ESRF	**UPGRADE PROGRAMME – PHASE I** **CONCEPTUAL DESIGN REPORT**

X-ray microscopy	
Current designated sector:	Facility goes to:
ID21	ID21

1.1 ID CARD

The X-ray micro-spectroscopy beamline ID21 enables identification down to a few ppm, and localisation with a submicron beam of various elements and with a higher sensitivity for low Z elements, from Na to Fe. These elemental mappings can be completed with spectroscopic analysis, performed on single points or also as 2D images. The submitted proposals are mainly in the field of Environmental Science, Life Science and Cultural Heritage. Typical scientific questions concern the co-localisation and/or speciation of trace elements in heterogeneous matrix at the micron scale.

1.2 SCIENTIFIC CASE

The ID21 beamline is dedicated to X-ray micro-fluorescence (µ-XRF) and micro-spectroscopy X-ray absorption near-edge structure (µ-XANES) over a spectral range from 2-7.5 keV. Despite this narrow energy range, ID21 provides access to absorption edges for a wide range of elements of interest for applications in life, materials and environmental sciences.

Micro-analytical techniques aim to screen samples at various levels of information, ranging from elemental and chemical to structural data. Most natural or man-made systems have properties that depend on the specific hierarchy of chemical components and their organisation at different scales of length. Therefore, an understanding of the macroscopic function requires insights into microscopic structure and dynamics on all length-scales down to the molecular and atomic levels. Ultimately, these investigations must address studies of realistic systems in their near-native environment using non-invasive techniques.

The above considerations underpin the need for a multimodal approach, where several analytical techniques are required to bridge the gap between a macroscopic function and its atomic or molecular origins, by probing chemical and structural information at various length-scales. In a more specific context, X-ray microanalysis techniques today follow the obvious trend in the development of nano-technologies by pushing spatial resolution further down to the nanoscale. Towards this perspective, synchrotron-based analytical techniques (diffraction, imaging and micro-spectroscopies) play an important role by offering unique capabilities in the study of complex systems. Both by extrapolation of the experience gained in the soft X-ray regime and by the development of new techniques, “tender” and “hard” X-ray microscopes offer today a unique analytical tool which contributes to a wide range of existing and new applications of X-ray microscopy in experimental research fields. In particular, compared to other techniques, synchrotron X-ray fluorescence microprobes display a unique combination of features. Associated with high detection efficiency, the radiation damage is minimised and accurate quantification is possible. Furthermore, the possibility of *in situ* experiments remains a unique attribute of synchrotron-based analytical methods. Photon-depth penetration of tender or hard X-rays enables specific sample environments to study realistic systems in their near-native environment rather than model systems. The ability to perform *in situ* analysis with controlled environments preserving, for instance, sample hydration, explains the increasing interest from various communities such as planetary and earth sciences, environmental science and microbiology. Finally, the need for combined morphological and chemical information is a clear trend, which is steering our strategy toward a coupled access to FTIR and X-ray microprobes.

In conclusion, ID21 aims to remain a method-oriented beamline offering a unique set of non-destructive analytical techniques which aim to satisfy the growing demand from experimental research fields such as materials science, medicine, geology, archaeology, earth, planetary and environmental sciences (1-3).

1.3 PROJECT HISTORY

The upgrade strategy of ID21 has to be seen in the overall context of the synergic development of the two X-ray microprobe beamlines at the ESRF (currently ID21 and ID22, and with the future UPBL4 project). It aims to best exploit the new possibilities offered by the ESRF Upgrade context: smaller probes, larger working distances and improved infrastructure with enhanced inter-connection and synergy between instruments. It is not only proposed to extend the intrinsic capabilities of the beamlines (e.g. ID21 and ID22 in the X-ray Imaging Group), in terms of spatial resolution and detection limits, but also to further develop a multimodal analysis platform based on the use of infra-red and X-ray micro-spectroscopies, micro-diffraction, and X-ray imaging techniques. This technique portfolio should be completed by electron and Raman microscopes. This CDR is an evolution of the proposal SMILE of the Purple Book: “Spectro-Microscopy and Imaging at Low Energies” for chemical mapping in the multi-keV range (2-10 keV) and mid-infra-red spectral range (1-20 µm).

1.4 BASIC TECHNICAL CONSIDERATIONS

The X-ray Microscopy beamline is currently installed on a 4.8 m long low-β straight section. The choice of a low-β section (small source but larger divergence) was driven by the following considerations: first, the small horizontal source size allows X-ray lenses to be used in a diffraction-limited regime even for long focal lengths; second, the larger horizontal divergence limits the heat load on the first optical components by spreading the power over a larger surface. The current performances of ID21 support this option. The flux and probe size of ID21 is not source-limited. Modifications of the source and front-end are therefore not foreseen for the time being. The proposed beamline Upgrade programme is articulated around four main components:

1. *Pre-mirrors in optics hutch:* The spectral range of ID21 includes long wavelengths and thus implies long longitudinal coherence lengths. The wave front quality is therefore rapidly affected by figure errors on optics. In particular the rather poor quality of the two horizontally-deflecting mirrors used as a high harmonic rejection device in this optics hutch limits the performances of the microscopy by increasing geometrically the apparent horizontal source size. The improvement of the polishing and figuring techniques allows today much better mirror quality. Those mirrors being flat, a figure with slope errors below 1µrad is a realistic target.
2. *Double-crystal monochromator:* It is proposed to improve the mechanical stability of the monochromator. The implementation of a KB mirror system as a focussing device makes the spatial stability of the microprobe even more sensitive to the stability of the monochromatic beam whilst scanning the energy. A cost-effective solution would be to revisit the mechanical concept of the crystal stage and its associated cooling system while recycling the vacuum vessel and associated support hosting the complete system.
3. Formation of a secondary source by means of a double focusing mirror system upstream the crystal monochromator.
4. Development of a highly efficient energy-dispersive device for XRF detection.

In summary, the most challenging technical issues of the project can be summarised as follows:

- Improved quality of the primary M0 mirrors.
- Stability of the fixed-exit double-crystal monochromator for X-ray absorption spectroscopy.
- Implementation of a high-collection X-ray fluorescence detector.
- Efficient and stable, vacuum-compatible KB mirror devices. The high quality KB mirror substrates will be crucial to achieve large numerical aperture optics providing simultaneously a large flux and the smallest focus.
- Development of a dedicated laboratory for sample preparation, sample inspection and visualisation will be of major importance for a successful development of this programme. Ancillary equipment such as electron and visible light microscopes, FTIR and Raman spectro-microscopes and sectioning/polishing apparatus are mandatory. Such a laboratory can be shared with other beamlines requiring similar instruments and equipment.

1.5 REFERENCES

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