OPTION]

2.3.4 CBO-f unit [OPTION]

(1) Optical device: Polycapillary

(2) Slit width:
- Divergence slit (DS) 0.01 – 7.00 mm

(3) Slit height:
- DS height (Zs) -3.0 – +1.5 mm

(4) X-ray beam footprint on sample: ~0.4 mm × ~0.4 mm (at 90° incident angle)

(5) Receiving optics: Angular acceptance angle 0.5°

2.3.5 Flexible optical system (for maximum SS window size: 7 mm)

(1) Type: Incident and receiving Soller slits

(2) Incident Soller slit: Axial divergence angle 5.0° [OPTION]

(3) Receiving Soller slit:
- Axial acceptance angle 5.0° [OPTION]
- Axial acceptance angle 2.5° [OPTION]

(4) PSA (for PB):
- Angular acceptance angle 0.5° [OPTION]
- Angular acceptance angle 0.114° [OPTION]
2.3.5 Flexible optical system (for maximum SS window size: 20 mm) [OPTIONS]

(1) Type: Incident and receiving Soller slits
(2) Incident Soller slit: Axial divergence angle 5.0°
   Axial divergence angle 2.5° [OPTION]
(3) Receiving Soller slit: Axial acceptance angle 5.0°
   Axial acceptance angle 2.5° [OPTION]
(4) PSA (for PB): Angular acceptance angle 0.5°
   Angular acceptance angle 0.114° [OPTION]
* These slits are for the variable receiving slit box with 20 mm windows.

2.3.6 Fixed diffracted beam monochromator for Cu [OPTION]

(1) Type: Diffracted beam monochromator
(2) Crystal*: Curved graphite (0002)
   *Can be used as a flat crystal for parallel beam geometry by rotating the crystal 90°.
(3) Monochromator slit: 0.80 mm

2.4 X-ray detector and controller

(1) Type: Scintillation counter (NaI)
(2) Detector high voltage (HV): 0 – 1500 V, computer-controlled
(3) Pulse height analyzer (PHA): Base line and window setting, computer-controlled
(4) Linearity: 700,000 cps or better with dead time correction
(5) Dead time correction: Computer-controlled and automated

2.5 Standard software

System Condition Setting: Changing configurations
XG Operation: Controlling shutter, kV-mA, aging
Manual Measurement: Manual operation of all axes and a detector
Automatic Alignment: Automated alignment of each of the goniometer and attachment axes as well as setting of the detector
PHA, HV, and dead time correction
Standard Measurement: Standard θ/2θ, θ, and 2θ measurements
Peak Search: Basic data reduction and peak search
Multiple-Recording: Multiple plot of raw data files Integrated Intensity
Calculation: Integrated intensity calculation
File Manager: Measurement condition display and editing
Data Converter: Data conversion of RINT binary format to RINT ASCII and general TEXT formats
2.6 Consumables

<table>
<thead>
<tr>
<th>Item</th>
<th>Quantity</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>Glass sample holder with 0.2 mm depression</td>
<td>2</td>
<td>Cat. No. 9200/2G</td>
</tr>
<tr>
<td>Glass sample holder with 0.5 mm depression</td>
<td>5</td>
<td>Cat. No. 9200/5G</td>
</tr>
<tr>
<td>Aluminum sample holder</td>
<td>2</td>
<td>Cat. No. 2100/AL</td>
</tr>
</tbody>
</table>
2.7 Application software [OPTIONS]

PDXL Basic Package
Basic data processing (Smoothing, background subtraction, Kα2 elimination, peak search, multi peak separation, crystallite size (Scherrer method), multiple plotting, task macros, ICDD access, file history and thumbnails, creation of various types of reports 2θ correction, pattern simulation from d-I lists, 3D multiple pattern display, ICSD access, crystal structure data (CIF) input and output, 3D crystal structure display, RIR quantitative analysis)

PDXL Options
PDXL Qualitative Analysis Hybrid search/match

PDXL Comprehensive Analysis
Crystallite size and Lattice strain (Williamson-Hall method), lattice constant refinement, % crystallinity, residual stress analysis, crystal system and lattice constant determination

PDXL Rietveld analysis Lattice constant, quantitative analysis, crystallite size, and lattice strain calculation using the Rietveld method

PDF-2 ICDD-PDF2 database for phase identification (CD-ROM)

CRYSTMET-X: Crystallographic data for metals, including alloys, intermetallics and minerals

Positive Pole Figure: Pole figure measurement, display, and analysis (for Multipurpose Attachment)

Inverse Pole Figure: Inverse pole figure display and analysis

Residual Stress: Residual stress measurement and analysis of iso- and side-inclination (for Standard or Multipurpose Attachment.)

Long Periodicity: Long-periodicity analysis

NANO-Solver: Particle/pore size distribution and correlation length analysis using curve fitting for both reflection and transmission SAXS data

Pore-Size Analysis: Particle/pore size distribution and diffuse scattering analysis using curve fitting of rocking curves and reflection SAXS data

GXRR: Multilayer modeling and layer structure analysis package for x-ray reflectivity data

3D Explore PF: 3D display and data process of pole figure data
2.8 Attachments [OPTIONS]

2.8.1 Sample spinner attachment

The sample rotation attachment allows continuous rotation at variable speed of the sample holder to improve particle statistics during powder diffraction measurements.

![Sample Spinner Attachment](image)

**Specifications**

**Rotating shaft**

- **Rotation speed:** Max. 120 rpm
- **Step:** Min. 0.72 °
- **Range:** -360 ° – +360 °

**Available sample holder:** Glass sample holder, Al sample holder
2.8.2 Sample spinner attachment with Z-axis

The sample rotation stage allows continuous rotation at variable speed of the sample holder to improve particle statistics during powder diffraction measurements. Fully automated sample height (Z-axis) alignment provides extreme ease in the positioning of samples for thin film measurement.

![Sample Spinner Attachment with Z-axis](image)

**Specifications**

**Rotating shaft (φ-axis)**

- Rotation speed: Max. 120 rpm
- Step: Min. 0.10°
- Range of movement: -360° – +360°

**Sample height (Z-axis)**

- Range of movement: -5.0mm – +2.0 mm
- Step: 0.005mm, Computer-Controlled
2.8.3 Capillary attachment

Automated sample stage for use with glass capillary tubes. Includes sample-spinning capability. Unique design simplifies capillary alignment.

Capillary Attachment

Specifications

Rotating shaft

<table>
<thead>
<tr>
<th>Specification</th>
<th>Details</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rotation speed</td>
<td>Max. 120 rpm</td>
</tr>
<tr>
<td>Step</td>
<td>Min. 0.72 °</td>
</tr>
<tr>
<td>Range of movement</td>
<td>-360 ° – +360 °</td>
</tr>
<tr>
<td>Capillary size</td>
<td>0.8 mm φ – 2.0 mm φ</td>
</tr>
</tbody>
</table>
2.8.4 Air tight attachment

Air tight attachment allows measurements to be performed in vacuum, or various gasses.

Air Tight Attachment

Specifications

- Measuring angle range: \(2\theta : 0^\circ - 158^\circ\) (\(2\theta/\theta\) coupled)
- Window size: Be (thickness = 0.25 mm)
- Tolerant gas pressure range: \(1 \times 10^{-3} - 2\) atm

Available sample holder: Glass sample holder, Al sample holder
2.8.5 Multipurpose attachment

Multipurpose attachment has four axes to enable thin film (grazing incidence diffraction, x-ray reflectivity, and rocking curve), pole figure, and residual stress measurements with a single attachment.

Multipurpose Attachment

Specifications

Rotating angle ($\phi$-axis)
- Range: $-360^\circ$ – $+360^\circ$
- Step: Min. 0.005 $^\circ$

Tilt angle ($\chi$ ($\psi$)-axis)
- Range: $-5^\circ$ – $+92^\circ$
- Step: Min. 0.002 $^\circ$

Sample height (Z-axis)
- Range: -6 mm – +1.5 mm
- Step: Min. 0.00025 mm

Sample Max. 100 mm $\phi$ x 10 mm thick
3. Acceptance test

Refer to the “customer acceptance test report” document.

4. Warranty

Warranty does not cover damages or defects caused by:

(1) Acts of God
(2) Improper application
(3) Tampering, alterations
(4) Installation contrary to this specification's recommendation, or in any event if the instrument has been altered, defaced, or removed
(5) Misuse, negligence, inappropriate on-site operating conditions
(6) Consumption wasting and reaching the end of their working life

After the warranty period expires, products and parts may be repaired and replaced for a reasonable service charge. The following secondary damage: a delay of research and development, receiving inspection, acceptance testing etc. caused by instrument failure, is not covered by warranty.