# PETRA III:
# A Low Emittance Synchrotron Radiation Source

# Technical Design Report

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# 6.7 High Energy Materials Science

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## 6.7.1 Current state of the scientific field

### 6.7.1.1 Introduction

Diffraction methods are well established in Materials Science and Engineering. From the 1930s on X-rays from laboratory sources have been used for non-destructive characterizations of the phase composition of various materials (Hauk, 1997). One of the early and still important applications regarding **phase analysis** in engineering materials is the determination of the retained austenite content in steels, which has a strong influence on steel properties (e.g. Wang et al., 2003a).
**Texture analysis** by diffraction enables the determination of the orientation distribution of the crystallites. Important technical applications of texture analysis, for instance, are optimizations of the directional properties of semi-finished material for forming processes, e.g. sheet materials, and optimizations of the directional properties of components (e.g. Juul Jensen & Kjems, 1983).
The development of methods for the analysis of **residual (internal) stress** by neutron diffraction started in several groups in parallel (e.g. Allen et al., 1981). The residual stress state of materials and of components is of high importance, since their stress state under working conditions is a superposition of the load and the residual stress state. Thus, for instance, in case of fatigue loading, in most cases compressive residual stresses in the component's near-surface zone proved to be beneficial while tensile stresses were detrimental for the component's lifetime. Reflection profile analysis reveals additional information about residual micro-stresses and, in general, about inhomogeneities of the crystal lattice (e.g. twinning, stacking faults). Engineering applications of profile analysis include e.g. the determination of solute nitrogen in high nitrogen steels and solute carbon in case-hardened steels as well as the determination of the effective depth of surface treatments such as shot peening.

### 6.7.1.2 Neutron diffraction

Neutrons have been widely used for phase, texture and residual stress analysis, applying well established techniques. Compared to laboratory X-rays the essential advantage of neutrons is their higher penetration depth. This enables phase, texture and internal stress analyses in the bulk of materials and components. For instance the three-dimensional residual stress state of the matrix and the particles of composite materials can be determined non-destructively (e.g.

Al - Si, Ti + SiC, Al + SiC) (e.g. Fiori et al., 2000). The application of neutron diffraction in materials science suffers from the comparatively low neutron flux and the high divergence of neutron beams. Due to the low flux the gauge volume element has a minimum size of app. 1 $mm^3$, which limits the achievable spatial resolution. Furthermore, the necessary data acquisition times, which are usually in the range of minutes, restrain the application of neutron diffraction for in-situ experiments. In addition, the high divergence of the neutron beam often leads to blurred volume element boundaries. This has also a strong influence on the reflection profiles and, thus, often rules out an accurate reflection profile analysis. Nevertheless, well established and standardized techniques are commonly used for texture and strain analyses and several dedicated instruments exist at various neutron sources. The complementarity of these techniques to X-ray diffraction techniques is of particular interest for the proposed project due to the vicinity of the neutron source at GKSS, Geesthacht.

#### 6.7.1.3 Synchrotron X-ray diffraction

The availability of synchrotron radiation has opened up a broad new field for the application of diffraction techniques in materials science (e.g. Liss et al., 2003). This is due to the extremely high photon flux, the high parallelism and the large range of photon energies available.
The high parallelism of the synchrotron radiation has opened up the possibilities to determine phase composition, texture, lattice defects, and strains / stresses in new materials such as nano-crystalline materials, nano-composites, and crystalline fractions of partially amorphous materials. Due to the high photon flux strain and texture scanning as well as in-situ experiments are possible. Within the area of materials science and engineering a distinction between experiments using monochromatic low and medium energy ($<$40 keV), monochromatic high energy, and white high energy synchrotron radiation is useful.

**Monochromatic low and medium energy synchrotron radiation**

As a result of the low penetration depth bulk investigations with low and medium energy radiation are limited to materials with low absorption such as polymers (e.g. Swartjes et al., 2003; Terry et al., 2003) or thin samples. An example from the field of energy storage is the in-situ investigation in transmission geometry of thin battery electrodes under operation (Morcrette et al., 2002a).
In the case of highly absorbing bulk materials, characterizations of the very-near-surface zone (penetration depths of a few hundred nanometers, e.g. in multi-layer-systems) and in the near-surface zone (penetration depths of a few hundred $\mu$m, e.g. in surface treated components) are commonly performed. Various techniques addressing these questions were developed in the past (e.g. Van Acker et al., 1993; Ruppersberg, Detemple & Krier, 1989; Genzel, 1994; Leverenz, Eigenmann & Macherauch, 1996).
A technique for three-dimensional structural microscopy with sub-micrometer resolution has been developed by Larson and co-workers (Ice & Larson, 2000; Larson et al., 2002). It is based on the Laue technique, the use of polychromatic low energy radiation and a scanning approach. It allows a micro structure and strain mapping in the near surface zone (Larson et al., 2003). Another method is due to Wroblewski and co-workers (Wroblewski et al.,

1999). It uses an 8 keV synchrotron beam and the setup consists of a 4 - circle diffractometer and a micro-channel plate in front of a CCD. The micro-channel plate serves as a 2D array of collimators. This so-called MAXIM setup provides extended maps with a reported resolution of 12 µm, e.g. of the position of various fine-grained phases (Pyzalla et al., 2001b) or the strain state (Wroblewski, 2002).

A number of so-called micro-focus beamlines, working at low photon energies, exist today. The focusing to 0.1–1 micrometer is achieved by a variety of optical components, such as focusing multi-layers (Ice et al., 2000), zone-plates (Tamura et al., 2000; MacMahon et al., 2003), refractive lenses (Snigirev et al., 1996; Lengeler et al., 1999b; Schroer et al., 2002a), and waveguides (Pfeiffer et al., 2002).

**Monochromatic high energy synchrotron radiation**

Due to the high photon flux and the penetration power of hard X-rays bulk investigations are commonly done in transmission geometry. The sample thickness ranges from several centimeters in case of Al samples to several millimeters in the case of samples containing large amounts of heavy elements like Ag, W or Pb. Three main fields where hard X-rays are used for the macroscopic characterization of materials can be identified. The first one is a powder diffraction approach where an average, macroscopic information about the full specimen is obtained. The works reported comprise e.g. the structure solving from polycrystalline specimens (Schmidt, Poulsen & Vaughan, 2003), high temperature powder diffraction (Kramer et al., 2002), and the processing of bulk metallic glasses (Yavari et al., 2002). A second field is the investigation of the local texture, initiated at HASYLAB (Garbe, Poulsen & Juul Jensen, 1996; Mishin et al., 2000) which later has led to a dedicated setup there (Wcislak et al., 2002). Thirdly, high energy X-rays have been applied by numerous groups for the characterization of the local strain state, mostly in transmission geometry, hereby integrating over the sample thickness or defining a fixed volume element for depth resolved investigations (e.g. Withers et al., 2002; Webster et al., 2001; Hanan et al., 2002).

The development and implementation of micro-focusing optics for hard X-rays (Lienert et al., 1998; Lienert et al., 1999) has opened the gate for the bulk characterization of the micro structure, texture, and strain on the mesoscopic length scale. Combined with optical elements such as a conical slit cell (Nielsen et al., 2000) or a spiral slit small gauge volumina down to 1.2 x 6 x 250 $\mu m^3$ are realized. 2D CCD detector systems provide in combination with the high photon flux a fast data acquisition (about one image acquisition cycle per second). Dynamic studies of the local texture and strain state in torsion samples during deformation were reported (Martins et al., 2002), using a slit imaging technique (Lienert et al., 2000) with a microfocused beam in combination with a conical slit cell and a large area CCD with fast readout. Non-destructive depth resolved phase and strain characterizations, e.g. of friction stir welds (Martins & Honkimäki, 2003), are possible with setups comprising a spiral slit and a large area detector. The development of the 3 Dimensional X-Ray Diffraction (3DXRD) microscope (Lienert, Poulsen & Kvick, 1999) has paved the way for investigation of mesoscopic phenomena such as e.g. the kinetics of individual bulk grains during recrystallization (Lauridsen et al., 2000), in-situ observation of the rotation of deeply embedded individual grains (Margulies, Winther & Poulsen, 2001) or the in-situ determination of the strain tensor in individual bulk grains (Margulies et al., 2002). The 3DXRD method is based on a ”tomographic” (meaning here a data acquisition at a series of different angular sample positions) approach in combination with a microfocused beam and area detectors. The use of high resolution detectors and appropriate data reconstruction routines leads to a spatial resolution down to 5 µm. The data acquisition in the ”tomographic” approach is considerably faster than in other 3D techniques, based on ”scanning” approaches. A fast data acquisition is the basis for the in-situ observation of dynamic processes.

A first experiment combining diffraction and a tomography data (acquired at two different beamlines), was reported by Preuss and co-workers (Preuss et al., 2002). In this combined experiment the progressive fragmentation process of a single SiC fibre embedded in a Ti-6Al-4V matrix was studied.

**White high energy synchrotron radiation**

In contrast to experiments with monochromatic X-rays, where the intensity of the diffracted photons is measured as a function of the Bragg angle (i.e. angle dispersive), the experiments using white radiation are energy dispersive. Usually an energy dispersive point detector with a sufficient energy resolution is placed at a fixed Bragg angle and the intensities of the energy spectrum are recorded. Residual stress analyses using white high energy synchrotron radiation were first reported by Reimers and co-workers (Reimers et al., 1999). Due to the short data acquisition times, in the order of seconds, in-situ investigations of particle reinforced Al alloys under tensile load at elevated temperatures became feasible (Pyzalla et al., 2001a). Another common application is the residual strain analysis of ceramics, e.g. of thermal barrier coatings (e.g. Gnäupel-Herold et al., 2000).

**Limiting factors**

Limitations in the applications up to now depend on the length scale the experiments are aiming for.

In the case of investigations on the macro scale, which usually means a powder diffraction approach, the limitations are given by the graininess of the material. To achieve a high spatial resolution parallel to the incident X-ray beam, slit systems are usually inserted in the diffracted beam and the incident beam has to be confined or focused horizontally and/or vertically. In the case of a coarse grained sample micro structure, macroscopic information is not ensured, because only a few grains are illuminated. (Due to the larger sampling volumes this problem usually does not occur in neutron diffraction experiments.) To maintain the high spatial resolution along the beam the samples can be, in some cases, oscillated or rotated perpendicularly to the beam to obtain a better grain sampling.

In the case of investigations on the mesoscale, i.e. on a level, where the aim is to obtain information about individual grains, the problems are different. The aim here is to observe simultaneously as many individual (possibly nano sized) grains as possible, in certain cases (e.g. grain boundary mapping) with a high spatial resolution. This is until now hampered by diffraction spot overlap, detection limit, and low detector resolution. Solutions to these problems are ,e.g. the development of detectors with higher spatial resolution (i.e. much better than 5 µm), a smaller beam size ($<1$ µm), new experimental approaches, and modified or new reconstruction algorithms. In-situ observations are up to now mostly limited to model cases. Characterizations of complex and highly dynamic processes are still limited by factors such as data acquisition rate and photon flux.

### 6.7.2 Science at PETRA III

Synchrotron radiation has gained an increasing interest in the materials science community within recent years. It has been shown that substantial new insight into material and component structures and relations between structures and properties can be obtained using synchrotron radiation. In particular materials science with hard synchrotron X-rays has been developing fast during the last 5 years and shown to be promising for investigations of bulk materials and components. Most experiments so far have been carried out at the ESRF (beamlines ID11, ID15A, ID15B, ID30, and ID31), at HASYLAB (beamline BW5 and PETRA II), and at the APS (beamline 1IDB). In principle, both angle and energy-dispersive measurement setups with monochromatic and white radiation, respectively, are possible. The materials science beamline at PETRA III will initially focus on angle-dispersive setups and take advantage of the small beamsize achievable there. The realization of an energy-dispersive setup will be highly emphasized at the HARWI-II beamline, projected by GKSS Geesthacht at the DORIS III storage ring (Beckmann et al., 2003). The HARWI-II beamline avails of the large beam size provided by the wiggler source (e.g. tomography).
The experiments performed up to now in materials science were, to a large majority, focusing on two fields: The investigation of engineering materials and components and fundamental studies in the field of metallurgy and crystallography. In both areas in-situ studies became more and more established. These studies were mostly comprising the (in-situ) investigation of thermally activated processes and / or the material behavior under different basic deformation modes such as tension, compression, shear, and torsion.
The experiments projected for the new beamline will focus on three main topics, which are partly intersecting and for which the instruments will be optimized for (discussed in detail in the following subsections):

- Experiments targeting the industrial user community which will be based on well established techniques with standardized evaluation routines.
- Applied research such as the investigation of power systems (e.g. fuel cells) or process optimization (e.g. manufacturing processes).
- Fundamental research where engineering, metallurgy, physics, chemistry, biology etc. are merging.

Large scientific progress in these fields will be ensured by three particular and unique features of the new beamline:

- Emphasis on complex in-situ studies, e.g. of highly dynamic processes
- Submicron spatial resolution at high energies for high resolution bulk investigations
- Merging of different analytical techniques such as diffraction, tomography, and small angle scattering

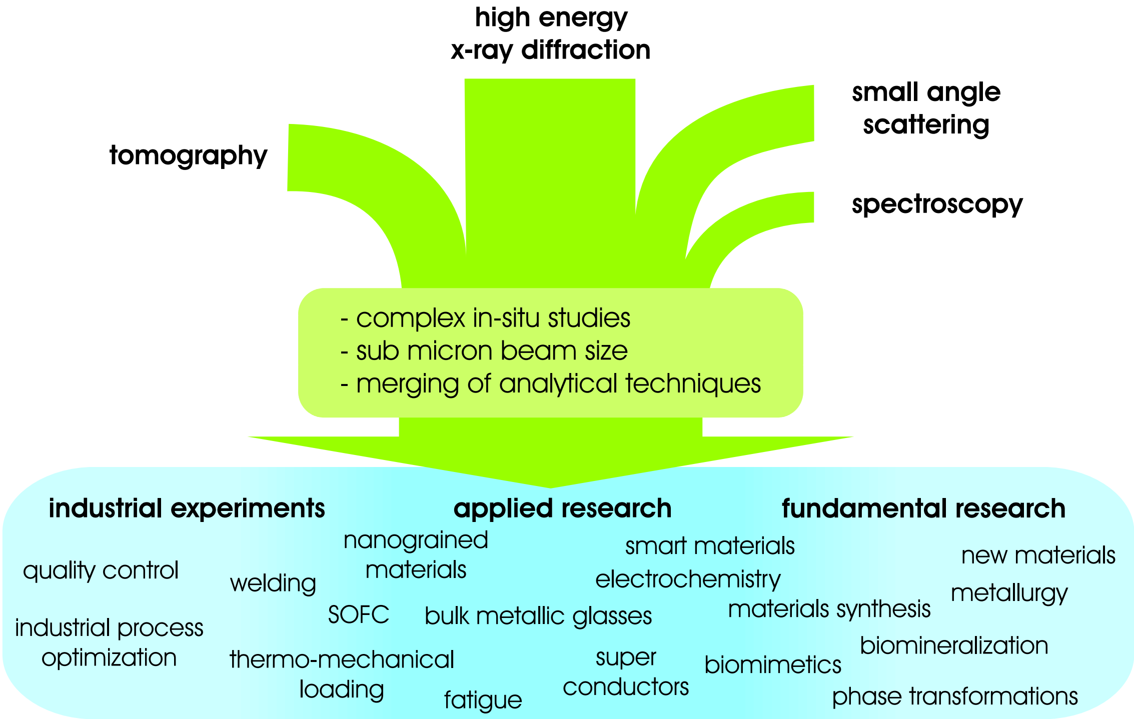


Figure 6.7.1: Overview of the main beamline features, analytical techniques, and research topics projected. (see subsections for details)

Fig. 6.7.1 summarizes the main beamline features, analytical techniques and research topics projected.
By providing the possibility to use this large range of different analytical techniques in parallel, this beamline will be the first one fulfilling one of the main requirements for future research in materials science as expressed in the *European White Book on Fundamental Research in Materials Science* (Rühle et al., 2001).

#### 6.7.2.1 Industrial user community

The intention is to respond to the increasing demand for ”full service” measurements. In other words, the industrial user wishes to send his samples and to receive the results in form of a report, without the need to have a deep understanding of the experimental details and the data evaluation. Moreover, industrial users often prefer measurements to be performed for a large number of samples. These investigations could concern, e.g. a process optimization or a process quality control. This type of experiment will be optimized for high sample throughput. It requires dedicated setups with fast data acquisition based on well established and robust experimental techniques for phase, strain, and texture analysis, and standardized online data analysis routines. They will be complying with the VAMAS standards (Versailles Project on Advanced Materials and Standards). Another type of industrial experiments is the non-destructive investigation of complete structural components or components in operation. This can be, e.g. the strain mapping in critical components of an engine in operation. The new beamline will provide the necessary infrastructure for heavy load sample and sample environment positioning (up to 1 t) and the operation of ”specimens” (e.g. dedicated air outlet for an engine investigated in-situ; cooling).

#### 6.7.2.2 Applied research

The experiments in this field will e.g. focus on the in-situ investigation of liquid / solid processes such as the melt spinning of bulk metallic glasses (e.g Yavari et al., 2002) or welding. These experiments will benefit from the high flux in combination with ultra-fast detector systems. In the field of power system development and optimization, which partly reaches into the field of electrochemistry, experiments of high importance are the investigation of the strain field and interface kinetics of operating Solid Oxide Fuel Cells (SOFC) or the in-situ phase monitoring of battery electrodes (Morcrette et al., 2002a).

The rapidly advancing miniaturization of components and the development of nano-materials will require powerful tools for the optimization of interface-engineering processes. Examples are the non-destructive micro structural analysis of nano-grained metals and ceramics or high resolution strain maps in functionally graded materials. These applications will benefit from the sub-micrometer focusing and the nano-detection capabilities of the new beamline. Furthermore, ”classic materials” (e.g. steel, Al-alloys), ”advanced materials” (e.g. metal matrix composites, Mg base alloys, TiAl), and new materials such as ”smart materials” (e.g. materials with embedded strain monitoring devices or ”self healing” materials), can be investigated. These investigations will excel in the new parallel design of the experiments. It enables e.g. the monitoring of the crack propagation by tomography and the development of the strain field by diffraction within a self healing component or a fiber reinforced material under thermo-mechanical loading. In addition, the installation of the proposed flight tube permits to characterize by small angle scattering *for the first time at high energies and simultaneously to diffraction* e.g. polymers, precipitates, voids, and phase separation (e.g. Fratzl, 2003; Kostorz, 1991) in bulk materials and complex components under thermo-mechanical loading. The use of high energy radiation will permit to perform these investigations in bulk samples of several millimeters to centimeters thickness of technologically important materials such as steel, Ni-base, and Al-base alloys. On a nanometer scale the technique will also allow to gain information on the internal grain microstructure (Fratzl, 2003) which is then directly related to the diffraction data from this specific volume element (cf. 6.7.2.3). Diffraction experiments employing different strain scanning techniques will allow to examine the complete strain state of a sample, from the very-near-surface region down to the bulk. Sample environment for thermo-mechanical loading will cover uni-, bi-, and triaxial deformation modes. The infrastructure will allow an easy accommodation of user provided equipment (e.g. for the in-situ monitoring of processes like friction stir welding). A unique feature of the projected beamline will be an external materials testing laboratory which will contain mechanical testing devices, furnaces, cryostats etc. so that samples during long-term mechanical, thermal and thermo-mechanical testing can be frequently studied using high energy synchrotron radiation.

### 6.7.2.3 Fundamental research

A large number of experiments will be related to metallurgy and the investigation of polycrystalline materials. The macroscopic properties of polycrystalline materials crucially depend on the behavior and interaction of the individual grains along the complex network of interfaces. The grain boundaries are the glue that hold polycrystalline materials together. Three-dimensional X-ray diffraction (3DXRD) microscopy provides a means for non destructive characterization of the internal structure of polycrystalline materials (Poulsen et al., 2001, Schmidt, Poulsen & Vaughan, 2003). The method is illustrated in Fig. 6.7.2. A monochromatic point-focused beam selects a local gauge volume that contains a sufficiently small number of grains for individual observation. Rotation about the $\omega$ axis is used to excite multiple reflections from each individual grain, from which the orientations of all grains in the gauge volume can be determined. Measuring the same reflection spot at different distances of the 2D detector, the position of the grains can be determined by backtracking. Finally, a complete map of the grain boundaries within the volume can be reconstructed (Lauridsen et al., 2001, Poulsen & Fu, 2003). The types of grain boundaries in a material and the way in which they are connected affect a wide range of material properties. The tailoring of these properties has triggered the emerging technology of 'grain boundary engineering'. Fundamental research in this field is essential to specify and produce the desired micro structures. High-energy X-ray microbeams provide a unique opportunity for the determination of internal structures. A scientifically very active area is the study of grain growth. The final micro structure of a material, for example, depends on the grain growth during thermal processing. For a direct engineering it is essential to understand the driving forces behind the change in grain size. The instrument proposed here offers a unique capability to access all

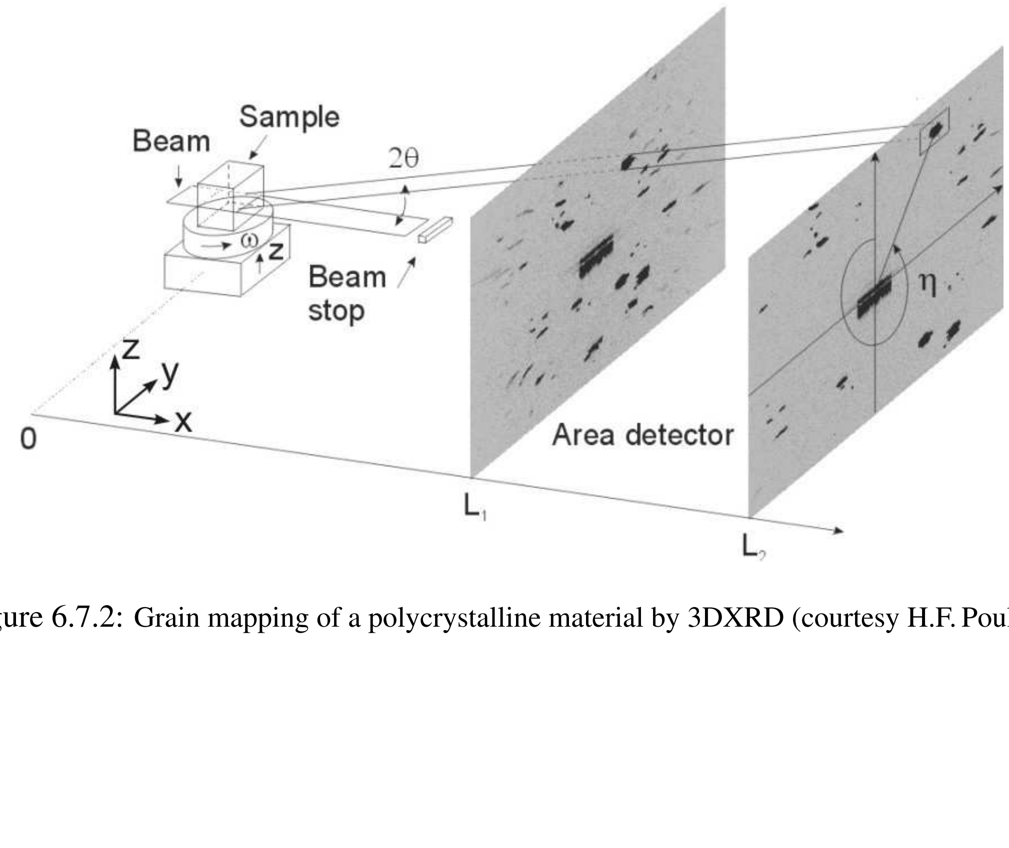


Figure 6.7.2: Grain mapping of a polycrystalline material by 3DXRD (courtesy H.F. Poulsen)

quantities to study grain boundaries and their dynamics (Poulsen & Schmidt, 2003). The development and numerous applications of 3DXRD have shown that a tremendous insight into mesoscale phenomena is possible with high energy X-rays. Up to now most of these experiments were related to model materials such as pure Cu or Al. Moreover, each experiment was focusing on one phenomenon at a time (deformation induced lattice rotation or plastic strain or elastic strain etc.). The aim is to fully characterize samples non-destructively in one experiment, i.e. to analyze all the aspects of deformation induced phenomena in parallel experiments, hereby merging different techniques (plastic strain measurement by tomography; elastic strain and lattice rotation measurement by diffraction, using a large area detector; grain mapping by diffraction, using a high resolution area detector; precipitation and void characterization by small angle scattering; spectroscopy (in the limits of the instrumentally given energy range) e.g. on single intermetallics or precipitations). The availability of a sub micrometer focus combined with high resolution detectors and optical elements like spiral and conical slits (see Fig. 6.7.3) will push this kind of non-destructive in-situ investigations to the subgrain and nano crystalline level.

The beamline will provide the necessary infrastructure to perform experiments in the field of physics under extreme conditions (high pressures, high magnetic fields, low / high temperatures) to investigate e.g. phase transitions at high pressure or extreme temperatures. This is of particular interest in the field of high temperature superconductors.

Furthermore, the beamline design permits an easy accommodation of sample environments

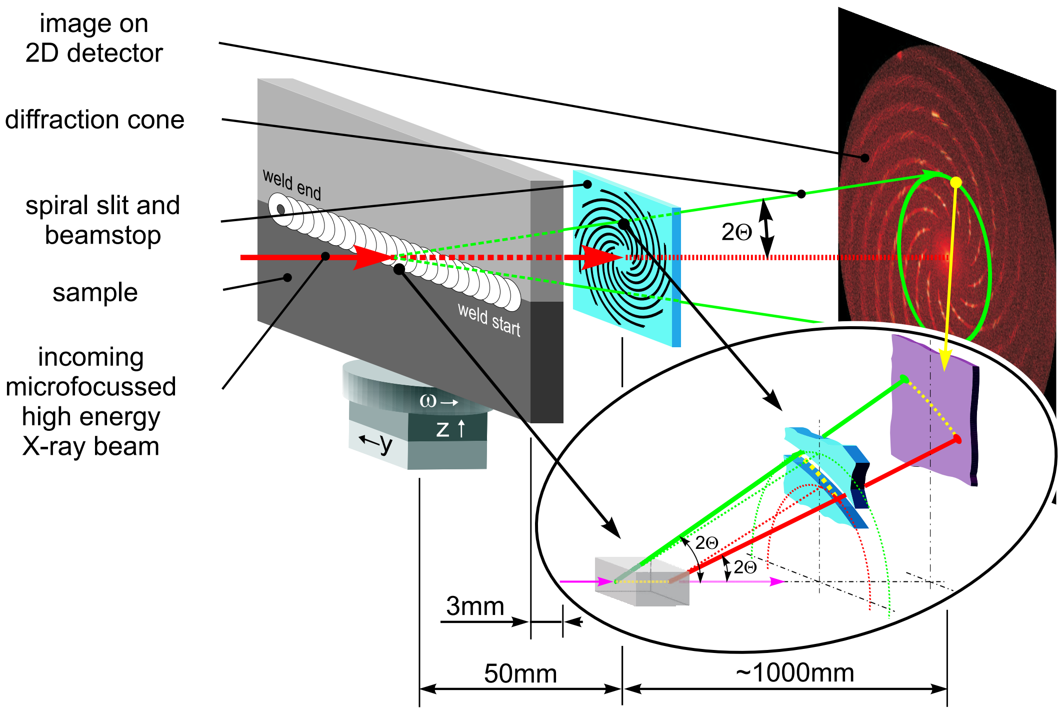


Figure 6.7.3: Schematic of setup with spiral slit system for non-destructive depth resolved phase and strain investigations. The inset visualizes the principle of the beam path which leads to the spatial resolution.

and optical components (e.g. monochromator) provided by the user, such as e.g. setups for the investigation of solid / solid and solid / liquid interfaces and diffuse scattering from binary systems (Reichert et al., 2000; Reichert et al., 2001). The beamline will be a powerful tool for analyses in rapidly emerging fields like biomimetics. Biomineralization and synthesis processes (e.g. nano crystalline bone material) can be investigated on a nanoscale due to the high resolution capabilities and low detection limit. The future development of new materials and materials processing will largely benefit from the high flexibility of this beamline.

### 6.7.3 Description of the beamline

The beamline layout as proposed at the moment is shown in Fig. 6.7.4. Characteristics valid for the whole beamline are:

- Temperature stability: experimental floor: 1 K, hutches: 0.1 K
- Beam height: 1.4 m above floor in order to accommodate heavy sample environments
- Pits with elevators in the experimental hutches to accommodate large diffractometers, samples, and sample environment
- Hutch height: experimental hutches: 5 m (cryostat), optics hutch 4 m
- Access to experimental hutches from the roof
- Manual cranes (> 1.5 t) below the ceiling in every hutch
- Media: biologically clean cooling water (15 °C), dry $N_2$, compressed air, He recovery line

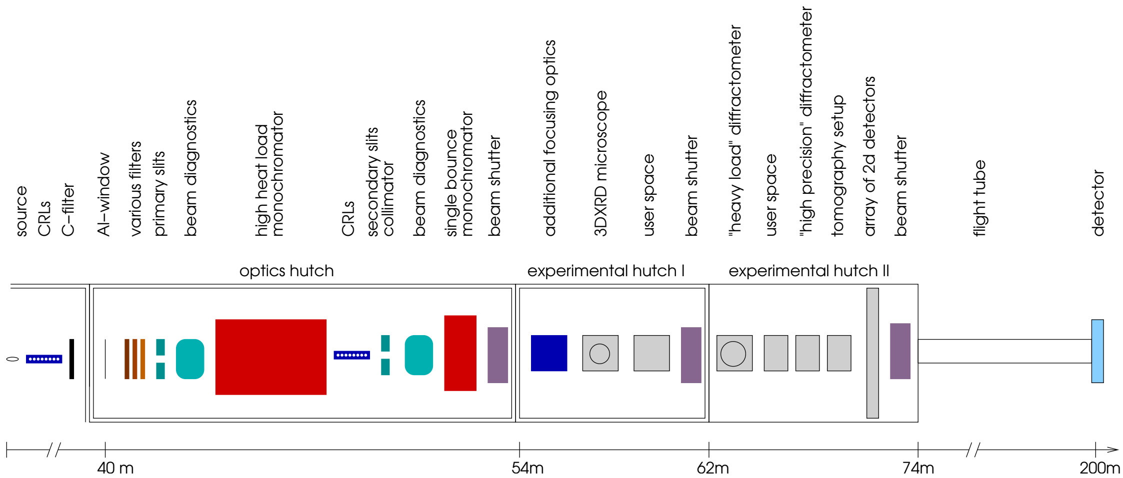


Figure 6.7.4: Schematic layout of the high energy materials science beamline.

- Special air outlet for engines (cf. 6.7.2.1), for experimental hutches only
- Decoupling of all hutches from PETRA hall floor
- Large hutch doors without barriers on the floor
- Network and power connections in every hutch

Besides the beamline itself, 40 $m^2$ for laboratory plus a storage area are needed. The lab has to be located in the direct vicinity. Office space for 4 scientists, 1 technician, and several students and postdocs is needed.

#### 6.7.3.1 Insertion device

The insertion device has to meet the following three high priority requirements of the beamline (discussed in detail below):

- Main energy of 120 keV
- Source suitable for sub-micron focusing at high energies
- Minimized heat load for optical elements

With an energy range between 50 and 300 keV the accessible energy range for hard X-rays will be substantially larger than for most of the existing beamlines. The X-ray energy is a compromise between the penetration depth necessary for bulk studies in most engineering materials (Al-, Fe-, Ni-, Co-base alloys) and the scattering angle respectively the angular resolution. Therefore, a main energy of 120 keV is proposed for the design of the insertion device.
The sub-micron focusing requires mainly a small source size, a small source divergence. Therefore the insertion device should have a source size below 57 x 10 $\mu m^2$ (H x V) FWHM (incl. source broadening) and a source divergence below 88 x 5 $\mu rad^2$ (H x V) FWHM.
A high flux is desirable for this device, but is of lower priority than the main energy, the source size, and the low heat load.
Typically a primary beam of about 1 $mm^2$ will impinge on the focusing optics, but in particular for fast grain mapping applications and tomography a primary beam with a substantially larger width (about 3mm) is favored.
Studies will be undertaken to examine the feasibility of a superconducting device.
Experiments using energies below 50 keV have to be performed at other beamlines.

#### 6.7.3.2 Beamline optics

Filters are foreseen to strongly suppress the energies below 50 keV and thus reduce heat-load problems on the optics. They will allow to operate all monochromators in air ambiance.
A unique specialty of the proposed beamline at PETRA III will be the high flexibility in shaping the high-energy X-ray beam, depending on the particular needs of the experiment. The beam type will range from a sub-micron focused pencil beam (e.g. for the investigation

of individual nanosized grains), over a line focus (e.g. for fast grain tracking) to box shaped beams with a homogeneous beam profile (e.g. macroscopic texture and strain analyses, tomography), hereby benefitting from the high photon flux, the small source size, and the very small beam divergence.
Beam focusing will be achieved with conventional bent monochromator and bent multilayer setup and with X-ray lens systems such as refractive X-ray lenses. The development of lens systems is currently progressing strongly and focusing spot sizes smaller than 1 μm are envisaged for hard X-rays. An important feature will be the availability of a fixed-exit focusing monochromator, thus allowing e.g. phase analyses with high spatial resolution using a conical slit cell. Compound Refractive Lenses (CRLs) that improve the parallelism of the beam will be installed in the front end, close to the source. For large beam sizes the shaping of the beam will be performed using slit-systems.
Optical elements that are part of the setups in the experimental hutches are conical and spiral slit cells, glas capillary arrays, and micro-channel-like plates made from tungsten. They ensure a high spatial resolution, within the sample, parallel to the beam (better than 50 μm). Further developments in this area are expected. Furthermore, refractive lenses can be used as analyzers in the diffracted beam.
Such a comprehensive system of beam types, slits and lenses for the different applications in materials science is so far not available at a single high-energy beamline.

Summarizing, several optics have to be considered:

- Slit optics: This is the simplest either in a white beam or through a monochromator setup.

- Scanning fixed-exit monochromator: The second crystal defines the incident beam condition by rotation to the appropriate Bragg angle only. The first crystal rotates and moves on a long translation stage and is fed back to the maximum transmitted intensity, thus eliminating the influence of rotational errors of the first crystal. The fixed-exit monochromator tunes through the whole energy range as defined above. Due to the small scattering angles, reflection condition is in asymmetric Laue geometry. A simple rotation of the inclination switches from the (111) to the (311) and eventually more reflections.

- Scanning fixed-exit focusing monochromator, maintaining focus size and position over a range of several keV, realized with bent Laue crystals.

- Several sets of crystals will be available such as perfect or oxygen precipitated and bent crystals. Different monochromators, placed besides each other, can be moved into operation by lateral translation.

- Single-bounce monochromator: This monochromator type in horizontal geometry will be foreseen for special purposes and the monochromatic beam will only go to the white beam hutch (experimental hutch I), allowing a high flexibility in the arrangement of the experimental setup in the hutch space as well as in focusing.

- Space will be foreseen for compound refractive lenses in the front-end, improving beam parallelism.
- For focusing purposes compound refractive lenses will be foreseen after the monochromator.

#### 6.7.3.3 Experimental hutch I

The first experimental hutch will be a white beam hutch with a space of $5 \times 8\,m^2$. It will permit the installation of user provided monochromators, increasing the flexibility for beam focusing and experimental setup design. All experiments using white beam can be carried out here. The main components of this hutch are:

- 3DXRD microscope.
- Additional focusing optics close to the 3DXRD microscope.
- A cooled beam stop at the end of the hutch.
- Beam available from single-bounce monochromator (see optics hutch).
- Space e.g. for a “user monochromator” and for user provided setups.

The single-bounce monochromator (situated in the optics hutch) will be operated in horizontal geometry. Its distance to the beam shutter has to be small in order to reduce the size of the horizontal shutter opening. The experiment itself can be set up in the first experimental hutch.
Even if the white beam hutch is initially not built at once, it has to be foreseen in the design for the static load for the whole beamline since from experience at the ESRF for safety reasons a radiation shielding thickness of at least 50–200 mm Pb is necessary which results in a significant floor loading. Preferential to the Pb shielding would be a concrete shielding of the white beam hutch, but the concrete shielding requires more space.

#### 6.7.3.4 Experimental hutch II

The second experimental hutch will have space for four experiments:

- A heavy load diffractometer which can carry a load up to 1000 kg for big samples and different sample environments (cryo magnet, press, tensile testing machine, furnace, etc).
- A smaller high precision diffractometer for micro beam applications.
- Space for user supplied experimental setups.
- A tomography setup which goes preferably to the end of the hutch using the largest possible beam size. The possible implementation of the flight tube (see fig. 6.7.4 and 6.7.3.5) will, therefore, also be of great advantage for these applications.

The heavy load goniometer will be optimized for materials science investigations. For analysis with a spatial resolution in the sub-μm range a high precision translation and rotation system will be installed. Until now (2003) there is no such instrument available at a synchrotron beamline. This device is supposed to enhance the precision in movement for larger samples and especially for large sample environments such as furnaces, devices for mechanical testing and thermo-mechanical testing. A further unique feature will be the combination of the synchrotron radiation laboratory with the above mentioned external materials testing laboratory (mechanical testing devices, furnaces, cryostats etc. ).
The second hutch will be at least 12 m long in order to achieve high angular resolutions and to accommodate all experiments. At the very end of the hutch 2-dim position sensitive detectors will be mounted. A novel multi-detector concept will enable simultaneous data collection for different diffraction angles.

#### 6.7.3.5 Flight tube

A unique and particular feature of the projected beamline will be the possibility to perform small angle scattering experiments at high energies on bulk samples, simultaneously with diffraction experiments. The feature distances in the micro structures to be investigated by small angle scattering typically range from 1 to 1000 Å (e.g. porous media, polymers, precipitates). To achieve a minimum angular resolution at 120 keV the area detector needs to be placed at least 200 m away from the source, i.e. at a distance of about 150 m from the first possible sample position. The detector chamber will be connected to the experimental hutch by a flight tube (see Fig. 6.7.4). Because of the resulting space constraints outside the experimental hall the beamline should occupy the third or fourth position in the experimental hall. Even if the flight tube is not part of the first construction phase the space required outside the experimental hall should be taken into account when decisions are made on the position inside the experimental hall.
With the option to perform on the same sample and the same volume element, parallel to diffraction experiments, in particular cases analyses by small angle scattering, tomography, and spectroscopy, the proposed beamline will be the first, where the boundaries between these analytical techniques are gradually disappearing.

#### 6.7.3.6 Detectors

The wide variety of experiments for this beamline is reflected in the particular need of various special detectors. In most cases the use of area detectors will be favored to realize the high data acquisition rates required for dynamic in-situ studies and to fully benefit from the high photon flux. Mainly three different types of area detectors for diffraction experiments can be distinguished:

- Large area detectors, allowing the monitoring of complete Debye-Scherrer-rings at large sample-detector distances (at least 2000 mm) for ultra fast strain and lattice rotation analyses.

- Array of large area wire-detectors which can be translated over a long range along the beam for high angular resolution, comparable to the HARWI II setup. In contrast to the other large area detector they can only monitor sections of Debye-Scherrer-rings.

- High resolution detectors (around 1 μm resolution) which, as a consequence, are placed close to the sample, e.g. for subgrain mapping.

For the small angle scattering experiments area detectors with an efficiency optimized for high energies are required Standard point detectors are required in case of spectroscopy analysis. Improvements in the field of detector development are expected within the next years.

#### 6.7.3.7 Redundant-beamline concept

The beamline will be equipped based on a ”redundant-beamline concept”, meaning that critical spare parts or a complete replacement exist for all crucial components of an experimental setup in case of failure. This concept relates to components such as critical sample translation and rotation units, detectors, fast beam shutters, sample environment, electronics etc. This spare pool does not necessarily need to be exclusively accessible to this one beamline. However, many of the critical parts will certainly be beamline specific such as detectors for high energy X-rays. The financial investment will be low in comparison to the loss of beamtime, considering the amount of lost manpower for several months of experiment preparation.

#### 6.7.3.8 Beamline control and data collection

To attract user communities, especially from the engineering sector, who are not familiar with synchrotron radiation, the concept for instrument control software and data evaluation will strongly involve the aspect of user-friendliness. Measurement techniques and analysis routines according to the VAMAS standards will be implemented, which will be of particular interest for industrial users. For scientists familiar with the techniques interfaces between the basic instrument control and data acquisition will be provided for the development of novel measurement and evaluation techniques. The use of SPEC as beamline control software is strongly favored. It ensures an easy compatibility of software and will facilitate the collaboration on this sector with other synchrotrons, e.g. the ESRF.

### 6.7.4 Capital investment and personnel

#### 6.7.4.1 Capital investment

The total investment costs for this beamline sum up to **5350** k€.

#### 6.7.4.2 Personnel

The projected beamline will be highly versatile in its application, merging different analytical techniques. This necessitates and justifies the employment of four scientists, one software engineer, one mechanical engineer, and one technician on a permanent basis.

Ideally the scientists are complementary in each others competences. Particular skills and knowledge are required in the fields residual strain analysis, texture analysis, powder diffraction, tomography, and high-energy X-ray optics.
A software engineer is a prerequisite for the smooth and efficient operation of the beamline. Most experiments carried out will highly rely on complex software for high-speed data acquisition and on-line data analysis. Furthermore, the beamline control requires a continuous improvement and maintenance.
The continuous improvement of beamline hardware, development and realization of new components and setups will be carried out by the mechanical engineer together with the technician, in close collaboration with the scientists.
The technical staff might be part of a pool for beamline software and hardware support. However, particularly in the start up phase of all the beamlines it is clear that these pools have to be sufficiently large (one engineer and technician per beamline) to avoid bottlenecks. It is emphasized here that dedicated PETRA groups of significant size will be highly appreciated for optics, metrology, detector support, computing, and beamline specific engineering tasks.