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Review article

A brief guide to synchrotron radiation-based microtomography in (structural) geology and rock mechanics

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ABSTRACT

This contribution outlines Synchrotron-based X-ray micro-tomography and its potential use in structural geology and rock mechanics. The paper complements several recent reviews of X-ray microtomography. We summarize the general approach to data acquisition, post-processing as well as analysis and thereby aim to provide an entry point for the interested reader. The paper includes tables listing relevant beamlines, a list of all available imaging techniques, and available free and commercial software packages for data visualization and quantification. We highlight potential applications in a review of relevant literature including time-resolved experiments and digital rock physics. The paper concludes with a report on ongoing developments and upgrades at synchrotron facilities to frame the future possibilities for imaging sub-second processes in centimetre-sized samples.

1. Introduction

Due to the complexity of natural rock deformation, structural geologists routinely gather three-dimensional information, from kilometre-scale seismic datasets to micron-resolution electron backscatter diffraction data. The need to image the spatial relationships amongst structural elements on multiple scales is reflected in the early use of block diagrams and serial geological cross sections (e.g., Heim, 1922, Tafel XXIV; Voll, 1961, Plate 24), detailed three-dimensional sketches (e.g., Cloos, 1948, Fig. 1), and stereographic projections (e.g., Sander, 1911).

While map-scale and outcrop-scale three-dimensional models can be developed and tested in the field, the three-dimensional imaging of rock fabrics on the smaller scales is more difficult. Traditionally, serial sectioning (or grinding) is employed to reconstruct the spatial distribution and alignment of fabrics and minerals in hand specimens, thin sections and clay models (e.g., Cobbold and Quinquis, 1980; Bryon et al., 1995; Marschallinger, 1998; Mock, 2005; Jerram and Higgins, 2007). More recently, focused ion beam tomography has advanced the serial sectioning technique, revealing important details of nano-scale structures (e.g., Holzer et al., 2006). However, serial sectioning suffers from the long data acquisition times as much as from being a destructive technique where all data acquisition on a particular section has to be completed before a new section can be added. Generally, a non-destructive imaging technique is therefore preferable.

Tomographic techniques enable the non-invasive imaging of a large range of materials, including rocks. Three-dimensional tomograms of rock fabrics can be acquired using photons, neutrons, ultrasonic waves and magnetic resonance, highlighting a variety of rock properties (see e.g., Brown and Reilly, 1996; Stock, 1998; Masschaele et al., 2004; Vontobel et al., 2006; Dierick et al., 2005; Carlson, 2006; Kaestner et al., 2008; Strobl et al., 2009; Wildenschild and Sheppard, 2013 for overviews). Here we report on the particular advantages of X-ray microtomography (*μ tomography*), with the additional refinement that we focus on the potential of synchrotron radiation based X-ray microtomography (henceforth *Sμ tomography*). This paper complements several comprehensive recent reviews on *μ tomography* (Betz et al., 2007; Stock, 2008; Mizutani and Suzuki, 2012; Baker et al., 2012a; Cnudde and Boone, 2013; Wildenschild and Sheppard, 2013; Kanitpanyacharoen et al., 2013, Maire and Witters, 2014) and aims to emphasize the potential benefits for structural geology.

Appendices 1–4 describe the process of getting access to a synchrotron beamline (various beamlines listed in Table 1), sample preparation, deal with the resolution of μ tomographic data and give tips towards creating useful visualisations.

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Table 1
Beamlines specialized in Sμ-tomography.

Synchrotron	Country	Beamline	Weblink
Advanced Photon Source (APS)	USA	2-BM	http://www.aps.anl.gov/Xray_Science_Division/Xray_Microscopy_and_Imaging/Beamlines/2_BM/
		13-BM GSECARS	http://www.aps.anl.gov/Beamlines/Directory/showbeamline.php?beamline_id=20
		32-ID	http://www.aps.anl.gov/Beamlines/Directory/beamline.php?beamline_id=84
National Synchrotron Light Source (NSLS)		X2B	http://beamlines.ps.bnl.gov/beamline.aspx?blid=X2B
		X27A	http://beamlines.ps.bnl.gov/beamline.aspx?blid=X27A
ANKA	GER	Image	http://ankaweb.fzk.de/website.php?page=instrumentation_beam&id=19
HASYLAB/ PETRA III	GER	Imaging P05	http://hasylab.desy.de/facilities/petra_iii/beamlines/p05_ibl/index_eng.html
		HEMS P07	http://hasylab.desy.de/facilities/petra_iii/beamlines/p07_high_energy_materials_science/index_eng.html
Australian Synchrotron (AS)	AUS	Imaging & Medical	http://www.synchrotron.org.au/index.php/aussyncbeamlines/imaging-medical/techniques-available
Diamond	UK	JEEP I12	http://www.diamond.ac.uk/Home/Beamlines/I12.html
		I13	http://www.diamond.ac.uk/Home/Beamlines/I13.html
European Synchrotron Research Facility	FRA	ID-19	http://www.esrf.eu/UsersAndScience/Experiments/Imaging/ID19/
		ID-15	http://www.esrf.eu/UsersAndScience/Experiments/StructMaterials/ID15
Spring8	Japan	BL20B2	http://www.spring8.or.jp/en/facilities/bl/list/
		BL20XU	http://www.spring8.or.jp/en/facilities/bl/list/
SOLEIL	FRA	PSICHÉ	http://www.synchrotron-soleil.fr/Recherche/LignesLumiere/PSICHE
Swiss Light Source	CH	Tomcat	http://www.psi.ch/sls/tomcat/tomcat

2. Data acquisition at synchrotron μ tomography beamlines

2.1. From X-rays to radiographs

X-ray tomography has become a readily available tool for the three-dimensional imaging of a wide range of materials, including rocks. Many drill cores sampled in the oil/gas sector are routinely scanned using adapted laboratory scanners, and X-ray tomographic images of samples up to several centimetres in size are easily acquired using commercial desktop scanners. Most desktop systems reach resolutions in the single-digit micron range on millimetre-sized samples (hence *micro*tomography). However, the X-ray μ tomographic data with the best image quality undoubtedly come from synchrotrons (Table 1 lists the most important synchrotron beamlines). The most important difference between *Sμ tomography* and conventional scanners is the way the X-rays are produced. Desktop- and laboratory-scale systems use X-ray tubes, in which an electron beam is accelerated by a large potential difference onto a small target. The target, when hit by the electrons, emits X-rays of a characteristic energy plus Bremsstrahlung with a wide electromagnetic spectrum (e.g., Ketcham and Carlson, 2001). In synchrotrons, X-rays are produced in a fundamentally different way (see Fig. 1a in Betz et al., 2007). In so-called storage rings, electrons that are travelling at near the speed of light are forced into a quasi-circular path by periodically installed 'bending magnets'. These separate straight sections of the ring. In the magnets, the electrons are accelerated and emit a continuum of very bright electromagnetic radiation tangentially to their quasi-circular path. Synchrotrons are, in contrast to other particle accelerators, designed to utilize this effect, and additional magnets (so-called undulators and wigglers) are installed in the straight sections of the storage ring to produce even brighter electromagnetic radiation.

In contrast to tube sources, synchrotrons produce very high photon fluxes over a wide energy spectrum (Fig. 1a), from which a monochromatic beam of an energy suiting the sample thickness and composition can be selected, or an energy bandwidth ('pink beam') can be tailored. In 'white beams', where most of the available energy bandwidth is used, the photon flux is increased up to 10,000-fold, reducing acquisition times of radiographic projections (see below) to the picosecond range (e.g., at beamline 32-ID at the Advanced Photon Source). More photons allow for rapid imaging, better count statistics and less image noise. The high flux allows managing 'beam hardening', a classical problem with tube-based μ-tomographs, by filtering the X-rays as necessary.

Sμ tomography also benefits from the geometry of the instrumental setup (Cloetens et al., 2002). At the magnets, electromagnetic radiation is emitted in a narrow cone tangentially to the storage ring. The large distance between the magnet and the sample, often tens of metres, yields a partially coherent beam with a very small cross section at the sample, which allows obtaining phase images by adjusting the sample-detector distance (Cloetens et al., 1996, 2002, see below). Where a monochromator is employed, the processed beam is also parallel, which greatly reduces beam artefacts in the radiograph and improves the quality of the resulting μ tomographic data (Fig. 1b, Davis and Elliott, 2006).

Despite the different characteristics of the X-rays used, synchrotron and desktop systems share that 3-dimensional tomographic datasets are based on 2-dimensional radiographs that are taken while a sample is rotated in between a stationary X-ray source and an X-ray sensitive detector[1] or a scintillator screen that converts the X-rays into visible light, which is then recorded by a detector (Fig. 1b). As they pass through the sample, X-rays are affected by photoelectric interactions and scattering. X-rays that exit the sample towards the detector carry information on these interactions and it is possible to map this information on the radiographs (e.g., Brown et al., 2006). Several *aspects* of photoelectric interaction can be mapped:

- The material-specific absorption of photons is utilized in *absorption contrast* tomography;
- Spatial variations in the refractive index, which is used in *phase contrast* tomography;
- The crystallographic structure of a sample using spatial differences in *X-ray diffraction*;
- The distribution of elements detecting spatial differences in *X-ray fluorescence*;
- Particle size and shape in a sample by detecting *small-angle scattering*;
- Information on the local bonding structure, using *spectroscopy contrast*.

This paper focusses on absorption and phase contrast tomography. However, it should be noted that both of these techniques can usefully be combined with any of the other techniques listed above.

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[1] In laboratory (and medical) systems, source and detector rotate around a stationary sample.