Synchrotron-based radioscopy with spatio-temporal micro-resolution using hard X-rays

Alexander Rack, Francisco García-Moreno, Oliver Betz, Simon Zabler, Christian Schmitt, Tomy dos Santos Rolo, Alexey Ershov, Tatjana Rack, Lukas Helfen, John Banhart, and Tilo Baumbach

Abstract–**The use of highly intense synchrotron light sources allows the next step in the fast imaging development: the use of hard X-rays. Micro-radiography as an established method to image the internal structure of an object with micrometer resolution can be extended to study its temporal evolution as well. While direct converting pixel detectors are known which can acquire images with high frame rates here detectors are needed with higher spatial resolution which can stand the highly intense synchrotron photon flux. Our approach is based on indirect pixel detectors which are already known for micro-imaging at synchrotron light sources. We combine those with CMOS cameras in order to achieve frame rates of up to 40 000 images per second, thus progressing to micro-radioscopy. Potential applications are studies of living insects with moderate frame rates up to 250 images per second (4 ms exposure time), velocity fields within a semi-solid alloy during a thixo-casting process and ruptures of individual cell walls in a liquid metal foam imaged with up to 40 000 frames per second (25 µs exposure time).**

I. Introduction

The outstanding scientific value of time-resolved imaging is known since the famous high-speed movies of living insects by Lucien Bull [1]. In order to make visible internal structures of opaque objects the use hard X-rays instead of visible light is required. For X-ray imaging, synchrotron light sources offer a photon beam that a) propagates quasi-parallel, b) allows one to exploit more sophisticated contrast modalities, and c) has flux densities that are higher by orders of magnitude compared to laboratory sources. The application of synchrotron radiation is therefore the next step in the fast micro-imaging development: hard X-ray radioscopy with high spatio-temporal resolution and different contrast mechanisms [2]-[7], [17], [28]. Due to the penetrating nature of the radiation imaging with X-rays offers the chance to investigate kinematics hidden in opaque objects. Examples are feeding and locomotion devices of animals or coalescence events in evolving foams.

In this article we will introduce experiments where we performed X-ray radioscopy with high spatio-temporal resolution up to the micro-scale.

II. Instrumentation

Experiments were carried out at the beamlines TopoTomo (ANKA light source, Germany), ID15a and ID19 (European Synchrotron Radiation Facility (ESRF), France) [4], [5], [8]. For all beamlines their corresponding white beam mode was chosen (filtered with Al or Si attenuators) in order to reach a photon flux density high enough to perform micro-radioscopy.

A suitable X-ray pixel detector for our applications should combine high spatial resolution, high detection efficiency and high acquisition speed. Hybrid pixel detectors or Charge-Coupled Device (CCD) cameras which are used in direct X-ray imaging are limited in terms of pixel size, radiation resistance and x-ray stopping power [9]-[11]. Therefore we apply indirect pixel detectors which were first introduced in 1975 for live X-ray topography [12]. During the 1990s the concept was developed further for synchrotron-based micro-imaging, allowing one to work with a spatial detector resolution up to submicrometer [13], [14], [23]. Here, the luminescent image of a scintillator screen is projected via visible light optics onto the chip of a digital camera. The numerical aperture (NA) of the front objective and the scintillator's wavelength of maximum emission determine the highest spatial resolution achievable.

For *in vivo* cine-radiography of organisms employing moderate acquisition rates of up to 250 images/s at ANKA's TopoTomo beamline we use an optical design which combines low-magnifying Rodenstock objectives (front objective) with photo objectives (tube lens). The large NA of the Rodenstock optics allows for an efficient light collection, resulting in a highly efficient detector with a large field of view. Further details of the design have been published already [15], [20]. Specially high efficiency is required in order to reduce the dose on the living organisms. Bulk LYSO:Ce (Ce doped

Manuscript received November 12, 2008. (Write the date on which you submitted your paper for review.)

A. Rack was with the Forschungszentrum Karlsruhe GmbH – ANKA, Karlsruhe, Germany. He is now with the European Synchrotron Radiation Facility, Grenoble, France (telephone: +33 (0)476 88 1781, e-mail: arack@snafu.de).

F. García-Moreno is with the Helmholtz Centre Berlin (Hahn-Meitner-Institut Berlin), Germany (e-mail: garcia-moreno@helmholtz-berlin.de).

O. Betz is with the Zoologisches Institut, Universität Tübingen, Germany, (e-mail: oliver.betz@uni-tuebingen.de).

S. Zabler is with the Max-Planck-Institute of Colloids and Interfaces, Potsdam, Germany (email: simon.zabler@mpikg.mpg.de).

C. Schmidt is with the Zoologisches Institut, Universität Tübingen, Germany, (e-mail: insektenfreund@web.de).

T. dos Santos Rolo is with the the Forschungszentrum Karlsruhe GmbH – ANKA, Karlsruhe, Germany (email: tomy.dossantosrolo@iss.fzk.de).

A. Ershov is with the the Forschungszentrum Karlsruhe GmbH – ANKA, Karlsruhe, Germany (email: alexey.ershov@iss.fzk.de).

L. Helfen is with the the Forschungszentrum Karlsruhe GmbH – ANKA, Karlsruhe, Germany (email: lukas.helfen@iss.fzk.de).

T. Rack is with the the Forschungszentrum Karlsruhe GmbH – ANKA, Karlsruhe, Germany (email: tatjana.rack@iss.fzk.de).

J. Banhart is with the Helmholtz Centre Berlin (Hahn-Meitner-Institut Berlin), Germany (e-mail: banhart@helmholtz-berlin.de).

T. Baumbach is with the the Forschungszentrum Karlsruhe GmbH – ANKA, Karlsruhe, Germany (email: tilo.baumbach@iss.fzk.de).

(Lu, Y)$_2$SiO$_5$) and CWO (CdWO$_4$) single crystal scintillators are commonly used with this detector [18], [19].

In case of dose considerations can be neglected, i.e. for materials science investigations on semi-solid or liquid metallic alloys, much higher time resolution can be achieved. Special white beam optics have been developed by the ESRF in order to stand the high heat load of a white beam emitted by a third generation ring's insertion device [4] , [5], [16]. We use those at the ESRF beamlines ID15a and ID19 to acquire X-ray movies with up to 40 000 frames per second (FPS). Bulk LuAG:Ce (Ce doped $Lu_3Al_5O_{12}$) and YAG:Ce (Ce doped $Y_3Al_5O_{12}$) are applied as scintillator screen [4], [5], [21], [24].

For the ultra-fast digitalization of the scintillator's luminescent image as projected via the optical system we use the novel high-speed camera Photron Fastcam SA-1 [22]. The camera's CMOS chip uses 1024 *x* 1024 pixels, each 20 µm in size. The peak quantum efficiency is 42% at 640 nm, the dynamic range 10bit (800:1) using a 12bit digitalization. The minimum shutter time is 2 µs. A charge of 5.5 electrons in the potential well of the chip corresponds to one signal unit (ADU). Up to 5 400 full frames per second can be acquired, up to 675 000 FPS when using a region of interest. For fast intermediate data storage a 32 GB onboard memory array is available which defines the maximum recording length.

III. *IN VIVO* CINE-RADIOGRAPHY OF INSECTS

Cine-radiography in phase contrast mode using monochromatic hard synchrotron radiation from a third generation ring's insertion device has already been reported [17]. The aim of our feasibility study is to show that the lower flux of a 2.5 GeV ring's bending magnet (e.g. the TopoTomo beamline at the ANKA light source, Germany) can be used as well when running in white beam mode. In this case, the radiation as coming from the source is only filtered by the 0.5 mm thick Be exit window of the beamline's flight tube and 1 mm Si in order to reduce heat load and dose on the insect and detector (the critical energy of TopoTomo is ~6.1 keV).

In our study, we imaged individuals of the cockroach *Periplaneta americana* (Linné), which is well known for its worldwide distribution and association with human dwellings. Cockroaches (Blattaria) have quite primitive chewing and biting mouthparts. Thus, they form adequate model systems for studying basic insect mouthpart coordination and function. We fixed the insects with their backs on a coverslip, so that they were still able to move the various parts of their mouthparts freely. This movement was stimulated by providing food adjacent to their mouthparts. In the long run, this research will help to compare quantitatively the kinematic patterns across major groups of insects and hence contribute to our understanding of the evolution of insect feeding.

In Fig. 1 frames of a movie consisting of several thousand images are shown. The data acquisition speed is 250 FPS at an effective pixel size of approx. 5.5 µm. The image quality we achieved is high enough to perform a semi-automatic 2D image analysis of the acquired radiographic sequences [3].

Fig. 1. *In vivo* image sequence of the feeding cockroach *Periplaneta americana*. 250 FPS data acquisition speed, ~5.5 µm effective pixel size.

Imaging with the X-ray beam only the head, the observed survival time of up to 1.5 minutes was sufficient to obtain good image sequences of naturally feeding cockroaches that made it possible to analyze several complete movement cycles of the mouthparts per individual. Additional Si attenuators increase the life time while reducing the maximum frame rate.

IV. *In situ* Spatio-Temporal Micro-Radioscopy of Metallic Foaming

In comparison to the previous paragraph we are not limited by dose considerations. Therefore, using brighter light sources allows us to progress to much higher data acquisition rates.

As example application we chose *in situ* X-ray radioscopy of metal foaming. First synchrotron-based *in situ* radiography with moderate frame rates around 1 FPS already showed the high potential of this approach in order to study pore coalescence and the corresponding stability of cell walls [24]. Recent experiments showed that frame rates above 10 000 FPS are required in order to temporally resolve a single cell wall collapse [4].

Foamable pre-cursor materials were produced using the powder-metallurgical route [25]. *In situ* foaming was performed using a dedicated furnace design [26]. Experiments were carried out at the ID15a beamline of the ESRF [5]. Using the intense white beam and running the Photron CMOS camera with a region of interest allowed us to record X-ray movies of the foaming process with 40 000 FPS (25 µs time sampling, 20 µm spatial sampling). With this spatio-temporal micro-resolution we were able to picture the collapse of a single cell wall including the relaxation of the novel pore. Selected frames of a time sequence consisting of 184 000 are displayed in Fig. 2 (left column: single frames, right column: major events sketched with the original image faded into the background). Frame 0 shows the two neighboring pores, 525 µs later (frame 21) the cell wall is collapsed, until 1800 µs (frame 72) the new cell wall relaxes into its final round shape. We were able to sample an event lasting around 1.8 ms with 72 frames. For watching the full movie see [27].

V. Summary & Outlook

Our experiments have shown that by using white synchrotron radiation and CMOS cameras on indirect X-ray pixel detectors it is possible to acquire X-ray image sequences with a spatio-temporal resolution up to the micro-scale. While for living organisms one is limited by the maximum dose the specimen can survive, application from e.g. materials research allow one to push the image acquisition speed much further.

For future imaging experiments of living organism, using a harder spectrum and relaxing a bit on the resolution should allow one to increase the data acquisition speed up to 1 000 FPS. Concerning imaging with the white beam of a third generation ring's insertion device such as on ID15a (ESRF): our experiments only used a fraction of the available photon flux. Otherwise the high heat load would destroy the scintillator screens. Thus, with more sophisticated detector designs it should be possible to reach frame rates above 100 000 FPS in the future.

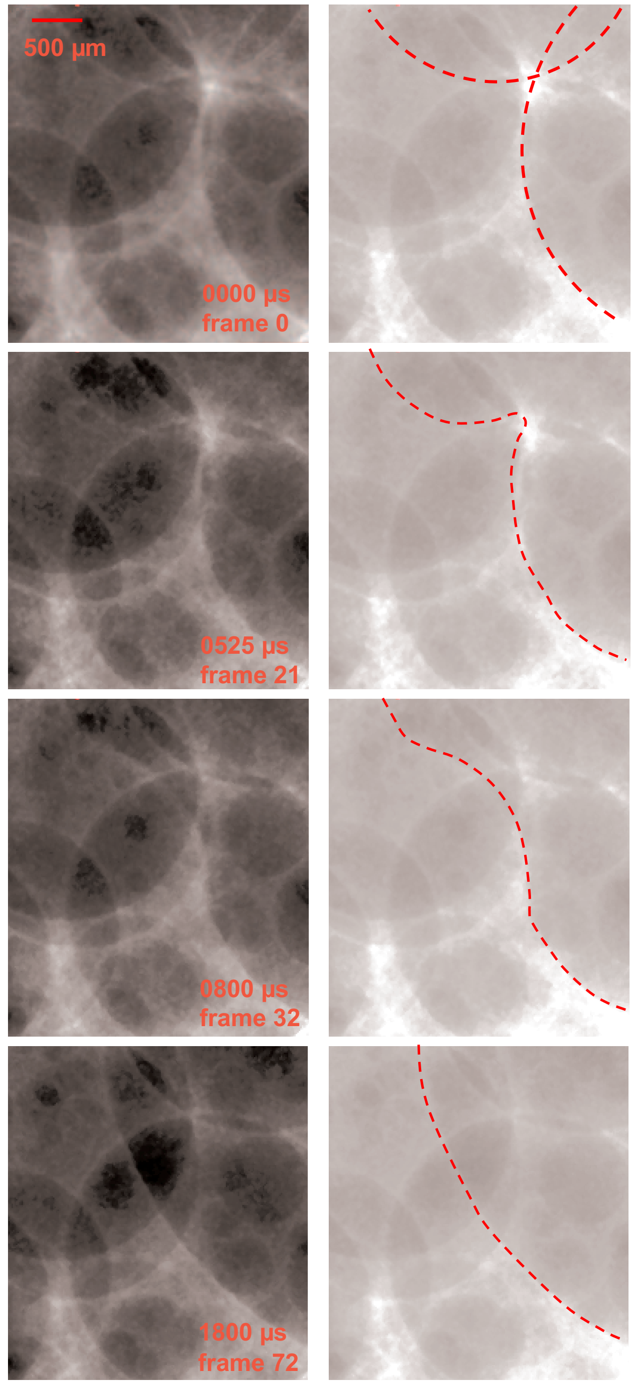

Fig. 2. Left column: image sequence taken out of a series of 184 000 which shows the merging of two pores (25 µs exposure time / 40 000 FPS, 20 µm effective pixel size); right column: sketch highlighting major features.

ACKNOWLEDGMENT

We thank T. Martin, C. Jarnias, M. Peele, M. Di Michiel, V. Honkimäki, T. Weitkamp (ESRF), A. Dieterich, L. Körner (Universität Tübingen), D. Haas, A. Cecilia, H. Schade, A. Volker (ANKA), N. Kardjilov, A. Hilger (HMI) for all the support and fruitful discussions. W. Tutsch (PCO AG, Germany) and A. Bridges (Photron Inc., USA) enlightened us with details about CMOS cameras.

REFERENCES

[1] L. Bull, *La Cinématographie*, Paris: Armand Collin, 1928.

[2] A. Rack, F. García-Moreno, T. Baumbach, J. Banhart, "Synchrotron-based radioscopy employing spatio-temporal micro-resolution for studying fast phenomena in liquid metal foams", J. Synchrotron Rad., submitted for publication.

[3] O. Betz, A. Rack, C. Schmitt, A. Ershov, A. Dieterich, L. Körner, D. Haas, T. Baumbach, "High-Speed X-ray Cineradiography for Analyzing Complex Kinematics in Living Insects", Synchr. Rad. News, vol. 21, no. 5, pp. 34–38, 2008.

[4] F. García-Moreno, A. Rack, L. Helfen, T. Baumbach, S. Zabler, N. Babcsan, J. Banhart, T. Martin, C. Ponchut, M. Di Michel, "Fast processes in liquid metal foams investigated by high-speed synchrotron x-ray microradioscopy", Appl. Phys. Lett., vol. 92, no. 13, pp. 134104-1 - 134104-3, 2008.

[5] M. Di Michiel, J. M. Merino, D. Fernandez-Carreiras, T. Buslaps, V. Honkimäki, P. Falus, T. Martins, O. Svensson, "Fast microtomography using high energy synchrotron radiation", Rev. Sci. Instrum., vol. 76, pp. 043702-1 – 043702-7, 2005.

[6] K. Uesugi, T. Sera, N. Yagi, "Fast tomography using quasi-monochromatic undulator radiation", J. Synchrotron Rad., vol. 13, pp. 403-407, 2006.

[7] Y. Wang, X. Liu, K.-S. Im, W.-K. Lee, J. Wang, K. Fezzaa, D. L. S. Hung, J. R. Winkelman, "Ultrafast X-ray study of dense-liquid-jet flow dynamics using structure-tracking velocimetry", Nature Phys., vol. 4, pp. 305-309, April 2008.

[8] A. Rack, H. Riesemeier, S. Zabler, T. Weitkamp, B. R. Müller, G. Weidemann, P. Modregger, J. Banhart, L. Helfen, A. N. Danilewsky, H. G. Gräber, R. Heldele, B. Mayzel, J. Goebbels, T. Baumbach, "The high resolution synchrotron-based imaging stations at the BAMline (BESSY) and TopoTomo (ANKA)", Proc. of SPIE, vol. 7078, pp. 70780X-1 – 70780X-8, 2008.

[9] S. M. Gruner, M. W. Tate, E. F. Eikenberry, "Charge-coupled device area x-ray detectors", Rev. Sci. Instr., vol. 73, no. 8, pp. 2815-2842, 2002.

[10] M. J. Renzi, M. W. Tate, A. Ercan, S. M. Gruner, E. Fontes, C. F. Powell, A. G. Mac-Phee, S. Narayanan, J. Wang, Y. Yue, R. Cuenca, "Pixel array detectors for time resolved radiography", Rev. Sci. Instr., vol. 73, no. 3, pp. 1621-1624, March 2002.

[11] G. Mettivier, M. Montesi, A. Sebastiano, P. Russo, "High frame rate X-ray imaging with a 256×256 pixel single photon counting Medipix2 detector", IEEE Trans. Nucl. Sci., vol. 53, no. 3, pp. 1650-1655, June 2006.

[12] W. Hartmann, G. Markewitz, U. Rettenmaier, H. J. Queisser, "High-resolution direct-display X-ray topography", Appl. Phys. Lett., vol. 27, no. 5, pp. 308-309, September 1975.

[13] U. Bonse, F. Busch, "X-ray computed microtomography (μCT) using synchrotron radiation (SR)" Prog. Biophys. Molec. Biol., vol. 65, no. 1/2, pp. 133-169, 1996.

[14] A. Koch, C. Raven, P. Spanne, A. Snigirev, "X-ray imaging with submicrometer resolution employing transparent luminescent screens", J. Opt. Soc. Am., vol. 15, no. 7, pp. 1940-1951, 1998.

[15] A. Rack, S. Zabler, B. R. Müller, H. Riesemeier, G. Weidemann, A. Lange, J. Goebbels, M. Hentschel, W. Görner, "High resolution synchrotron-based radiography and tomography using hard X-rays at the BAMline (BESSY II)", Nucl. Instr. & Meth. in Phys. Res. A, vol. 586, no. 2, pp. 327-344, 2008.

[16] A. Koch, "Lens coupled scintillating screen-CCD X-ray area detector with a high quantum efficiency", Nucl. Instr. & Meth. in Phys. Res. A, vol. 348, pp. 654-658, 1994.

[17] M. W. Westneat, O. Betz, R. W. Blob, K. Fezzaa, W. J. Cooper, W.-K. Lee, "Tracheal respiration in insects visualized with synchrotron X-ray imaging", Science, vol. 299, pp. 558–560, 2003.

[18] I. G. Valais, I. S. Kandarakis, A. Konstantinidis, D. N. Nikolopoulos, I. Sianoudis, D. A. Cavouras, N. Dimitropoulos, C. D. Nomicos, G. S. Panayiotakis, "Evaluation of the light emission efficiency of LYSO:Ce scintillator under X-ray excitation for possible applications in medical imaging", Nucl. Instr. & Meth. in Phys. Res. A, vol. 569, no. 2, pp. 201-204, 2006.

[19] L. Nagornaya, G. Onyshchenko, E. Pirogov, N. Starzhinskiy, I. Tupitsyna, V. Ryzhikov, Y. Galich, Y. Vostretsov, S. Galkin, E. Voronkin, "Production of the high-quality CdWO4 single crystals for application in CT and radiometric monitoring", Nucl. Instr. & Meth. in Phys. Res. A, vol. 537, no. 1-2, pp. 163-167, 2005.

[20] A. N. Danilewsky, A. Rack, J. Wittge, T. Weitkamp, R. Simon, H. Riesemeier, T. Baumbach, "White Beam Synchrotron Topography Using a High Resolution Digital X-ray Imaging Detector", Nucl. Instr. & Meth. in Phys. Res. B, vol. 266, no. 9, pp. 2035-2040, 2008.

[21] J. Touš, M. Horváth, L. Pína, K. Blažek, B. Sopko, "High-resolution application of YAG:Ce and LuAG:Ce imaging detectors with a CCD X-ray camera", Nucl. Instr. & Meth. in Phys. Res. A, vol. 591, pp. 264-267, 2008.

[22] T. Inoue, S. Takeuchi, S. Kawahito, "CMOS active pixel image sensor with in-pixel CDS for high-speed cameras", Proc. of SPIE, vol. 5580, pp. 293-300, 2005.

[23] J. Banhart, *Advanced Tomographic Methods in Materials Research and Engineering*, Oxford University Press, Oxford, 2008.

[24] J. Banhart, H. Stanzick, L. Helfen, T. Baumbach, "Metal foam evolution studied by synchrotron radioscopy", Appl. Phys. Lett., vol. 78, no. 8, 1152–1154, February 2001.

[25] C. J. Yu, H. Eifert, J. Banhart, J. Baumeister, "Metal foaming by a powder metallurgical method: production, properties and applications", Mat. Res. Innovat., vol. 2, pp. 181-188, 1998.

[26] F. García-Moreno, N. Babcsan, J. Banhart, "X-ray radioscopy of liquid metalfoams: influence of heating profile, atmosphere and pressure", Colloids Surf. A, vol. 263, no. 1-3, pp. 290-294, August 2005.

[27] http://www.alexanderrack.eu/ieee_movie.avi - last visit November 2008.

[28] O. Betz, U. Wegst, D. Weide, M. Heethoff, L. Helfen, W.-K. Lee, P. Cloetens, "Imaging applications of synchrotron x-ray micro-tomography in biological morphology and biomaterial science. I. General aspects of the technique and its advantages in the analysis of arthropod structure", J. Microsc., vol. 227, pp. 51-71, 2007.