



	Experiment title: Ultra fast micro-imaging in two and three dimensions	Experiment number: MI-935
Beamline: ID15a	Date of experiment: from: 11th Feb 2009 to: 17th Feb 2009	Date of report: 10th Mar 2010
Shifts: 18	Local contact(s): M. Peele	<i>Received at ESRF:</i>
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Report:

The proposal MI-935 was submitted as Long Term Project in order to develop high speed imaging in two and three dimensions. The LTP was not approved but 18 shifts were allocated as a standard proposal. Due to this circumstances, all aspects which relate to a long term development could not be covered. We focused therefore our efforts on several selected feasibility studies as basis for potential further developments. Splitting the beam time into two sessions was not possible as stated by our local contact.

During our previous experiments (MA-485 as well as ME-1237) we successfully applied CMOS cameras in combination with indirect high resolution X-ray imaging detectors in order to perform microradioscopy. We reached a spatio-temporal micro-resolution, applied to study ultra-fast processes in metallic foams (cell wall collapses). A known drawback of CMOS cameras in comparison to CCD-based cameras is their poor dynamic range (commonly less than 1000 graylevels for CMOS, more than 10 000 graylevels for scientific CCDs). Their application for microtomography is discussed due to this circumstances. It is not proven if the combination of CMOS cameras and white beam will allow one to study multi-constituent specimens in a senseful manner.

In order to proceed further the high speed imaging using a white, hard and intense beam plus the corresponding heat load, dedicated indirect detection optics as well as scintillating screens have to be available. We used the beam time to perform extensive tests on a novel scintillating material based on a modified LSO-crystal (Lu_2SiO_5) doped with Tb. The

material was developed as part of the EC FP6 project ScinTax (STRP 033 427, <http://www.scintax.eu/>). Furthermore as part of this project, a novel white beam optic was developed which we could intensively test as well, leading to several major modifications.

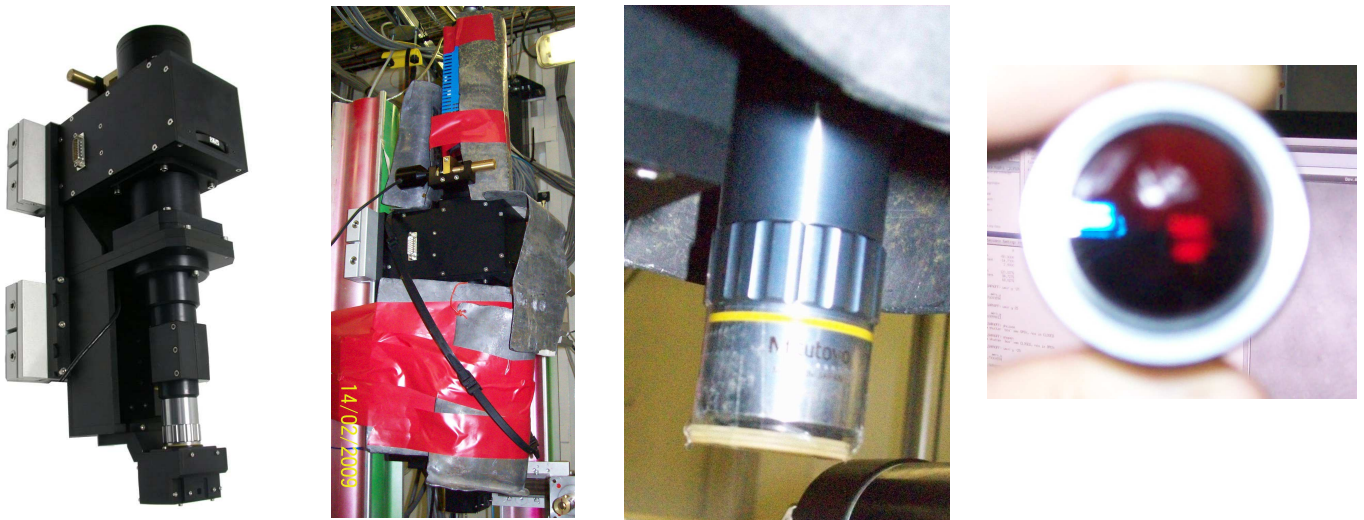


Fig 1: From left to right: the white beam optics as designed by OptiquePeter during the ScinTax project; the installed system at ID15a incl. the required radiation protection shielding; lead glass to protect the optics from radiation scattered by the mirror; burned objective due to the high amount of hard X-ray photons scattered by the mirror.

For testing the OptiquePeter optics developed for X-ray imaging with hard spectrum and a high heat load, we noted that the current radiation protection is not sufficient in order to apply the system as it is. Lead shielding have to be installed as well as protective lead glass to prevent damages from the visible light optics – cf. Fig 1.

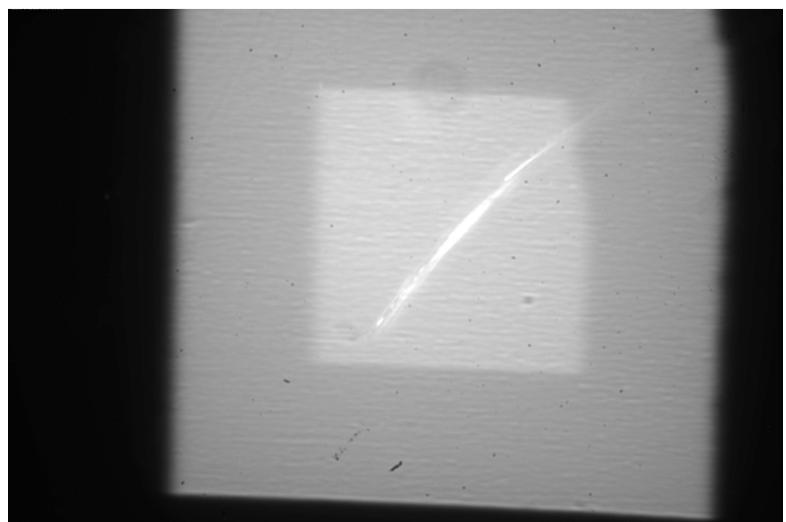
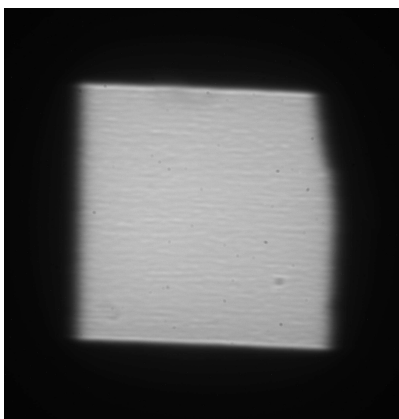


Fig 2: Left: Flat field image with LSO 25 and 10x objective plus 0.5 x 0.5 mm² slits. Right: Flat field image with LSO 25 and 10x objective plus 1 x 1 mm² slits.

The LSO:Tb material seems to be fitted for high heat load X-ray white beam imaging. The material does not stop to scintillate despite the high heat load, it does not crack as well nor does it shows the creation of color centers.

During the radiation damage test with the LSO an interesting effect was observed: after 48 minutes of irradiation with 0.5 x 0.5 mm² open slits we have opened the slits up to 1 x 1 mm². Then, after 7 minutes more we have further opened the slits up to 1.1 x 1.1 mm². As we have enlarged the X-ray beam we could observe the image of the preceding used slits (Fig. 2).

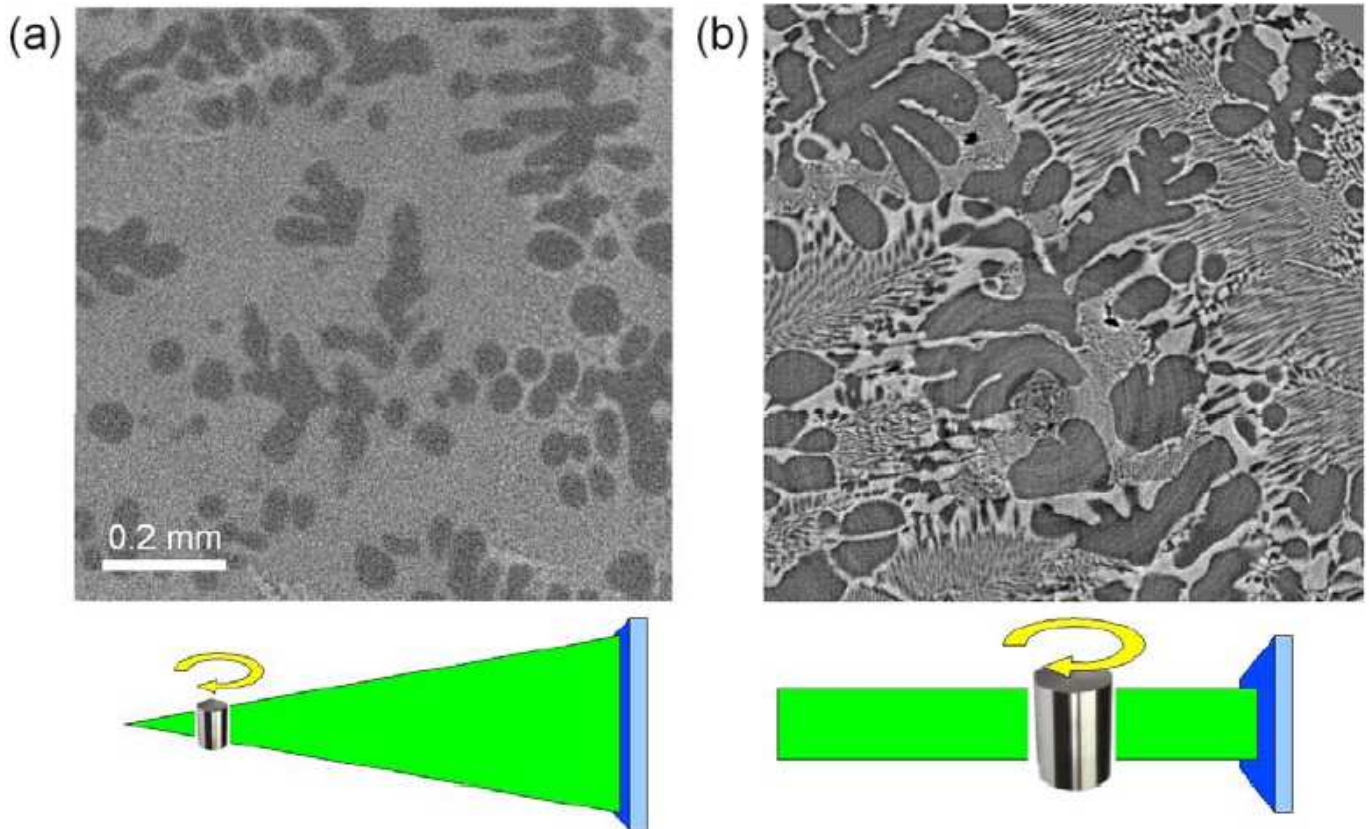


Fig 3: Comparison of (a) microtomography with cone beam geometry using a laboratory X-ray source (W anode, 100 kV, 1.56 μm pixel size) and (b) microtomography with a parallel beam and synchrotron radiation (ID 15A, ESRF). Shown is a Al-Cu alloy (30 mas%), 0.2 mm diameter. Sample kindly provided by L. Ratke and G. Kasperovich (DLR Köln) [1,2].

For our future applications in materials research we need detailed knowledge about the quality of image information when using polychromatic hard X-ray synchrotron radiation. A comparison between a lab investigation and a test image done at ID15a is displayed below. It demonstrates the superior high image quality of a synchrotron volume image despite the usage of white beam (Fig. 3).

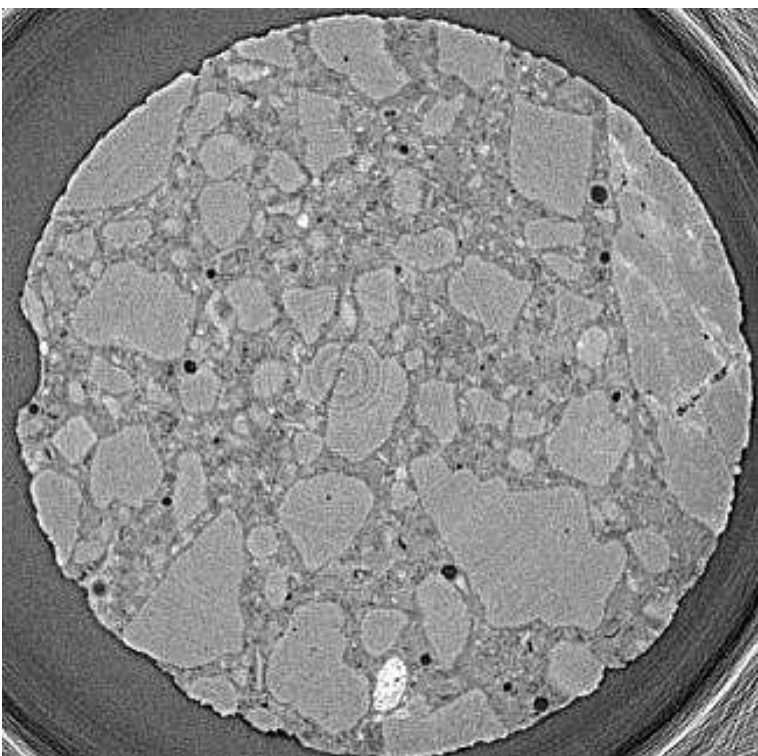


Fig 4: MicroCT of a concrete sample using a CMOS camera with 800 graylevels dynamic range as detector.

In order to display the potential of CMOS cameras for time-resolved microtomography, despite of their poor dynamic range, a CT scan was performed employing a Photron CMOS camera SA1 (1000 x 1000 pixels, each 20 μm in size, 800 gray levels dynamic range). The result is displayed in Fig 4. 1000 radiographic projection images were acquired during 4 s exposure time with a continuous rotation of the sample (no triggering involved). Despite the poor dynamic range, different material constituent are distinguishable clearly. We note that with CMOS cameras microCT is performable. Hence, CT with time-resolutions below 1 s is feasible.

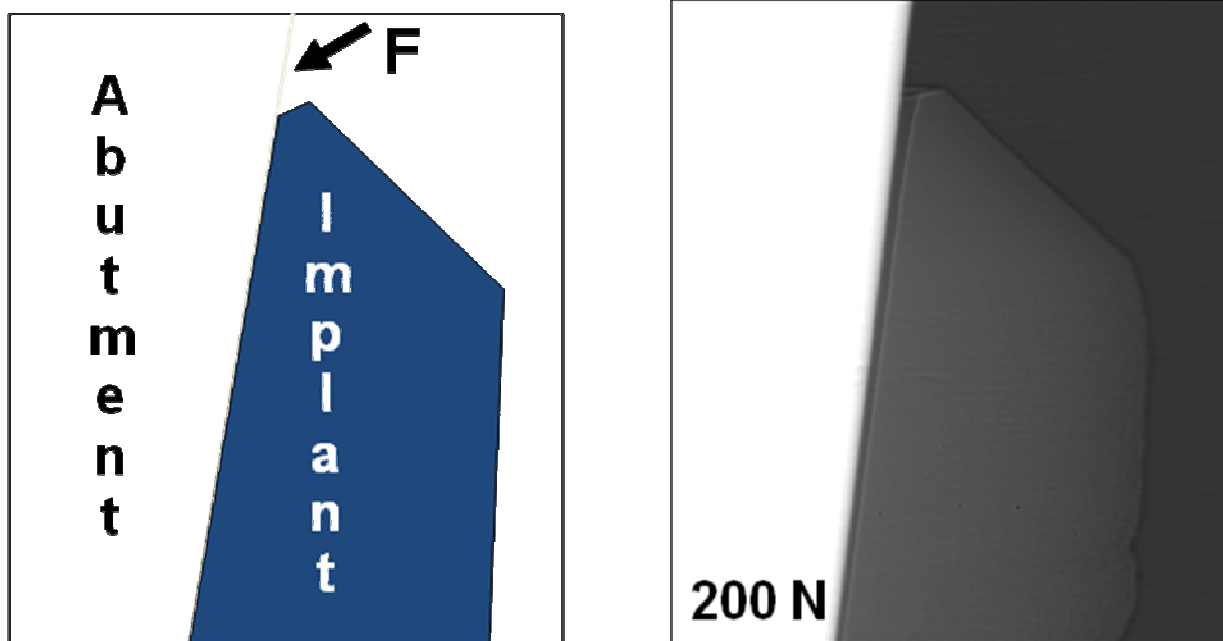


Fig 5: High-resolution radiographic image of the micro-gap formation at the implant–abutment interface for 200 N mechanical load.

Furthermore, micro-gap formation at the implant–abutment interface of two-piece dental implants was investigated in vitro using high-resolution radiography in combination with hard X-ray synchrotron radiation. Images were taken with the specimen under different mechanical loads of up to 200 N. The aim of this investigation was to prove the existence of micro-gaps for implants with conical connections as well as to study the mechanical behavior of the mating zone of conical implants during loading. The first illustration of a micro-gap which was previously indistinguishable by laboratory methods underlines that the complex micro-mechanical behavior of implants requires further in vitro investigations – Fig 5. Results obtained were not publishable but initialised further successful studies [3].

A continuation of our high speed radioscopy work was not possible as we found the required detector optics fully dismantled / out of order when we arrived at the beamline. Performing microCT with higher speed as shown in Fig 4 was not possible due to an intervention of the beamline staff.

References:

- [1] S. Zabler, H. Riesemeier, A. Rack
Röntgenbildgebung am Synchrotron: Radiographie und Tomographie
Proc. DGZfP-Jahrestagung Münster, Germany, DGZfP Proceedings BB 115-CD (2009)
- [2] S. Zabler, A. Rack, F. Garcia-Moreno, A. Ershov, T. Baumbach, J. Banhart
Imaging fast processes in liquid metal foams and semi-solid alloys using synchrotron radioscopy with spatio-temporal micro-resolution
Proc. 1st International Workshop "In-situ Studies with Photons, Neutrons and Electrons Scattering" (submitted 2009)
- [3] A. Rack, T. Rack, M. Stiller, H. Riesemeier, S. Zabler, K. Nelson
In vitro synchrotron-based radiography of micro-gap formation at the implant-abutment interface of two-piece dental implants
Journal of Synchrotron Radiation, vol. 17, part 2, 289-294 (2010)