IRMA-2 at SOLEIL: a set-up for magnetic and coherent scattering of polarized soft x-rays

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Abstract. We have designed, built and tested a new instrument for soft x-ray scattering experiments. IRMA-2 is a UHV set-up for elastic and coherent scattering experiments developed at the SEXTANTS beamline of the SOLEIL synchrotron. Applications will be in the field of solid state physics, with emphasis on the investigation of the magnetic properties of artificially structured materials.

1. Introduction
Resonant scattering of polarized soft x-rays is a field that encompasses several techniques, notably inelastic scattering, Raman spectroscopy, reflectivity, diffraction and coherent scattering; the SEXTANTS beamline [1] of the SOLEIL synchrotron is among the beamlines [2] that comprehensively implement the ensemble of these techniques. The IRMA-2 instrument is a new UHV set-up that, although mobile, has been developed for the SEXTANTS beamline specifically. It is intended for elastic and coherent resonant x-ray scattering experiments, with focus on the investigation of the magnetic properties of nanostructured materials. In this paper we report on the design, construction, technical performance and first results of the instrument.

2. IRMA-2 vessel and goniometer
2.1. Goniometer
The core element of the IRMA-2 reflectometer is a two-circle UHV goniometer defining the sample and detector coaxial rotations. The goniometer axis (X direction) is horizontal and normal to the incident x-ray beam (Y) and to the vertical scattering plane (YZ). The goniometer design was presented before [3]. It is composed of two stainless-steel hollow shafts that can rotate independently, defining the angular position of the sample (inner shaft) and of the detector; the sample shaft can also slide along the rotation axis. Two stainless-steel sleeve-bearings placed between the shafts maintain them coaxial. All parts, whose working surfaces are ground to a 0.8 µm roughness and a 5 µm
mechanical tolerance, underwent an anti-sticking treatment to avoid seizing in vacuum [4]. The rotation of each shaft about their common axis is actuated by rotary-stages, driven by high-precision encoded stepper-motors, which are bolted to a CF160 UHV flange that serves to connect the goniometer to the vacuum chamber. Differentially-pumped feedthroughs maintain vacuum during rotations. The combined accuracy of the angular positioning of sample and detector is within ±0.005° over the 0°-160° range of the detector rotation.

2.2. Sample holder
In addition to the rotation $R_X$ defined by the goniometer, three orthogonal translations $T_{X'}$, $T_{Y'}$ and $T_{Z'}$ are provided for aligning the sample ($X'=X$ is the rotation axis, $Y'$ lies within to the sample surface and $Z'$ is normal to it). An optional azimuthal rotation $R_z'$ can be implemented via a piezoelectric positioner. $T_X$ is achieved by translating ±35 mm the entire sample shaft, using a motorized linear manipulator. $T_Y$ and $T_Z$ rely on in-vacuum stepper motors and translation tables, mounted between the sample shaft and the sample holder, capable of positioning the sample to within ±300 nm over a range of 26 mm. Custom-designed sample holders can be attached to the translation tables according to specific needs. The default one features an electromagnet (±500 Oe static and ±1500 Oe pulsed field along two orthogonal directions within the sample surface) and temperature control (either via a thermoelectric cooler or by liquid-N$_2$ circulation + Cu-braid).

2.3. Detector holder
The detector holder can host up to six elements (diodes, CEM, YAG imager, etc.) at a radial distance of either 150 mm or 200 mm from the rotation axis. Its position can be adjusted along the X direction. We currently employ a 10×10 mm$^2$ Si diode-detector and GaAs diodes with an active surface of 4×4 mm$^2$ and 2×2 mm$^2$. The diodes and the CEM can be fitted with slits of 0.1 mm to 1 mm vertical aperture (vertical accepted angle between 0.03° and 0.3°).

![Figure 1. Sketch (a-c) and pictures (d-f) of the vacuum chamber. Pictures show details of the wide-access door (d), of the goniometer (e) and of the CCD detector (f).](image)

2.4. Vacuum chamber
The vessel hosting the goniometer is a 600 mm diameter UHV chamber [5], designed to fit at the working position B of the SEXTANTS beamline [2]. A 520 mm door (Fig. 1d) guarantees an easy access for implementing specific sample and detector holders on users' request. The chamber can be aligned by three translations and two rotations ($R_X$, $R_Z$), with $T_X$, $T_Z$ and $R_X$ movements motorized. The ±5° $R_X$ rotation takes place around the same axis of the goniometer. Seven ports (four CF100 and three CF150) are located within the YZ scattering plane, with their axes pointing at the center of the chamber; they are available for hosting fixed-scattering-angle (eventually 2D) detectors that wouldn't fit within the vessel (Fig. 1f). Since the entire chamber can rotate around the goniometer axis, these
ports can cover scattering angles from -15° to +160° continuously. In addition to service flanges (wiring, fluids, etc.), CF40 and CF63 flanges are available for inserting beam conditioning elements (pin-holes, filters, beam-stops, etc.) along the path of the incoming and of the outgoing (transmitted or scattered) beams. The chamber design is such that the XZ vertical plane containing the goniometer axis is a mirror plane; this allows the incoming beam to enter from either one of the CF150 flanges along the Y axis (see Fig. 1a,c), according to specific space constraints.

3. Coherent scattering set-up

Within the IRMA-2 project, we have developed two set-ups for coherent scattering and X-ray holography, both offering the possibility of a separate alignment of the sample and of the holographic mask with respect to the X-ray beam [6]. One set-up consists of high precision tables driven by stepper motors [7] and of piezoelectric positioners [8], all assembled on a single plate that fits within the vacuum chamber (Fig. 2a). The sample holder can be aligned by three translations (T_X, T_Y, T_Z), the holographic mask, the coherence pin-hole, the guard-hole (limiting the illuminated area) and the diagnostic diode by two translations (T_X, T_Z) each. This set-up is for measuring in transmission mode, using a back-illuminated CCD camera [9] mounted on a 300 mm travel manipulator for adjusting its distance from the sample (Fig. 1f). An Al-coated parylene filter and a beamstop are placed in front the CCD in order to block visible light and the intense transmitted x-ray beam, respectively. The standard sample holder features an electromagnet (500 Oe static and 1500 Oe pulsed field, oriented at choice) and liquid-nitrogen + Cu-braid cooling (sample T_min ≈ 120 K).

The second coherent scattering set-up (Fig. 2b) consists of a small plate that can replace the goniometer sample-holder and can be aligned with respect to the x-ray beam using all the degrees of freedom described in 2.2. The coherence-mask is fixed to the base-plate, while the sample holder is mounted on three linear and, optionally, two rotary piezoelectric positioners [8] for an independent and precise alignment with respect to the mask. As the previous one, this set-up can be used in transmission geometry; being mounted on the sample shaft of the goniometer, it is also readily available for coherent scattering measurements in reflection geometry, with the 2D detector placed on one of the flanges within the scattering plane.

4. Results

![Figure 2. Pictures of the coherent scattering set-ups, mounted on the optical plate (a) and on the goniometer sample arm (b). In (b), main elements and movements are listed.](image)

![Figure 3. (a) specular reflectivity from a (Co_{1 nm} / Gd_{0.5 nm})_{60} multilayer, using 777eV circularly polarized x-rays. (b) zoom showing the magnetization dependent phase of the oscillations.](image)
Both reflectivity and coherent scattering experiments [6] were performed at SOLEIL using IRMA-2. Fig. 3 shows specular reflectivity from a Co/Gd multilayer measured at the Co-2p resonance using circularly polarized x-rays. At a core resonance, the optical index depends on the relative orientation between local magnetization and photon helicity; therefore, in Fig. 3 both the scattered intensity (a), spanning 5 decades over the 0-40° angular range, and the phase of the oscillations (b) vary strongly with the photon helicity. We characterized the degree of transverse coherence at the sample position using a test mask (Fig. 4a) consisting of eleven 200 nm diameter holes spaced in such a way that cross-correlation images (Fig. 4c) do not overlap [10]. Scattering diagrams were collected as a function of the beamline angular acceptance, defined the 4-jaw aperture placed 10 m downstream of the undulator source, without implementing any coherence pinhole. Fig. 4d shows data obtained with full vertical and horizontal acceptance of the beamline. The two-dimensional Fourier transform (2D-FT) shows cross-correlations up to ~8 µm and ~15 µm in the horizontal and vertical directions, respectively of (Fig. 4e); this asymmetry is related to the anisotropy of the source. Limiting the angular acceptance to 40 µrad in both directions produces a much sharper diagram (Fig. 4f) and better defined cross-correlation points in the 2D-FT (Fig. 4g), which extend beyond 25 µm in both directions, indicating a larger and more symmetric transverse coherence length.

![Figure 4. Sketch of the mask used for coherence tests (a). Calculated diffraction diagram (b) and corresponding 2D-FT (c) are compared with experimental results obtained at 778 eV. The vertical and horizontal beamline angular acceptance values are 150 µrad in (d)-(e) and 40 µrad in (f)-(g). The same scale of greys apply to (d)-(f) and to (e)-(g).](image)

5. Conclusion
We presented the main characteristics of the IRMA-2 scattering chamber, which is now available to users of the SEXTANTS beamline for reflectivity and diffraction measurements, as well as for coherent scattering and x-ray holography experiments.

References
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