Industrial Applications with Synchrotron Radiation

J. Homeyer, A. Webb, T. Wroblewski and K. Wurr

Introduction

Synchrotron radiation and the variety of experiments available at HASYLAB provide in many cases the key to solve problems arising from technological challenges in industrial research or development. It is the aim of DESY-HASYLAB to support applications of synchrotron radiation in collaboration with industrial partners. An industrial service group co-ordinates access to the laboratory and the wide range of techniques available. The group provides contacts for the companies, defines the technique to solve the problem in question and helps in to carry out the measurements. To organise industrial collaborations, DESY has created the *HASYLAB co-operation model*.

In the co-operation model, rapid access to beam time can be provided and the publication of the results, which is otherwise mandatory for all experiments at HASYLAB, may be delayed or fully omitted. The service group co-ordinates the preparation of the measurements, can provide help during the experiment and in many cases can provide data analysis. To offer highest flexibility and best service HASYLAB promotes the idea of long-term engagement with the industrial partners. Within the three-year contracts, more knowledge is gathered about the specific problems under investigation and about synchrotron radiation techniques and their possibilities and limitations. In this way the collaboration becomes more effective. Furthermore, we can guarantee full service and fast access to beam time for long-term co-operation projects.

The *HASYLAB co-operation model* distinguishes between industrial basic research, applied research and proprietary research.

For basic research companies can apply for beam time in the same manner as universities or research institutes. The proposal will undergo peer reviewing, beam time will be allocated on a long-term planning basis and the results have to be published. For basic research, no fees for the usage of the facility will be charged. The service group provides help in the selection of the technique and the preparation of the proposal.

For applied research projects companies may request rapid access to beam time. The proposal is not peer reviewed and only safety issues must be declared in advance. Results or reports of these measurements have to be published. In a long-term co-operation contract, the publication can be delayed. For applied research projects the use of the facility and its instruments is charged on an hourly basis.

In proprietary research projects, publication of results is not required. Approval and rapid access to beam time are handled as for applied projects. The charges for beam time are higher than for applied research projects.

Current projects

Industrial research comprising three long term projects, two continued and one started in 2005, using X-ray absorption spectroscopy were carried out in 2005. The long term projects are all dealing with the *in-situ* characterization of catalysts and were carried out at the beamline X1. The projects used both *in-situ* and *ex-situ* measurements under a wide range of conditions. Measurements were done from liquid nitrogen temperature to over 500°C. Samples were in air or under pressure with mixtures including H_2O, H_2, H_2S and inert gases. One project was working on MoS_2 based hydrotreating catalysts, supported NiRh catalysts for catalytical partial oxidation of methane, and CuPt supported catalysts for water gas shift. The projects were interested in the treatment of fuels for energy production and use in automobiles. The companies involved are interested in areas such as oil and gas, automotive catalysts, biofuel, precious metals products and catalysts and environmental issues such as the treatment of fuel and the exhaust gases.

Three short term projects have been performed at beamline G3 using the set-up for diffraction imaging [1]. A micro channel plate in front of a CCD detector acts as 2D-collimator suppressing crossfire of the radiation diffracted at different locations of the polycrystalline sample which is illuminated by a broad X-ray beam. In this way, images of the scattered intensity in the direction of the channels are obtained yielding spatial resolved information of the structural properties.

One of these projects triggered the development of a novel measuring strategy. The samples, which are normally used in a tension jig, were cylindrical shaped and treated by sand paper to simulate wear. This treatment produced grooves around the circumference where residual stresses were expected. Due to the cylindrical shape the angle ψ of the incident radiation to the surface normal varies continuously. This effect was used to perform a $\sin^2\psi$ measurement of the entire sample in a single 2θ scan.

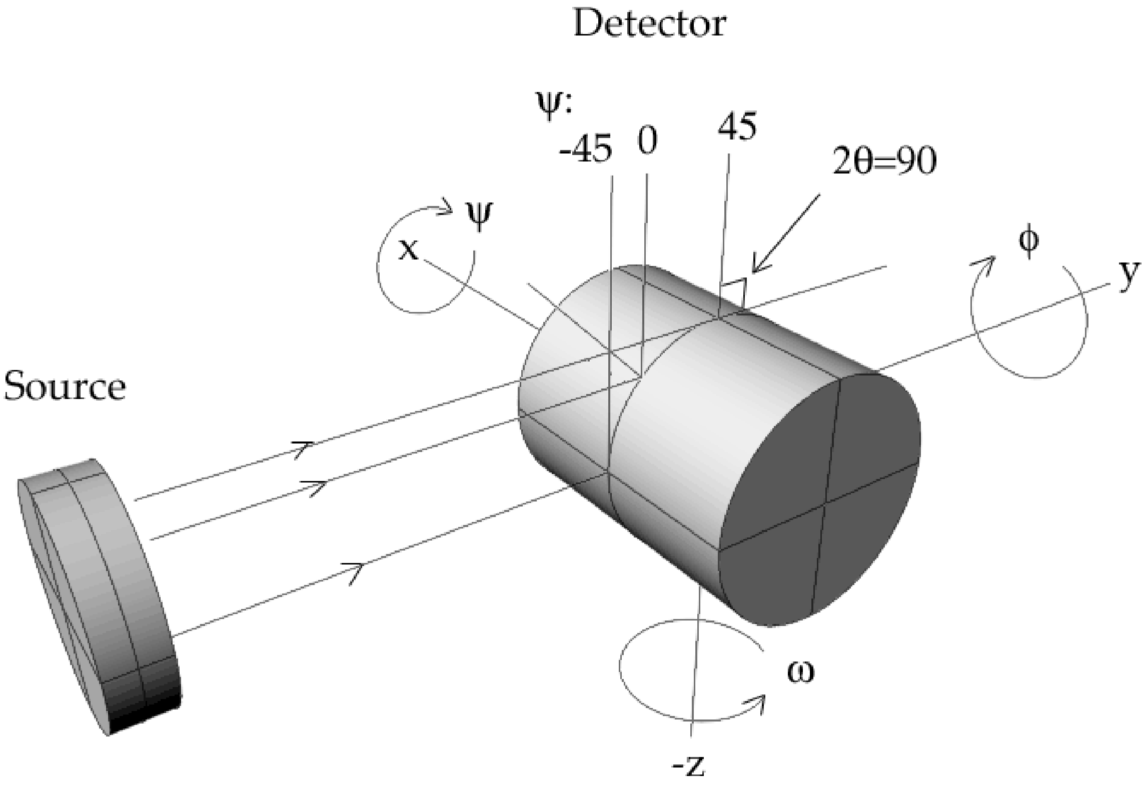

Figure 1: Scheme of the experimental geometry at G3. The sample is continuously rotated around the x-axis. Variation of the ψ-angle is automatically achieved by the cylindrical sample shape. The CCD detector collects the diffracted intensity from the upper front quarter illuminated by the beam yielding an image as function of the y coordinate (and thus as function of ψ) and the x coordinate along which the variation of residual strain is measured. (Figure by courtesy of S. Ås)

In the second project the influence of irradiation on novel fuel elements for the high flux reactor in Munich was tested. These samples which had been irradiated with heavy ions at the GSI in Darmstadt showed phase transformations dependent on the position in the ion beam. The data evaluation was done by André Rothkirch applying remote sensing strategies on the diffraction imaging data and by Carsten Baehtz doing Rietveld refinement.

The third experiment at G3 complemented investigations on mineralogical samples and standards performed at the fluorescence spectroscopy beamline L. Measurements at beamline L have a high sensitivity and allow the detection of very low elemental concentrations but require time consuming scans for spatial resolved investigations. The imaging method at G3 has only medium sensitivity (due to limited angular acceptance and no energy discrimination by the detector) but can be applied for mapping of the major components and in cases where impurities show high local concentration. It could therefore be used to check the homogeneity of the standards and to map the distribution of the main elements in geological samples. The investigations at beamline L also included XRF and XANES studies on fluid inclusions which were performed by Karen Rickers.

After a decline in the number of projects in industrial application of protein crystallography by EMBL and MPG/ASMB in 2004, during this year again several projects at the EMBL beamlines have been carried out. However, the industrial applications in this area at HASYLAB have been decreasing over the last years. This is in contrast to the growing use of 3rd generation synchrotrons, like the ESRF, where protein crystallography for industrial customers meanwhile accounts for a relatively large share of the activities. The high brilliance of such sources allows for a much faster throughput of protein samples. Thus, structure determination of proteins seemingly may be achieved much more efficiently than at 2nd generation light sources. This could mean on the one hand that the number of projects in industrial application of protein crystallography at HASYLAB will only increase again significantly with the availability of PETRA III. Other options towards the aim of carrying out more industrial projects might include actions directed at improving the cost effectiveness of these measurements.

Further activities

Several feasibility studies were performed, including topography for the semiconductor industry and residual stress analysis for the aerospace industry.

Julia Wienold, who was responsible for X-ray spectroscopy, left the service group in the beginning of 2005. This part has now been taken over by Adam Webb, who joined the group in September. Edmund Welter and Karen Rickers took care of the industrial users in the interim. Peter Kappen, a former member of the service group, has now joined the industry group of the Australian synchrotron radiation project Boomerang. The fluorescence investigations at beamlines G3 and L and a feasibility study on the de- and remineralisation of teeth have been performed in collaboration with this group.

The ‘Industrieforum’ series, organized in collaboration with ANKA GmbH and BESSY GmbH, is an event targeted at industrial interests. It was initiated in November 2003 at the DESY site and continued at the BESSY site on 12th November 2004 with a session dealing with ‘Nanofabrication’. A continuation of this series is planned for spring 2006 at ANKA on the topic of ‘Imaging’.

Members of the service group participate in the activities of the virtual institute ’Photons and Neutrons Research on Advanced Materials’ which is strongly focussed on problems relevant to industry. In the framework of this institute, an autumn school (see this annual report) was organized with speakers and participants from industry including a presentation of results from the collaboration of DESY with the company Framatom by H.J. Sell.

Further information about industrial applications of synchrotron radiation at HASYLAB, its conditions and selected examples can be found in the leaflet “Service Group Industry – Your Partner for Research” or on our web presentation: www-hasylab.desy.de/industry-cooperation/main.htm.

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References

[1] T. Wroblewski et al., A new diffractometer for materials science and imaging at HASYLAB beamline G3, Nucl. Instrum. Meth. **A428** (1999) 570-582