IMAGING MATERIALS ON THE RUN: DEVELOPMENTS IN FAST SYNCHROTRON TOMOGRAPHY

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Summary: Many technically relevant dynamic processes like crack propagation in composites, bubble formation in foams, liquid transport in porous media, etc., occur on microscopic length scales on a sub-second time scale. Time-resolved synchrotron microtomography is pushing the achievable time resolution into the multi-100Hz regime, while allowing for ever more complex environments to produce realistic sample conditions.

1. INTRODUCTION

Recent years have witnessed a constant increase in the achievable time-resolution of synchrotron tomography measurements, pushing firmly into the sub-second regime and reaching scan rates of up to 20 – 25 Hz [1, 2, 3]. This paves the way to image materials dynamically and on relevant time scales while they are undergoing structural changes, for example, during production processes or when a sample experiences external forces or changes in its environment. While progress to push the temporal resolution has been steady and fast, a number of challenges remain when aiming to make these techniques easily applicable to a wider range of material science topics: Many of the technologically interesting processes proceed on even shorter timescales, necessitating a further push towards even shorter acquisition times for full tomographic data sets. However, access to scan times below about one second is only possible when allowing for a continuous rotation of the sample during the measurement. At the same time, the duration of the sample evolution can span hundreds to thousands of individual scans, requiring very fast and sustained data acquisition of large amounts of data. Relevant sample conditions are often far from ambient equilibrium, and require sample environments that are capable of subjecting the sample to a multitude of different physical, mechanical, thermal, and chemical conditions, such as high pressure, low or high temperature, reactive atmospheres, tensile or compressive strain, flows of gases or liquids, electrical potentials, etc. More often than not, a few of these have to be combined together simultaneously. The construction of sample cells able to provide these conditions while remaining compatible with a continuous rotation and unobstructed view of the sample to the X-ray beam is thus crucial for these studies. Furthermore, the amount of data produced during extended 4D studies (3 spatial dimensions + time) needs to be transferred, stored, and analyzed, calling for highly scalable workflows and dedicated IT infrastructure. Lastly, most experiments are currently carried out semi blindly in that the dynamic of the system needs to be known beforehand in order to choose the correct acquisition parameters. Ideally, one would thus like to obtain a nearly realtime visualization and basic quantification of the acquired data in order to implement direct feedback mechanisms to dynamically control the experimental conditions during a measurement.

2. EXPERIMENTAL METHOD

A number of recent developments at the TOMCAT beamline X02DA of the Swiss Light Source have helped to push the achievable time-resolution for microtomography measurements significantly. The availability of the GigaFRoST camera system [4] allows for extended and sustained high-speed data acquisition, while the acquisition of a new white beam-compatible high numerical aperture microscope with 4x magnification has boosted the light yield by nearly a factor of 5 compared to the previous model available at TOMCAT, while delivering far superior image quality [5]. High sample rotation speeds are achieved with a custom-built rotation table [3].

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3. RESULTS

Making use of the newly available infrastructure, we have performed time-resolved measurements on a large variety of samples relevant for various research fields. Among the highlights are investigations of catalyst slurry deposition and water evaporation from industrial-grade ceramic catalyst structures (Figure 1(a)), or the observation of the combustion process of carbon fiber-based spacecraft heat shield materials under planetary re-entry conditions (Figure 1(b)). Pushing for higher temporal resolution, we have recently achieved tomographic acquisition speeds of just over 200 full tomograms per second on Al-based metal foams at 5 μm voxel size [6] (Figure 1(c)). First experiments further demonstrate the feasibility of performing nearly real-time reconstructions of selected arbitrarily oriented slices through the tomographic volume in less than a second, allowing for a live preview of the interior structure of a sample during measurements.

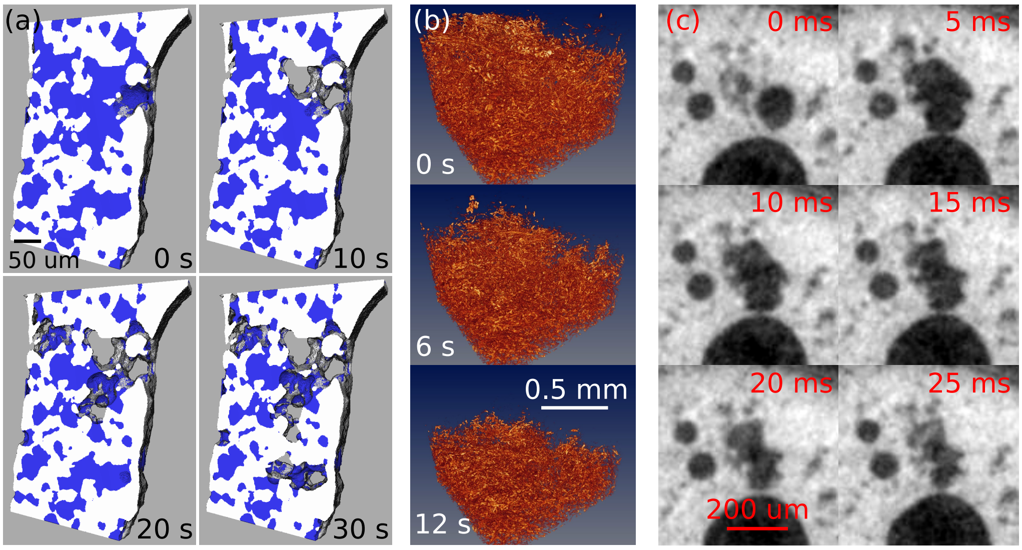

Figure 1: Time-resolved tomography on various time scales: (a) Water evaporation from a ceramic catalyst structure. Courtesy of V. Novak *et al.* (b) Combustion of carbon-fibre-based heat shield materials for planetary re-entry of spacecraft. Courtesy of F. Panerai *et al.* (c) Reconstructed tomograms of an Al-based metal foam showing bubble collapse on the millisecond time scale during foaming. Courtesy of F. García-Moreno *et al.*

References

[1] T. dos Santos Rolo, A. Ershov, T. van de Kamp, and T. Baumbach. In vivo X-ray cine-tomography for tracking morphological dynamics. *PNAS*, 111(11), 3921–3926, 2014.

[2] E. Maire, C. Le Bourlot, J. Adrien, A. Mortensen, and R. Mokso. 20 Hz X-ray tomography during an in situ tensile test. *Int J Fract*, 200(1), 3–12, 2016.

[3] F. García-Moreno, P. H. Kamm, T. R. Neu, and J. Banhart. Time-resolved in situ tomography for the analysis of evolving metal-foam granulates. *J Synchrotron Rad*, 25(5), 2018.

[4] R. Mokso, C. M. Schlepütz, G. Theidel, H. Billich, E. Schmid, T. Celcer, G. Mikuljan, L. Sala, F. Marone, N. Schlumpf, and M. Stampanoni. GigaFRoST: the gigabit fast readout system for tomography. *J Synchrotron Rad*, 24(6), 1250–1259, 2017.

[5] M. Bührer, M. Stampanoni, X. Rochet, F. Büchi, J. Eller, and F. Marone. High numerical aperture macroscope optics for time-resolved experiments, 2019. Under review.

[6] P. Kamm, T. Neu, C. M. Schlepütz, F. Bülk, R. Mokso, M. Stampanoni, B. John, and F. Garcia-Moreno. Revealing the dynamics of foaming metal by x-ray tomoscopy, 2019. Under review.