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Advanced synchrotron diffractometer

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Abstract

The result of the work for developing a new high precision and multipurpose diffractometer able to work in a third-generation synchrotron facility by using various modern diffraction techniques is presented. The advanced modular concept consists from a combination of several pre-aligned manipulation modules – Detector arm (Da), Sample positioning (Sp) and Detector (Dc) performing correlated high-precision motions on diffraction physics principles and rules specific to each application. Sample manipulation module has been especially (re)designed to work for both, horizontal and vertical positions by carrying relative heavy loads - samples and instruments (e.g. vacuum chambers, cryo, etc). A newly developed interchangeable device, called OCTOGLIDE on Parallel Kinematic Mechanism (PKM) based topology was designed to deliver high-accuracy and stable motion in all Dof (6) of the confined allocated space using mainly standard precisision components (e.g. stages, joints, etc). With this new diffractometer design, is expected that all customer's requirements are fulfilled including the costs, as well. After the customer approval, the first prototype will be released and tested. Here, the most important kinematics and design (CAD) aspects of the final concept are presented.

Synchrotron, Positioning, Diffractometer, PKM

1. Introduction

Synchrotron research is an advanced and actual active field of research. By using the X-ray beam tool as investigation, the atomic and molecular structures of various - solids, liquids and biological materials under normal (air) or extreme (vacuum, cryo, etc) conditions have been (or, will be further) investigated.

The dedicated large-scale facilities for production of X-ray beams called synchrotrons [1] are highly complex and costly constructions. They are located especially in developed countries, but several are now under the development and/or upgraded process in Asia. Particulary, some of them are now benefiting from higher specialized beams being in the same time more versatile.

The scientific investigative methods have been developed, accordingly. A constant increased interest has been observed in recent years for researchers to understand the corelated phenomena between micro and macro worlds, at the surfaces and interfaces between liquid and other materials (solid, gas).

The applications are especially coming from the thin films, multilayers, polymers, lubrication, etc with applications from semiconductor, pharmaceutical, oil and/or tissues engineering. The X-ray diffraction techniques, using traditional and modern methods e.g. XRR, GIXD, GISAXS, etc are providing the base [2].

However, it is large accepted that the succesfully obtained results are in the direct relation with the precision of machines, instruments and devices used here, too. Therefore, a specific equipment have to be designed accordingly.

The Diffractometers (Dm) machines are the best example. They are one of the most used machines in a synchrotron facilities. Even if, they fund their place here even from the beginning, however, their complexity evolved during the years. For example, the recent advancement in X-ray technologies (methods & instruments) have made possible to develop a new class of diffractometers [3]. Various measurements are now possible inside, by applying several techniques. By this, the available user time and diversity of applications will increase through a reduced switching time. In turn, their configurations are becoming more complex. They include for example, several additional (sub)systems, e.g. Double Cristal Deflector (DCD), etc. and, the old ones, e.g. sample manipulation (sub)system necessary to pose various instruments (spectrometers, vacuum chambers, cryo, etc) devices to accomplish with the tasks must be adapted. Shortly, a single Dm product must be able to work in different configurations (horizontal & vertical scattering), by being able to carry heavy-loads, too. Thus, the product became a heavy load multipurpose (universal) Dm.

Consequently, the specific instruments and/or devices here often must work with higher accuracy. The (sub)system for load manipulation - samples (and/or instruments) is built on a succession of linear and/or angular precision positioning units (stages), materializing one of the motion axes (X,Y,Z, Rx,Ry,Rz). This traditional design occupies an appreciable space inside Dm.

The Parallel Kinematic Manipulators (PKM)[4], e.g. Hexapods [5], [6], etc has been recently in increasing use instead. They are providing high capabilities regarding the load and the accuracy manipulated. However, they are still in a few numbers of standard typo-dimensions and by this, sometimes difficult to fit inside of the Dm's wokingspace. In order to overcome these aspects, the designers look for alternative solutions. There are several key players in the market: HUBER, NEWPORT, KOHZU, etc. HUBER [7] is a well-known name in synchrotron positioning delivering high-quality products and pushing the precision at its high limits.

A new high precision multipurpose Diffractometer (Dm) able to work for various investigations (mainly, at the liquids surfaces /interfaces), by applying several modern diffractive methods including horizontal or vertical scattering has been developed. Inside, a new specific PKM (sample) positioning device fitting the working space requirments has been designed.

After the presentation of the general kinematic and design concept by taken in to account the working requirements, the paper focused on the newly PKM development. The paper ended with some conclusions and further steps to be done.

2. Liquid Diffractometer

The study of liquid-liquid interfaces with X-ray scattering methods requires special instruments to be considered [8].

A Diffractometer (Dm) has been proposed to work in a dedicated new Surface Diffraction Beamline (SDB) from a third-generation synchrotron facility [9].

The construction of (optimized) beam line of this intermediate energy light source was coming from Phase-II project [10] development and is expected to investigate the phenomena produced at surfaces (liquid/gas) and interfaces of liquids (liquid/liquid, liquid/solid) under several environmental conditions (air, vacuum, etc) using adequate surface scattering techniques (GIXD, GISAXS, etc).

The required specifications including the working, installation and commissioning issues and parameters, as well as the tests and timescale have been previously specified [11]. Most important design parameters are presented in Tab. 1.

Table 1 Design specifications (Dm)

Pm	Circles (C)	Range (°)	Rep. (m°)	Acc. (m°)	Wobble (μrad)
Da	$C_1(\Upsilon)$	135/-40		0.5	≤10
	$C_2(\delta)$	15/-195	0.1		
Sp	$C_3(\theta)$	±180			≤15
	$C_4(\alpha)$	±180			
Dc	$c_1(\rho)$	±130	0.1	0.5	≤10
	$c_2(\varphi)$	±180	0.05	0.2	

A 'liquid diffractometer' machine has been built on a previously 4(2+2)C (C-circles) basic concept [12]. It consists from several sub(systems), as Detector Arm (Da), Sample positioning (Sp) and the X-ray (Xray) called Deflector (Dc) manipulation modules (Mm), Fig. 1.

The main constitutive components, their roles and the precision issues are shortly presented below.

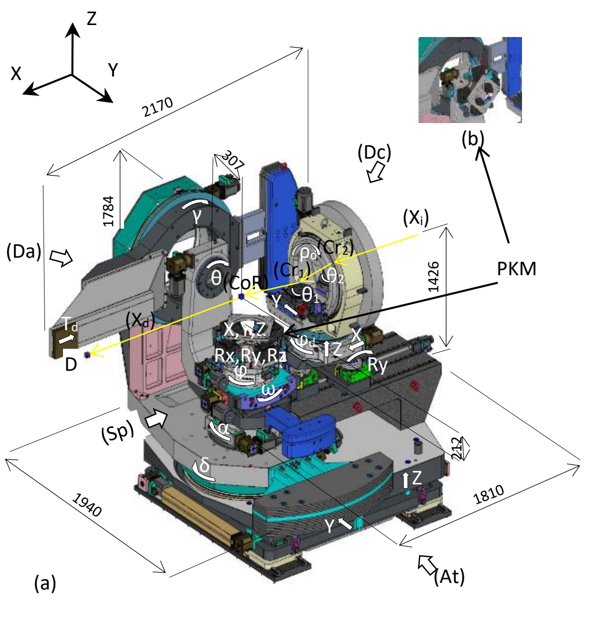

Figure 1. Diffractometer (Dm) : a) horizontal and b) vertical sample positioning

2.1. Sample Positioning

The Sample Positioning (Sp) (sub)system should provide the means to pose (positioning and orientating) a sample with the required precision towards the incoming beam (X_i, i-incident), for two working/scattering modes - a) horizontal and b) vertical, respectively.

Two kinds of samples are foreseen to be investigated: a) liquid (surface), using Langmuir instrument horizontally mounted on a fixed anti-vibration stage (590x390 mm) and in addition b) solid(surface), mounted either horizontally or vertically, respectively.

Two types of independent subsystems have been required based on two perpendicular circles - a) C3(θ) ⊥ C4(α) and b) C5(φ) ⊥ C6(ω), respectively. The afferent loads have been estimated to be 150 (horizontal) and 60 (vertical) kilograms, respectivelly.

Thus, in the design concept is including for the first system (a) two precision actuation rotation units (Gonio 430-X3W2, 420-X3W1/modified/ INA/ YRT) and for the second one (b) two (stacked) Gonio (5202.8HL, 420-X3W1) for partially orienting the sample. The first one is performing full (360°) and the second one small (Ry/ω=±6°) and larger (Rz/φ=±160°) rotations, respectively.

In addition, an auxiliary interchangeable unit working for both, the horizontal and/or vertical investigations has been introduced. It is based on a special Parallel Kinematics Mechanisms (PKM) providing stable and full motion(6dof) along / around all axes (X, Y, Z, Rx, Ry, Rz) for the afferent load of 100(H)/30(V) kg.

PKMs are special kind of mechanisms offering high accuracy, high stability and high-speed capabilities for heavy load manipulation tasks by their inherent feature (stiffness). They fund out their well-deserved place in many applications, including the synchrotron ones, especially as hexapod positioners. However, when forced to fulfil the heavy load in small available spaces (especially, height), other features (e.g. redundancy) must be taken in to account, too.

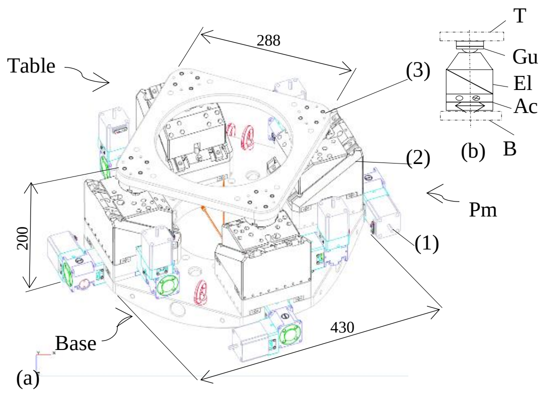

Figure 2. Positioning Table Device (PTD)

The newly developed PKM, Fig. 2 is a Positioning Table Device (PTD) providing high precision full (6dof) positioning capabilities including the support, alignment and motion, for the sample manipulation, in both, the horizontal (100kg) and vertical (30kg) working conditions.

It is based on a Quadropod structure and consists from two pairs of vertical pillars having orthogonal coplanar actuators each (2dof), symmetrically arranged around a vertical axis. When XY motorized stages are used for the actuation, the synchronized work of the eight actuators, namely the OCTOGLIDE [13] principle offers stable, fast and intuitive symmetric heavy load positioning capabilities.

A compact design (height) has been achieved by using on a base(B) the Positioning modules (Pm) with integrated motorized stages (1) for actuation (Ac) - modified XY/5102.15, customized wedges (2) – with IKO rails for elevation (El) and spherical rolling joints (3) - (SRJ/016C [14]) for table (T) guidance (Gu). As can be seen, in order to minimize the costs, standard and commercially available components have been choosen and/or modified.

The most important expected features for the whole PTD are specified in Tab. 2.

Table 2 Design specifications (PKM)

PKM	Range (mm or °)	Acc. [μm or m°]	Rep. [μm or m°]
OG	X=Y=±25, Z=±14	3	1
	Rx=Ry=5, Rz=6	3	0.5

2.2. Detector arm

The Detector arm (Da) subsystem or module is responsible for moving the Detector(D) apparatus in a required position in order to catch the resulted diffracted / scattered beam(spots) - X_d (d-diffracted) after coming from the sample.

Mainly, it must provide fast precision orientational motions and support for the D (80kg) and, other optics in both, the horizontal and vertical scattering processes, with set-up adjusting capability (500-1100mm). Specifically, it must materialize the two circles (2C) - δ(-195/15°) and γ(+135/-40°) with high accuracy (repeatability - 0.0005° / 0.0001° having ≤ 10 μrad wobble motion error, as Tab. 1 shown.

Therefore, a stiff manipulator arm based on two high precision motorized actuation units (Gonio 480-X3W1) orthogonally arranged each other, together with a stiff linear motion support manually actuated have been conceived, accordingly.

2.3. Deflector

The X-ray manipulation module called Deflector (Dc) is located upstream of the Dm. It should be able to deflect the incoming (X_i; i-incindent) beam with the highest precision towards the sample and to perform the necessary motion to form an incident conical shape. For this purpose, two mirrors / deflectors using Si(111) & Si(220) cristals working in air are used, respectively.

Each crystal should have a parasitic motion of its Bragg angle of no more than 5 μrad when turning (ρ) to full range (360°), Tab.1. Moreover, the position of the two crystals should remain aligned and within the Darwin width of the crystal (Ge 111 / 220 from 7 to 28keV is min. 18 μrad). Therefore, the max. parasitic tilt motion (Ry) has to be max. 1.8 μrad , and the total parasitic motion max. 5 μm.

Shortly, the Deflector(Dc) must materialize a) the orientation / alignment motions of crystals with high-precision and b) provide stable support to cope with the vibrations coming from underground. Especially, the support of these two crystals deflectors has to be very stiff.

Consequently, the whole deflector system was conceived as a combination of two parts: a) Double Cristal Deflector (DCD) and b) Positioning (Pd) devices, respectivelly.

DCD was conceived on three circles (3C) structure, perpendicular on each other: c1(ρ) ⊥ c2(φ) ⊥ θ (θ1 ∥ θ2; θ1 fixed) and designed on an improved Euler cradle (Ec) unit by using a very stiff guide (Gonio 440/INA/YRT, Gonio 430), two precision deflectors (Gonio 410) units and a transversal linear alignment stage (5101).

Positioning device (Pd) was designed from a PKM (hybrid) - parallel / serial device supporting the whole system above (DCD). It has the capability to be moved / aligned in four directions - 4dof (Tx,Ty,Tz,Ry), beeing mounted on a granite table (bridge) element. Thus, a PKM redundant (TRIPOD) structure based on three (3) stiff vertical actuators (Gonio 5103) has been choosen.

It is expected to provide 2dof (Tz,Ry) - alignments and precision stable motion (Tz=±10mm, Ry=±0.5 m°). In addition, Tx (60/-500mm) and Ty (+10/-10mm) translational motions are performed through standard and integrated linear guides.

2.4. Alignment Table

All above subsystems (or, modules) must be fixed on a stable support, having in addition the possibility to be roughly adjusted relative to the location and direction of the X-ray beam (Y, Z, Rx).

Therefore, a stiff Alignment table (At) positioning module based on a Parallel Kinematic Mechanism (PKM3), able to be moved (and, fixed) on two rails through adjustable feet (4) was conceived. It can provide the vertical and rotational displacements (Z==±25mm, Rx=±1.5°) with high precision (Res=0.5mm, ≤0.5m°) and the possibility to perpendicularly be moved/removed on the beam (Y=±50mm), respectively. In addition, the roughly adjustment(setup) along Z can be performed through feet(washers), too.

The Diffractometer will be supplied as separated pre-aligned modules and will be operated in an Experimental Hutch (EH). It includes the electronics (drivers and controllers) with easy Ethernet communication to standard computers (PC) networks, as well.

An operational time life of 30 years is expected with the actual designed solutions; much more suitable for the repairment, than the replacement.

The general and relative motion accuracy are to be done by several adjusting techniques (screws, eccentric pins, etc) included in the designs. The expected specific parameters of precision (with load) are included in Tab. 3.

Table 3 Precision parameters (Dm)

Component	Para (II) [μrad]	Perp (⊥) [μrad]	SoC [μm]
(δ,γ)	-	≤ 50	≤30
(θ,ω) (α,φ)	≤ 50 (δ) ≤ 50	≤ 50 -	≤30
ρ,θ(1,2)	-	≤ 50	-

3. Conclusions

The results of a work for developing a new Diffractometer (Dm) has been presented.

It includes the standard design (CAD) and challenging issues regarding the concept, functionality and precision for a liquid diffractometer (Dm) to work with the required parameters, by using modern scattering techniques, at liquid surfaces/ interfaces in a beam line from a modern synchrotron facility, having also the possibility to be used for a variety of other related tasks.

In its final design stage, the modular advanced Diffractometer it's a complex multi-axis (>20) diffraction machine (6Circles) based on a redesigned conventional structure (4C) with now full integrated Deflector (Dc) module and an interchangeable PKM device.

The new PKM (OCTOGLIDE) delivers stable and precise sample positioning (Sp) in the confined allowable space for both, the horizontal and vertical loading positions.

Is expected that by this, all the Dm's specifications have been fulfilled, and the first product (prototype) will be start and tested soon.

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