An Overview of the ESRF's ID13 Microfocus Beamline

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The ID13 beamline at the European Synchrotron Radiation Facility specialises in the delivery of micrometre and sub-micrometre X-ray beams, primarily for soft condensed matter studies. The techniques available include high-resolution scanning experiments using wide- and small-angle X-ray scattering techniques, protein microcrystallography and grazing incidence scattering geometries. ID13s modular design is compatible with a number of different focussing optics which allows the X-ray beam's characteristics to be tailored to individual experimental requirements. This flexibility also makes ID13 ideally suited for *in situ* studies such as investigating chemical reactions, probing the effect of local hydration or temperature changes, and monitoring deformation micromechanics within complex materials.

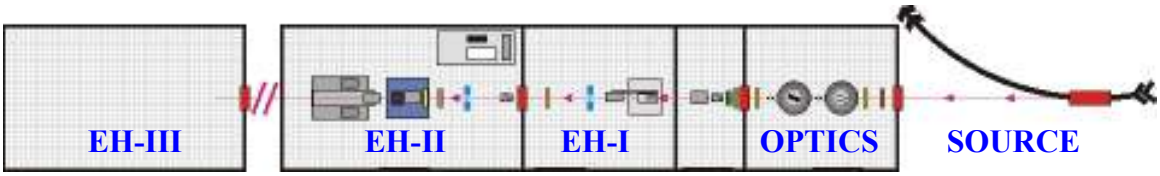

Figure 1. General beamline layout.

1. Introduction

The ID13 beamline of the European Synchrotron Radiation Facility (ESRF) specialises in the delivery of microfocussed X-ray beams for wide- and small-angle scattering experiments. Although primarily operating within the field of soft condensed matter, its use covers a wide range of different scientific applications and sample types. Offering high-brilliance X-ray beams on micrometre and sub-micrometre length scales, ID13 is ideal for studying small sample volumes, or probing microscopic morphologies.

ID13 was one of the first beamlines to be commissioned at the ESRF and it remains one of the most heavily oversubscribed today. Its origins can be traced back to the first microfocus workshop held in 1989 [1], although it wasn't until 1992 that construction of the beamline officially started [2]. Since then, literally thousands of users have capitalised on the unique facilities offered by ID13. Indeed, in the past six years alone around 230 scientific publications have been authored as a direct result of work carried out on the beamline (2000-2005).

Microfocussed X-ray beams are popular experimental tools for a wide range of different reasons. Their higher flux density can be used to probe small sample volumes whilst they allow an increased spatial resolution for scanning experiments on heterogeneous materials. In many ways they offer all of the benefits of other highly localised probing techniques such as electron diffraction (ED), although they do so without many of the drawbacks. For example, there is no need for special sample preparation which risks artefact introduction and precludes *in situ* studies whilst beam damage is also less of an issue. In addition, compared to ED methods, microfocussed X-rays can be used to access structural information over a much greater range of length scales.

2. Optics & Infrastructure

ID13 is a linear beamline with its main elements comprising an optics hutch and three experimental hutches, each specialised in a specific area. The general beamline layout is shown schematically in *Figure 1*. ID13's primary source is an 18 mm period in-vacuum undulator optimised for energies suitable for soft condensed matter studies (in the range of 12-13 keV). However, for special applications ID13 also offers a second fully-tuneable ex-vacuum undulator with a 46 mm period (approximately 5-17 keV).

The ID13 optics hutch houses two monochromators positioned in series, their use being mutually exclusive. The first is a liquid nitrogen cooled Si-111 double crystal (bounce) monochromator. The second, a relatively new addition to the beamline, is a channel cut monochroma-

tor employing a single liquid nitrogen cooled Si crystal. This monochromator has been specifically developed for nanobeam applications, requiring particularly high downstream stability.

The first experimental hutch (EH-I) houses a dedicated goniometer for protein microcrystallography, which is also used for small unit-cell crystallography and fiber diffraction [3]. This unit, which was originally designed collaboratively between the European Molecular Biology Laboratory (EMBL) and the ESRF, is the prototype of the commercial microdiffractometer available at other ESRF beamlines and SR facilities. The microgoniometer offers X-ray beam spot sizes ranging between 5-30 μm, and includes all of the features which are now considered standard in such devices (integrated on-axis sample and beam visualisation, centre of rotation correction, cryostream integration *etc*.).

The second experimental hutch (EH-II) is dedicated to scanning microfocussed X-ray diffraction (μXRD) experiments. A range of different focussing optics are available, allowing the beam characteristics to be altered as a matter of routine (in response to experimental requirements). These include compound refractive lenses (CRLs) [4,5], Kirkpatrick Baez (KB) mirrors [6], crossed Fresnel zone plates [7] and waveguides [8,9]. The different optics, split between short and long focal length configurations, provide both a range of different beam spot sizes and beam divergence options. For example, long focal length CRLs can be coupled with a collimator to provide a low divergence 5 μm beam ideal for small-angle X-ray scattering (SAXS) applications. Fresnel and KB optics provide medium-range solutions for combined SAXS/WAXS experiments with beam sizes in the range 300-700 nm. Spot sizes of 100 nm or less have been reached with X-ray waveguides or refractive lenses. *Figure 2* shows a number of different ID13 beam spot sizes that are currently in routine use, superimposed to scale over an SEM of a single fibre.

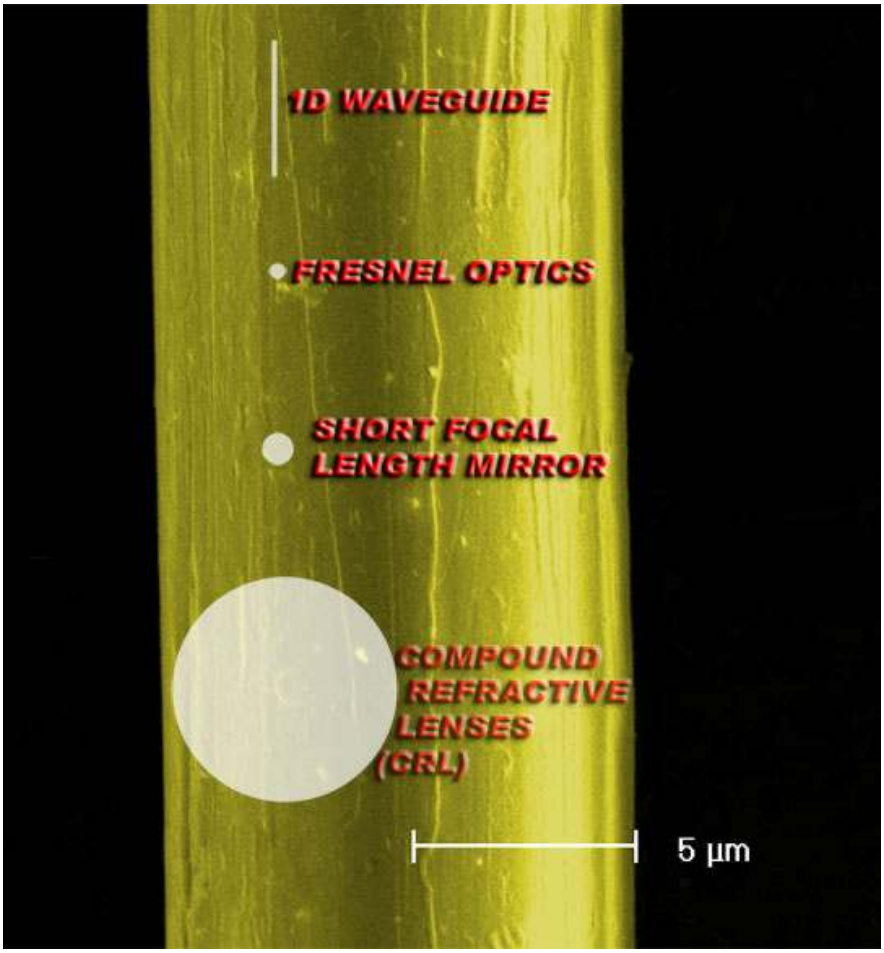

Figure 2. Routine ID13 beam spot sizes superimposed to scale over an SEM image of a single fiber.

Most experiments carried out on ID13 employ SAXS and/or WAXS to investigate periodicities existing over different length scales. However, various other techniques are also used somewhat less frequently for special applications. For example, it is possible to perform microfocussed X-ray fluorescence experiments on the beamline in a scanning acquisition mode [10]. Alternatively, grazing incidence SAXS can be used to investigate interfacial geometries using a microbeam (so-called μGiSAXS) [11,12].

In addition to its flexibility in terms of optics, EH-II also offers a modular sample environment ideal for the integration of complex experimental setups. For this reason, ID13 is also popular, and indeed specialises in so called '*in situ*' studies. These frequently involve either physical or chemical modifications during data collection, such as deformation, changes in temperature or hydration. Such studies also benefit from the availability of a number of detectors, including a MARCCD 165 and a 16 bit FReLoN camera (2000 model). For example, the 200 ms readout time of the FReLoN camera is attractive both in terms of time resolution (for *in situ* studies) and for covering large scan regions rapidly. Meanwhile the MARCCD has a lower effective point-spread-function and larger window area for combined SAXS/WAXS studies and structural refinement.

The final experimental hutch (EH-III) is a new addition to the beamline, current still under construction. When completed in Summer 2007, this will offer high-stability nanofocus X-ray beams for SAXS/WAXS applications, well beyond the capabilities of any existing SR facilities. In addition, its position at about 100 m from the source opens up broader SAXS possibilities due to the lower beam divergence when using longer focal length optics.

3. Scientific Applications & Examples

The modular nature of ID13's instrumentation is reflected in the wide range of scientific applications studied on the beamline. Single fibre studies feature heavily in ID13's scientific output, primarily because there are very few other techniques which are suitable for studying the internal microstructure of such small sample volumes (typically <10 μm). For example, morphological studies have investigated the skin-core morphology of high-performance polymeric fibers using 200 nm waveguide beams [13,14]. Using Monte Carlo simulations it has been possible to develop structurally-defined models of radial anistropy which convincingly explain the results obtained experimentally [14]. Biopolymer fibers such as silk [15,17], hair [18], and cellulose [19-21] also feature in a number of ID13 investigations, many of which are on-going studies. However, in addition to fiber-related studies, a wide selection of other sample types are routinely studied on the beamline. These include archaeological specimens [22], biological samples [23-25] and minerals [26]. Furthermore, the microdiffractometer in EH-I has been used extensively for solving a range of crystallographic structures [27-30].

There are many different examples of *in situ* studies on ID13, one of the most widely known being the *in situ* extrusion of spider silk [31-32]. However, the most commonly used *in situ* technique is mechanical deformation. This has been used extensively to investigate stress-induced changes in the microstructure of a wide range of different materials. The information these experiments provide can be used to identify structure-property relationships which have both a scientific and commercial value. Several ID13 studies report on high-performance polymeric fibre micromechanics [33-35]. Meanwhile compressive deformation modes have also been investigated, either by bending single fibres [36] or by performing *in situ* microindentation [37]. However, studies involving the deformation of materials are not confined exclusively to fibres. The influence of deformation on bulk polymers has also been studied *in situ* in order to assess the impact of different processing parameters [38,39]. Similarly, a microfocussed X-ray beam has proved to be a novel way of monitoring stress transfer *in situ* in deformed composite materials [40].

As well as deformation there are several other types of *in situ* study carried out on the ID13 beamline. *In situ* hydration is one such example using microdroplet technology to deliver targeted picolitre-volume droplets. Starch granule and *β*-chitin hydration are two recent examples which have been studied using this delivery technique [41,42]. In addition, a microbeam has obvious applications for probing within microfluidic cell channels. This could be used to monitor small-scale chemical reactions or for investigating flow dynamics within microscopic systems.

In addition to the physical or chemical modification of samples, there is another class of *in situ* experiment which is particularly important for facilities such as ID13. This is the *in situ* combination of complimentary experimental techniques. With microfocussed X-ray beams of micrometre and sub-micrometre dimensions, *in situ* combinations are the only reliable method of probing the same sample region using multiple techniques. For example, ID13 has recently commissioned an *in situ* microRaman spectroscopy system with a laser beam size similar to the X-ray beam size which is delivered simultaneously on-axis [43]. This offers the possibility of simultaneous microRaman and μXRD in a single experiment, providing a range of complimentary information which is inaccessible using other techniques or performing experiments sequentially *off-line*.

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