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Materials Science Forum, Mechanical Stress Evaluation by Neutrons
and Synchrotron Radiation VI (2013)
Trans Tech Publications

DOI: 10.4028/www.scientific.net/MSF.772.57

Materials Science Forum Vol. 772 (2014) pp 57-61
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doi:10.4028/www.scientific.net/MSF.772.57

The High Energy Materials Science Beamline (HEMS) at PETRA III

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Keywords: beamline instrumentation, synchrotron radiation, high energy x-rays

Abstract. The HEMS beamline at PETRA III has a main energy of 120 keV, is tunable in the range 30-200 keV, and optimized for sub-micrometer focusing with Compound Refractive Lenses. Design, construction, and main funding was the responsibility of the Helmholtz-Zentrum Geesthacht, HZG. Approximately 70 % of the beamtime is dedicated to Materials Research, the rest reserved for “general physics” experiments covered by DESY, Hamburg. The beamline P07 in sector 5 consists of an undulator source optimized for high energies, a white beam optics hutch, an in-house test facility and three independent experimental hutches, plus additional set-up and storage space for long-term experiments. HEMS has partly been operational since summer 2010. First experiments are introduced coming from (a) fundamental research for the investigation of the relation between macroscopic and micro-structural properties of polycrystalline materials, grain-grain-interactions, recrystallisation processes, and the development of new & smart materials or processes; (b) applied research for manufacturing process optimization benefitting from the high flux in combination with ultra-fast detector systems allowing complex and highly dynamic *in-situ* studies of microstructural transformations, e.g. *in-situ* friction stir welding; (c) experiments targeting the industrial user community.

Introduction

PETRA III is the new German high-brilliance synchrotron radiation source on the DESY site in Hamburg with 14 new beamlines [1]. After the conceptual design in 2002 and the final approval of the project in May 2005, the reconstruction of the storage ring began July 2007. By September 2011, all beamlines started commissioning and the majority had already regular external users.

Several organisations collaborated to build and now run the various beamlines and enhance the infrastructure at the site. Design, construction, operation and main funding of the High Energy Materials Science Beamline HEMS is the responsibility of the Helmholtz-Zentrum Geesthacht, Centre for Materials and Coastal Research, HZG (formerly GKSS) [2]. Roughly 2/3 of beamtime is dedicated to Materials Research, the rest to “general physics” experiments covered by DESY.

Fundamental research encompasses metallurgy, physics, chemistry, biology. First experiments have investigated the relation between macroscopic and micro-structural properties of poly-crystalline materials, grain-grain-interactions, re-crystallisation processes, and the development of new and smart materials or processes. *Applied research* for manufacturing process optimization benefits from the high flux in combination with ultra-fast detector systems allowing complex and highly dynamic *in-situ* studies of micro-structural transformations. Experiments targeting the *industrial user community* are based on well established techniques with standardised evaluation, allowing "full service" measurements, as well as automated investigations of large sample numbers, e.g. for texture determination and tomography.

Beamline Characteristics

Source. Design parameters of PETRA III are an energy of 6 GeV, a current of 100 mA, and an emmittance of 1 nmrad (horizontally) and 0.01 nmrad (vertically). The source for HEMS will be a *4.5 m long in-vacuum undulator* (U19-5) *at a high-*β *position* optimized for high energies with *tunability 30-200 keV* [3]. Currently a 2 m long standard PETRA undulator is installed.

Floorplan. HEMS consists of a main optics hutch (OH1), an in-house *test facility* (EH1) and *three independent experimental hutches* (EH2, EH3 and EH4) working alternately, plus additional focussing optics hutches (OH2, OH3) with set-up and storage space for long-term experiments as sketched in Fig. 1. All experimental stations have their own *control cabins*, allowing independent experimental set up down the beam while experiments further up run with live beam. *Two side laboratories* (machine shop and cleaning facility) and *office space* in the 1st floor hall gallery complement the infrastructure.

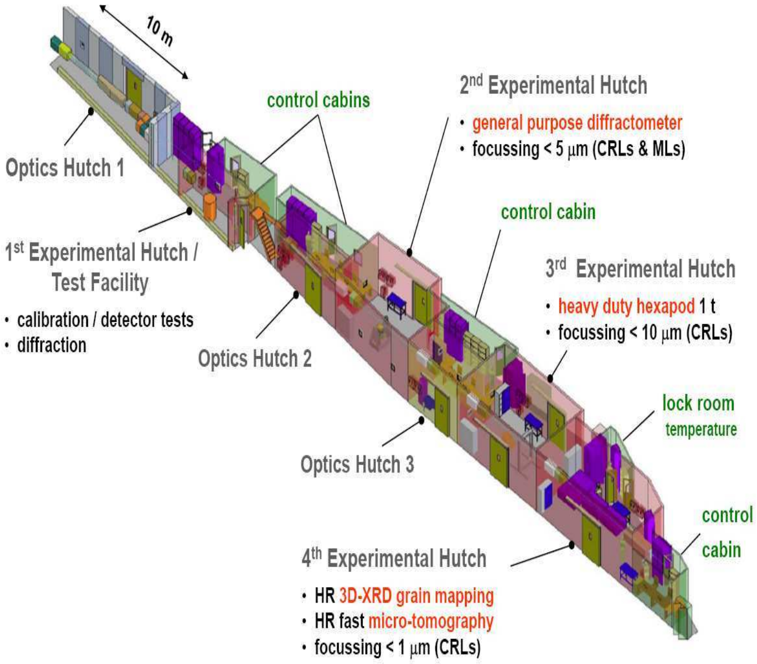


Fig. 1: 3D-view with main experimental stations of HEMS.

Optics. The optics for the side station (Test Facility EH1) consist of *two flat water-cooled Laue crystals Si(111) and Si(220)* 15 mm (wide) x 30 mm (high) x 1.5 mm (thick) on a lateral slide, with asymmetric angle 35.36°. The energy with this single bounce monochromator (SBM) can thus be changed between 53 and 87 keV (scattering angle fixed to horizontally 4.25°). The maximum beam size is 0.3 x 0.5 mm^2 with an integrated flux of approximately 7x10^9 photons / sec. 0.01% bw.

For all other experimental hutches, i.e. the main beam, the optics consist of *water-cooled bent Si(111) Laue crystals* on Rowland geometry (35.36° asymmetric cut, triangularly shaped with base 35 mm, length 89 mm and each 1.25 mm thick [4,5]) in fixed exit (horizontal deviation 21 mm) keeping the beam at 1400 mm height above the floor. The energy is tunable between 30 and 200 keV with this double crystal monochromator (DCM) in horizontal scattering geometry. At 100 keV the σ-sub-mm large beam showed an integrated flux of 4x10^{10} photons / sec. 0.2% bw. Both SBM and DCM had been custom-built by FMB-Oxford [6].

A multilayer filter box inside the ring tunnel cuts out low-energy harmonics. Lens change-boxes for Al Compound Refractive Lenses [7] in OH2 allow variable focusing with easy handling.

Instrumentation

The in-house Test Facility EH1 consists of versatile set-ups for general testing, detector calibration and "minor experiments". This can be done while experiments in the preferentially served main beam are running (absorption by the SBM crystals is negligible). EH1 is in high demand for quick access test experiments of all kinds and also serves an educational purpose by offering student tutorials. In practice there can always be found a gap position for an optimized main beam experiment which leads only to minor intensity reduction for the parallel beam use in EH1.

Experimental Hutch EH2 allows *"general physics" experiments*. It is equipped with a versatile diffractometer for up to 300 kg load from HUBER [8] in an improved design as set-up by H. Reichert *et al.* [9] at ID15A at ESRF, Grenoble, for the study of deeply buried interfaces. An additional tilt monochromator enables optionally precise structural investigations of free liquid surfaces [10]. An analyzer and detector stage on rails allows quick change for variable detector geometries with energy dispersive scintillators for high resolution and low background and a 2D 15 Hz read-out Perkin Elmer XRD 1621 [11], see Fig. 2 above.

Fig. 2: HEMS general purpose diffractometer with optional pre-tilt-monochromator (courtesy U. Rütt).

Experimental Hutch EH3 satisfies the *engineering community* with its demand for handling large and heavy samples or sample environments (welding and fatigue-loading machines, furnaces, cryostats). Due to the key property of hard x-rays, i.e. a large penetration length, this community can methodically profit from previous experience in neutron scattering. The much higher photon brilliance at HEMS, however, facilitates *in-situ* investigations of structural transformations during manufacturing processes. The work-horse is a custom built hexapod from PI [12] for heavy load of 1 t with positioning resolution of +/-1 µm (travel ranges tens of cm, tilt angles 15^{o}) in combination with an in-house built detector portal for various 2D-detectors, e.g. mar345 image plate and mar555 selenium based flat panel [13], or PE XRD 1621 [11], see Fig. 3.

In the last Experimental Hutch EH4 two specialized instruments share space (access via a *temperatur lock*): a *3D-XRD microscope for stress and strain mapping* of polycrystalline materials – its principle analogous to the pioneering work of H.F. Poulssen [14], and a dedicated *micro-tomography* set-up identical to the one at the Imaging Beamline P05 which will operate in the complementary energy range 5-50 keV. All stations use rotary air-bearing stages with a wobble of sub-microradians [15]. The location and separate climatisation should allow a stable focus down to the 200 nm range. Figure 4 shows the final design for the micro-structure mapper which will be commissioned in the winter shut-down 2011/2012. First tomograms had been taken in April 2011. By switching to low-β mode a FWHM beam size of 0.9 (v) x 6 (h) mm^{2} should be reachable at the tomography station at the end of the beamline (100 m distance to source).

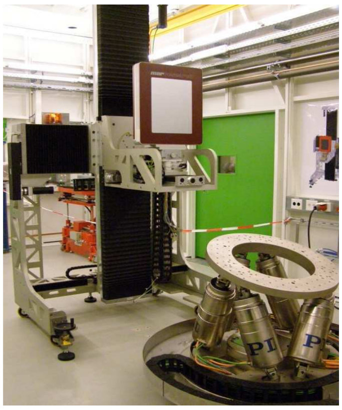
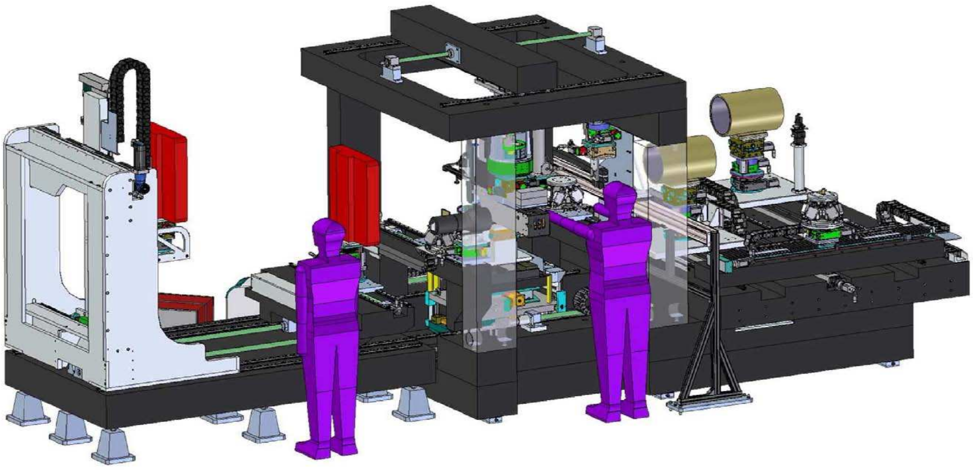


Fig. 3 (left side): HEMS heavy load hexapod with detector portal.

Fig. 4 (below): 3D-XRD micro-structure mapper with sample-handling in the center part, shielded optics to the right (with free space shown for Kirkpatrick-Baez Multilayer mirrors or Compound Refractive Lenses) and various near-field (3D x-ray detector from Risø [16] or Photonic Science VHR 11 megapixel CCD [17]) and far-field detectors to the left (mar345, mar555).



Experiments

HEMS "saw first light" in 12/2009, performed first experiments in 05/2010, took external reviewed users in 06/2011, and has been in regular user mode since 08/2011. Typical experiments comprise residual stress mapping of various welds in steels (also depth resoved via conical slits), texture studies of all kinds in heavy atom materials, structure studies of metallic glasses during fast heating, and exotic applications of diffraction investigations of dinosaur eggshells or combined diffraction and fluorescence studies of bronze artifacts. Fig. 5 shows exemplary the first tomogram of HEMS and the first grain map in pre-tests for the mapper instrument.

Fig. 5: First tomogram at HEMS of a Nb3Sn superconductor with scan volume 1 mm^3 at 70 keV (left), first centre-of-mass grain map of undeformed Al at 60 keV (right) – 14 cuts á 50 µm, measured with beam size of 0.05 x 1.0 mm^2. 128 indexed grains are shown as spheres with diameters reflecting relative volumes and colours corresponding to orientation (position accuracy 30 µm, orientation accuracy 0.15^o, relative error of volume 35 %, courtesy J. Oddershede).

Summary

The HEMS beamline at PETRA III is optimized for the key properties of hard x-rays in the tunable range 30-200 keV – large penetration depth, negligible extinction and Bragg scattering, large Ewald spheres – to investigate non-destructively bulk properties or deeply buried structures mainly in the context of Materials Research, but versatile enough to allow experiments in the merging fields of physics, chemistry and biology. Fast 2D-detectors together with varying focus spots down to 200 nm in the future will allow highly dynamic *in-situ* experiments of micro-structural transformations. It can handle large and heavy user provided equipment. Two specialized experiments will allow the detailed characterization of grains, their interaction and their stress and strain states with sub-micrometer resolution, as well as tomographic imaging.

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