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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

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### 1. Introduction

During the past few years, applicative research 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 correlated phenomena between physical and chemical properties of functional oxide materials, such as those included in *semicon* nanostructures has been constantly increasing [4].

Several discoveries have examined nanomaterials (nanostructures) 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 highly 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 period 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 being used in synchrotron facilities, for rough manipulation (alignment base) and precision positioning





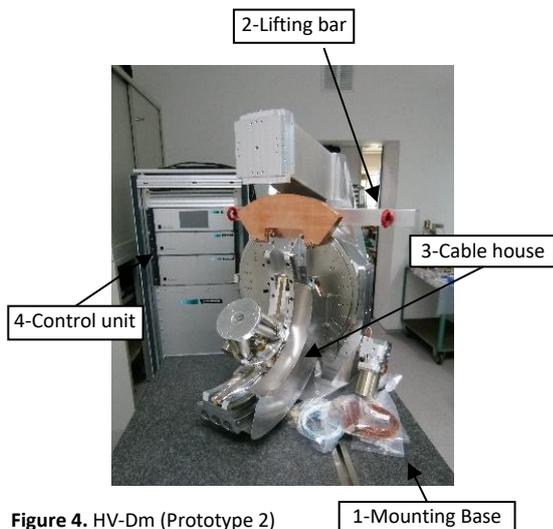
**Table 3** Design specifications (Gonio)

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  $\mu\text{m}$  maximal sphere for all full rotational motions and charged with loads. Tests were performed using calibration sphere ( $\phi 15$  mm, 25 nm sphericity) and sensitive dial gages (2  $\mu\text{m}$ ). As can be extract from the measurement protocol, the maximal individual errors (radial run-outs) occurred as the segment (and cables) is moving (e.g.,  $\varepsilon = -25\mu\text{m}$ ,  $\theta = -120^\circ$ ). The

maximal errors (axial run-out) for Da happened in vertical position ( $\varepsilon = 24\mu\text{m}$ ,  $\delta = 90^\circ$ ).

### 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.

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