euspen's 23rd International Conference & Exhibition, Copenhagen, DK, June 2023
www.euspen.eu

Diffractometer platform for X-ray synchrotron reflectometry

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Abstract
The recent development of a set of highly integrated diffractometers for advanced X-ray structural investigations of materials at their surfaces, working inside of a synchrotron research facility, after the upgradation process will be ended is here presented. In-situ hard X-ray scattering technique (XRS), as reflectometry (XRR) will be mainly used for each of the three (3) precision multipurpose diffractometers (Dm) machines built to be shared with each of the branches (B, D, G) of a Coherent Hard Energy (CHEX) beamline located in a dedicated sector (28-ID). Dms (-B,-D,-G) machine configurations are slighty different each other and is uses a previously succesfull common concept derived from 6-circle diffractometer (Dm6) type based on the combination of stacked and in-parallel (hexapod, tripod) motion principles. With the additional improvements for the main modules – sample (Sp), detector (Dt) and the table (Tb) the functional features, e.g. versatility and safety use of the entire platform (CHEX-Dm) consistently increased. The kinematic motion concept, design issues and precision motion aspects for the manufactured prototypes are in detail described.

Synchrotron positioning, diffractometers, parallel kinematics, concept, kinematics, design, accuracy

1. Introduction

Over the time, X-ray scattering/diffraction technique proved to be a powerfull tool for investigating the structure of materials, typically for physical / chemical properties point of view [1].

When a synchrotron X-ray radiation (tool) and in-situ (real) experiments are combined, a structural process can be detected even in the real time. Thus, the electronic properties at surfaces/ interfaces of materials (e.g. thin films, catalyst, etc) dedicated for some crucial energy applications (e.g. semicon, solar cells, bateries, etc) can be in-process detected / manipulated [2].

X-ray reflectometry/reflectivity (XRR) as an experimental technique emerged in the last years, as a modern X-ray technique to apply for the purpose [3].

Till now, the investigations have been carried out using classical diffraction machines, called diffractometers (Dm) located in at the end of beamlines (endstations) built on a traditional stacked stages concept.

However, the required functional and precision features for surface-scattering instruments strongly evolved in the last years. New (or, improved) Dms are to be built to cope with the actual/ prospective stringent research interests [4], applying modern techniques, e.g X-ray reflectometry (XRR).

Advanced Photon Source (APS) [5], as a well-known X-ray synchrotron research facility is now undertaking a massive upgradation process (APS-U) [6]. Apart of the new beam lines with increased investigative power, as reduced emitance and/or increased coherence physical parameters, the enginering performances of machines inside the experimental facilities (hutches) will be also enhanced.

The new Coherent High Energy X-ray (CHEX) beamline from X-ray Science Division (XSD) included in its plan the development of several new instruments for advancing XRR investigations, for each of the four (4) selectable/tunable energy branchlines.

An overview of this development including the fore mentioned instruments (Dms), forming an integrated, but versatile multipurpose platform (CHEX-Dm) is further presented.

2. CHEX-Dm

CHEX beamlines [7], in the dedicated sector (28-ID) will operate with a combination of: a) integrated and b) shared instruments to perform various tasks related with high energy coherent X-ray scattering measurements. Each of the instruments have to include a central diffraction machines (Dm) and additional optical components (table, flight path, etc) located in B (tunable) and D,F,G (selectable) beamlines experimental hutches. Dm(s) should accomodate with a relative large spectrum of experimental devices (e.g. vacuum/cell chambers) and the posibility to be easly and reliably transfered from one instrument to another [8].

In this respect, a previously succesfully customized 6-Circles Dm single structure [9] was proposed, as the basic structure (Dm-G). By the addition of two functional modules (Linear stage and Euler cradle), other configurations (Dm-B and Dm-D) can fastly and stably be built. In addition, most of the precision parameters have been reviewed and improved, togheter with the introduction of new transportation and safety means.

This platform of Dms, forming an integrated multipurpose platform of diffractometers (CHEX-Dm) was recently shortly described in [10]. It included some general aspects related with the kinematics and design of motions, only. Here a more detailed description, as kinematics structure analysis, challenging design issues, together with the manufacturing aspects to accomplish the goal during the entire process of producing them is presented. *Note: For the moment, the platform is including only a set of three (3) components (Dms) in an issued order, instead of four (4) as initially planned.*

2.1 Kinematics

Generally, the Dm(s) machines inside of the platform have to perform two types of motions (positioning and scaning) with required precisions and speeds, carrying sometimes an appreciable load (100kg) - the sample itself and the chambers (cells). In addition, the entire system have to maintain an imposed level of mechanical stability, during the investigations.

In order to solve the evident dilemma of simultaneous high precision & speed working conditions, a combination of devices based on serial & parallel motions principles has been proposed, Fig. 1. The final concept is expecting to provide a good compromise between the two above required parameters.

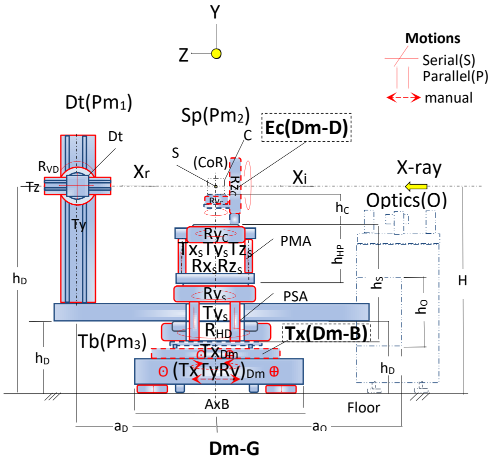


Figure 1. CHEX-Dm kinematics – Dt, Sp & Tb modules

Following this, in a modular aproach, each Dms is proposing to have a subset of two positioning modules ($Pm_{1,2}$), as main components: a) detector (Dt) and b) sample (Sp), respectively. Their role is to work together in an interconected way towards the incoming X-ray beam which is penetrating (Xi) first and then refracted/ reflected (Xr) from the sample to be investigated.

The detector (Dt) positioning module (Pm_1) mainly consists of a serial combination of two - orthogonal rotations (R_{HD},R_{VD}) and translational (T_{YD},T_{ZD}) motion mechanisms to pose the image detector (Dt) / 20kg in the right position relative to Xr. The specific motions are materialized by afferent single axis positioning units (Pu) performing each, (T) and (R) motions.

The samples(Sp) positioning module (Pm_2) includes a combination of - serial and parallel positioning devices(Pu) which have to move the sample and, possible an experimental chamber (Ch) relative to Xi. Their role is to perform the requested motions with the sample, as specified for the investigations. As a previous motion, to position the sample rotation center (CoR) of the machine to coincide with the beam line (alignment).

As recently, the parallel positioning technology (PPT) fastly evolved, the related products fund their right place in synchrotron positioning, e.g. diffractometers, as well. Therefore, due to their evident avantages (load, precision, etc), the devices using parallel kinematic mechanisms (PKM) [11] as tripods, quadropods or hexapods were ones of the options to work

Table 1 CHEX-Dm motion specifications

Type (T) (R)	Range [mm] [rad]	Acc. [μm] [μrad]	Rep. [μm] [μrad]	Res. [μm] [μrad]
T1(X_{Dm})	±20	<2	<2	1
T2(Y_{Ch}) R2(θ_{YCh})	±25 ±180	<2 <150	<2 10	1 <5
T3(X_{Sp},Y_{Sp},Z_{Sp}) R3(θ_{XSp},θ_{ZSp})	±10 ±5±10 30 ±5	<5 2 <5 <150	<2 0.3 2 <10	1 0.3 1 <5
R4(θ_{YSp})	±180	<150	<10	<5
T5(Y_{Dt},Z_{Dt}) R5(θ_{HDt},θ_{VDt})	1050 ±250 ±180	<10 <150	<20 <10	10 <5
R6(θ_{ZCh},θ_{YCh})	±157 ±180	<150	<10	<5

inside of the sample module. Finally, two parallel motion mechanisms – a) single-axis (PSA) tripod and b) multiaxis (PMA) hexapod based on parallel actuation principle, have been proposed to be used, as positioning units (Pu).

However, we must note that tripod PSA (M) is a qvasi-PKM in the right sense of PKM theory, providing a single dof, with the help of three actuators (redundancy). Nevertheless, it can be included in the general, wider class of PKMs.

Comercial precision quadropods [12] are exhibiting very good power and precision parameter inside of a compact small space (height), beeing perfect adapted to Dm working conditions. However it fails to provide one of the required parameters - R3 (θ_{XSp})=25°.

Finally, depending of the Dm version (Dm-D), the chamber, holding the sample have to perform two orthogonal rotations ($R\theta_{Ch}$,$R\theta_{Ch}$). This last rotations will be done in an interchangeable way, by a specific positioning unit called Euler cradle (Ec).

All above components inside (Dt) and (Sp) modules have to be supported by an aditional module (Pm_3), beeing stable enough in the fixed position, but having in the same time planar mobility (TxTzRy) for the transportation purpose.

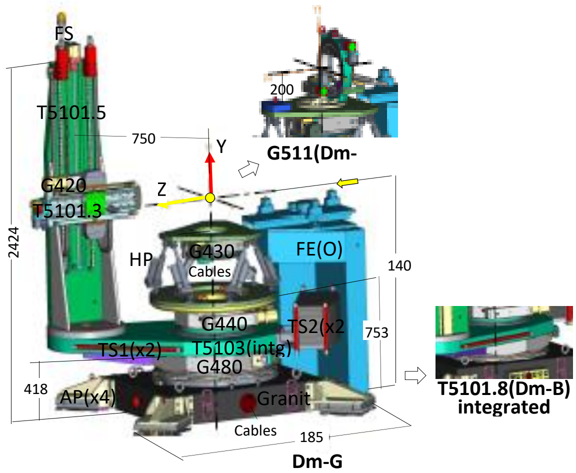
An overview of all types of motions, including the range and precision parameters are shown in the Table 1.

In addition, the values for the required speed of motions have to be as: a) translational (T=3mm/s) and b) rotational (R=1°/s), respectively. In addition, the required stability have to be bigger than : a)T= 1 μm/h, b) R= 30 μrad/hr (hr-hour) values.

From kinematics point of view, as easy can be noted from Figure 1 (concept) and Table 1 (parameters) the platform is including an appreciable number of precision motorized (single) axis of motions, Dm-B (16), D (17), G (15). This is qualifying the system to belong to the highly precise complex kinematic class. *Note: More details about the front-end optics (O) can be found in [13].*

2.2 Design

Following the above proposed modular kinematic concept, inside the design process most of the components in motion have been materialized with commercially conventional (standard / customized) precision positioning units. Thus, for: a) simple motion axis, with linear/rotation stages [14], and b) multi-axis motion, with more complex positioning devices, as



precision hexapods [15], Fig.2 .

Figure 2. CHEX-Dm design-rotational (G) & translational (T) & multiaxis (TP,HP) positioning devices

Table base (Tb) module as to has been made from a granite block with tight values of geometrical parameters (e.g. planarity < 5μm), having a central/orthogonal hole for an appreciable number of (electric) cables from above to go through. It can be moved by the use of four (4) foots, having each of them, powerful air pads. In addition, depending of the Dm version, in an integrated way, a translational device (5101) is moving horizontally (X) a strong and higly precise rotational goniometer (G480), shortly called in the “synchrotron language” gonio.

Sample module (Sp) is by far the most complex components of Dm. It consists of a combination of several serial (T,R) and in-parallel (HP, TP) actuated positioning units (Pu) with single or multiaxis scope.

As it was evident the necessity of stable and precise vertical translation to be done, by all the equipments superposed on the table a (quasi)tripod positioning unit has been choosen (T5103) to be integrated inside of the first gonio. It stands as one of the main components of the (Sp) module.

In order to fulfill the required complex spatial motions of the sample (and, chamber), a medium size (h_{HP}=350mm) hexapod (BREVA) has been preferred, as it provides 6dof (TxTyTzRxRyRz), beeing able to carry an appreciable load (<200kg) with high resolution (0.5µm), allowing to scan/pose the sample with high repeatability (±1µm/±5µrad), performing large rotations traveling strokes (±20°). However, for the required full rotations of the sample, a Gonio (G430) device has been attached beneath the platform. *Note: The third rotation, around the vertical axis(Y) beeing substituted by the addition of a device performing full rotation, the hexapod rotation (Ry) becomes redundant.*

The Euler cradle (G511) positioning device is providing means for performing the last orthogonal rotations of the (sample) ($R\theta_{Ch}$,$R\theta_{Ch}$).

An overview of all motion axis, together with the prefered choosen design solutions are shown in the Table 2 below.

Table 2 CHEX-Dm design solutions for motion axis

Axis (A)	Type (T,R)	Module (Pm)	Unit (Pu)	Diffractometer (Dm)
M1	T1	Tb	T5101.80	Dm-B
M2-M4 M5	T2 R2	Ch	T5103.F30 G440	
M6-M11	T31-T33 R31-R33*	Sp	HP (BREVA)	Dm-G
M12	R4	Sp	G430	
M13, M14, M15, M16	T51,T52 R51,R52	Dt	T5101.50/30 G480/420	
M17, M18	R61-R62	Ch	G511	Dm-D

** R33(not used)*

Most of the motorised axis above are using VEXTA(PKP)/ ORIENTAL stepping motors which are controlled in a closed loop way, through a dedicated control box (SMC9000) and software for which sensors, as absolute RESOLUTE(RKLA/RESA) RENISHAW encoders(E) compatible with EPICS existent control systems have been provided, together with electrical limit switches and mechanical stop/end components. In addition, from safety reasons, to avoid collision with front end optics (FE), two pairs of touch sensors TS1, TS2 (TS28/TAPESWITCH) have been attached to Dt arm. Finaly, a central lamps flash sensor (FS/WERMA) will warn about the system when it is in the working state (motors on). *Note: The length of the cables have been around 10m long, as requested from the cable management point of view (motor electronics envisaged to be on the roof of each enclosure/hutch).*

2.3. Accuracy

In order to fulfill with the final required functional and precision parameters, as specified in [6], the platform has been tested before the delivery.

In this respect, each of the three Dms have been separately undergone some specific tests at the factory premises. The results were included in a Factory Acceptance Test (FAT) report [16]. The conclusion was that all specified requirements have been met the customer expectations.

Before, all motorized individual axis (M) of the components mentioned above have been individually tested, as well. The procedure consisted in to carefully determination of: a) functional (range, homing, collisions, etc) and b) precision (accuracy, repeatability, resolution, etc.) behaviour of the units. For precision, the metrological setup included a granite base, interferometric instrument [17], dedicated devices (supports), data manipulation(PC) and recording means (printer).

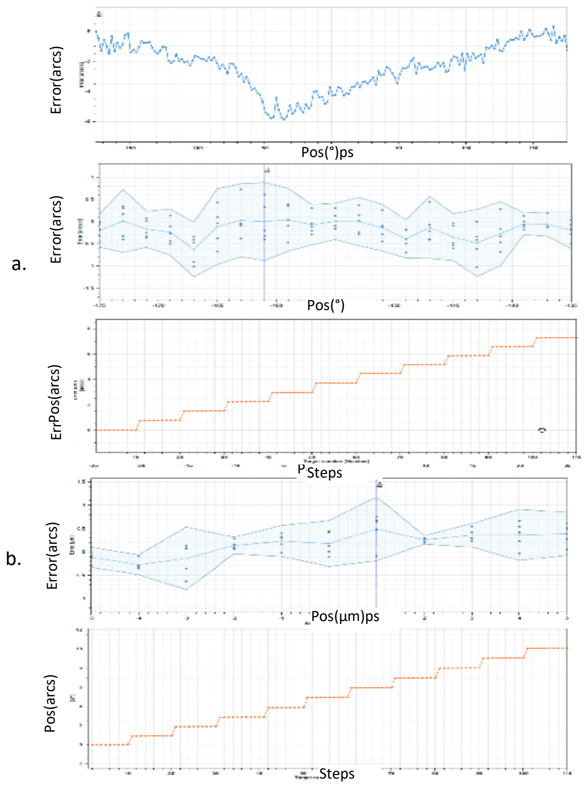


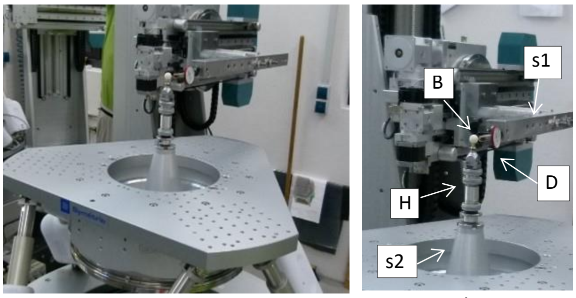
Figure 3. CHEX-Dm motion accuracy (single axis): a) G480 & b)T5101

As an example, for the main individual axis (M15) of the Dt, defined by the gonio unit (G480) and the (M2-M4) of Sp defined by linear axis device (T5103) the results, as diagrams are shown in Fig. 3 a,b. *Note: Several other measurements (precision tests) have been performed for the integrated devices, including the hexapod, to be sure they are fulfilled the general specifications.*

One of the most important parameters of the accuracy for a Dm is its Sphere of Confusion (SoC). The value represents all the accumulated errors of the machine around the center of rotation (CoR), during the combined motions (Sp&Dt).

However, from practical pov the SoC are often separately evaluated. The obtained values for all Dm(s) related with a) sample (SoC_S) – B (15µm), D (18µm), G (16µm) and b) detector (SoC_D) – B (21µm) D (22µm), G (23µm) motions fall well inside of the specifications (SoC_S=40µm, SoC_S=50µm).

The (SoC) metrological setup included several standard and customized artifact - calibrated ball (B/d=14mm,o25nm) with adjusting head (H1002) and supporting plate (s2) and the dial gage (D/res=2µm) with supporting bar (s1), as shown in Fig. 4.



a. b.

Figure 4. CHEX-Dm motion accuracy (SoC) : a) layout & b) details setup

3. Prototypes

Finally, based on the proposed kinematic concepts and the design choices for the components, expecting that the required specifications of the final motions are all fulfilled, the complete set of Dm(s) have been manufactured. The products (prototypes) – P1 (Dm-B), P2 (Dm-D) and P3 (Dm-G), inside of the system (platform) have been already delivered to the dedicated customer, waiting now for the installation / commissioning.

Special attention was given at machining and assembly operations to match with prescribed final precision. In addition, care has be taken for transportation and packaging, as well.

As most of the components were in-house manufactured, for which the necessary experience already existed, care has been taken for the outsourcing elements (granite, connectors, cables, etc), especially at their common contact with the existent (in house) products. Among others, the procedures involved the qualities (surfaces) and tolerances (dimensional/geometric) inspection for key elements (e.g. centering holes/pins, etc).

The functional sensitive surfaces (e.g hexapod platform/gonio plates) with the experimental devices (chambers), allowing various configurations of samples / instruments to be rapidely fixed on them, the process involved also a (pre)aligned step, using specific and general related procedure and metrological devices (levelling, autocolimators, etc).

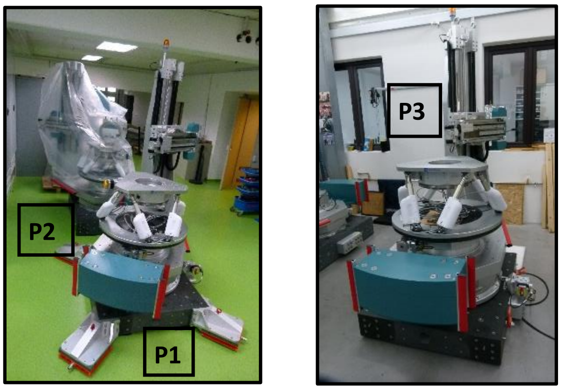


Figure 4. CHEX-Dm components: a) P1 (Dm-G) & P2 (Dm-D) and b) P3 (Dm-B) prototypes

4. Conclusions

A new highly integrated platform of dedicated diffractometers (CHEX-Dm), using mainly X-ray reflectometry (XRR) technique, as the investigative tool has been presented.

Each of the three components (Dms-B, D, G) of the platform are exhibiting an enhanced functional capabilities, by using in an interchangeable mode, a large variety of common and specific instruments (chamber) and apparatus (optics) for manipulating an appreciable number of different samples.

Each of the diffractometer fully serves one of the branches of main beamlines. Thus, first Dm (Dm-B) is serving the branch B, the second (Dm-D) the D branch, and third (Dm-G) the G branch of the main beam line 28-ID.

The kinematics, design and manufacturing aspects have been revealed in detail for each of the above Dm(s) components, and the platform as an whole.

From kinematic point of view, the Dm(s) structures are using a previously successful combination of stacked and in-parallel high precision positioning mechanisms, performing simple axis (translation/rotations) or multiaxis (hexapod, tripod) motions based on a common 6-circle structure of diffractometer (Dm6).

From design point of view, single or multiaxis positioning devices, as linear/gonio stages and tripod/hexapod have been used. In addition, for transportation reason, airpads have been provided and for safety reasons, means as warning lamps and touch sensors have been included.

From precision point of view, high precision positioning components have been used, as single axis units (e.g gonio) belonging to XP (extra) class of precision and high precision multiaxis (tripod, hexapod) devices. With the addition of in-parallel actuated axis the precision of moving parameters, together with the stability, consistently increased.

Thus, the prototypes of the platform are offering both, high stability and reliable versatility, for performing complex research investigations, by cutting the (auxiliary) times, and using several methods and instruments in the same place (or, nearby).

Next step will include the integration of the fourth component (Dm-F) dedicated to the last branch (F) of the main beam line.

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