Status of the hard X-ray microprobe beamline ID22 of the European Synchrotron Radiation Facility

Gema Martínez-Criado, Rémi Tucoulou, Peter Cloetens, Pierre Bleuet, Sylvain Bohic, Jean Cauzid, Isabelle Kieffer, Ewelina Kosior, Sylvain Labouré, Sylvain Petitgirard, Alexander Rack, Juan Angel Sans, Jaime Segura-Ruiz, Heikki Suhonen, Jean Susini and Julie Villanova

J. Synchrotron Rad. (2012). 19, 10–18

Copyright © International Union of Crystallography

Author(s) of this paper may load this reprint on their own web site or institutional repository provided that this cover page is retained. Republication of this article or its storage in electronic databases other than as specified above is not permitted without prior permission in writing from the IUCr.

For further information see http://journals.iucr.org/services/authorrights.html

Synchrotron radiation research is rapidly expanding with many new sources of radiation being created globally. Synchrotron radiation plays a leading role in pure science and in emerging technologies. The Journal of Synchrotron Radiation provides comprehensive coverage of the entire field of synchrotron radiation research including instrumentation, theory, computing and scientific applications in areas such as biology, nanoscience and materials science. Rapid publication ensures an up-to-date information resource for scientists and engineers in the field.

Crystallography Journals Online is available from journals.iucr.org
Status of the hard X-ray microprobe beamline ID22 of the European Synchrotron Radiation Facility

Gema Martínez-Criado,* Rémi Tucoulou, Peter Cloetens, Pierre Bleuet, Sylvain Bohic, Jean Cauzid, Isabelle Kieffer, Ewelina Kosior, Sylvain Labouré, Sylvain Petitgirard, Alexander Rack, Juan Angel Sans, Jaime Segura-Ruiz, Heikki Suhonen, Jean Susini and Julie Villanova

European Synchrotron Radiation Facility, Experiments Division, 38043 Grenoble, France.
E-mail: gmartine@esrf.fr

The ESRF synchrotron beamline ID22, dedicated to hard X-ray microanalysis and consisting of the combination of X-ray fluorescence, X-ray absorption spectroscopy, diffraction and 2D/3D X-ray imaging techniques, is one of the most versatile instruments in hard X-ray microscopy science. This paper describes the present beamline characteristics, recent technical developments, as well as a few scientific examples from recent years of the beamline operation. The upgrade plans to adapt the beamline to the growing needs of the user community are briefly discussed.

Keywords: X-ray microprobe; X-ray nanoprobe; X-ray fluorescence; microspectroscopy.

1. Introduction

Among the 40 beamlines in operation at the European Synchrotron Radiation Facility, ID22 is fully dedicated to hard X-ray microanalysis consisting of the combination of X-ray fluorescence (XRF), X-ray absorption spectroscopy (XAS), X-ray diffraction (XRD) and X-ray imaging (XRI) techniques in the hard multi-keV X-ray regime (Somogyi et al., 2005). The beamline is composed of two experimental stations, which permit studies in several research fields such as medicine, biology, earth and planetary sciences, environmental science, archaeometry and materials science. These disciplines seek non-destructive investigation of the spatial distribution, concentration and speciation of trace elements to be correlated to the morphology and crystallographic orientations at the (sub)micrometre levels. Both stations share a common instrumental set-up: an X-ray focusing device, a high-precision stage to raster the sample on the beam, a visible-light microscope (VLM) to visualize the regions of interest of the samples, as well as some detection schemes and 2D/3D XRI approaches.

After several years refining the analytical methods, hard X-ray focusing devices, positioning stages and detection schemes, two hutchies are clearly defined today by their spatial resolution: EH1 devoted to microanalysis and EH2, also known as ID22 nano-imaging station (ID22NI), exclusively used for nanoanalysis (see Table 1). The stations offer a large variety of well established approaches:

(i) EH1: scanning-XRF and XRF-tomography, micro-XAS and XANES imaging, X-ray excited optical luminescence, linear dichroism, scanning XRD, absorption/phase contrast tomography, and diffraction-tomography.

(ii) EH2-ID22NI: scanning-XRF and XRD, XRF- and XRD-tomography, X-ray projection microscopy, full-field magnified tomography, and coherent scanning X-ray diffraction.

The flexible design, long working distances and high penetration powers also allow the integration and development of different controlled sample environments in EH1. A few examples include anvil cells, microfurnace, He chamber, cryostreams as well as other environments routinely integrated in the beamline (LINKAM HSF91 stage for heating and freezing applications, He mini-cryostat, etc.). An additional development to be shared between both stations is the confocal XRF mode using a polycapillary half-lens pioneered by the MiTAC group (Vincze et al., 2004). In the next section the major technical upgrades recently performed at ID22 are summarized.

2. ID22 instrumentation

2.1. X-ray source

Currently the high-β straight section of ID22 is equipped with two insertion devices: an in-vacuum U23 and a revolver U35/U19. Table 2 summarizes the main parameters for both undulators. The photon flux emitted by both devices is presented in Fig. 1, calculated at 30 m from the source through a 0.5 mm × 0.5 mm pinhole (the insertion device U42 is depicted for reference purposes only). The electron beam characteristics included a current of 200 mA, an energy of 6 GeV and a relative energy spread of 0.001. The vertical (horizontal) emittance, β values and dispersion are 39 pm (3.9 nm), 3 m (37.2 m) and zero (0.137 m), respectively. The
A revolver device was chosen to give maximum photon output in a very narrow energy range centred on 17.5 keV that is the principal working energy at ID22NI. It has at least the same performance as a conventional U42 undulator in terms of gap reproducibility and speed. The device is equipped with a tunable undulator (U35) and a dedicated (optimized) low-K undulator specific to the needs of the beamline (U19). The switching between one undulator to the other takes about 2 min including the opening of the gap to 250 mm, the rotation of the girders, and the gap closure with the revolver undulator to 11 mm. It is almost transparent to the user. The availability of two interchangeable magnetic structures (35 and 19 mm period) combined with the U23 in-vacuum undulator allows for better optimization of the X-ray photon flux for various energy ranges, overcoming the old configuration based on a U42 undulator, which created an energy gap between 15 and 18 keV.

### 2.2. End-station EH1

#### 2.2.1. Microprobe set-up

An overview of the experimental arrangements of ID22 end-stations is depicted in Fig. 2. The end-station EH1 has two parts: the full-field tomography table and the microprobe set-up. In order to explore the merits of high energy (up to 65 keV), a special pair of crossed mirrors in Kirkpatrick–Baez (KB) configuration is installed at the microprobe set-up (Borchert et al., 2010). It comprises two elliptically shaped Si mirrors, a 170 mm-long mirror focusing at a distance of 390 mm from the centre of the mirror in the vertical direction, and a 92 mm-long mirror with a 190 mm focusing distance in the horizontal direction. They are coated with graded multilayers (B4C/[W/B4C]40/Cr), playing both monochromatization and focusing roles. Four actuators (μ-Focus picomotors) bend the flat polished mirrors (CoastLine Optics) into the elliptical figures required for imaging the X-ray source. Both arms of each bender are equipped with linear encoders (Mercury 3500). This design provides reflectivity of 96% at 65 keV and 75% at 8 keV. Thus, we can exploit both pink and monochromatic beam operations based on

![Figure 1](image1.png)

**Figure 1**

The output spectra of the undulators of ID22 shown as photons s⁻¹ (0.1% bandwidth) through a 0.5 mm (H) × 0.5 mm (V) pinhole at 30 m (equivalent to the position and normal slit gaps of the primary slits) of the centre of the undulator. U42 is shown for reference purposes only.

<table>
<thead>
<tr>
<th>Table 1</th>
<th>Main characteristics of ID22 end-stations.</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>EH1</td>
</tr>
<tr>
<td>Spatial resolution (μm)</td>
<td>1 × 4</td>
</tr>
<tr>
<td>Focusing optics</td>
<td>KB mirrors</td>
</tr>
<tr>
<td>Maximum flux (photons s⁻¹)</td>
<td>5 × 10¹¹</td>
</tr>
<tr>
<td>Energy range</td>
<td>6.5–65 keV</td>
</tr>
<tr>
<td>Techniques</td>
<td>XRF, XAS, XRD</td>
</tr>
<tr>
<td></td>
<td>XRI-2D/3D</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Table 2</th>
<th>Summary of the relevant parameters of the revolver undulator U35/19 and the in-vacuum undulator U23.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Insertion device</td>
<td>U35/19</td>
</tr>
<tr>
<td>Period (mm)</td>
<td>35/19</td>
</tr>
<tr>
<td>Length (mm)</td>
<td>1.6</td>
</tr>
<tr>
<td>Magnet material</td>
<td>NdFeB</td>
</tr>
<tr>
<td>Minimum gap (mm)</td>
<td>11</td>
</tr>
<tr>
<td>Peak field at minimum gap</td>
<td>0.74/0.32</td>
</tr>
<tr>
<td>Power density at 30 m, minimum gap</td>
<td>97/82</td>
</tr>
<tr>
<td></td>
<td>I = 200 mA (T)</td>
</tr>
</tbody>
</table>

![Figure 2](image2.png)

**Figure 2**

Overview of the experimental arrangements of ID22 end-stations EH1 and ID22NI. The upper part illustrates the EH1 end-station: on the right is the full-field tomography set-up, and on the left is the microprobe. The lower part depicts the ID22NI end-station. The direction of the X-ray beam is also indicated. KB represents the Kirkpatrick–Baez mirrors, 13-ED the 13-element detector, SDD the Si drift detector, and VLM the visible light microscope.
Bragg diffraction and total external reflection modes, respectively. The pink beam approach uses the standard multilayer configuration to increase beam divergence (numerical aperture), producing a very high photon flux \(10^{12} \text{photons s}^{-1}\). The second strategy optimized for spectroscopic acquisitions relies on grazing incidence to provide a monochromatic beam flux of about \(5 \times 10^{10} \text{photons s}^{-1}\). In the high-energy range, the beamline is well equipped. First, the in-vacuum undulator provides a high photon flux, and, second, the Kozhu double-crystal monochromator, which can cover an angular range from 2.6 to 32.5° [i.e. 3.7 to 43.5 keV energy range for Si(111), and 7.1 to 83.5 keV for Si(311)]. The resulting spot size at the focal plane of about 1 μm × 4 μm (V × H) is shown in the upper part of Fig. 3.

2.2.2. Full-field tomography set-up. Fig. 4 shows the full-field micro-tomography set-up, which is located upstream from the focusing optics on EH1 (Weitkamp et al., 1999). This retractable stage provides complementary information and is often used preliminarily to investigate specimens (e.g. to select a specific region of interest within a larger object and/or to select the most representative of a number of samples). The full set-up is mounted on a table and a high-precision linear stage that guarantees not only repeatability but also an easy and quick switch with the microprobe (Artioli et al., 2010). The system includes the high-precision air-bearing rotation stage UPR-160/Air (miCos GmbH), the tilt, the vertical translation stage, and the CCD camera ESRF standard FReLoN 2k 14-bit (Labiche et al., 2007). The readout speed of the FReLoN detector depends drastically on the operation mode (full-frame, frame transfer, kinetics pipeline), binning, dynamic range and the region of interest. The most frequently employed mode gives about 100 ms readout time without region-of-interest or binning (Labiche et al., 2007). Similarly, the spatial resolution depends on the scintillator screen (Martin et al., 2009) and the numerical aperture of the objective, as well as the effective pixel size used. Frequently, it is adapted together with the desired field of view and can reach up to the submicrometre range. For the reconstruction of the tomographic images the filtered-backprojection algorithm is used via the ESRF software package PyHST (http://www.esrf.eu/UsersAndScience/Experiments/TBS/SciSoft/).

2.3. End-station EH2-ID22NI

2.3.1. Nanoprobe set-up. Located at 64 m from the source, the nanofocusing optics consist of two graded multilayer coated surfaces mounted in crossed KB configuration (Morawe et al., 2006). It is composed of a 112 mm-long mirror focusing at a distance of 180 mm from the centre of the mirror in the vertical direction and a 76 mm-long mirror with an 83 mm focusing distance in the horizontal direction. Four actuators (μ-Focus picomotors) bend the flat polished mirrors (CoastLine Optics) into the elliptical figures required for imaging the X-ray source. Both arms of each bender are equipped with linear encoders (Mercury 3500). This design provides reflectivity of 73% at 17 keV and 74% at 8 keV. The resulting spot size at the focal plane of about 60 nm × 60 nm (V × H) is shown in the lower part of Fig. 3. The vertical mirror images the undulator source (≈25 μm FWHM), whereas a virtual source is created in the horizontal direction using the high-heat-load slits (depending on the spatial resolution and photon flux required by the experiment, from 10...
Table 3
Summary of the relevant characteristics of the KB systems.

<table>
<thead>
<tr>
<th>KB system</th>
<th>EH1</th>
<th>EH2-ID22N</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lengths, V × H (mm)</td>
<td>170 × 92</td>
<td>112 × 76</td>
</tr>
<tr>
<td>Material</td>
<td>Si</td>
<td>Si</td>
</tr>
<tr>
<td>Coating</td>
<td>B₄C/[W/B₄C]₄₀/Cr/Si</td>
<td>B₄C/[W/B₄C]₂₅/Cr/Si</td>
</tr>
<tr>
<td>Source distance, p (m)</td>
<td>41</td>
<td>64</td>
</tr>
<tr>
<td>Focal lengths, V × H, q (m)</td>
<td>0.390 × 0.190</td>
<td>0.180 × 0.083</td>
</tr>
<tr>
<td>Incidence angles</td>
<td>2.5 × 3.5 at 65 keV</td>
<td>8.1 × 8.2 at 17 keV</td>
</tr>
<tr>
<td>V × H (mrad)</td>
<td>10.7 × 15.1 at 15 keV</td>
<td>4.8 × 4.8 at 29 keV</td>
</tr>
<tr>
<td>Spot size (μm)</td>
<td>1 × 4</td>
<td>0.060 × 0.060</td>
</tr>
</tbody>
</table>

up to 25 μm). The multilayer mirrors play both the role of focusing device and monochromator, resulting in a very high flux of about 5 × 10¹² photons s⁻¹ and medium monochromaticity of ΔE/E ≈ 10⁻². Invar, as material of choice for the benders, has improved the thermal stability (Tucoulou et al., 2008) and, in particular, the stability of the incident angles and curvature of the elliptically shaped mirrors. A complete description of the nanofocusing optics and nano-imaging station can be found elsewhere (Barrett et al., 2011; Hignette et al., 2007; Zhang et al., 2010; Cloetens et al., 2012).

The main characteristics of previous focusing systems are listed in Table 3.

2.3.2. Polycapillary optics. Polycapillary optics in confocal detection geometry can be used as a spatial filter for all applications in which background radiation, from areas not in the region of interest, interferes with the signal under study. XOS monolithic polycapillary optics optimized to a working distance of 2.5 mm and a cut-off energy of 15 keV is available at the beamline with a transmission efficiency of about 25% at 15 keV. Thus, the spontaneous radiation background is practically eliminated from the spectrum and therefore the detection sensitivity and accuracy is greatly improved. Also, buried structures can be studied by depth-sensitive X-ray absorption spectroscopy in fluorescence detection mode at the micrometre scale. In summary, these lenses can be used in our applications in which background radiation, from areas not in the region of interest, interferes with the signal under study. XRD positioning within the micrometre length scale (MICOS and Huber motors of high repeatability in the micrometre range, with also long travel and submicrometre resolutions). XRD acquisitions in transmission configuration are also suitable over the same high pressure–temperature range.

2.4. Sample environments

2.4.1. HP and HT diamond anvil cell. Within the framework of a close collaboration with the Laboratoire des Sciences de la Terre (ENS-Lyon, France), a diamond anvil cell dedicated to XRF analysis under high pressure and high temperature was drafted, built and tested at EH1 (Petitgirard et al., 2009), allowing in situ geochemical studies of heavy elements, rare earth elements (REE), and first transition metals at p.p.m. concentration levels. The designed system enables XRF detection at 90° from the incident beam using the thermally isolated 13-element Si(Li) solid-state detector located 50 mm from the sample position. Elements like Rb, Sr, Y and Zr with concentrations as low as 50 p.p.m. were detected with the cell operating at 5.6 GPa and 1273 K. Its vacuum chamber (10⁻¹² mbar) presents an optimized shielding and collection geometry that significantly reduces the background radiation (Fig. 5). As a result, for the above-mentioned elements, minimum detection limits of about 0.3 p.p.m. were estimated using such a set-up (Petitgirard et al., 2009). In order to properly handle its 15 kg weight, special translation and rotation stages are incorporated, allowing a precise and robust sample change time (60 min), on the other hand, is determined by the long thermal response to warm the system up. Finally, the choice of the window material depends on the wavelength and intensity of radiation, and whether polarization is required. The mini-cryostat not only allows substantial access but also reduces X-ray scattering by eliminating air path (very important for XRF). In addition, electrical contacts are available when transport- and/or electric-field-dependent studies are required.

2.4.3. Linkam stage. Commercially available heating–freezing stages also provide accurate and stable temperatures.
To operate in the 77–873 K range, a HSF91 stage (Linkam Scientific Instruments) compatible with our microprobe set-up is available. The scheme is optimized for vertical mounting and has high temperature stability (<0.1 K). With a compact and versatile design for easy mounting, it is supplied with a thermal jacket for tighter control of the sample environment (kapton or mica windows). The pure silver heating element has even a transverse aperture to accept a quartz capillary loaded with sample. This guarantees the sample is heated from all sides ensuring temperature homogeneity. For operation below room temperature, there is an automated cooling pump with 2 l dewar and 80 cm tube that tolerates a minimum stage temperature of 173 K. The system includes a stand-alone T95-LinkPad system controller with data sampling of 20 times per second. Heating rates can reach up to 150 K min⁻¹. The controller has RS232 connectivity control and programmable outputs for synchronization purposes with our beamline devices.

2.5. Detection schemes

2.5.1. 13-element detector. New requirements in terms of detection limits and acquisition rates fostered the installation and commissioning of a liquid-nitrogen-cooled multi-element Si(Li) detector (Gresham Scientific Instruments, UK) (Letard et al., 2006). Thirteen Si(Li) crystals mounted on a spherical holder form a close-packed array, each element being equidistant from the centre of the sphere. The collimated active area of each crystal is 50 mm². It provides a large total active surface (650 mm²) in optimized compactness (95 mm diameter) without any observable cross-talk effect. The thickness of the crystals is 3.5 mm which preserves the efficiency over the 8–20 keV energy range. The efficiency falls off above 25 keV, with 60% at 30 keV. Each crystal is individually protected by a 12 μm-thick Moxtek DuraBeryllium vacuum window. The digital signal-processing system was manufactured by X-ray Instrumentation Associates (XIA, CA, USA). It is made of four-channel Digital X-ray Processor XMAP modules, designed specifically for quick X-ray mapping (continuous scans). The theoretical maximum throughput is 10⁶ counts s⁻¹ channel⁻¹. However, detection dynamics are significantly reduced by the detector linearity as well as scattering effects. The peaking time can be set between 0.1 and 100 μs. In a high-counting-rate configuration (1 μs peaking time), the linearity measurements showed less than 80 kcounts s⁻¹ for a dead-time of about 30% (much lower for a low counting rate, 12 μs peaking time). External triggering can be used for synchronization with other processes such as energy scans or sample motions. The average energy resolution is 150 eV at 5.9 keV (for a peaking time of 12 μs and 1000 counts s⁻¹). The detection limits (for 10 s integration time) are below 0.1 p.p.m. for elements heavier than Mn (Letard et al., 2006).

2.5.2. Silicon drift detectors. Another alternative detection often used in EH1 is the silicon drift detector (SDD) technology. The use of the 13-element detector has been proved to be efficient in many cases (e.g. experiments requiring high energy resolution or elemental traces); however, often the relatively low photon count rate of such Si(Li) detectors limits the acquisitions (e.g. in XRF tomography). Furthermore, the combination of the high photon flux (>10¹⁵ photons s⁻¹ in the focal spot) and a large variety of sample thicknesses and matrices makes scattering radiation frequently one of the saturation sources. In that context, the complementary SDD technology offers not only lower detection limits and photon count rates at the expense of a slightly decreased energy resolution (150 eV) but also compactness owing to the absence of liquid-nitrogen cooling. In consequence, based on the XIA electronics, two SDDs (Vortex-EX, SII Nano-Technology) are available. The 50 mm² single-element SDD produced from high-purity silicon using state-of-the-art CMOS production technology operates with thermoelectric cooling. The drift structure ensures very low capacitance and low noise. In principle, at a peaking time of 0.25 μs, output count rates up to 600 kcounts s⁻¹ are achievable. The real count rate measured with 1 μs peaking time is about 175 kcounts s⁻¹.

2.5.3. FReLoN camera for X-ray diffraction. For (powder) diffraction experiments, commonly a large field-of-view camera with low resolution compared with XRF but high quantum efficiency is required. Accordingly, the taper version of the ESRF FReLoN camera (Labiche et al., 2007) is used at the beamline. It consists of a FReLoN F_K4320T (Kodak) equipped with 3.3/1 demagnifying fibre optics taper hardly bonded to the CCD chip (46 μm effective pixel size, 94 mm × 94 mm field of view, sensitivity 1 a.d.u. per incident 20 keV X-ray photon, 0.5 DQE at 20 keV). A 50 μm-thick Gadox powder scintillator screen converts the X-rays into visible-light photons. The use of a Kodak chip offers a high sensitivity of about 3.9 a.d.u. per incident 20 keV X-ray photon and a 0.6 DQE at 20 keV. A microphotodiode is also integrated in the beamstop to record simultaneously the transmitted intensity.

3. Examples of recent scientific applications

The beamline’s potential for simultaneous trace-element detection and mapping, quantitative fluorescence analysis, chemical state specificity and structural probe make it ideal for a wide range of disciplines: biology, medicine, environmental and earth sciences, art and archaeology, as well as material sciences. The versatile instrumentation of ID22 offers an excellent scheme to carry out unique projects. The following sections illustrate some of the research activities that have been carried out recently, focused mainly on, but certainly not limited to, the following fields: biomedical, earth and environmental, and materials sciences.

3.1. Biomedical sciences

Various examples of applications include cellular physiology, pharmacology, and toxicology of metal ions involved in biological processes, often called biometals (Bohic et al., 2011; Lewis et al., 2010; Carmona et al., 2010; Bacquart et al., 2010; Ortega et al., 2009; Corezzi et al., 2009). For instance,
workers indicates that the Golgi apparatus plays an important role in the cellular detoxification of Mn.

### 3.2. Earth and environmental sciences

The investigations in this area cover exploration from the earth interior up to stellar particles: homogeneity of the deep mantle, fluid–mineral relationships in the upper mantle, tracking elemental speciation in crustal melts and fluid sources in hydrothermal settings, as well as the nature of extra-terrestrial materials (Carbone et al., 2011; Simionovici et al., 2011; Borchert et al., 2010; Petitgirard et al., 2009; Reith et al., 2009). Carbone and co-workers (2011) recently used micro-XRD, micro-XRF and micro-XAS to investigate metal speciation in mine wastes and soils. The authors studied Ferich hardpans within waste-rock dump and show that the authigenic iron-rich phases generally contain significant amounts of hazardous elements such as Cu, Zn, Mo and Se. Moreover, a significant mineralogical control on the mobility of these elements was observed; in particular, the goethite-rich assemblages show high affinity for Cu and Zn, whereas hematite-rich assemblages selectively concentrate As, Se, Mo, Cu and Zn.

On the other hand, Borchert et al. (2010) have examined the partitioning of Ba, La, Yb and Y between haplogranitic melts and aqueous solutions under in situ conditions in EH1. Their findings show a strong influence of the composition of the starting fluid and melt with no dependence on temperature and only weak dependence on pressure. For chloridic fluids, there was a sharp increase in the Ba, La, Y and Yb partition coefficients with the alumina saturation index. Their results imply that both melt and fluid compositions have a strong influence on trace-element behaviour, while the complexion of Ba, REEs and Y is not controlled by the presence of Cl in the fluid only, but likely by interaction of these elements with major melt components.

The cycling of rare and precious metals, such as gold, has been also analyzed in ID22NI. In previous studies, researchers reported the presence of bacteria on gold surfaces, but never clearly elucidated their role. Recently, Reith et al. (2009) found that the bacterium Cupriavidus metallodurans catalyses the bimineralization of gold by transforming toxic gold compounds to their metallic form using an active cellular mechanism. So, there may be a biological reason for the presence of these bacteria on gold grain surfaces. The distribution of gold and other elements was mapped in individual deposits. On the other hand, Borchert et al. (2010) have examined the partitioning of Ba, La, Yb and Y between haplogranitic melts and aqueous solutions under in situ conditions in EH1. Their findings show a strong influence of the composition of the starting fluid and melt with no dependence on temperature and only weak dependence on pressure. For chloridic fluids, there was a sharp increase in the Ba, La, Y and Yb partition coefficients with the alumina saturation index. Their results imply that both melt and fluid compositions have a strong influence on trace-element behaviour, while the complexion of Ba, REEs and Y is not controlled by the presence of Cl in the fluid only, but likely by interaction of these elements with major melt components.

The cycling of rare and precious metals, such as gold, has been also analyzed in ID22NI. In previous studies, researchers reported the presence of bacteria on gold surfaces, but never clearly elucidated their role. Recently, Reith et al. (2009) found that the bacterium Cupriavidus metallodurans catalyses the bimineralization of gold by transforming toxic gold compounds to their metallic form using an active cellular mechanism. So, there may be a biological reason for the presence of these bacteria on gold grain surfaces. The distribution of gold and other elements was mapped in individual deposits. On the other hand, Borchert et al. (2010) have examined the partitioning of Ba, La, Yb and Y between haplogranitic melts and aqueous solutions under in situ conditions in EH1. Their findings show a strong influence of the composition of the starting fluid and melt with no dependence on temperature and only weak dependence on pressure. For chloridic fluids, there was a sharp increase in the Ba, La, Y and Yb partition coefficients with the alumina saturation index. Their results imply that both melt and fluid compositions have a strong influence on trace-element behaviour, while the complexion of Ba, REEs and Y is not controlled by the presence of Cl in the fluid only, but likely by interaction of these elements with major melt components.

The cycling of rare and precious metals, such as gold, has been also analyzed in ID22NI. In previous studies, researchers reported the presence of bacteria on gold surfaces, but never clearly elucidated their role. Recently, Reith et al. (2009) found that the bacterium Cupriavidus metallodurans catalyses the bimineralization of gold by transforming toxic gold compounds to their metallic form using an active cellular mechanism. So, there may be a biological reason for the presence of these bacteria on gold grain surfaces. The distribution of gold and other elements was mapped in individual deposits. On the other hand, Borchert et al. (2010) have examined the partitioning of Ba, La, Yb and Y between haplogranitic melts and aqueous solutions under in situ conditions in EH1. Their findings show a strong influence of the composition of the starting fluid and melt with no dependence on temperature and only weak dependence on pressure. For chloridic fluids, there was a sharp increase in the Ba, La, Y and Yb partition coefficients with the alumina saturation index. Their results imply that both melt and fluid compositions have a strong influence on trace-element behaviour, while the complexion of Ba, REEs and Y is not controlled by the presence of Cl in the fluid only, but likely by interaction of these elements with major melt components.

The cycling of rare and precious metals, such as gold, has been also analyzed in ID22NI. In previous studies, researchers reported the presence of bacteria on gold surfaces, but never clearly elucidated their role. Recently, Reith et al. (2009) found that the bacterium Cupriavidus metallodurans catalyses the bimineralization of gold by transforming toxic gold compounds to their metallic form using an active cellular mechanism. So, there may be a biological reason for the presence of these bacteria on gold grain surfaces. The distribution of gold and other elements was mapped in individual deposits. On the other hand, Borchert et al. (2010) have examined the partitioning of Ba, La, Yb and Y between haplogranitic melts and aqueous solutions under in situ conditions in EH1. Their findings show a strong influence of the composition of the starting fluid and melt with no dependence on temperature and only weak dependence on pressure. For chloridic fluids, there was a sharp increase in the Ba, La, Y and Yb partition coefficients with the alumina saturation index. Their results imply that both melt and fluid compositions have a strong influence on trace-element behaviour, while the complexion of Ba, REEs and Y is not controlled by the presence of Cl in the fluid only, but likely by interaction of these elements with major melt components.
3.3. Materials sciences

In this broad field, several scientific issues have been addressed using the beamline stations. The recent research comprises many materials with potential applications in spintronics, catalysis, optical sources, renewable materials like solid oxide fuel cell and silicon solar cells, etc (Sancho-Juan et al., 2011; Basile et al., 2010; Mino et al., 2010; Palancher et al., 2011; Kwapil et al., 2009; Martinez-Criado et al., 2009). For example, the combined use of micro-XRF, micro-XRD and nano-XRF techniques has been applied to the characterization of active-phase-coated metallic supports, structured catalysts, at different scales in both scanning and tomographic modes by Basile et al. (2010). In particular, coatings of FeCrAlY foams were examined, which are gaining attention because they improve heat transfer. The results show that the morphology of the coating depends on the synthesis conditions and that the catalyst may be described as Ni metal crystallites dispersed on $\gamma$-Al$_2$O$_3$, homogeneously coating the FeCrAlY foam.

Another recent experiment applied XRD scanning tomography to an annealed $\gamma$-U$_{0.8}$Mo$_{0.15}$ multiphase particle. UMo/Al dispersion fuel is one of the prospective materials for a high-uranium-density fuel for high-performance research reactors owing to its excellent stability during irradiation. The results published by Palancher et al. (2011) revealed a micrometre-scale layered structure morphology, the presence of an embedded 5 µm-thick interdiffusion layer, and an unexpected phase at trace levels which plays a protective role by inhibiting thermally activated Al diffusion into UMo.

The structural characterization of multi-quantum wells in electroabsorption-modulated lasers by Mino et al. (2010) is an excellent example of application in the microelectronic industry. The structural gradient (in both strain and barrier/well widths) that allows this system to operate as an integrated device has been determined with a 2 µm × 2 µm beam, scanning both laser and modulator regions. The investigated material is used for 10 Gb s$^{-1}$ telecommunication applications up to 50 km propagation span. In the same way, the application of hard X-ray nanoprobe techniques to the structural analysis of pyramidal defects in Mg-doped GaN, a potential material for optoelectronic devices, has been recently reported (Martinez-Criado et al., 2009). Fig. 8 shows the XRF data collected at ID22NI. The presence of elemental traces of Cr and Fe is revealed. A blue–red plot displays the Cr- and Fe-K intensity distributions. While the Ga arrangement presents equally spaced and periodic planes sequentially stacked from the hexagonal base (not shown), Cr and Fe exhibit a close correlation on their spatial locations without the three-dimensional pyramidal shape. The observations emphasize the
underlying diffusion mechanism, indicating local impurity agglomeration predominantly on the hexagonal base, supporting the occurrence of such pyramids by the kinetics of additional impurities that accompanied Mg incorporation. On the other hand, the strong polarization-dependent XAS features showed the preservation of the hexagonal crystalline structure in both defect-free and hexagonal pyramids. The X-ray linear dichroism (XLD) shows no preferential disorder in the direction parallel or perpendicular to the crystal growth.

Special thanks are due to the machine, instrumentation and technical services of the ESRF for their continuous support. In particular, the authors are very grateful to Joel Chavanne, Yves Dabin, Robert Baker, Eric Gagliardi, Cyril Guilloud, Alejandro Homs, Armando Solé, Jérôme Kieffer and Ricardo Steinmann for their useful and excellent help. GM-C thanks Dr Michael Reynolds for the critical reading of the manuscript.

References


