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HIGH-PRECISION SYNCHROTRON KAPPA DIFFRACTOMETER

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Abstract

A new research product aiming to work in a 3rd generation synchrotron facility has been developed. Based on increased energy X-ray synchrotron radiation tool and well-known Kappa geometry, the product is expected that will investigate atomic and molecular structures of materials at nanoscale level using X-ray diffraction (XRD) technique. The Kappa Diffractometer (KDm) machine is maintaining the common structural principle of its family, but working with an extreme precision, which is far of the competition. The main body is consisting of a customized Kappa goniometer (KGm) device with vertical axis of rotation for high-precision sample (cryostat) manipulation, versatile detector arm (Da) for manipulating in horizontal plan different detectors (optics, slits, etc.) after X-ray beam is scattered and stable alignment base (Ab) for roughly adjusting the product towards the X-ray beam. In addition, a new XYZ cryo-carrier inside of the KGm is included for fine(submicron) sample adjustments. The kinematic, design and precision concepts applied, together with the obtained test results are all in detail presented.

INTRODUCTION

Synchrotron radiation is one of the most powerful investigative tools available today for exploring internal structure of the matter. Last generation (4th) synchrotrons are being currently on the way to be built and several other (3rdgeneration) took a modernization process. However, advanced investigations are requiring not only new modern techniques [1], but dedicated instruments adapted to the specificity of the applications, as well.

Korean Pohang Accelerator Laboratory (PAL) research facility is managing the 3th generation accelerator, which was under an upgradation process (2009-2012). In the actual configuration (PLS II) it is including a portfolio of 40 beamlines [2] from which an appreciable number are dedicated to X-ray diffraction (XRD). 1C beam line is managed by Institute of Basic Science (IBS) being currently under the development. After its completion, it will investigate the properties of advanced functional materials lattices dynamics using time-resolved scattering technique (TR-XRS).

A growing interest is seen today to discover new materials with improved quantum features. A research centre at IBS (CALDES) is focusing on investigating such properties based on untapped potential of low-dimensional electronic materials [3]. An international request to develop a specific diffractometer has been launched [4] and attracted several proposals. In one of them [5], the intention was to use a four-circle diffractometer with Kappa geometry (horizontal), to provides a better access and large Bragg diffraction angles for preferred crystallographic orientations. However, after the proposal was accepted, based on a more detail analysis of required precision and load - sample and specific instruments manipulation, it has been concluded that a new solution must be adopted to cope with all the specifications. The proposed diffractometer hoping to offer not only an improved manipulation capability, maintain at the same time the intrinsic advantages of the architecture, but an increased precision, as well.

The main features of the first product (prototype) are described below, including most important aspects related with kinematics, design and precision concepts.

DIFFRACTOMETER

Generally, diffractometers have been conceived till now based on two type of architectures- Eulerian (E) and Kappa (K), respectively. These are deriving from the way a sample is manipulated; specifically, the working principle chosen for the manipulation mechanisms. There are several well-known companies producing both of such machines (HUBER, NEWPORT, KOHZU).

Euler Diffractometer (EDm) is using a combination of three orthogonal gonio (G) stages, forming Euler goniometer (EGm) device. It delivers high precision positions, able to carry instruments with appreciable weight and size because of its intrinsic high stiffness of the mechanism (closed loop). However, it is providing a limited access of X-ray (incident, scattered) to the sample, and to operator (setup, maintenance). On contrary, Kappa diffractometers (KDm) are based on an open loop angular device to manipulate the sample, called Kappa goniometer (KGm). By this, the access at the sample is almost entirely free. However, the precision of the manipulated load and size are limited, because of intrinsic flexible (open loop) working principle of the mechanism. A good overview of Kappa diffractometer capabilities is given in [6].

Requirements

One of the express requirements for the new Kappa Diffractometer (KDm) was to manipulate a specific atmosphere & temperature-controlled instrument (cryostat) having a maximum - weight (20kg) and size (500mm) with highest possible accuracy (SoC<30μm). In addition, the orthogonality of the last rotation axis has to be less than a maximum value (20″). It should accommodate the use of several type of detectors - in-line and area, weighing a value of about (40kg) for related processes use. An overview of the most important motion parameters and their values for precision are included in Table 1.

Table 1: Motion Parameters (Sample)

Range (°/mm)	Acc. (″/μm)	Rep. (″/μm)	Res. (″/μm)
φ=κ=θ=±180	20	1	0.36
X,Y,Z=±5,5,3	2,2,2	1	<1

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Kinematics

From kinematic point of view, the new diffractometer is based on a classical four-circle (4C) geometry using Kappa concept with vertical axis of motion [7]. It can be seen as a well-established combination of two independent manipulators – sample(S) and detector(D), each of them being composed from joints and links, forming distinct kinematics chains (K_i, i=1,2), involving a basic set of rotative actuation called circles (C_i, i=1,..,4), Fig. 1.

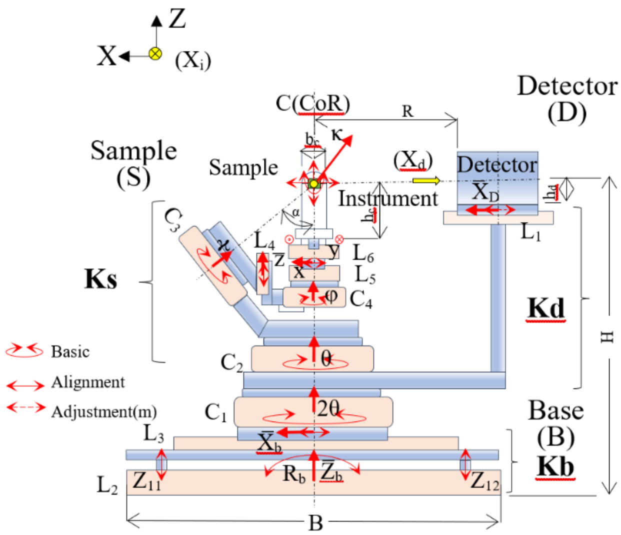

Figure 1: Kinematics (4C-1D3S).

All the experimental investigations are based on above corelated motions(positioning) relative to X-ray (fixed) beam, respecting the diffraction law (Bragg) and specific procedures.

The detector manipulator (D) is based on a simple kinematic chain (Kd) mechanism, consisting of an active rotational joint $C_1(2\theta)$ and an arm, supporting linear sliding guides (L1), accommodating the use of different detectors, covering a large region in the reciprocal space (time-resolved), catching the scattered X-rays. A detector is performing a plan-parallel circular motion (R=1000mm, Xd=±150mm). The corresponding (pseudo) vector of rotation (2θ) is vertical with positive direction upwards and null position when D is in the YZ plan (-Y). The angular motion range (±160°) has the same precision parameters as in Table 1.

The sample manipulator (S) is an open loop kinematic chain (Ks) mechanism, consisting of three actuation circles (C_i, i=2,..,4) linked together at a fixed angle (α=50°). C_2 (θ) is supporting the two others - $C_3(\kappa)$ and $C_4(\varphi)$; last one holding the sample. It performs spherical motions around a fixed point (C), called centre of rotation (CoR).

As the specific instrument (cryostat) is with an appreciable weight and size and the final precision was always related before with Z carrier stage source of errors, a new carrier mechanism has been included for fine adjustments, between the circles, instead of the classical one at the end (XYZ). By this solution, is providing not only an increased access to easily mount and operate (setups, maintenance) the afferent instruments, but is opening the way for an increased weight (load) manipulation. However, attention must be paid to the stiffness of the arm.

The nominal position of the mechanism is defined by $\theta_0=\kappa_0=\varphi_0=0°$ and $2\theta_0=0°$ angles. Then, θ and φ (pseudo) vectors coincide (support, direction), and κ lies in XZ plane (second quadrant); φ direction can be arbitrary chosen, often null coinciding with OX(-X) axis. Positive rotations for 2θ, θ and φ are consisting in moving X towards Y (right hand rule), and for κ to be always out(upper)side of horizontal plane (XY); K resultant orientation vector being upside, as well. All vectors must intersect in C point. Generally, the convention follows the basic rules included in [8].

Note: The OXYZ reference system is a right-handed set of orthogonal axes. It has OY axis against the incoming X-ray beam.

Design

A modular approach has been applied in the design process [9], consisting of adopting the detector, sample and base manipulation subsystems, as the main positioning modules (Pm_i, i=1,2,3), Fig. 2.

The core of sample module (Pm)s is a modified standard Kappa goniometer (KGm) TS70712. It is based on a combination of two active positioning units (Pu_i, i=1,2) called Goniometers (Gm) or simply gonio(G) linked together in an angular way, as described before. The first one is a precision gonio (G430/X2W2) able to carry the second one (G420/X2W2) and the linear in-parallel redundant actuated unit Z (Z1,Z2) located in-between; both gonios being integrated in the supporting arms (θa, κa) for increasing stiffness. This solution is coping not only an increased load manipulation but could eventually solve the arm deflection error compensation.

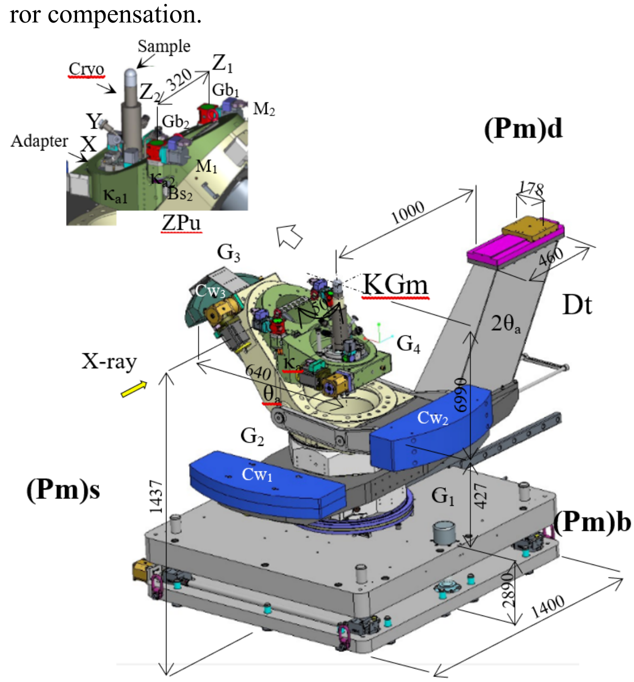

Figure 2: CAD layout (ZPu).

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The whole device is being carried by a precision (G440/X2W2) gonio for which a compact solution has been applied for its static balance (CW2-counterweight). On top (and, inside) of the last gonio, a cryostat (CS202SK) is being held through a dedicated interface. It is a relatively simple (reliable) and efficient (economical) instrument for low temperatures (4.2K ÷325K) experiments [10].

(Pm)d was built on precision gonio (G440/X2W1), having a very light (welded) arm, but with stiff structure. The detector is manually moved and fixed (dovetail slide).

The alignment base module (Pm)b was designed based on the existent table type (6204), providing stiff (1000kg) and short motions (Z, X, Rx) support for roughly alignment against the X-ray beam.

For the whole KDm design, different types of materials (e.g. steel, Al) has been carefully chosen as to be a compromise between own weight and stiffness

As precision was the main concern, modelling and simulations using Finite Element Analysis (FEA) have been performed iteratively to estimate the deflections and by this reducing (or, eliminating) them. Kappa arm being one of the critical components, simulations have been performed for both parts (κa1,κa2) in several critical configurations (0°-120°). Figure 3 is showing von Misses stress distribution for second part(a2) in nominal position (0°), with maximal value $(\varepsilon\kappa)_2$max=12.5 µm occurring on the upper site of the surface adjacent to the rails. The variation across of the rail width (l) is given in a diagram (b).

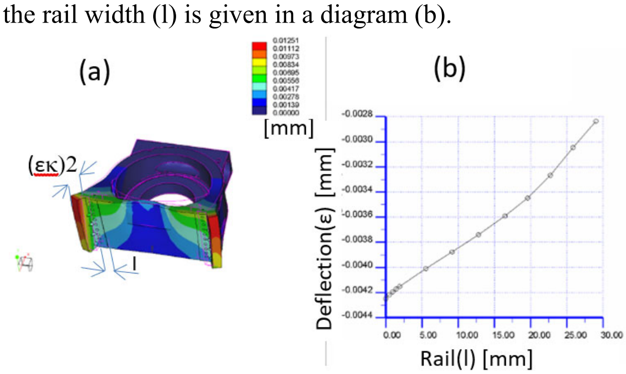

Figure 3: κarm2 deflection-a) distribution & b) variation.

Prototype

A first product based on above design considerations has been manufactured. Attention was given in the machining process for obtaining high quality of functional surfaces, respecting geometrical tolerances, as those in contact with important components (e.g. gonio, rails, etc). Then, in the assembly process care has been taken to mounting the components with highest precision, from earlier to the final stage, by performing fine adjustments. The control of the system with eleven (11) motorized axes has been realized using closed loop method (motors, gears, mechanism, encoder) based on a dedicated programable hardware (SMC9300) with Ethernet interface and a PC with a pre-installed operation system (LINUX) and specific software (SPEC/C-PLOT).

The prototype was tested at factory site from functional and precision point of view. A factory acceptance test (FAT) report was issued before the installation at the indicated premise. An overview of the set-up(a) and obtained results(b) are given in Fig. 4. The main source of runout errors values (negative) is coming along Z axis (14µm) when rotation angle is between (190°-210°); the smallest ones (positive) encountered along X axis (3µm) is corresponding to 180°. As errors are with substantially increased values (negative) along Y axis around (150°-240°) interval, we suspect that the effect is coming from a combined deformation result of components under an increased moment of own weight(s), especially in the maximum extension (180°). Runout errors for theta (ε_θ) and 2theta ($\varepsilon_{2\theta}$) rotations have been around one (ε_θ=±1µm) and few micrometres ($\varepsilon_{2\theta}$=±2.5µm) and, for phi (ε_φ) below 3µm (ε_φ<3µm). The above values conducted us to consider a maximal value for the (sample) sphere of confusion (SoC=15µm) which is well behind the specification (SoC=30µm). In addition, precision measurements have been performed in relation with the geometry of axes. The orthogonality of (φ) vector was inside of eight arcsec interval ($\varepsilon_{\varphi\perp}$=±8″, $\varepsilon_{(\varphi\perp)min}$=-8″/90°, $\varepsilon_{(\varphi\perp)max}$=8″/270°) being below the requested value ($\varepsilon_{\varphi\perp}$=20″). All values included the motion of a dummy cryostat (10kg) and the measurements have been performed with an electronic dial gage instrument (TT60/TESA/1µm) and a calibration ball (Ø14mm). In addition, an electronic autocollimator (ELCOMAT 3000 / MÖLLER/ 0.01″) with plan-parallel mirror was used.

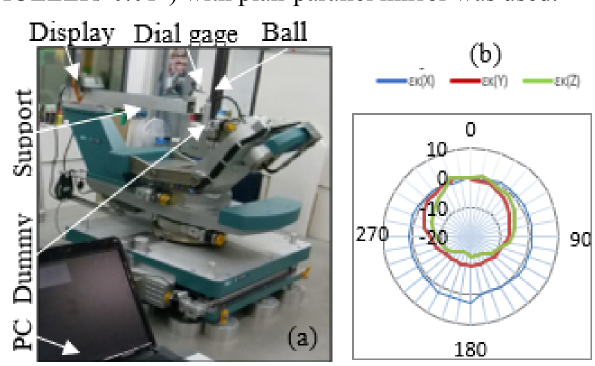

Figure 4: Measurements a) set-up and b) errors (εXmax=4µm, εYmax=10µm, εZmax=15µm, κ=±180°).

CONCLUSION

A new kappa diffractometer (KDm) with improved precision capabilities for X-ray scattering investigations (XRS) of materials, to work in a 3rd generation upgraded synchrotron has been developed. Built on a classical four-circle (4C) concept, it has resulted as a fine balance between robustness and manipulability, including an innovative concept for fine alignments (Z) and compact (integrated) design solutions (Gonio, CW) for Kapa goniometer (KGm). Mainly, it offers an improved solution to manipulate instruments specific to cryo-conditions (4K) with appreciable load (20kg) and size (500mm) with highest available precision (SoC=15µm), using several types of detectors (1D/2D). Due to its modular design is prone to be adapted at other similar applications.

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