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The New GKSS Materials Science Beamlines at DESY: Recent Results and Future Options

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Abstract. GKSS is currently investing heavily into new beamlines at DESY in Hamburg, Germany. After the completed installation of the wiggler beamline HARWI II at DORIS III GKSS is now building two new undulator beamlines at the new PETRA III storage ring. The High Energy Materials Science Beamline (HEMS) will allow high resolution diffraction experiments using samples and sample environments with masses up to 1 t, 3DXRD measurements, and high-energy micro-tomography experiments. The Imaging Beamline (IBL) will provide a nano-tomography as well as a micro-tomography station for X-ray energies up to 50 keV. Examples of typical experiments in the field of residual stress analysis, micro-tomography, and high-energy small-angle X-ray scattering will be given.

Introduction

In engineering materials science X-ray scattering and micro-tomography using synchrotron radiation have over approximately the last 10 years evolved as an increasingly accepted and utilized technique for characterizing the internal structure of materials. This development is supported by the higher flux and increasing resolution in reciprocal and real space, which synchrotron radiation offers compared to laboratory sources. To make full use of these new possibilities, GKSS is currently constructing new beamlines at the neighboring DESY in Hamburg, Germany.

The GKSS wiggler beamline HARWI II at DORIS III is in routine operation since 2006 providing a large beam with a cross section in the range of several cm^2 for the investigation of large objects. At the new PETRA III storage ring two new undulator beamlines are currently under construction by GKSS. Due to the world record brilliance of PETRA III these beamlines will provide unique opportunities for ultrafast experiments at high resolution in real and reciprocal space using small beams down to the nm-range. The High Energy Materials Science Beamline (HEMS) will allow high-resolution diffraction experiments using samples and sample environments with masses up to 1 t, 3DXRD measurements yielding e.g. the 3D grain structure and strain map of metallic alloys, and a micro-tomography experiment optimized for X-ray energies above 30 keV. The Imaging Beamline (IBL) will provide a nano-tomography as well as a micro-tomography station for X-ray energies up to 50 keV. The two new PETRA III beamlines are scheduled to commence operation in 2009; routine user operation will begin in 2010. In the following, the three beamlines are briefly described and typical examples of experiments in the field of residual stress analysis, micro-tomography, and high-energy small-angle X-ray scattering are given.

The GKSS Wiggler Beamline HARWI II@DORIS

Description of the Beamline. The new high-energy beamline HARWI II is dedicated to texture, strain, and imaging measurements for materials science (Fig. 1). The monochromator tank in the optics hutch accommodates two different types of monochromators. The first is a double Laue monochromator in horizontal geometry for strain and stress analysis, which delivers beams of 10 mm × 10 mm in size. Using Si (111) crystals an energy range of 58–250 keV is available. In addition, a Si/Ge gradient crystal can be used in order to achieve a large bandwidth, i.e. high photon flux at high energies. The second monochromator is optimized for imaging experiments. It produces a beam size of up to 70 mm × 10 mm in vertical diffraction geometry. The energy range currently is 20–150 keV with an option for a range of 20–250 keV.

In the first experimental hutch (EH1) three pits are lined up along the beam. In the first pit there is a heavy-load diffractometer, which is designed for carrying heavy samples and sample environments up to 600 kg. The maximum sample–detector distance is 10 m. Thus, measurements with high angular resolution can be performed. An energy-dispersive Ge-detector, a scintillation counter, an image plate scanner, and a flat panel detector are available. A hydraulic INSTRON stress rig (100 kN) is available for performing in-situ experiments under load.

The second pit houses a complete micro-tomography setup. In the third pit a setup for simultaneous diffraction and tomography (DITO) is installed. The experiments in pits 2 and 3 can be lifted up into the beam within minutes, thus enabling a fast change between different experiments. In the second experimental hutch (EH2) there is a large press operated by the Geoforschungszentrum Potsdam.

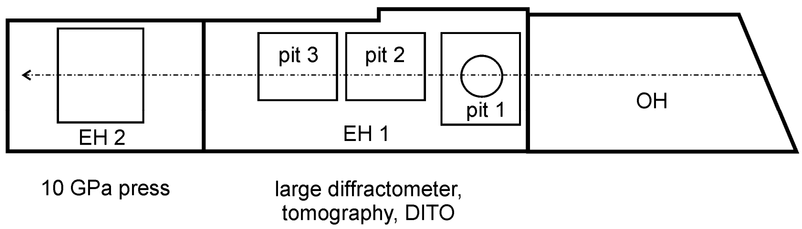


Fig. 1: Layout of the HARWI II beamline. There are one optics hutch and two experimental hutches.

Residual stresses in laser beam welded T-joints. The use of high-energy X-rays enables penetration of thick samples. Thus, investigations of real production processes and large parts become feasible. One example is the investigation of laser beam welds. Replacing rivets by laser beam welded (LBW) joints of aluminum alloys is a current topic in the aircraft industry, which has been particularly realized for skin-stringer joints in the Airbus A380, A318, and A340. The major advantages of LBW joints are reduction of weight and faster manufacturing. However, extension to T-joints of so-called clips to the skin is not yet achieved. Start (run-in) and end (run-out) of such short-distance welds are most prone to crack initiation. Therefore, the knowledge of residual stresses in these welds is important [1].

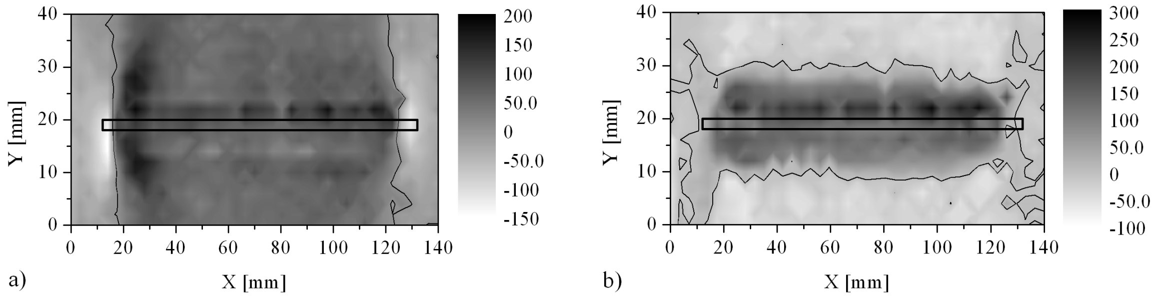


Fig. 2: RS maps for a laser beam welded T-joint, a) transverse direction; b) longitudinal direction. The rectangle marks the position of the clip; the solid line separates tensile from compressive stresses.

A sample with a 4.5 mm thick AA2139 base plate and a 2 mm thick AA6013 clip was measured using a photon energy of 78.6 keV and a beam cross section of 1 mm × 1 mm. The Al (311) reflection was analyzed at 41 × 21 = 861 points in an area of 140 mm × 40 mm. From these measurements, maps of residual stresses in longitudinal and transverse directions could be calculated (Fig. 2). Nevertheless, these maps reveal details of the stress distributions, e.g. at both weld ends, that can be compared with predictions of finite element models. Finally, ways of reducing residual stresses can be tested by modifying welding parameters and sequences.

The Micro Tomography Setup. Synchrotron radiation based micro computed tomography (SRµCT) is an established imaging method in the field of materials science. It combines the advantages of a non-destructive 3D visualization with high spatial resolution and a high density resolution due to the high-flux monochromatic X-ray beam [2].

The SRµCT setup is installed on a lift table in the second pit. It mainly consists of three parts: A CCD detector (14 bit, 1536×1024 pixels), a fluorescence screen converting hard X-rays into visible light, and a high-precision sample manipulator stage for positioning and rotation of the sample. The system is designed to operate with photon energies from 20–150 keV. The field of view can be adapted to the diameter of the investigated sample using optics with variable focus. Spatial resolutions up to 2 µm can be achieved.

As an example, SRµCT of a laser beam weld (LBW) of an aluminum alloy T-joint produced using a Nd:YAG laser [3] is shown. A small sample (∅ 6 mm × 30 mm) was prepared by cutting the weld region cylindrically out of the T-joint as shown in Fig. 3a. The field of view of the detector was adapted to cover the whole sample. The used X-ray energy was 40 keV and a spatial resolution of 10.5 µm and an effective pixel size of 6.8 µm were determined by the measured modulation transfer function for this measurement.

The tomogram showed an excellent density resolution and the different aluminum alloys present in the weld could be clearly distinguished [4]. The density difference between the aluminum alloy in the clip (top of the weld) and the alloy in the skin (bottom region of the weld) was below 5% and, thus, produced only a weak absorption contrast. Even the weld itself could be separated and made transparent for visualizing the pores inside the weld (Fig. 3b). These results demonstrate the possibility to visualize structures in relatively high absorbing materials with very high density resolution without artifacts.

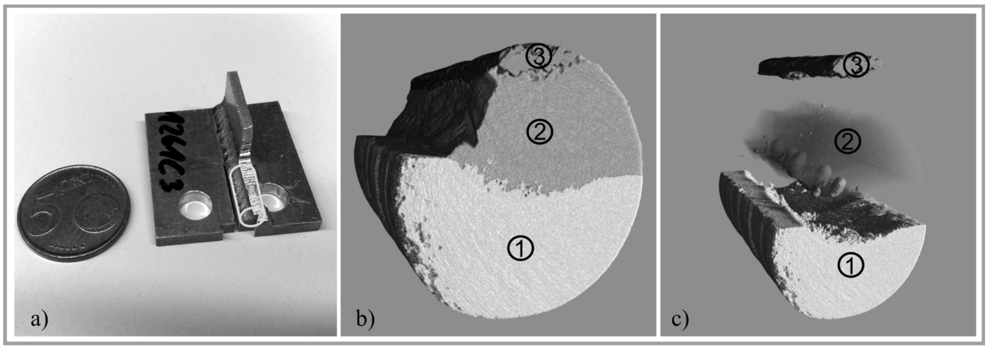


Fig. 3: a) Part of the LBW T-joint from which the cylindrical sample was cut. b) Volumetric rendering of the tomogram of the cylindrical sample. The different gray values represent the different alloys (bottom: AA2139-T3, top: AA6013-T6). c) The weld was made transparent to make the pores visible.

The New GKSS Undulator Beamlines at PETRA III

The HEMS Beamline. The future High Energy Materials Science Beamline HEMS at the new German high brilliance synchrotron radiation storage ring PETRA III will be fully tuneable in the range of 50–300 keV and will be optimized for sub-micrometer focusing with compound refractive lenses and Kirkpatrick-Baez multilayer mirrors [5]. The beamline will consist of a five meter in-vacuum undulator source optimized for high photon energies, a general optics hutch (OH1), an in-house test facility (EH1), and three independent experimental hutches working alternately (EH2–EH4), two with additional small optics hutches (OH2, OH3), see Fig. 4. The main optics are two monochromators – one Single Bounce Monochromator, one Double Crystal Monochromator with water-cooled bent Si (111) Laue crystals (40° asymmetric cut) in fixed-exit geometry – both horizontally deflecting with 1.4 m beam height above the floor. Lens change-boxes for compound refractive lenses will allow variable focusing with easy handling.

Experimental hutch 2, operated by DESY, allows general physics experiments. It will be equipped with a versatile diffractometer from HUBER in an improved design as set-up by H. Reichert et al. [6] at ID15A at ESRF, Grenoble, for the study of surfaces and deeply buried interfaces.

Experimental hutch 3 will serve the engineering community with its demand for handling large and heavy samples (turbine blades, motor blocks, etc.) or sample environments (welding and fatigue-loading machines, furnaces, or cryostats). The high brilliance at high X-ray energies at HEMS will facilitate the use of sample-environments for in-situ investigations e.g. of structural transformations during manufacturing processes. An in-situ friction stir welding machine is already in use at HARWI II and will also be used at HEMS; further in-situ experiments are planned. The work-horse for sample positioning is a hexapod for heavy loads of 1 t from Physik Instrumente with travel ranges of laterally 40 cm and vertically 20 cm, tilt angles up to 20°, and a resolution of 1 µm. As 2D-detectors, a mar345 image plate is available and a mar555 selenium-based flat panel direct conversion detector is currently tested.

In the last experimental hutch (EH4) two specialized instruments will share the space: a 3DXRD microscope for stress and strain mapping of polycrystalline materials [7] and a dedicated micro-tomography set-up. It will complement the existing set-up at the wiggler beamline HARWI II, which can cover the energy range 20–250 keV, and the imaging beamline IBL, which will operate in the range 5–50 keV. All stations will use air-bearing rotary stages with a wobble of less than 10^{-6} rad. The location and air conditioning will allow a stable beam in the 100 nm range.

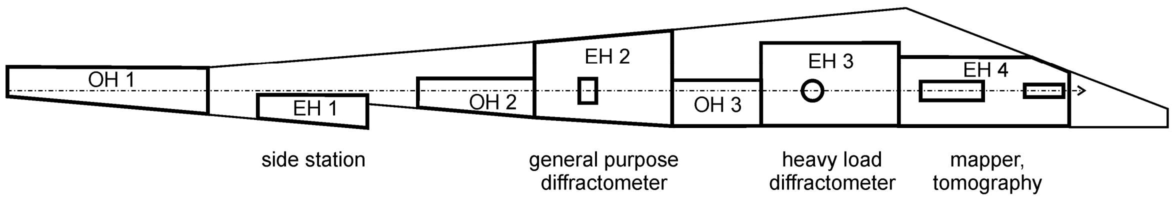
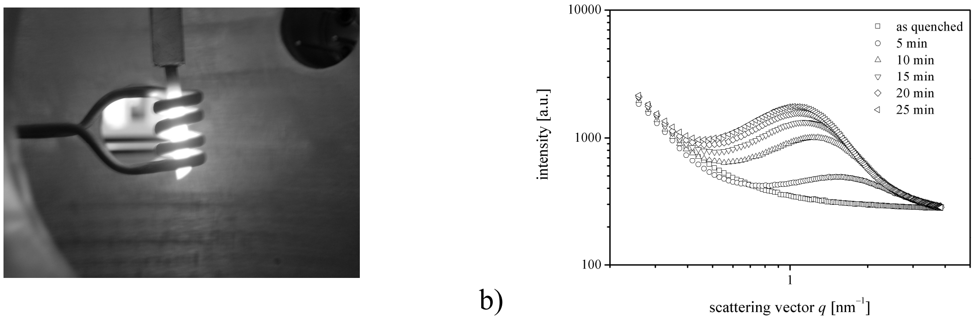


Fig. 4: Layout of the HEMS beamline. There are three optics hutches and four experimental hutches.

It is also planned to perform small-angle X-ray scattering (SAXS) experiments at HEMS using high X-ray energies. At higher energies, higher angular resolution is required for accessing small scattering vectors q. The accessible q-range can be increased by either using focusing elements to concentrate the beam on the beam stop or by using large sample–detector distances. The latter will be possible through large beam shutters enabling distances of up to about 30 m by putting the sample in EH2 and the detector in EH4. In combination with a fast induction furnace, which was built in cooperation with the Department of Physical Metallurgy and Materials Testing of the University of Leoben (Fig. 5a), such a setup will make studies e.g. of the early stages of decomposition reactions possible.

Experience in this field has already been gained at HARWI II. An example is the study of precipitate formation in a Fe-Co-Mo alloy [8]. Fe-Co-Mo alloys show a characteristic hardening mechanism, which is characterized by low hardness in the solution annealed state and a significant increase of hardness up to 70 Rockwell C during subsequent aging treatments. These extraordinary mechanical properties make the alloy a potential candidate for various high-performance applications. It is assumed that the strengthening is caused by precipitation of the nanometer-sized intermetallic μ phase from a supersaturated matrix. The formation and growth of precipitates can be monitored by an in-situ SAXS experiment as shown in Fig. 5b. The cylindrical sample had a diameter of 5 mm and a photon energy of 100 keV was used. The shift of the intensity maximum in the scattering curves indicates the growth of precipitates. With the higher brilliance at HEMS and a fast flat panel detector, also the early stage of this reaction will be accessible.



a) b)

Fig. 5: a) Heating coil of the induction furnace built in cooperation with the University of Leoben. b) Azimuthally averaged scattering intensity for a ternary Fe-Co-Mo alloy in the as-quenched state and isothermally aged at 450 °C for various times.

The IBL Beamline. Due to the extraordinary high brilliance of the new storage ring PETRA III, the extremely low emittance of 1 nm rad, and the high fraction of coherent photons even in the hard X-ray range, an extremely intense and sharply focused X-ray light will be provided. These unique beam characteristics will promote novel applications of tomographic techniques enabling ultra-fast in-situ measurements as well as highest spatial and density resolutions. Additionally, the highly coherent beam enables the application of phase contrast methods in an exceptional way.

To generate monochromatic X-rays, two types of monochromator will be installed in the optics hutch. A silicon single crystal monochromator designed by DESY will be used for tomographic methods, which need a very high monochromatization (e.g. vector tomography, absorption edge tomography, diffraction tomography). For applications that need particularly high flux (e.g. for fast in-situ experiments) a double multilayer monochromator will be designed in house. The energy range for both types of monochromator will be tunable between 5 and 50 keV.

The field of view in a large distance from the source will be large enough to investigate samples of some millimeters diameter in (sub-) micrometer resolution. To achieve spatial resolutions near the physical limit, high precision air bearing stages will be used for the sample rotation as well as for the camera movement. In addition a special sample positioning system was designed, which guarantees extremely low tilt errors of the samples.

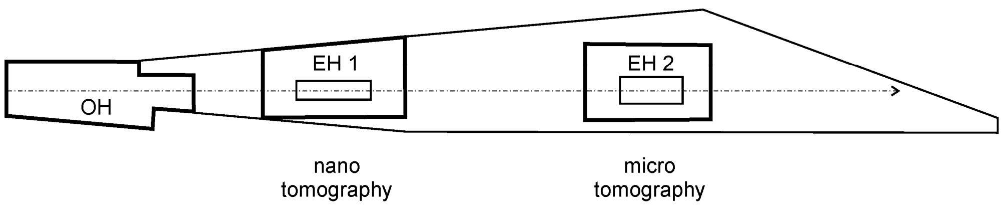


Fig. 6: Layout of the IBL beamline. There is an optics hutch and two experimental hutches.

The possibility to focus the X-ray beam on a nanometer scale enables nano-tomographic imaging. Two different dedicated nano-tomography setups are planned (Fig. 6) using compound parabolic refractive lenses and crossed cylindrical lenses [9, 10] to achieve a beam geometry for magnified imaging (Fig. 7). In both cases a spatial resolution below 100 nm is expected. Fields of application for this beamline will be materials science (e.g. imaging and quantitative analysis of pores, cracks, precipitates, grains, small components, phase transitions, and morphological transitions); biology and geology (e.g. plants, insects, stones, soil), as well as medicine (e.g. implants, structures of bones, tissues, and teeth).

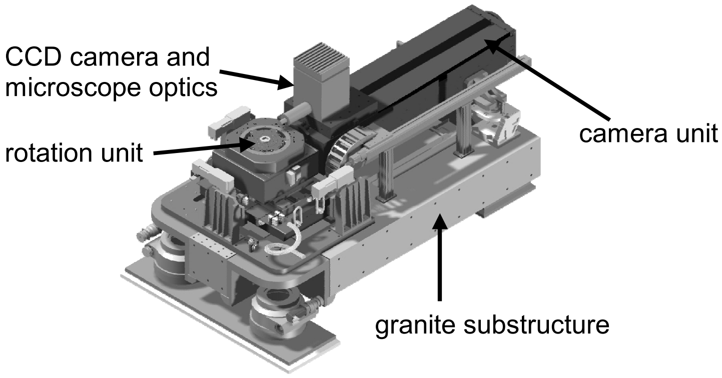


Fig. 7: Micro tomography setup composed of a rotation stage, a camera translation stage, and a granite substructure which carries the setup.

Summary

The new high-energy materials science undulator beamlines HEMS and IBL at the new third generation synchrotron source PETRA III, which provide beams in the mm to nm range, are optimized for high resolution experiments and will start user service in 2010. These are complemented by the wiggler beamline HARWI II offering a large beam cross section. Thus, GKSS is offering a suite of beamlines and corresponding experimental environments with a strong focus on the engineering materials science community.

References

[1] W. Machold, P. Staron, F. Bayraktar, S. Riekehr, M. Koçak, A. Schreyer: Mater. Sci. Forum Vol. 571–572 (2008), pp. 375–380.

[2] F. Beckmann, R. Grupp, A. Haibel, M. Huppmann, M. Nöthe, A. Pyzalla, W. Reimers, A. Schreyer, R. Zettler: Adv. Eng. Mat. Vol. 9 (2007), pp. 939–950.

[3] F.S. Bayraktar, P. Staron, M. Koçak, A. Schreyer: Mater. Sci. Forum Vol. 571–572 (2008), pp. 355–360.

[4] J. Herzen F. Beckmann, S. Riekehr, F. S. Bayraktar, A. Haibel, P. Staron, T. Donath, S. Utcke, M. Koçak, A. Schreyer: Proc. Society Photo-Optical Instrumentation Engineers (SPIE) Vol. 7078 (2008) 70781V.

[5] N. Schell, R.V. Martins, F. Beckmann, H.-U. Ruhnau, R. Kiehn, A. Schreyer: Mater. Sci. Forum Vol. 571–572 (2008), pp. 261–266.

[6] H. Reichert, V. Honkimäki, A. Snigirev, S. Engemann, H. Dosch: Physica B Vol. 336 (2003), p. 46.

[7] H.F. Poulsen: *Three-Dimensional X-Ray Diffraction Microscopy* (Berlin, Springer, 2004).

[8] G.A. Zickler, E. Eidenberger, H. Leitner, E. Stergar, H. Clemens, P. Staron, T. Lippmann, A. Schreyer: Materials Characterization Vol. 59 (2008), pp. 1809–1813.

[9] E. Reznikova, T. Weitkamp, V. Nazmov, M. Simon, A. Last, V. Saile: Journal of Physics: Conference Series (IOP), 9th Conf. on X-ray Microscopy, July 21–25 (2008) Zürich, Switzerland.

[10] V. Nazmov, E. Rezinkova, A. Somogyi, J. Mohr, V. Saile: Proc. Society Photo-Optical Instrumentation Engineers (SPIE) Vol. 5539 (2004) pp. 235–243.