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Versatile high precision synchrotron diffraction machine

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Abstract

A newly developed instrument intended to be used for advanced experimental investigations in a modern upgraded synchrotron research facility has been recently completed and delivered. The dedicated diffraction machine (diffractometer), working on X-ray diffraction principle consists of a basic structure with five circles 5C(2D+3S) geometry. By adding some specific components related with the enlargement of functional spectrum, as different techniques (polar dependent spectroscopy and magnetic scattering) applied on various samples (such as magnets and thin films), the machine became a complex research instrument, including the rigorous requirements of precision. In this respect, two interchangeable positioning modules have been provided, opening the way of comparing the results of various samples with the same technique, avoiding the associated errors related with their transfer (reproducibility). A stiff alignment base (B) supports a heavy dovetail detector (D) and sample (S1, S2) modules, each including several high precision positioning units (Pu) with linear and/or rotational motions (goniometers/translational) stages. (S1) includes a stiff high precision customized Euler cradle (Ec) for positioning of a magnet (2T) and (S2) several precision gonios (G), together with a nano positioning piezo device. The paper gives a complete final view of this versatile instrument, including the proposed kinematic structure, design concept selected solutions, together with an analysis of precision data collected during the final tests.

Synchrotron, diffractometer, positioning, kinematics, design, precision

1. Introduction

Synchrotron radiation is one of the most powerful investigative tools available today for exploring the internal structure of various matter states. It is expected that in the future, advanced research will be based not only on newly built or, improved (upgraded) modern facilities with increased functional beam parameters, but on new dedicated instruments /machines, working inside for specific applications [1].

A consistent upgradation process started few years ago at Advanced Photon Source (APS-U) [2]. After that, the primary characteristics of the beam e.g., emittance, coherence, etc will be improved and several new beam lines will be constructed. As such, the afferent instruments in the experimental hutches will be new ones or partially renewed.

Several beamlines from the X-ray Science (XRS) division have been allocated for the Magnetic Materials (MM) group investigations. Here, 4-ID (POLAR) beamline will be focused on emergent topic of electronic/magnetic effects of inhomogeneities in magnetic and/or ferroelectric materials relevant to actual quantum and energy requests. In this purpose, several techniques will be used, as X-ray spectroscopy (XRS) and/or X-rax magnetic scattering (XRMS) [3].

For one of the experimental stations, hutch (G) a request to develop a dedicated machine, called diffractometer (Dm) has been released [4]. Based on this, the new (Dm) intended to be used has to be adapted for horizontal scattering (Q-range) access, using both heavy load (superconducting magnets) and low vibration smaller samples, under extreme conditions (low temperature, high pressure) investigations. (Dm) has to offer not only the possibility of using the above-mentioned X-ray techniques for a single sample, but to be prepared for high precision (interchangeable) working capabilities of the samples.

A short overview, including the main features of the final instrument (prototype), during its finalized process have been revealed [5]. However, here more details are to be presented, especially taking into account the relevant challenging and solutions aspects related with its precision based on raw collected data in the final step of tests.

2. Diffractometer

For a relative long time, diffractometers have been one of the key research instruments in any synchrotron facility. Based on X-ray diffraction principle and using well-known classic or modern specific techniques [6,7], various types have been developed since their first proposal. Actually, there are several well-known companies producing them; see, for example [8].

2.1. Geometry

Generally, Dm(s) include two types of geometrical configurations, in which various actuated rotation stages, called circles (C) are arranged relative to their motion axes.

In the first group (orthogonal), the basic types include machines with four to six circles (Dm-nC, n=4-6) arranged at the right angles (α=90°). However, (n) can increase, depending on the number of auxiliary devices, and sample manipulation setups, reaching eight (n=8), or more (n>8). As the actual tendency is to use – a) several X-ray techniques to investigate b) different types of samples in the same place, the number and complexity of (Dm) machines constantly increased. Note that the working precision didn't change, but even decreased. In the second group (Kappa), each axis of the circles is arranged in an angular way (α=50°-60°), as Kappa letter. Mainly, the (Dm) working principle consists of a corelated motions of detector and sample circles, relative to a fixed beam (X-ray), based on diffraction law (Bragg) principle.

Based on the specificity of ID-4G beamline [9], the new requested diffractometer was necessary to be able to use two – spectroscopy (polarized dependent resonant) and magnetic scattering X-ray techniques. In addition, it has to provide the possibility to manipulate - a) heavy cylindrical (200x400) mm 2T magnet (120kg) and b) smaller (30, 2, 0.2 kg) samples under extreme conditions– high pressure (HV) and/or low temperature (cryostat) devices. A 2D pixel array detector (5kg), together with the afferent vacuum tube (12kg) and polarization analyser (15kg) with optics devices, totalling (100kg) must be manipulated, as well.

Following these, and others explained further, the POLAR-Dm machine was proposed on a basic five circles (n=5) orthogonal geometry, Dm-5C(2D+3S), where C_i, i=2 for detector (D) and C_i, i=3 for the sample (S). However, by the intention to use two sets of sample manipulators (S1, S2), each with additional circles (C_i, i=6), together with those included in the auxiliary devices (polar analyser) the subsequent total number of circles became much more (Ci, i=9), as Fig. 1 shows. Thus, the (POLAR-Dm) machine, belongs to a complex multi-circles (Dm) family. More details featuring the chosen kinematic structure are given below.

2.2. Kinematic structure

From the kinematic (K) point of view, a (Dm) structure results from the selected geometry and the auxiliary devices necessary to be used. Each of the components from inside must fulfil the required functional and precision working parameters, e.g., range, accuracy, etc of motions. The type of their arrangements (serial and/or parallel) has also a significant importance.

In the POLAR-Dm case, the proposed structure consists of a combination of three main kinematic chains (Ki, i=1,2,3), corresponding to detector (D), sample (S) and base (B) manipulation subsystems (modules), Fig. 1.

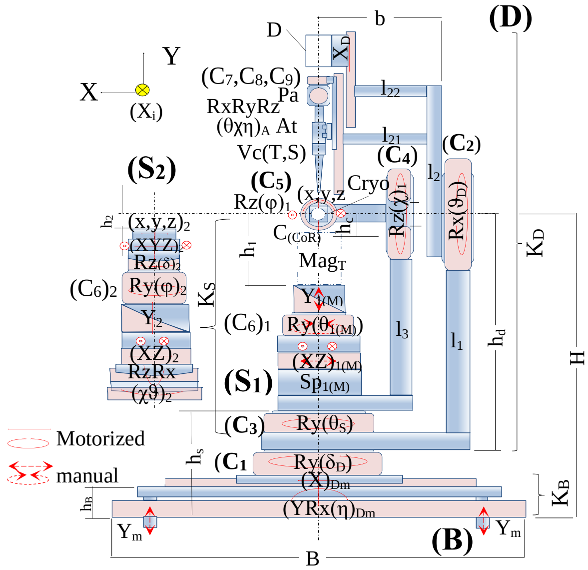


Figure 1. Kinematic structure (K)

The first kinematic chain (K_D) of the (D) manipulator consists of two active rotational joints (circles) –$C_1(\delta_D$=±180°) and $C_2(\vartheta_D$=-30°+180°) orthogonally linked together by an arm (l_1). In addition, arm (l_2) is supporting two linear guides (L_1, L_2) to accommodate with the use of a detector (D) and optics, as the polar analyser (An), attenuator (At) and vacuum tube (Vc) to catch the scattered X-rays. (An) is based on a combination of three orthogonal circles - $C_7(\theta_X$=±5°), $C_8(\chi_y$=±5°) and $C_9(\eta_Z$=+30°-110°). Basically, a detector point performs a spherical motion around the fixed point of the machine, called center of rotation (CoR), which in turn, coincides with beam line (sectional) center. The (D) nominal position ($\delta_D=\vartheta_D$=0°), otherwise when the polar analyser arm is collinear with the beam.

The kinematic chains of the sample manipulation (Ks) afferent of two types of manipulators (S1, S2) consist of various in-series (stacked) arranged actuated devices, performing rotation or translational motion, with $C_3(\theta_S$=±180°), as a common base.

In its first configuration (S_1), two orthogonal circles $C_4(\chi)$ and $C_5(\phi)$ form the Euler cradle (Ec) mechanism. (Ec) is providing an enlarged access (setup, maintenance) to the samples, especially for the large sample (magnet). It includes also a small translational motion (X=Y=±2, Z==±3) mm device, holding the cryostat. Nominal position, χ=0° when $C_5(\phi)$ along X(+). A separate support (Sp), and two (manually) driven translational (XZ=±10, Y=±5) mm and rotational $C_6(\theta_{1M}$=±180°) devices performing the alignment of (M), will be sometimes removed.

In the second configuration (S_2), several motion devices are stacked one after the another, starting with one performing arc circles RzRx $(\chi=\vartheta)_2$=±7°, following the translational ($X_2=Z_2$=5, Y_2=2.5) mm ones. On top of these, performing full $C_6(\phi_2$=±180°) and partial $Rz(\delta_2$=±7°) circular motions, carrying a course ($X_2=Z_2$=5, Y_2=2.5) mm and fine $(xyz)_2$=0.1 mm translational motions devices is forming the precision gonio (Pg) mechanism.

An alignment base (B) subsystem has to support the entire machine structure, providing reliable stability and short motions - $(X,Y)_{Dm}$= 20 mm and $Rx(\eta)_{Dm}$=±0.5° for the rough alignment toward X-ray beam longitudinal axis. (K_B) was chosen as a four legged (quatro) in-parallel mechanism (PKM) [10], from which two pairs of them are actuated. (Y_m) are manually driven feet are short motions levelling with the floor.

The main motion parameters, as the range – stroke (St) and precision -repeatability (Rp), resolution (Rs) required for the basic circles are included in Table 1. The indicated values are the lowest (highest) allowed ones. A short simulation video of the motions can be seen [6].

Table 1 Dm basic motions parameters

Circles (C_i)	St (°)	Rp* (“)	Rs (“)
$C_1(\delta_D)$	±180	8	3.6
$C_2(\vartheta_D)$	-30+180	8	3.6
$C_3(\theta_S)$	±180	0.5	0.36
$C_4(\chi_S)_1$	±100	8	3.6
$C_5(\phi_S)_1$	±180	8	0.36

* Bidirectional

The O-XYZ reference system is a right-handed set of orthogonal axes, with the origine in CoR point (O=C); the positive orientation of Z being along the incoming X-rax beam and Y vertically upward.

2.3. Design concept

Following the kinematic structure, a design process [11] started to select the suitable components, based on load supported, actuation type, shape and precision criteria. Most of them are standard with modified/improved features in-house manufactured. The resulted concept (layout) is shown in Fig. 2.

As a modular design approach has been applied, the entire machine architecture has been divided into three main positioning modules (Pm_i, i=1,2,3), each of them built from a serial and/or parallel combination of multiple positioning units (Pu_i, i=1,...,n) (Pu_i), performing basic rotational/translational motions. (n) varies from module to module, being in a strong correlation with the motion actuated axis Ai (i=1,...,n). In addition, there is a fourth module (Pm_4), dealing with the wire/cable manipulation.

Mainly, the first positioning module (Pm_1), corresponds to the detector manipulator (D) includes two powerful motorized Pu goniometers (G) linked with two stiff arms. (Pm_1) was built for the first time on two heavy load and high precision gonios (G480) able to work in both – horizontal (W1) and vertical (W2)

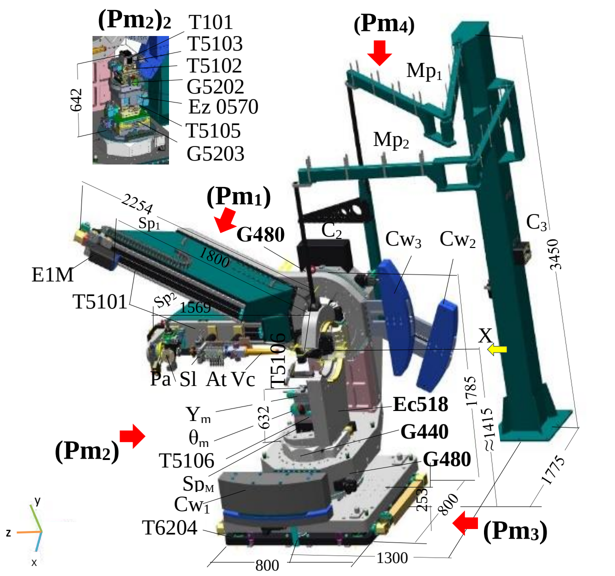


Figure 2. Design concept (CAD)

This extreme solution was adopted based on the fact that the (D) arm must carry several heavy instruments at an appreciable distance (2m) and apart (0.63 m) from the machine center point. Thus, it must provide the necessary actuation moment (force) carrying a) detector (EIGER1M) and b) Polar analyser (customized), pneumatic attenuator (3002.60M), slit (IB-C30-HV) and vacuum tube (Vc), together with their supports (linear stages). In this purpose, a dovetail concept including two stiff, but light spacers (Sp) from sheet metal (welded) was provided. And, for the static balance three counterweights (Cw_i, i=1,2,3), as well. (Pa) was built on a combination of two gonios (G410A, G409 - XEW2), one linear (X5101/XE) and head (H1005).

The positioning module $(Pm_2)_1$, corresponding to (S_1) configuration consists of a customized Euler cradle (Ec518), combining a stiff base and carrier, holding a translational stage (5106), which must carry the cryostat (ARS DE-202G).

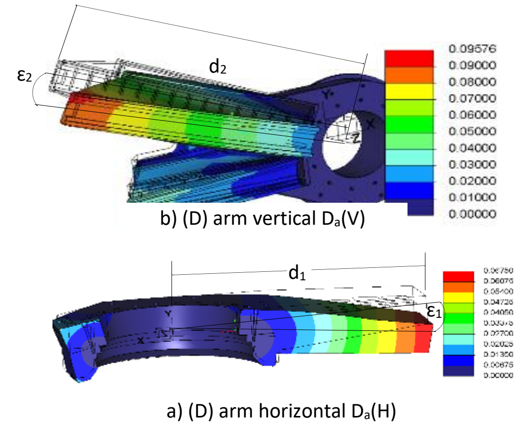
The positioning module $(Pm_2)_2$ corresponding to the second configuration of the sample manipulator (S_2) is a high Precision gonio (Pg) system built on a (gonio) segment (S5203) with required precision (XE), supporting a standard XYZ translation stage (T5105) on which a high precision air bearing stage (EZ 0570) is located. On top of them, a segment (gonio) stage (S5202) is fixed where a combination of two linear stages (X5101/T5102) and a nano(piezo) positioning stage (TRITOR 101 CAP, JENA) are supported. $(Pm_1)_2$ and $(Pm_2)_2$ modules are moved by a common precision gonio stage (G440, X2W2).

The alignment base (B) module (Pm_3) was designed on a standard table type (T6207), providing stiff and stable support of loads (<1000 kg) and precision motions for all the necessary working modules above it. An overview of selected (Pu) types, together with their available loads and precision provided Tab.2.

Table 2 Dm design parameters

Axis (A_i)	Modules (Pm_i)	Units (Pu_i)	Load (H/V)	Prec. (X)
A_1	Pm_1	G480	W2V	XE
A_2	Pm_1	G480	W2H	XE
A_3	$(Pm_2)_{1,2}$	G440	W2V	XE
A_4, A_5	$(Pm_2)_1$	Ec518	H&V	XE

As precision was also one of the main requirements, modelling and simulations have been performed, using finite element analyse (FEA).
The estimation of the deformations (deflections) and stresses (von Misses) helped a better design. By improving the stiffness, the deformation causing the geometric errors of positioning was reduced, or even totally eliminated. (D) and (Ec) components were identified as the critical ones, and the process focused mainly on them. An example is given in Fig. 3 for (D) deformation, where (ε_2) must be less than 0.02 mrad around Z



(θ_z), at d=2m and with ϑ_D=0-30°.

Figure 3. Modelling and simulation (FEA)

(Dm) machine design included not only an appreciable number of motorized circles, but the linear ones, as well. The total amount of axes reaching a high number (A_i, i=1,...,30). Generally, from the control of motion point of view, this multi-axes machine is driven by commercially available stepping motor (VEXTA/ORIENTAL, PK/P), gears boxes and incremental encoders (VIONICS/RENISHAW, RKLC/RESM). In addition, four power drives (POWERPACK) and two driving (SMC9300) electric /electronic boxes have been provided for basically, a closed loop approach applied. As expected, the management of the appreciable number of the cables from the actuated axis- wires (electric) and pipes (air), together with their necessary length for the roof (>10m) was an issue. To solve it, a specific design of a manually actuated (planar) manipulator (Pm_4) was provided. (Pm_4) mainly consists of two Brinkman incorporated manipulation components – one for detector (Mp_1) and other for Euler cradle (Mp_2), respectively. In addition, the connections have been performed through three main connexion boxes - $(Cb)_i$, i=1,3. Sleep rings were sometimes used, and the wires directed through central holes of the components. Additionally, for preventing any crushing hazards of large components in motion, electric warning stickers have been included, as well.

2.4. Precision

Precision positioning is a permanent concern in the synchrotron industry. The successful management involves not only knowledge and extensive expertise from several fields (mechanics, electric, metrology), but a permanent control (tests) during the entire manufacturing process.

Based on the above design considerations and the final adopted solutions, a first POLAR-Dm instrument (prototype) has been manufactured. It is shown in Fig. 4 (left), about to undergo the last round of motion errors tests. Fig. 4 (right) includes the measurements setups details, for both (Ec) and (Pg), submodules. However, it does not include the real cryostat device (Ec) and nor the piezo units (Pg) in order to make possible the process. In addition, the slits and vacuum tubes were already shipped before to the facility. All functional and precision motion parameters were tested at factory premises and then included in a report [12] issued to the facility. For the measurements purpose several instruments (interferometers, autocollimator, dial gages) and auxiliar devices (dummies, calibrated balls) have been used.

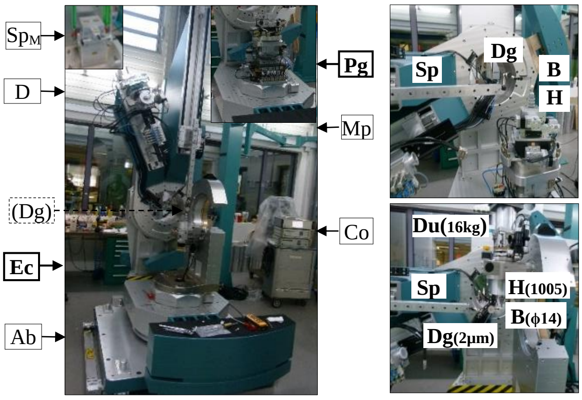


Figure 4. Prototype and measurements (SoC)

Sphere of Confusion (SoC) parameter is a global indicator of the accuracy of positioning of any (Dm). It takes in to account all types of geometric errors during the complex motion of (D) and (S). However, from practical point of view, the separate partial values - $(SoC)_D$ and $(SoC)_S$ or even those of individual axis $(SoC)_i$ could be from real interest. The required values for POLAR-Dm are displayed, Table 3.

Table 3 Runout errors (Max)

C_i [i=1,5]	$C_1(\delta_D)$ [μm]	C_2 (ϑ_D) [μm]	C_3 (θ_S) [μm]	$C_4(\chi_S)$ [μm]	$C_5(\phi_S)$ [μm]
$(SoC)_i$	15	50	5	50	5

As easy can be seen from the table, the maximum values must be less than fifty micrometers. In addition, as specified the value of reproducibility, following the interchangeable actions of individual and/ or combined modules (Ec)/(Pg) should comply with fifty microns interval (Rpr<50 μm). At a short survey, these values are qualifying the instrument, as with high precision one.

For the detector module (Ci, i=1,2), the error determination consisted in the measurements of radial (ε_r) and axial (ε_a) runout values in the two cases, with following conditions: a) (Ec) - ϕ_S=0°, χ_S=90° and b) (Pg) – χ_2=υ_2=ϕ_2=0°, through the basic gonio stage (θ_S=0°). In the first case (a), the horizontal arm performing full rotations (δ_D=±180°), and the second arm being vertical (ϑ_D=295°), the max/min values reached: a) $\varepsilon_r \in$ (-3,3) μm and b) $\varepsilon_r \in$ (0,8) μm intervals. Note, that ε_a=0 μm. When the second arm performs partial rotations (ϑ_D=-180°+30°), and the horizontal arm fixed (δ_D=25°), the obtained values were: a) $\varepsilon_r \in$ (-25,8) μm and b) $\varepsilon_r \in$(-13,0) μm and $\varepsilon_a \in$(-20,18) μm and b) $\varepsilon_a \in$(-25,12) μm.

For the sample module (Ec) with (Ci, i=3,5), the same type of errors has been collected, under the condition of the detector arm being horizontal (δ_D=25°), in two cases of payloads: a) 5 kg and b) 16 kg, Fig. 4(right). Thus, for the first case of the motion (χ_S=±100°), with (ϕ_S=0°), the obtained values were: a) $\varepsilon_r \in$(-5,7) μm, $\varepsilon_a \in$(-6,6) μm and b) $\varepsilon_r \in$(-13,12) μm, $\varepsilon_a \in$(-10,11) μm. Note: For the second determination, (ϕ_S=±180°) all the errors fall inside $\varepsilon_r \in$ (0,2) μm and ε_a=0 μm, identically with the individual gonio stage values. The graphical representation is given (Fig. 5.)

The measurements in the case of basic gonio (θ_S=±100°), for both types of sample (sub)modules – (Ec) and (Pg), the values reached: a) $\varepsilon_r \in$ (-4,0) μm and b) $\varepsilon_r \in$ (0,4) μm intervals, with (χ_S=90°), (ϕ_S=0°) and the detector arm vertical (ϑ_D=295°).

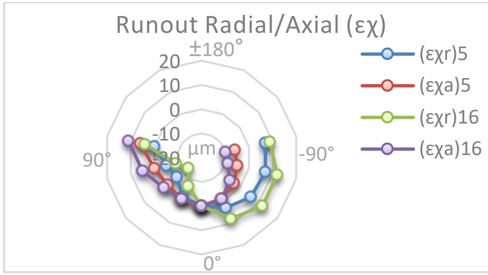


Figure 5. Positioning errors (Ec)

The max values from all measurements above are summarized in Table 4.

Table 4 Runout errors (Max)

S_i (i=1,2)	$C_1(\delta_D)$ ε_r[μm]	C_2 (ϑ_D) $\varepsilon_r/\varepsilon_a$[μm]	C_3 (θ_S) ε_r[μm]	$C_4(\chi_S)$ $\varepsilon_r/\varepsilon_a$[μm]
S_1(Ec)	6	25/38	5	25/21
S_2(Pg)	8	13/33	4	-

Shortly analysing the obtained results and to corroborate with the specificity of measurement conditions, qualifying us to affirm that the complex multi-axes (Dm) machine was able to deliver an outstanding precision, overall SoC<50μm.

3. Conclusion

A dedicated diffractometer (Dm-POLAR) with flexible research investigation capabilities to work in an upgraded 4th generation synchrotron facility has been developed. Able to support different types of X-ray techniques applied on large spectrum of (electromagnetic) materials, under extreme conditions (pressure and temperature), the machine has resulted, as a successful combination of commercial and customized components improving a basic structure. Two selective chosen specific precision modules for samples are preserving the necessary accuracy during the interchangeable process. Based on its specific features and precision data obtained, during the tests, we are entrusted to believe that a versatile high precision diffraction machine has been produced, expecting to enhance the experimental synchrotron investigation capabilities in the aforementioned fields.

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