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Synchrotron-based X-ray Tomographic Microscopy for Rock Physics Investigations

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SUMMARY

Synchrotron radiation X-ray tomographic microscopy is a non-destructive method providing ultra-high-resolution 3D digital images of rock microstructures. We describe this method and, to demonstrate its wide applicability, present 3D images of very different rock types: Berea sandstone, Fontainebleau sandstone, dolomite, calcitic dolomite, and three-phase magmatic glasses. For some samples, full and partial saturation scenarios are considered using oil, water, and air. The rock images precisely reveal the 3D rock microstructure, the pore space morphology, and the interfaces between fluids saturating the same pore. We provide the raw image data sets as online supplementary material, along with laboratory data describing the rock properties. By making these data sets available to other research groups, we aim to stimulate work based on digital rock images of high quality and high resolution. We also discuss and suggest possible applications and research directions that can be pursued on the basis of our data.

Introduction

Three-dimensional (3D) information of rock microstructures is important for better understanding physical phenomena and for rock characterization at micro-scale (Madonna et al., 2012). Various methods for obtaining a 3D image of the rock microstructure exist (Madonna et al., 2013, and references therein). They can be separated into two major groups: destructive and non-destructive methods. If possible, the latter is preferable because the same rock sample can be used for further investigations after imaging, for example in laboratory testing. This allows a direct comparison between laboratory tests and calculations based on a digital rock image. The most common non-destructive 3D imaging method for earth sciences is X-ray computed tomography (CT). There is a clear trade-off between sample size and resolution (Figure 1a). For each single material sample, it has to be clarified if the chosen sample size is representative for the given task to be considered. In the last decade, the X-ray micro-computed tomography (micro-CT) method became widely available and many modern studies have made use of it to obtain 3D rock images. The resolution of micro-CT is high enough to image the spatial distribution of grains, pores, and pore fluids.

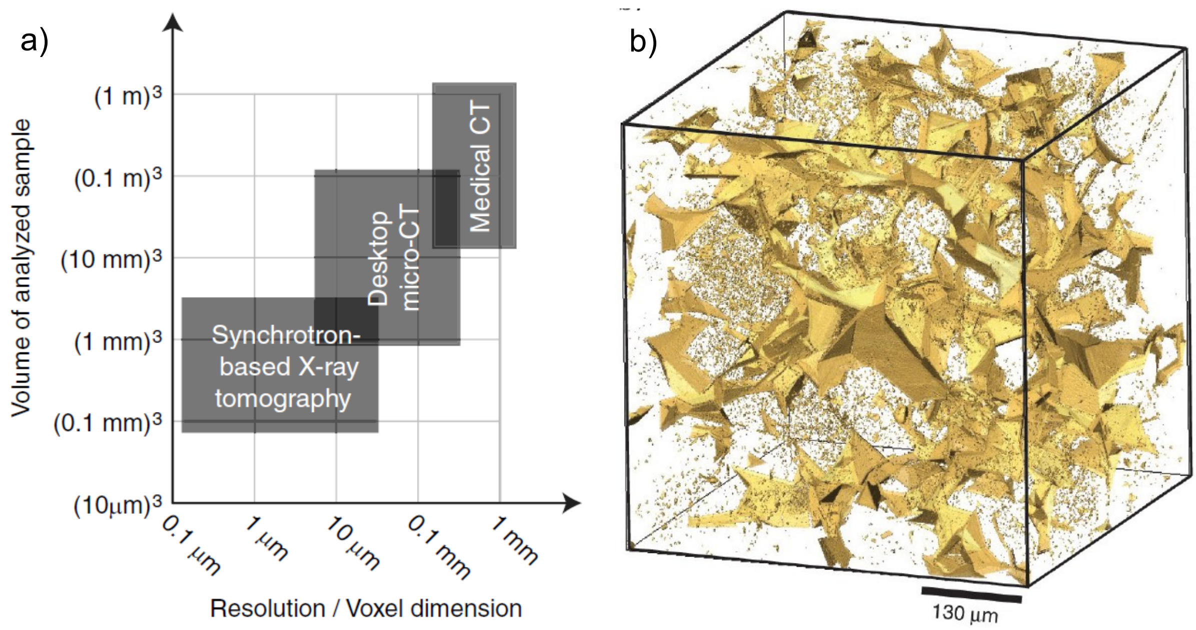

Figure 1 *a) Schematic representation of sample size versus resolution of the most common X-ray computed tomography methods. Other methods are available, but are not plotted here. b) 3D visualization of the SRXTM-volume of dry Fontainebleau sandstone. Only the pore space is shown in yellow; the quartz grains are not shown.*

Additionally, 3D rock images (Figure 1b) can be used for predicting properties such as porosity, permeability, pore size distribution, effective elastic moduli, and electrical conductivity. For example, permeability can be successfully predicted by numerically simulating fluid flow through 3D rock models, with the numerical results being in reasonable agreement with laboratory measurements. In this case, the resolution of the micro-CT technique is sufficient because fluid pathways predominantly follow larger pores. However, if the porosity is much smaller than 1 µm (e.g., shale) the agreement might be less satisfactory because of resolution limitations. On the other hand, mechanical properties, such as the effective elastic moduli, strongly depend on the microstructural details of the rock, which are unresolved by the micro-CT technique. The inability to fully characterize the microstructural details of a rock can lead to disagreements between numerical estimates of mechanical properties based on micro-CT images and laboratory data. Dvorkin et al. (2011) suggested considering trends formed by data points from computational and laboratory measurements, instead of direct point-to-point comparisons.

The limitations of traditional X-ray micro-CT instruments in fully characterizing the microstructural details of rocks can be significantly reduced by using third-generation synchrotron sources, thanks to their exceptional photon density. The high brilliance of third-generation synchrotron radiation provides increased spatial and temporal resolution. Brilliance (i.e., light intensity) is defined as the number of photons per second per unit source area, emitted per solid angle within a certain wavelength band. The detection of details as small as 1 micron in millimeter-sized samples is routinely possible within only a few minutes. In addition, the monochromaticity of the X-ray beam allows for quantitative measurements of material properties (e.g., density) and simplifies the identification of the different X-rayed phases, because beam hardening artefacts, distinctive for micro-CT setups, can be avoided. Increased contrast and reduced noise are also promoted by the monochromatic beam and the high photon flux. All of these factors result in high-resolution images of astonishing quality, which allow a much more detailed analysis compared to micro-CT images.

For this paper, we have employed synchrotron radiation X-ray tomographic microscopy (SRXTM) to obtain high-resolution 3D images of samples of:

- Dry and water-saturated Fontainebleau sandstone.
- Dry and partially saturated (with two or three fluid phases) Berea sandstone.
- Dry dolomite and calcitic dolomite.
- Three-phase magmatic glass and high-temperature vesiculating magma.

We chose such a wide range of different rock samples to demonstrate the power and versatility of the SRXTM-method. On one hand, we deliver images of two standard rocks, Berea and Fontainebleau sandstones, which are often considered as analog rocks for siliciclastic reservoirs and are therefore intensively studied and characterized. On the other hand, imaging magmatic glass and magma demonstrates the wide applicability of the method also to nonporous materials. The SRXTM-methodology and the resulting images are described and shown in the following sections.

We provide the raw data of all the images (http://www.rockphysics.ethz.ch/downloads) as an incentive to the development of rock physics research using high-resolution digital rock images, which have been of scarce access to a broad scientific community. More details about the described datasets can be found in Madonna et al. (2013).

Case Studies

The presented rock images exploit the absorption contrast of the synchrotron beam in gray values. The brighter the gray value is, the higher the absorption of the beam was. To a first order, the absorption is proportional to the material density. Sometimes, the single phases can be easily detected by eye when scrolling through neighboring slices. For a quantitative analysis, each phase of interest has to be identified in the data. This is normally done by attributing a certain range of gray color to a particular phase and is called segmentation. The process of segmentation itself is a subject of current research and is discussed, for example, by Gao and Wong (1989). Here, our objective is only to provide and explain the raw data sets. Due to the experimental setup at the TOMCAT beam line, the data in an inner volume of the data cube will provide highest quality. This region is marked as a white dashed circle in the figures showing raw data (e.g., Figure 2). The corners contain artifacts due to less complete data related to the rotation symmetry inherent in tomography. For an accurate analysis, we suggest to preferably use the data within the marked region.

Case 1: Fontainebleau sandstone
A dry sample of Fontainebleau sandstone was imaged with the experimental conditions given in Table 1. Fontainebleau sandstone is often referred to as an analog rock for siliciclastic hydrocarbon reservoirs and has already been investigated in other digital rock studies. The average porosity of our sample is 5.1 vol%. A 3D view of the segmented pore space is shown in Figure 1b.

***Table 1** Experimental conditions and characteristics of the raw data.*

Case	Beam energy	Exposure time	Size of datacube	Magnification	Voxel size	Pixel depth
1	26 keV	500 ms	1024×1024×1024	10×	$(0.74\ \mu m)^3$	16 bit
2	26 keV	300 ms	1024×1024×1024	20×	$(0.38\ \mu m)^3$	16 bit
3	26 keV	500 ms	1024×1024×1024	10×	$(0.74\ \mu m)^3$	16 bit
4	26 keV	300 ms	1024×1024×1024	20×	$(0.38\ \mu m)^3$	16 bit
5	26 keV	500 ms	1024×1024×1024	10×	$(0.74\ \mu m)^3$	16 bit
6	22.6 keV	300 ms	2048×2048×2048	20×	$(0.38\ \mu m)^3$	8 and 16 bit
7	22.6 keV	300 ms	2048×2048×2048	20×	$(0.38\ \mu m)^3$	8 and 16 bit
8	20 keV	100 ms	2048×2048×2048	10×	$(0.74\ \mu m)^3$	16 bit
9	Polychromatic	1.4 ms	1008×1008×2016	~3.7x	$(2.96\ \mu m)^3$	8 bit

Case 2: Water-saturated Fontainebleau sandstone
The second image of Fontainebleau sandstone is from the same rock sample as the first image. The specific 2 mm diameter specimen was saturated through imbibition.

Case 3: Dry Berea sandstone
For the imaging and laboratory experiments of Berea sandstone, a sample from the Berea Sandstone™ Petroleum Cores (Ohio, USA) was used. Berea sandstone is also frequently used as analog rock for siliciclastic hydrocarbon reservoirs and, therefore, is well studied and characterized. The connected porosity of the used sample is around 20%. Permeability as provided by the company is between 200 and 500 mD.

Case 4: Different saturation scenarios of Berea sandstone
In this experiment, another specimen of Berea sandstone from the same block is imaged for three subsequent saturation scenarios. With the same imbibition technique, we saturated the Berea sandstone sample with Angiofil oil. After the first scan of the almost fully oil-saturated Berea sandstone, our aim was to create partially saturated stages with the same specimen. A four-step technique was employed:

- The specimen was enveloped in a ~2 mm inner diameter polyolefin shrink tube.
- The specimen was placed inside one end of a 3 mm inner diameter, ~10 cm long latex pipe.
- The other end of the pipe was connected to a 10 ml syringe filled with the new liquid.
- Finally, acting on the syringe piston, a flux of liquid was forced to flow through the specimen.

This technique was subsequently applied twice to our specimen. First, we injected air, which pushed the oil out of the pore space. As a result, two phases, air and oil, occupied the connected pores. Second, we injected water. Hence, three phases were expected in the pore space; water, air, and oil (Figure 2, left).

Case 5: Another saturation scenario of Berea sandstone
Using a small vacuum pump, a dry sample of Berea sandstone was first saturated with the same Angiofil oil as in Case 4, and subsequently with water, which leads to a partial saturation of oil and water. This procedure completes the different saturation scenarios described in the previous case. In contrast to Case 4, we have the two saturating phases, oil and water, and a lower magnification. Hence, a bigger field view as shown in Figure 2 (right).

Cases 6 and 7: Trigodonusdolomit, Upper Muschelkalk (northern Switzerland)
Cases 6 and 7 provide image data sets for two dry carbonate rock specimens that originate from a core drilled in northern Switzerland.

Case 8: Three-phase magmatic glass
A synthetic three-phase magmatic glass was deformed in torsion configuration at 200 MPa and 450°C using a Paterson gas-medium apparatus. A small core (2 mm diameter; 4 mm length) was subsequently drilled from the external periphery of the deformed and was analyzed using SRXTM.

Case 9: Magma foaming

To constrain in 3D the effect of water on the process of volatile exsolution (nucleation, growth and coalescence of gas bubble), a natural water-poor, crystal-free phonolitic obsidian from La Cañadas Caldera (Tenerife, Spain) was imaged during an in situ, real-time, high-temperature, room-pressure experiment using the ultrafast end-station of the TOMCAT beam-line.

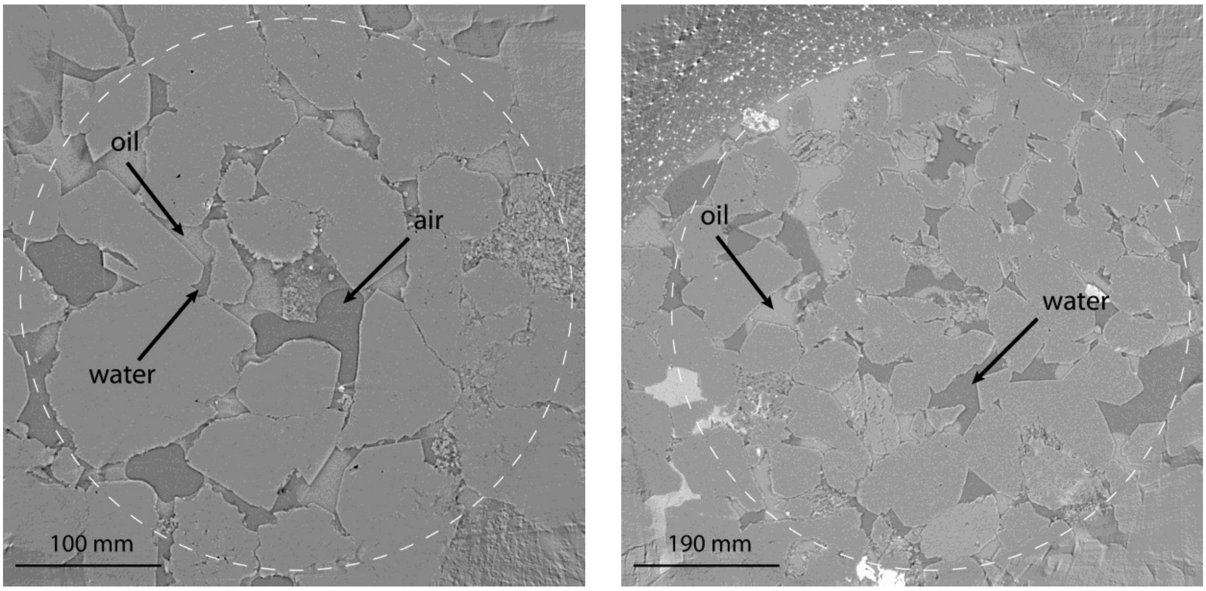

Figure 2*: Raw SRXTM-images of Berea sandstone partially saturated with water, air, and oil (left) and with water and oil (right). The full data cubes contain 1024^3 voxels each with a voxel size of 0.38 μm^3 (left) and 0.74 μm^3 (right).*

Conclusions

SRXTM is a non-destructive imaging method providing ultrahigh-resolution 3D volumes of rocks of any type. The images from the TOMCAT beam-line at the Swiss Light Source are characterized by resolutions down to 0.38 µm, allowing for a detailed study of the rock microstructure. SRXTM adds another level of detail compared to micro-CT. Unfortunately, synchrotron facilities are not easily available to a broad scientific community. Therefore, we provide SRXTM raw data (in TIF format) of various rock types to the scientific community in the online repository of this publication (http://www.rockphysics.ethz.ch/downloads), together with descriptions of imaging conditions and characterization of the rock samples (through laboratory measurements). The provided data should stimulate many types of further investigations, for example, on image analysis methods, segmentation algorithms, numerical fluid flow calculations, or studies on mechanical rock properties.

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