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# Synchrotron vacuum diffractometer

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**Abstract**
An international joint collaboration between several research institutions and companies working in synchrotron industry has led in to the development of a new research product. The high-vacuum diffractometer (HV-Dm) will be able to perform X-ray investigations, by applying several - standard and/or modern synchrotron techniques under vacuum conditions and serves to quench the increasing demands of studying the materials (nanostructures) at nanoscale. On the basis of first HV-Dm, a second prototype, with minor improvements was manufactured and delivered to a large-scale next generation (4th) synchrotron facility. Basically, both diffractometers have had similar structures on a classical four-circle geometry (4C) based, comprising two manipulation/positioning modules - sample (Sp) and the detector arm (Da) – performing high-precision correlated motions on diffraction physics, in an allocated confined vacuum space of a vessel (chamber). The sample manipulation (positioning) module derived from a general Euler cradle includes a customized large opened circle (1/4), small full rotation circle and vacuum-compatible hexapod device. The design features of the components, together with their integration are described. With its compact specific architecture, using mainly precision standard components (e.g. rotation stages), the diffractometer is expected to deliver the necessary motions and accuracy by doing various tasks. The most important aspects of its kinematics, design (CAD) and precision in relation with high-vacuum conditions are presented in this paper.

Synchrotron, positioning, X-ray diffraction, manipulation, diffractometer, hexapod, vacuum

## 1. Introduction

During the past few years, applicative researche using synchrotron radiation visible has increased. This cutting-edge technology served at the base to many discoveries in natural sciences (physics, chemistry and materials). Several industrialized countries have built large-scale facilities based on this technology [1].

X-ray diffraction (XRD) is a well-established technique for characterization of material structure [2]. It is commonly used in research laboratories, as well as in large facilities, as synchrotrons. While some investigations are trying to answer the fundamental questions regarding the atomic-molecular structures of solids, liquids, and biological materials under normal conditions, others are attempting to discover or examine the behaviours of new materials for specific purposes, by using suitable advanced techniques [3]. Especially, the interest to understand the corelated phenomena between physical and chemical propierties of functional oxide materials, such as those included in *semicon* nanostructures has been constantly increasing [4].

Several discoveries have examined nanomaterials (nanostructers) by using surfaces sensitivity analyses and characterizations. Many of these techniques require extreme environmental conditions, such as high-pressure, high/low temperature or high magnetic field, or a combination of them.

Vacuum technology [5] appears to support the synchrotron science in many aspects, such as being higly used along beamlines (front-end) to facilitate X-ray production and at the end (end-stations) of the beam line in the vicinity of a specimen for investigative purposes.

However, in addition to the development of new methods and techniques, the equipment has to fit, accordingly. Recent advancements in X-ray technologies have placed considerable pressure on diffraction machine (diffractometers) designers and causing the development of a new class of diffractometers. Thus, various measurements are now possible to be performed on one site with high precision, offering the users the possibility to investigate different kinds of samples (applications) in a short periode of time[6], thereby decreasing the switching time and increasing the accuracy. In turn, the configurations of the equipment are becoming increasingly complex, by using several methods and techniques (e.g. horizontal and vertical scattering) and including additional instruments (e.g., vacuum, cryogenic and/or spectrometer chambers). Most often these instruments are large with complex architecture, making their use cumbersome. Thus, the positioning devices inside must be able to carry them in different positions. By this, the final product becomes a complex heavy-load and multipurpose equipment, such as [7].

It is important to simplify the diffractometer design, to increase its versatility offering a solution to work inside of a chamber containing the extreme conditions (e.g., vacuum). However, such a specific machine must fulfill the specific design related conditions (high pressure) and (smaller) existent spaces.

To date, the whole system of a diffractometer is built on a serial (stacked) architecture, materializing the rotation axes (circles), as a succession of precision positioning units (gonio) and linear stages, linked together; e.g., products from HUBER, NEWPORT or KOHZU companies. However, this traditional simple design occupies a large space and is prone to accumulated errors.

The Parallel Kinematic Manipulators (PKM) [8], e.g., Hexapods are recently beeing used in synchrotron facilities, for rough manipulation (alignment base) and precision positioning (sample) in diffractometers, providing an effective solution to load manipulated, working space and motion accuracy.

A joint research collaboration between two well-known international research institutions (synchrotrons) [9,10] and two specialized company (diffractometers [11], hexapods [12]) has led to the development a new equipment (diffractometer) for X-ray investigations of nanostructures. Inside, a dedicated four-circle simple diffractometer device has been proposed to work. It is expected that this high-vacuum diffractometer (HV-Dm) is able to work by using several standard and modern XRD techniques. An overview of the prototypes, including similarities and differences, challenges and rules from kinematic, design and fabrication points of view are now presented.

## 2. High-Vacuum Diffractometer

The diffractometers (Dm) are measuring machines used for structural identification and characterization of materials. It can be used for various physical (solid, liquid and gas) and biological (tissues) forms of matter. From engineering view point, the Dm are precision systems posing in a relative positions both, the sample (and environmental instruments) and detector(s) on diffraction laws basis. Typically, their structures, are a combination of two manipulators one, manipulating the sample (and instruments) toward the incoming X-ray (incident beam) and another manipulating the detector to catch the bunch of scattering spots (diffracted beam). As mentioned previously, with the necessary (vacuum) instruments Dm are becoming complex machines (equipment) involving the collaboration of several companies. In addition, they must be able to stop with high-precision in the required positions.

A first dedicated high-vacuum diffractometer (HV-Dm) to work for a beamline (SIRIUS) [13] in a well-known synchrotron facility (SOLEIL) was developed (2016). SYMETRIE company was acting as integrator for the whole diffractometer equipment (DmEq). The second HV-Dm device (2017) is expected to work for another beam line (FemtoMAX) [14] and will be located at a fourth-generation (4th) radiation source facility (MAX IV).

The first equipment (FORTE) started to work recently (2018) at one of the two endstations. In the first experimental results have been investigated the DmEq capabilities and phenomena produced in compound nanostructures under conditions of about 2keV (tender X-ray) and $10^{-6}$ mbar using several surface scattering techniques (SXS) [15]. The technical specifications for the second (HV-Dm) product was strongly related with those presented in this work. The most important kinematic and design features are given below. Note: The minimum spot size of beamline is set to be about 50-100 µm (focused mode).

### *2.1. Kinematic structure*

The high-vacuum diffraction equipment (DmEq) has been built around a diffractometer (Dm) device working in a dedicated high-vacuum chamber (Vc), both supported and moved by a parallel manipulator alignment base (Ab), Fig. 1.

The vacuum chamber (Vc) [16] is where the Dm has to work comprises from a rectangular base ($A_{Vc}$x$B_{Vc}$=1500x1300mm) and a sophisticated half-cylinder (R=$C_{Vc}$=700mm) bell vessel manufactured from stainless steel, with an appreciable number of flanges for various connections, including viewports for fast opening, transfer, detectors, exit or visualization and others in relation with electrical/electronic connections (feedthroughs) for turbo pump, gages and safety valves. Some of these ports are interchangeable and most of them point toward the sample (C-center of rotation point). It has bar vacuum capability$10^{-6}$-$10^{-8}$. The diffractometer (Dm) itself is based on a classical four-circles - 4C(1+3), 1–detector and 3–sample geometry [17], where $C_i$ 1,…,4 are full or partial circular motions / circles.

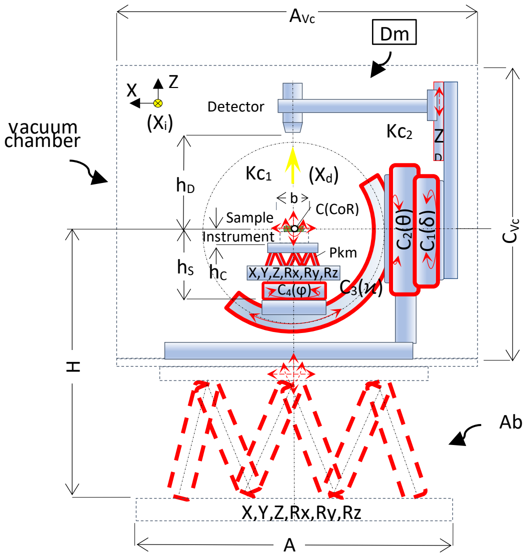

**Figure 1** HV-Dm Kinematics

The fundamental motion equations of this geometry can be found in [18] and the operational setting angles in [19].

From kinematic point of view, the structure consists of two (2) interconnected chains: 1) $Kc_1$ - sample positioning (Sp) and 2) $Kc_2$ - detector arm (Da), having in common a horizontal axis of rotation, coinciding with X-ray beam (H). The $Kc_1$ is an orientation mechanism based on orthogonal arrangement of three (3) active circles, $C_i$, i=2,..,4 ($C_4 \perp C_3 \perp C_2$), known as Euler cradle. However, here is an opened configuration ($C_3$=1/4 circle). Note: Dm is shown in nominal position with all components at their initial positions ($\theta$=$\varkappa$=$\varphi$ =0°), except the Da ($\delta$=90°); incident X-ray ($X_i$) coincides with Y-axis / negative direction.

The HV-Dm functional principle is as follows: $C_1(\delta)$ circle is responsible for partial rotations of the detector (Dt) around a horizontal axis (X), perpendicular on X.ray beam (Y). For the specifity of the investigations, the values must be in the range of -10 to up 180 grades. ($\delta$ = 0°, corresponds to the arm in horizontal (scattering) position / negative Y axis direction). $C_2(\theta)$ circle is responsible for bidirectional half-circle rotations of the sample (Sp) around the same horizontal axis (X). It has to be performed inside of a ±180° range. ($\theta$=0°, corresponds to $C_3$ circle / segment in lower position). $C_3(\varkappa)$ circle is responsible for rotations of the sample (Sp) around the second orthogonal axis (Y), perpendicular to $C_2$. These (partial) rotations must be performed inside of -3 to 100° range. More specific, the sliding plate of the circular segment must be able to be vertically oriented ($\varkappa$=90°) for surface XRD investigations. ($\varkappa$=0°, sliding plate horizontal). $C_4(\varphi)$ circle is responsible for half-circle rotations (±180°) of the sample (Sp) around a third orthogonal axis (Z). ($\varphi$=0° is corresponds to one of the base symetry axes colinear with X ).

Note: $C_1$ belongs to Da, $C_i$, i=2,…,4 to Sp manipulation systems.

The speed was not a specific requirement issue but the values has to be around 1 °/s.

In addition to above main (rotational) motions resulting from diffraction (Df) principles, other (auxiliary) motions must be performed. These are including the linear adjusting motions – a) X,Y and Z of the sample (Sp), being performed by PKM device (see the values in Section 2.2.1) and b) $Z_d$ (± 150 mm) of the detector (Dt), by a linear manual stage.

The maximum / minimum values for the travel range of most important kinematic parameters - circular motion/circles ($C_i$, i = 1,…,4) together with the required accuracy are presented in Table 1.

**Table 1** Kinematic specifications (HV-Dm)

| Circles ($C_i$) | Range (°) | Acc. (m°) | Res. (m°) |
|---|---|---|---|
| $C_1(\delta)$ | 180/-10 | 6 | 0.2 |
| $C_2(\theta)$ | ±180 | 10 | |
| $C_3(\varkappa)$ | -3/100 | 9 | |
| $C_4(\varphi)$ | ±180 | 21 | |

The alignment base (Ab) system consists of a large, but a precision heavy load customized manipulator (hexapod) supporting the vacuum vessel (and diffractometer) [20]. It has a good accessibility (aperture) for connecting the necessary vacuum devices (e.g., turbopump) performing 6dof motion, including small rotations around Z axis (Rz=±10°) through a bellow. This choice allows for the use of Dm in both, bulk and surface XRD configurations.

### *2.2. Design concept*

Positioning in vacuum for XRD experiments imply specific fundamental (theoretical) knowledge of and practical experience in designing such systems, from both, the diffraction and the vacuum technologies. Primarly, it consists of choosing the adequate solutions for diffraction mechanisms (motions) and their actuations (motors, sensors and cables), including materials for components and their maintenance (e.g., grease) selection, for a room with high-pressure ($10^{-6}$) and sever space constraint, for the proposed HV-Dm kinematic scheme.

The complete structure is divided in two modules - sample (Sp) and detector arm (Da), each of the positioning modules (Pm) having one or several positioning units (Pu) components. These can be, standard or customized rotation / linear stages, adapted to vacuum specifications. However, most of their parts is comming from commercially available sources. The resulting architecture is shown in Fig. 2.

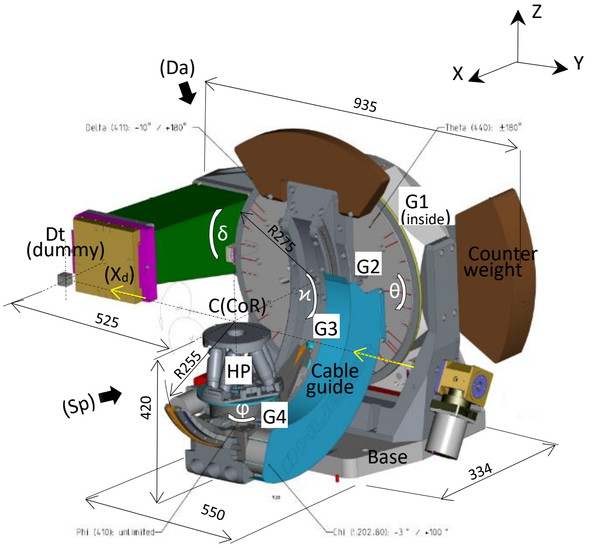

**Figure 2.** HV-Dm Layout (CAD)

The constitutive parts, their roles, together with the available precisions will be presented below.

### *2.2.1. Sample Positioning*

The sample positioning (Sp) module should provide means to pose (translate and orientate) the sample with the required precision toward the incoming X-ray beam ($X_i$, i-incident). Basically, it can be designed on EULER cradle (E500[21]) device principle. However, the actual Sp module design, includes two subsystems: a) customized opened EULER cradle and b) parallel kinematic device (hexapod). These are materialized by the following components: 1) Gonio 2 (440X2W2), 2) Gonio 3 (5202.80HL), 3) Gonio 4 (G410-X2W1) and 4) precision hexapod (BORA). The first three are performing rough (coarse) – rotations (Rx, Ry, Rz) and the last one fine (small) - rotation (Rx,Ry,Rz) and translations (Tx,Ty,Tz). The manipulated load (sample) was estimated to be around 6 kg, including additional instruments (e.g., heater). Note: G3 is a gonio segment supporting the G4 (and hexapod) and the afferent electrical cables (motors and sensors). It has to be particularly stiff. It consists of a main body (wormgear) mechanism and two lateral roller arc guides (THK) for sliding a plate and a special electric cable house.

BORA product is a standard, compact and stiff positioning hexapod (HP) with a good ratio of performance/manufacturing costs [22]. It can carry of approx. 3.5 kg in any direction, Fig. 3.

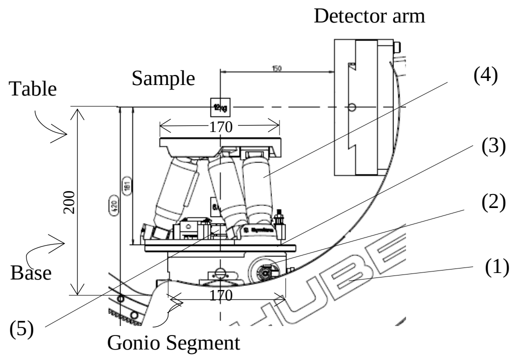

**Figure 3.** Sample Positioning Module (Sp)

The hexapod is fixed on Gonio 4 (2) through an adapter plate (3) and the transfer of electric cables to the central connector box done through slip-ring connection (5) fixed on plate. Further, the cables are guided through a guiding means consisting of a blade (stainless steel) and protective house (Al) especially when (HP) is moving in horizontal position ($\varkappa$=90°) or the segment (G3) rotated ($\theta$=90°). The most important precision features of the hexapod are specified in Table 2.

**Table 2** Design specifications (PKM)

| PKM | Range<br>(mm / °) | Acc.<br>[µm / m°] | Res.<br>[µm / m°] |
|---|---|---|---|
| BORA | X=Y=±20, Z=±10 | 13,10,3 | 0.1 |
| | Rx=Ry=10,Rz=15 | 7,5,10 | 0.1 |

### *2.2.2 Detector arm*

The detector arm (Da) subsystem or module is responsible for moving the detector (Dt) apparatus to a required position to catch the resulted diffracted beam - $X_d$ (d-diffracted) after it is comes out from the sample. In our case, it must provide precision orientational motions and support for different type of detectors (1D/2D) owing maximum 12kg weight. In addition, it must be able to have adjusting capabilities (± 150 mm) for Dt around nominal/fixed position (525 mm). Thus, it is conceived as a very stiff bilateral arm (1 dof) manipulator, by using high precision motorized gonio stage (Gonio 411-X2W2) as the actuation joint, integrated in the Sp gonio (G440). In addition, a (mannually) actuated ball-screw linear stage was provided at one end, and a counterweight pack of plates (12x1kg) from copper (Cu) included, for static (and dynamic) balance. Note: Dt was expected to be a light and compact two-dimensional (2D) Hybrid Photon Counter (HPC) product (PILATUS 100K-M / Dectris) with 172 x 172 µm pixels size area compatible for high-vacuum [23]. An overview of the precision parameters, including accuracy and resolution of motions for all above actuation (gonio) stages is presented in Table 3.

**Table 3** Design specifications (Gonio)

| Pu | Type | Precision | Acc. [“] | Res. [“] |
|---|---|---|---|---|
| G1 | 411HV | X2W2 | 30 | 3,6 |
| G2 | 440HV | X2W2 | 20 | |
| G3 | 5202HV | HL | 10 | |
| G4 | 410HV | X2W1 | 30 | |

All the actuations of motorized stages are performed using stepping motors (VSS/VSH, PHYTRON) [24], gearboxes (2083.10/2056.10(20), HUBER) and (RESOLUTE/RESA/RTLA, RENISHAW) [25] absolute encoders, eventuelle limit switches, all of which are compatible with high-vacuum. At the factory premises, the control was performed using a dedicated control box (SMC9300) for Dm and specific controller (SYMETRIE) for hexapod.

## 3. Prototypes

Based on the accomplished design work, the first high-vacuum diffractometer (HV-Dm) product was manufactured at the end of 2016. After carrying out factory tests, the prototype (Prototype 1) was installed at the allocated place, at one of the two endstations. The second prototype (Prototype 2) from Fig. 4 (shipped at the beginning of 2017) follows soon the first one, at the next location. In addition to a lifting bar (2) with two eye bolts for transportation and fast setup in the vacuum chamber (or, in other places), it consists as previously mentioned, from new component related with the connections and protection of electrical cables (3). Both prototypes were tested for precision and vacuum capability (bake out) before shipping.

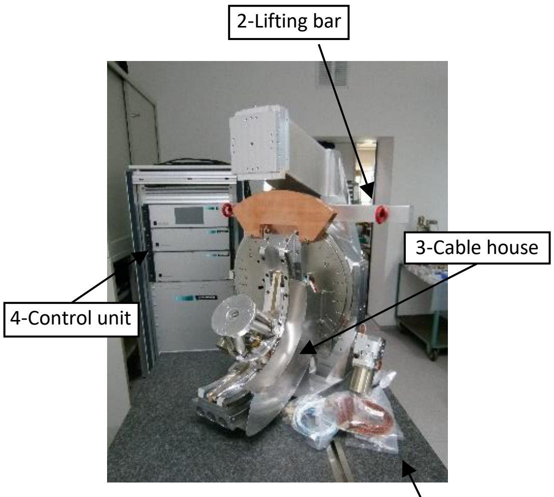

**Figure 4.** HV-Dm (Prototype 2)

Sphere of confusion (SoC) is the most important precision parameter for a diffractometer. It consists of the registered values of a combined (spherical) motion involving main components (sample, detector) with / without the loads (instruments, detectors). The maximum obtained values expressed all possible accumulated errors during fabrication (machining, assembly) and control (geometric, kinematic) points of view. These values have to be correlated with the detector pixel area and the beam size to decide if the product will be good enough for the desired experiments. For HV-Dm the SoC values were inside of 80 µm maximal sphere for all full rotational motions and charged with loads. Tests were performed using calibration sphere (ϕ15 mm, 25 nm sphericity) and sensitive dial gages (2 µm). As can be extract from the measurement protocol, the maximal individual errors (radial run-outs) occured as the segment (and cables) is moving (e.g., Ɛ=-25µm, θ=-120°). The maximal errors (axial run-out) for Da happened in vertical position (Ɛ=24µm, δ=90°).

## 4. Conclusions

The development of a synchrotron research equipment relevant to investigations at the nanoscopic level has been presented. This involved a strong collaborative work between several institutions and companies. The expertise to manage the vacuum technology has been among the competences of the members. The core of the development was a compact but versatile high-vacuum diffractometer (HV-Dm). It consisted of a four-circles (4C) structure able to perform various standard and specific X-ray investigations, using the actual standard and modern scattering techniques for nanostructured materials. The challenging issues regarding its work in vacuum, together with kinematic and design concepts and factory precision tests have been revealed. With the new architecture, including a PKM (hexapod) device, the multipurpose HV-Dm is expecting to deliver stable and precise positioning motions in the allocated small space of a vacuum chamber. It can be adapted for other tasks under similar conditions.

## References

[1] Light Sources Organization 2019 *http://www.lightsources.org*
[2] Klug H.P. 1974 X-Ray Diffraction Procedures: For Polycrystalline and amorphous materials, *John Wiley & Sons*, 2nd ed., ISBN: 978-0471-49369-3
[3] Seek O.H. and Murphy B.M. 2015 X-Ray Diffraction: Modern Experimental Techniques *CRC Pres* ISBN 9789814303590
[4] Lamberti C., Agostini G. 2013 Characterization of Semiconductor Heterostructures and Nanostructures *Elsevier* ISBN: 978-0-444-59551-5, 2 ed.
[5] Nagamitsu Y. 2008 Vacuum Technology *Springer* Berlin ISBN 9783540744320
[6] NSF 2017 Multipurpose X-ray Diffractometer, *MRI* 1725016, US
[7] Olea G. et al 2015 High Precision multipurpose diffractometer *Proc. of euspen* ICE 2015 391-392
[8] Merlet J-P. 2006 *Springer* 2 ed. ISBN-10-1402041322
[9] SOLEIL Synchrotrpon 2019 *https://www.synchrotron-soleil.fr/en*
[10] MAX IV Facility 2019 *https://www.maxiv.lu.se/*
[11] SYMETRIE 2019 *https://www.symetrie.fr/en/home/*
[12] HUBER Diffraction and Positioning GmbH & Co. KG 2019 *https://www.xhuber.com/en/products/2-systems/21-diffractometers/*
[13] G. Ciatto et al. 2016 SIRIUS: A new beamline for in situ X-ray diffraction and spectroscopy studies of advanced materials and nanostructures at SOLEIL *Thin Solid Films*, 617(A), 48-54
[14] FemtoMAX 2019 *https://www.maxiv.lu.se/news/new-worlds-open-up-in-billionths-of-a-microsecond/*
[15] G. Ciatto et al. 2019 FORTE-a multipurpose high-vacuum diffractometer … *J. Syncr. Rad.* 26(4), 1374-1387
[16] SDMS-La Chaudronnerie blanche 2019 *https://www.sdms.fr/en /our-skills/*
[17] Hamilton W.C. 1974 Angle settings for four-circle diffractometrs, *Int. Tables for X-ray Cristallography*, IV 273-284
[18] Busing W.R. Levy H. 1967 A Angle calculations for 3-4-Circles X-ray and Neutron Diffractometers *Acta Cryst.* 22 457-464
[19] Mochrie S.G.J. 1987 Four-Circle Angle calculations for Surface Diffraction *J. Appl. Cryst.* 21 1-3
[20] JORAN Hexapod 2005 *Data sheet* SYMETRIE 4p
[21] HUBER Euler Cradle 2019 https://www.xhuber.com/en/products/ 1-components/12-rotation /eulerian-cradles/
[22] BORA Hexapod 2017 *Factory acceptance tests* SYMETRIE 49p
[23] PILATUS3 100K-M 2019 Compact and variable hybrid photon Counting detector *Technical sheet* DECTRIS Ltd. Switzerland 4p
[24] VSS/VSH Stepper Motor 2018 *Brochure* PHYTRON GmbH Germany 15p
[25] RESOLUTE$^{TM}$ absolute optical encoder 2018 *Data sheet* L-9517-9399-03-B RENISHAW Ltd. UK.