MEASUREMENT PARAMETERS AND RESOLUTION ASPECTS OF MICRO X-RAY TOMOGRAPHY FOR ADVANCED CORE ANALYSIS

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ABSTRACT

Micro X-ray Tomography (µ-XCT) is an emerging non-destructive imaging technology with large potential for application in advanced core analysis. This scanning technique provides 3-D images and quantitative information in the micron range, i.e. at pore scale level. µ-XCT will assist to gain fundamental insight on rock topology structure, in-situ physicochemical processes as well as grain contacts and micro-fractures. Especially in the Carbonate arena (e.g., Middle East), where pore structures are extremely complex, µ-XCT can provide a step change in our understanding of multi-phase flow properties of rock. The knowledge gained will lead to more accurate scaling relationships between micro- and macro- (field) scales enabling better estimation of hydrocarbon recovery.

Tabletop µ-XCT scanners are envisaged to provide optimal flexibility for advanced core analysis research. In this paper we discuss the various measurement parameters that play a role in µ-XCT. Signal-to-noise ratio and spatial resolution are considered as the most important parameters for µ-XCT. Some results will be presented of an assessment study towards the performance capabilities of a number of tabletop µ-XCT scanners. In this study different reservoir carbonate rock samples as well as synthetic rock samples have been used. The spatial resolution of current commercial tabletop µ-XCT scanners is in the order of 5-8 microns, which is not sufficient to probe micro pores for carbonate rock. We have also considered technical innovations to enhance the spatial resolution in tabletop µ-XCT. Possibly the spatial resolution in µ-XCT can be improved using nano-focus X-ray sources with sub-micron focal spot. Further improvement in spatial resolution is expected from using emerging tabletop laser plasma X-ray technology. For applications in tight carbonates, synchrotron beam line based µ-XCT offers spatial resolutions below 1 micron.

INTRODUCTION

Currently µ-XCT scanning is operated at a laboratory tabletop scale as well as at synchrotron beam lines. Synchrotron beam lines offer advantageous high beam intensities enabling realization of spatial resolution in the sub-micron range. However, beam lines are operated at third parties and are considered inflexible because experiments need to be planned ahead. The focus of this paper is on tabletop µ-XCT systems. These systems are small, portable and flexible and therefore are ideally suited for advanced core analysis research. Various measurement parameters and sensitivity to the imaging performance that play a role in tabletop µ-XCT are discussed. The photon intensity originating from commercially available micro focal X-ray sources is specifically discussed because it affects the accuracy of the measured attenuation signals. In addition, we have assessed the imaging performance capabilities of tabletop µ-XCT scanners, discussed scientific aspects involved with X-ray generation, and identified technology opportunities for realization of an ultra-bright point X-ray source in support of tabletop sub-micron µ-XCT imaging.

MICRO-XCT PRINCIPLE & PARAMETERS

Micro X-ray Computerized Tomography (µ-XCT) is a non-destructive analytical technique providing a cross sectional image of an object using a computer and a reconstruction algorithm to calculate the image from X-ray attenuation profile data. Figure 1 shows the basic set-up of a µ-XCT system. An X-ray source, an opposite placed X-ray detector and an object rotation table in between form the main parts of a µ-XCT scanner. During a scan the object is irradiated with a collimated fan-beam of X-rays being attenuated when penetrating materials. The absorbed intensity depends on the material composition, thickness and density along the X-ray path. The resulting transmission intensities are projected on a 1-D linear detector or on a 2-D detector screen. Next the object is rotated at minimal over 180 degrees to obtain attenuation profiles at different angles providing three-dimensional information. The entire system is computer controlled. During scanning the attenuation profile data are continuously measured and transferred in real-time to a high-speed processor for image reconstruction of the cross-section of the object. In order to be able to measure the attenuation profile, a linear µ-XCT detector should consist of large number of discrete detector elements. In 3-D cone beam µ-XCT the scan slice thickness is determined by the detector pixel size. Furthermore, a large number of projections should be taken at various angles.

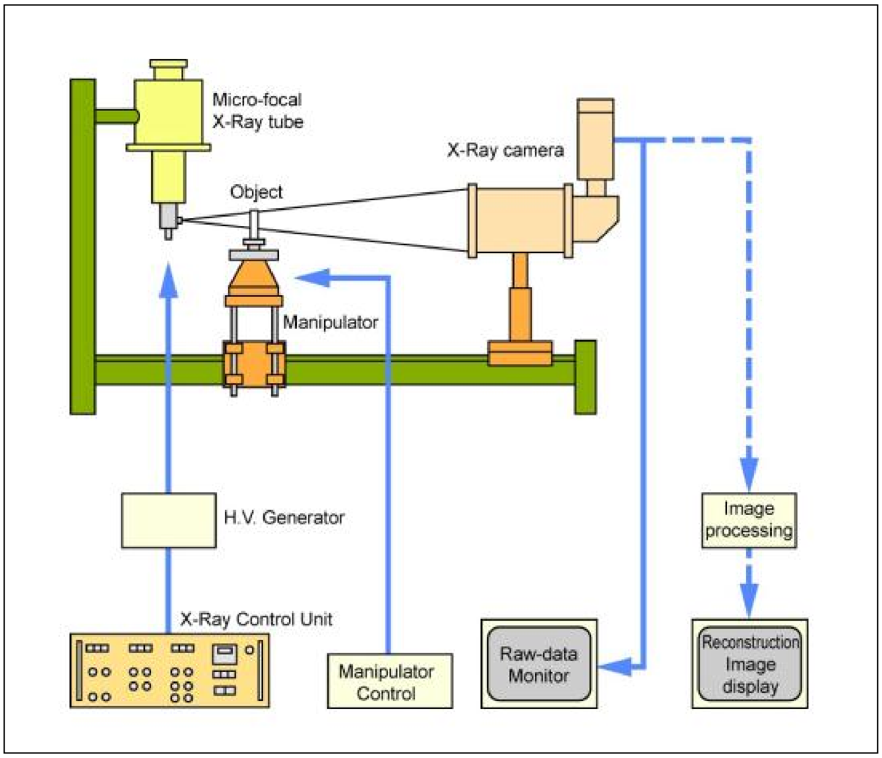

Figure 1: Illustration of the principle of Micro X-ray Tomography

Below a summary overview is given on the most important parameters that affect the quality of the end-result in μ-XCT [Ref-1]:

A Machine dependent factors

- X-ray source focal spot size
- X-ray energy, intensity and spectral shape
- Geometrical magnification
- Center of rotation
- Size and number of detector elements
- Detector Point Spread Function quality
- Number of projections

B Other determining factors

Ray spacing
Angular view spacing
Mechanical accuracy of rotation table.
Reconstruction filter function
Reconstruction matrix
Display matrix

SPATIAL RESOLUTION AND GEOMETRICAL MAGNIFICATION

An important parameter for the quality in μ-XCT imaging is spatial resolution. In μ-XCT high demand is put to the reconstruction of sharp μ-XCT images with large detail thus obtaining large spatial resolution. Basically μ-XCT scanning exploits the advantage of geometrical enlargement by straight X-rays emanating from a virtual “point source” (see Figure 2). The factors determining the spatial resolution in μ-XCT reconstruction images are: the geometrical magnification factor (M), the detector resolution (d) and the focal spot width (f) of the X-ray source. Quantitative under-standing for the parameters playing a role in the end-quality for the reconstruction image is obtained via the contributing factors in the effective beam width at the center of the object.

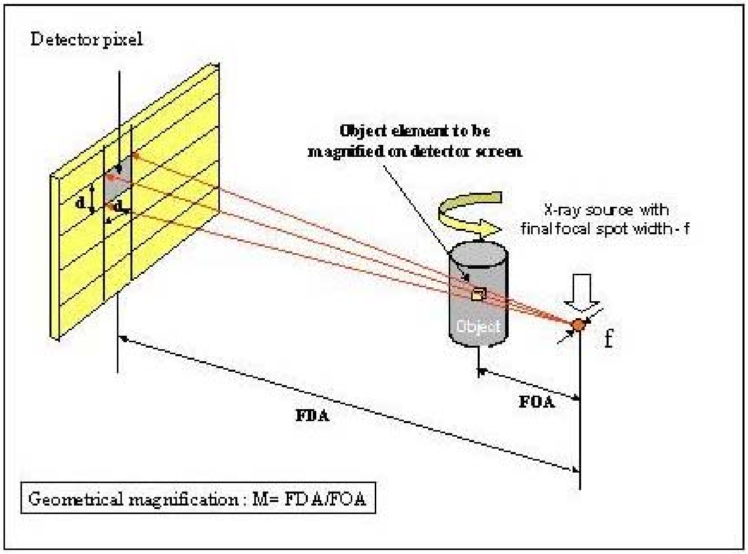

Figure 2: Geometrical magnification in 3-D cone beam µ-XCT (see Glossary of symbols).

The effective beam width at the center of rotation [Ref-2] expressed by:

$$U_{str} = \sqrt{\left(\frac{d}{M}\right)^2 + \left(\frac{f}{\frac{M}{M-1}}\right)^2} \qquad 1$$

In practice in µ-XCT, the geometrical magnification factor M varies between 40 and 110. Equation 1 has been validated for typical µ-XCT scanning conditions. Figure 3 shows the effective beam width (U_{str}) for a practical situation during a µ-XCT scan of a small, 10 mm diameter core. We use a large M factor of 90 as well as a fine detector resolution of 25 µm/pixel, currently provided by modern CCD detectors.

An important finding is that under large geometrical magnification (M>40) and fine detector resolution, the effective beam width (U_{str}) is completely determined by the size of the focal spot (f). Therefore, for high-resolution µ-XCT we need an X-ray source with an ultra small, point-like, focal spot. Another observation from this analysis is that the ratio d/M, which equals to the object element size (d'), is the main determining factor for µ-XCT rather that the actual size of the detector pixel element (d).

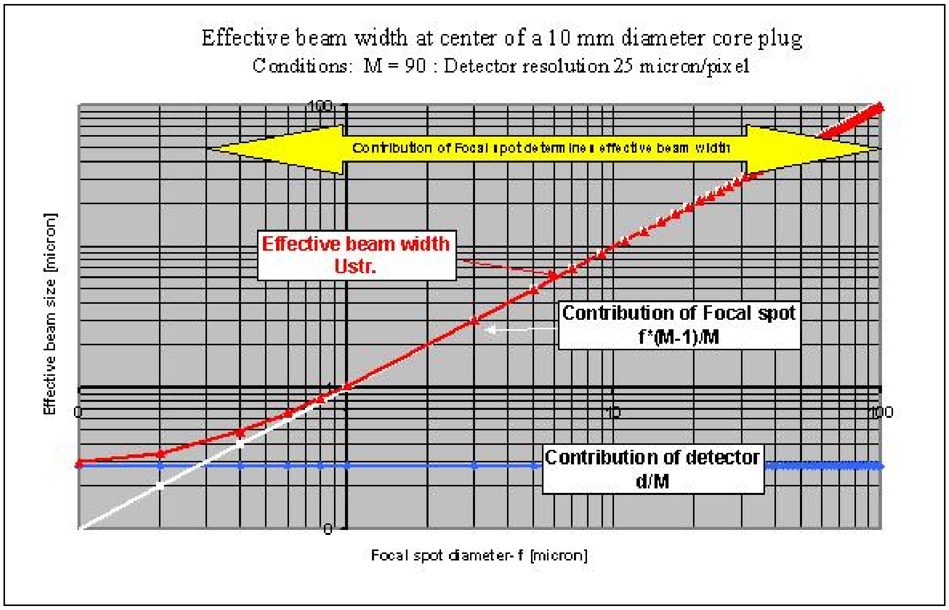

Figure 3: Effective beam width for large magnification and large detector resolution. Note that the applied distances and magnification are for imaging of a relative small 10 mm diameter object. (The X-scale units are printed in the graph).

From a geometrical point of view it makes no difference whether one puts a high resolution detector with small pixel elements at short distance to the object or a crude resolution detector with large pixel elements at large distance to the object. Thus in µ-XCT the main parameter in fact is the smallest volume element in the object (d’) one wishes to visualize. This determines the µ-XCT machine configuration or vice versa. Equipment parameters such as distances FDA and FOA have to be tuned in accordance to the given pixel size (d).

X-RAY SOURCE PARAMETERS

For high resolution µ-XCT scanning with large Signal-to-noise ratio (SNR) we should apply an X-ray source offering large flux and a small focal spot.

Drift in source flux and focal spot position

In µ-XCT one usually aims for a stable flux. During normal µ-XCT where the full object is in the beam, the zero-beam is measured at the detector edge channels. Because the main-beam is log normalized to the zero-beam and both signals suffer in the same way the drift in X-ray flux, the effect of source flux drift is canceled. However an exception forms the case of local tomography where the boundaries of the object are outside the scan field and we miss the zero-beam for log normalization. A concern however is with a drift in focal spot position inside the source. During long-term operation of an X-ray source, the focused electron beam will erode the anode and the focal spot will change in position [Ref-3]. This is a source of error in µ-XCT because the reconstruction software assumes a fixed position of focal spot position during a scan. Thus, for µ-XCT X-ray tube operation should be appraised on stability of the position of focal spot rather than on drift in flux.

Effect of wide focal spot

In 2-D and 3-D µ-XCT image reconstruction the mathematics are based on assuming ray paths emanating from an infinite small point. Rays that emanate from different positions at the line source traverse the object along different directions towards the same detector element. Then the detector intensities yield a blurred attenuation profile. Image reconstruction using these blurred attenuation profiles result into an image with considerable un-sharpness. Again it follows that for high resolution µ-XCT imaging a bright X-ray source should be used with ultra small focal spot.

Intensities of micro-focal X-rays sources

The noise and signal-to-noise ratio (SNR) in µ-XCT reconstruction images depend very much on the brilliance of the X-ray source. In order to get an understanding of the expected level in SNR in µ-XCT scanning it is important to have quantitative information on the photon flux produced by micro-focal X-ray sources. Many vendors present data with regard to output energy flux in Watt. We however would favor the use of photon flux in terms of [photons/s.cm^2] at a specific distance from the source such that we can use these intensities in the SNR equations. Therefore we have developed a procedure, similar to Callendar & White [Ref. 4], whereby the measured radiation output, for instance, given in Röntgen per minute or Sievert per hour units, have been transformed to X-ray beam intensity at a fixed reference distance from the focal spot. It turns out that X-ray beam intensities from micro-focal X-ray sources are in the order of 10^{10} to 10^{11} [photons/s.cm^2] at 20 cm reference distance to the focal spot. To our knowledge currently the best micro focal X-ray sources available offer a minimum focal spot diameter of 5 µm. However, new nano-focus X-ray sources are coming to the market offering ultra fine focal spot width (<1 µm). The data from two commercial nano-focus sources show beam intensities at 10-Watt load of about 8 10^{10} [photons/s.cm^2] at 20 cm distance. This radiation yield is only marginally smaller than for micro focal X-ray sources considering the ultra small focal spot of ~ 0.75µm. Therefore nano-focus X-ray sources would provide a resolution improvement for tabletop µ-XCT performance.

TABLETOP MICRO-XCT APPRAISAL TESTING

Each tabletop µ-XCT system provides a certain quality for the final reconstruction image. For tabletop µ-XCT appraisal testing we choose the direct route of providing well-defined test samples to vendors of commercial µ-XCT systems and judging the quality of the reconstruction images by comparison to prior information on the structure of these test samples. The samples are predominantly carbonate reservoir rock samples but also well-defined synthetic rock samples such as a core made up from sintered spherical Quartz beads. Pore throat distributions from duplo sets of these test samples have been obtained by the petrophysical “autopore” method. The auto pore method is a standardized petrophysical characterization method involving stepwise injection of mercury into a small (unstressed) rock sample under vacuum. We have also applied high-resolution electron microscopy (SEM) scanning on thin sections from duplo samples. The purpose of these analyses is to obtain an independent evaluation of the porous structure at sub-micron scale. The SEM images give prior information to the ideal appearance of the reconstruction images to be obtained from µ-XCT scans. Small cylindrical cores were drilled from the various dry rock samples to a diameter of 4 mm and a length of 10 mm. In a second test series the core diameter was reduced further to 2 mm diameter.

The market study on tabletop µ-XCT systems that we have performed are based on 17 vendors of tabletop µ-XCT systems and 4 scientific institutes with in-house built systems distributed over 8 countries. Another domain in µ-XCT systems is that of micro CT-scanners for biomedical research viz. for in-vivo animal pet scanning. Basically these scanners are downscaled medical CT-scanners whereby the source and detectors are statically connected and rotate 360^0 around an object. In our view µ-XCT systems with an open structure (see Figure 1) allowing variable geometric magnification are best suited for special core analysis studies. We have appraised a limited number of commercial tabletop µ-XCT scanners. Thereby we asked vendors to apply the best setting on their machines with the aim to produce reconstructions with the best spatial resolution for the given test samples. Qualitative assessment has been carried out on the produced reconstruction images. First, a display of the full (1024x1024) pixel reconstruction image of the complete sample provides a total view of the internal morphology and contrasting features. Next, a small region-of-interest (ROI) of the same scan image is maximally enlarged. Thereby, we have a better chance to study the effect of spatial resolution on µ-XCT image quality.

In this paper we focus in general terms on the capability of commercial tabletop µ-XCT scanners. With kind permission from vendor-A we discuss the results obtained with its µ-XCT scanner as being typical for tabletop systems. Scanning is carried out on our samples using a micro focal X-ray source with 90 kV energy and a focal spot size ~5 µm. The number of projections is 1500 projections over 360^0. Maximal geometrical magnification and resolution could be obtained by using 2 mm diameter samples in combination to a focal spot – object distance of 10 mm, producing a geometrical magnification factor of 60. The 2-D scan images comprise1024x1024 pixels, with a reconstruction diameter of 2.72 mm and a reconstruction pixel size of 2.65 µm/pixel. 2-D reconstruction images are presented in Figures 4-5. The resulting scan images obtained on our test cores show excellent resolution. The spatial resolution however is in the order of ~5µm. This is the same for a scan image made on a typical Middle East carbonate core (sample–2) provided by another vendor-B (see Figure 6). Vendor-C has done its utmost to maximize on the spatial resolution using a 2 mm diameter core. Figure 7 shows a µ-XCT scan image of the sintered Quartz core using an undisputed large geometrical magnification factor of 103. The spatial resolution in this case is estimated to 3-5 µm/pixel. The morphology of a porous structure from spherical grains can be recognized.

Based on performed appraisal testing of a number of commercial µ-XCT scanners we have come to the general observation that the spatial resolution is in the order of 5-8 microns and larger. This resolution is sufficient for a wide range of sandstones- and vuggy carbonate rock but insufficient to probe micro pores in tight carbonate rock.

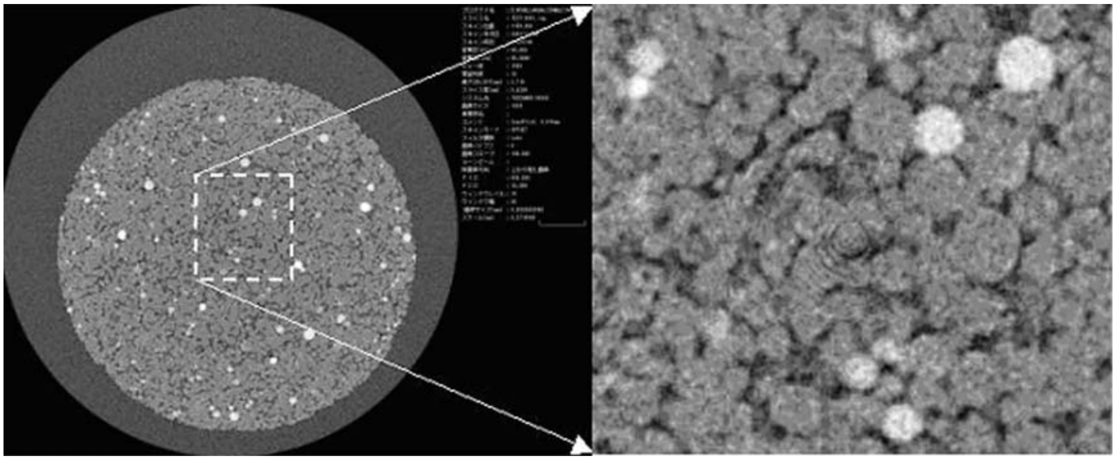

Figure 4: µ-XCT scan of sintered Quartz core sample, 2 mm diameter. Full core overview at left. At right a detail from the image at left is maximally zoomed-in and displayed. 90 kV scan, 1024x1024 pixels, recon circle diameter 2.72 mm, 2.65 mu/pix, geometrical magn.=60. (Courtesy of vendor-A).

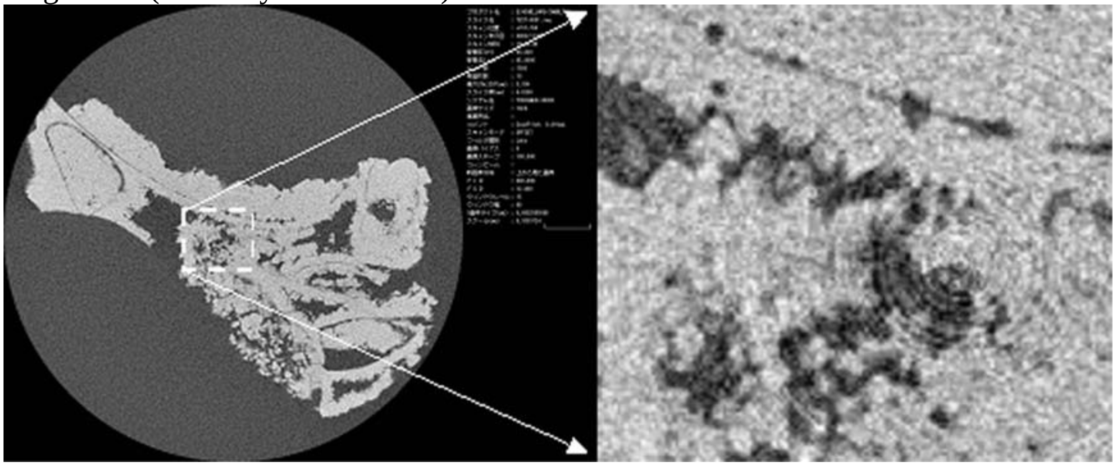

Figure 5: µ-XCT scan of carbonate sample-1, 2 mm diameter. Full core overview at left. On theright a detail from the left image is maximally zoomed-in and displayed. 90 kV scan, 1024x1024 pixels, recon circle diameter 2.72 mm, 2.65 mu/pix, geometrical magn.=60. (Courtesy of vendor-A).

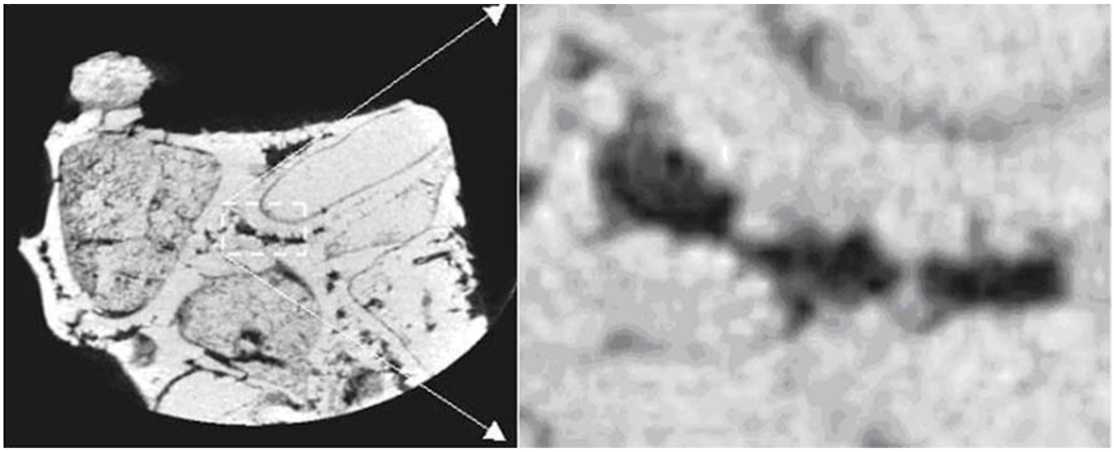

Figure 6: µ-XCT scan of carbonate sample-2, 2 mm diameter. Full core overview at left. At right a detail from the image at left is maximally zoomed-in and displayed. 80 kV scan, 512x512 pixels, recon circle diameter 2.12 mm, 4.3 mu/pix, geometrical magn.=87. (Courtesy of vendor-B).

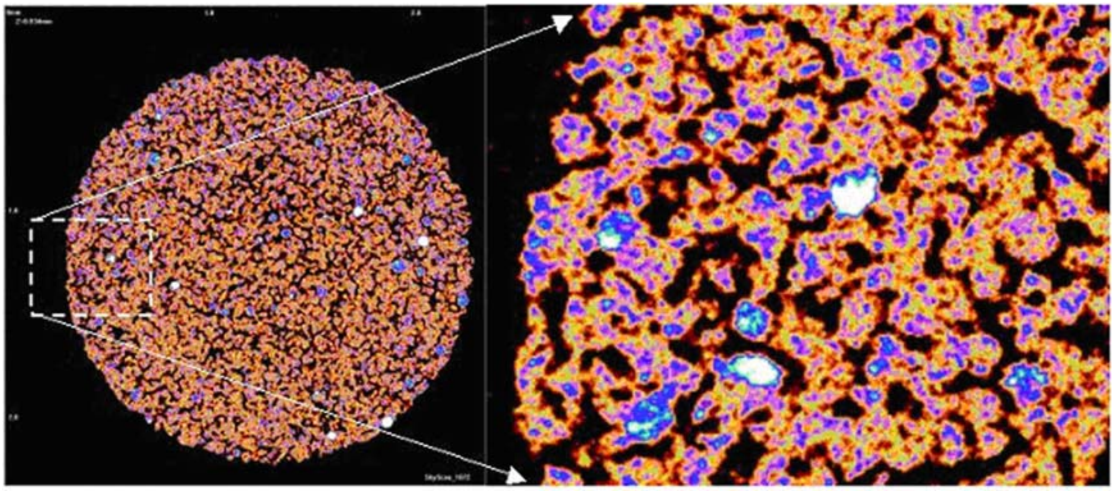

Figure 7: High resolution scan overview on sintered Quartz core sample, with 2 mm diameter. Full core overview at left. At right a detail from the image at left is maximally zoomed-in and displayed. 80 kV scan, 1024x1024 pixels, recon circle diameter 2.33 mm, 2.28 mu/pix. (Courtesy of vendor-C).

SYNCHROTRON BEAM LINE µ-XCT

In order to identify the current limit in spatial resolution offered by third generation beam lines and for bench marking in this study we carried out micro tomography scanning tests on our set of test samples at the European Synchrotron Radiation Facility (ESRF, France) [Ref-6]. The synchrotron beam line tomography scanning has been carried at ESRF using 20.5 keV monochromatic X-rays. The reconstructions were carried out on a 2048*2048 matrix grid. Our test were done with the following scanning modes:

- Full object scanning using a field of view of 2.8 mm. The spatial resolution is 1.4µm.

- Local tomography using a field of view of 0.7 mm. The spatial resolution is ~0.3µm.

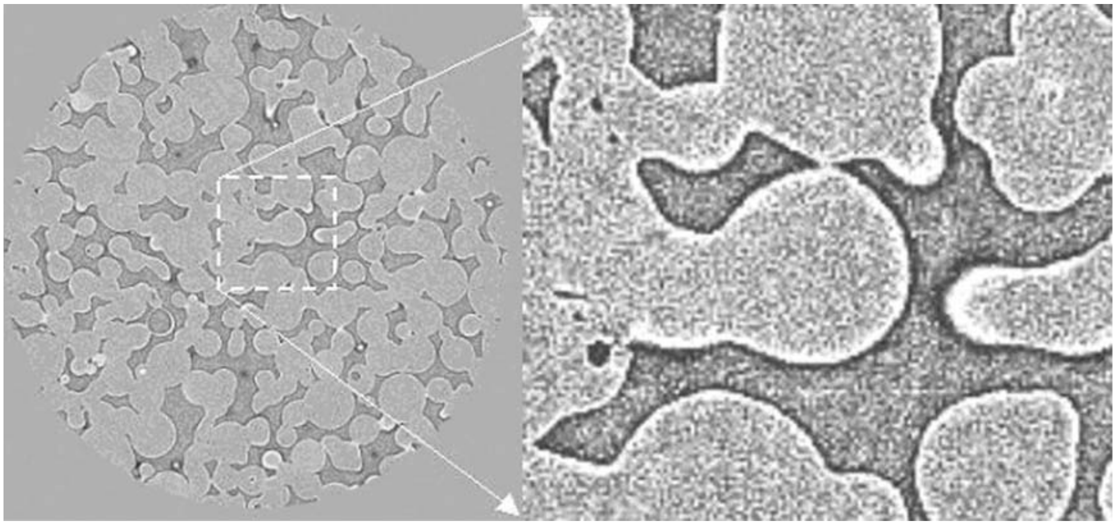

Figure 8: Synchrotron beam line scan overview on sintered Quartz core sample, 2 mm diameter. At left local tomogram from the center (0.7 mm diameter) of Quartz core. At right a detail from the image at left is maximally zoomed-in and displayed. 20 kV scan, 2048x2048 pixels, recon circle diameter 0.7 mm, 0.33 µm/pix.

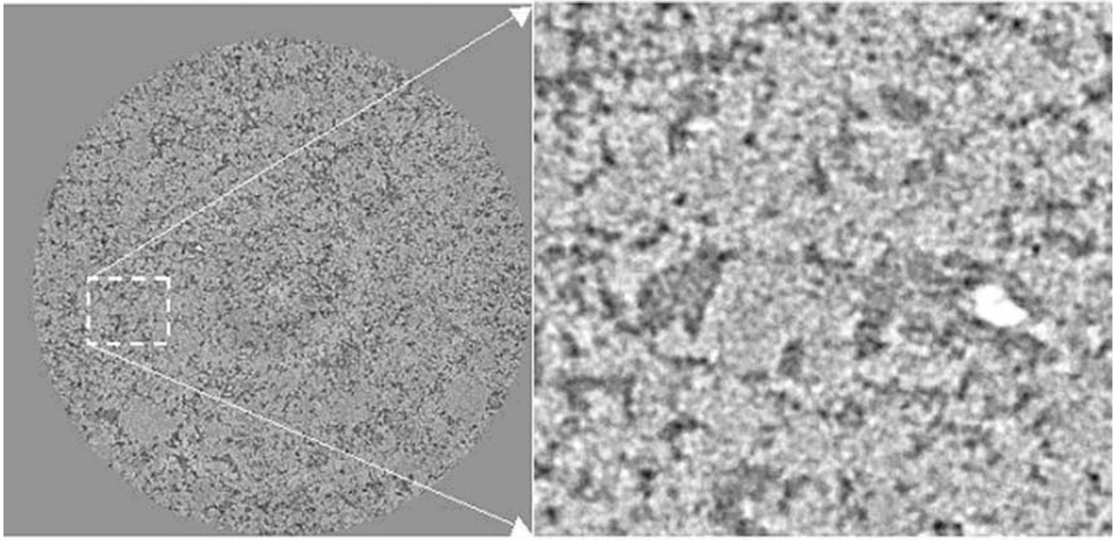

Figure 9: Synchrotron beam line scan on carbonate core sample-3, 2 mm diameter. At left local tomogram from the center (0.7 mm diameter) of the core. At right a detail from the image at left is maximally zoomed-in and displayed. 20 kV scan, 2048x2048 pixels, recon circle diameter 0.7 mm, 0.33 µm/pix.

The µ-XCT images obtained from full object scans on the 2 mm diameter carbonate cores (not shown) are reconstructed on a 2048x2048 pixel grid with 1.4 µm/pixel spatial resolution. This resolution is sufficient to observe all lithology features relevant to this rock type. This improves further for the local tomograms i.e. reconstructions of a 0.7 mm diameter region in the center. The local core scan images are reconstructed on a 2048x2048 pixel grid with 0.33 µm/pixel spatial resolution. This is an unprecedented low spatial resolution. Figure 8 shows the local tomogram from the sintered Quartz core. Evidently the 0.3 µm spatial resolution enables to visualize and to recognize the spherical quartz grains and inverted open voids. However, the quantitative CT values are a source of concern. Figure 9 shows the local tomogram from another Middle East carbonate core (sample 3) indicating a spatial resolution of 1.4 micron on the 2 mm diameter specimen. A better spatial resolution of ~0.3 µm/pixel on a 0.7 mm diameter local tomogram of this core is obtained. Note that local tomography applies filtering disregarding intensity data from places outside the 0.7 mm diameter reconstruction circle. This may affect the accuracy for the CT data inside the reconstruction circle. The contrast resolution offered by the ESRF images could be improved upon. Possibly errors are introduced by a thresh holding and CT-data truncation procedure.

In conclusion the synchrotron beam line scan reconstruction images show superior spatial resolution. However, when using local tomography only small part of the core is visualized.

NEW TECHNOLOGY FOR PERFORMANCE IMPROVEMENT

We have investigated the possibilities to push further current performance boundaries in spatial resolution for tabletop µ-XCT imaging from 5-8 µm to below 1µm. To this aim we would need an ultra-bright, point-like X-ray source.

Anode target optimization

We have investigated possibilities for performance improvement of current micro focal X-ray sources by anode target optimization. The best target material for obtaining a small focal spot with high X-ray yield should possess the best combination of atomic number, specific density (X-ray generation), thermal conductivity (heat disposal), melting and boiling temperature (life time). We have carried out a simple benchmarking study towards material properties. Heavy metals such as Tungsten, Iridium, Platinum and Osmium could be good target candidates. To support this study the Technical University Delft (The Netherlands) has carried out Monte Carlo radiation transport simulations on transmission anode targets such as used in transmission micro-focus X-ray sources using GEANT4 [Ref-6]. The Monte Carlo calculations show that an X-ray anode of Iridium with a thickness much smaller than the electron range (<1 µm) may be a feasible approach to minimize the spot size of a trans- mission type X-ray tube. The thin target restricts the physical boundaries of the focal spot to very small sizes.

Laser X-ray technology

We have also investigated a number of industry innovations that possibly could provide ultra bright point X-ray sources other than via incremental improvement of transmission micro focal X-ray sources. A potential new X-ray technology for µ-XCT could be by using femto-second pulsed laser plasma X-ray sources as bright, point-like X-ray sources [Ref-7]. Note that in standard X-ray tubes electrons inside the anode repel each other with electrostatic forces while the light photons from laser beams can be focused by optical mirrors to very small spot and high intensities. A promising research study on the usefulness of a Laser X-ray Source for micro tomography has been carried out for us at the Rutherford Appleton Laboratory (UK). Also, other researchers like Grätz et. al. [Ref-8] have demonstrated hard X-ray generation 50 – 68 keV using a pico-second terawatt laser system using liquid droplet targets. Laser X-ray generation seems promising for µ-XCT imaging. However, more R&D efforts should be devoted in this direction to achieve the goal of a tabletop ultra-bright laser X-ray systems producing 20 – 50 keV X-rays.

CONCLUSIONS

This paper has addressed the important measurement parameters and resolution aspects related to 2-D and 3-D µ-XCT scanning. Micro X-ray tomography can be carried out by means of tabletop (desktop) µ-XCT scanning equipment or by synchrotron µ-XCT systems. Tabletop µ-XCT scanners are flexible and well suited for in-house advanced core analysis. Synchrotron µ-XCT systems on the other hand offer superior spatial resolution but are considered inflexible for R&D on a routine basis.

In this study, we have presented the fundamentals of tabletop µ-XCT scanning. The relevant scanning parameters that determine spatial resolution and contrast resolution are: geometrical magnification, focal spot size of the source, detector PSF, KV and mA (flux) setting of the source.

- Key point in µ-XCT is the size of the smallest volume element viz. spatial resolution that is desired inside the object of investigation. This can be adjusted via the geometrical magnification. At large magnification the focal spot size of the source sets the limit in spatial resolution. Detector resolution is of lesser importance.
- In order to judge the brilliance of current micro-focal X-ray sources and for SNR calculation in µ-XCT we have transformed the radiation output reported on commercially available X-ray sources in terms of a more practical photon intensity units. X-ray beam intensities from micro-focal X-ray sources are in the order of 10^{10} to 10^{11} [photons/s.cm^2] at 20 cm reference distance. Nano-focus sources show beam intensities of about 8 10^{10} [photons/s.cm^2] at 20 cm distance. This radiation yield is acceptably large considering the ultra small focal spot of ~ 0.75 µm.
- We have assessed the imaging performance capability of a number of commercial µ-XCT machines. Commercial tabletop µ-XCT scanners usually apply a 1024^2 pixel reconstruction grid and show spatial resolution of 5-8 µm. For 2 mm diameter samples 2 µm is attainable. This resolution is sufficient for some sandstone- and vuggy carbonate rock but insufficient to probe micro pores in tight carbonate rock. For reasons of benchmarking and scouting the limit in µ-XCT additional tests has been carried out at the European Synchrotron Facility (ESRF) in Grenoble (France). Spatial resolution in the order of 0.3 µm is attainable using “local tomography” and a 2048^2 pixel reconstruction grid.
- We have investigated the possibilities to push further current performance boundaries in spatial resolution for tabletop µ-XCT scanners from 5-8 µm to below 1µm. At short term using nano-focus transmission X-ray sources seem to be the best option for performance improvement in µ-XCT. Target optimization can help to further reduce the focal spot size. We have also considered other technical innovations to provide an

ultra-bright, point X-ray source. In the next decade table top laser plasma X-ray sources may become available to provide bright, point-like X-ray sources for µ-XCT. This is because optically light beams can be collimated to very narrow proportions.

- Micro CT-scanning is in our view a key enabler for new opportunities in E&P research and development. Promising applications of µ-XCT are in the field of reservoir engineering, specifically relating to pore network modeling and in geochemistry and geomechanics.

GLOSSARY OF SYMBOLS

d : detector resolution or pixel width
FOA : distance between focal spot and center of object
FDA : distance between focal spot and detector
f : focal spot diameter
M : geometrical magnification factor M= FDA/FOA
U_{str} : Effective X-ray beam width at the center of the object
PSF : Point Spread Function
MTF : Modular Transfer Function
SNR : Signal-to-Noise-Ratio

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