

X-ray MicroCT in Conjunction with Other Techniques for Core Analysis

I.V. Yakimchuk¹, A.S. Denisenko¹, B.D. Sharchilev¹, and I.A. Varfolomeev^{1,2}

¹Schlumberger, 119285, 13 Pudovkina str., Moscow, Russia,

²Moscow Institute of Physics and Technology, 141700, 9 Institutskiy per., Dolgoprudny, Russia

Aims

Over the last decades X-ray micro computed tomography (microCT) has become well accepted in the petrophysics community as a convenient nondestructive technique for studying internal core structure. Being effective separately, X-ray microCT has the potential to support and/or combine to be productive with other material study methods. In this work, we demonstrate several examples of such complementation in the area of core analysis.

Method

In accordance with the above, X-ray microCT is one of the methods used in this work, namely the SkyScan 1172 [1]. Other techniques used to obtain the presented results are nuclear magnetic resonance (NMR) and scanning electron microscopy (SEM) including energy dispersive X-ray analysis (EDX). In particular, for geological core sample studies, these methods may be used together with X-ray microCT to overcome limitations in spatial resolution and mineral identification. Both points are essential for an adequate representation of the rock structure in terms of multiscale pore space and multiminerals solid matrix. Typical rock samples (mini-plugs) used in the study are cylindrical with a diameter of approximately 8 mm and height of approximately 10 mm. A description of used methods is presented below.

X-ray MicroCT. As rock mini-plugs are rather dense, the mode of X-ray tube was 100 kV and 100 μ A. Reconstructed datasets have the size of approximately 4000 \times 4000 \times 2000 with an effective pixel size of approximately 2.2 μ m. In some cases (e.g. tight rocks) it is not sufficient and the higher detailing is required. For image processing and analysis we used both commercial and internally developed software.

NMR. One of the critical pieces of information derived from laboratory core analysis is a pore size distribution. In contrast to X-ray microCT, laboratory NMR machines observe hydrogen nuclei of fluids constrained by pores of any size and geometry. Using T_1 or T_2 relaxation spectra one could simply construct a reliable pore size distribution within a rock by several minutes [2]. All presented NMR measurements were carried out using an Oxford Instruments relaxometer, which contains a MARANi-Pharmasence magnet block and DRX HF electronic control system. The resonance frequency of 20.6 MHz corresponds to hydrogen nuclei spin precession in a magnetic field of roughly 0.5 T [3].

NMR is an indirect method for pore size evaluation based on proportionality between the relaxation times and surface-to-volume ratio (or approximately a pore diameter) of each pore. The coefficient of proportionality is relaxation activity (relaxivity) or ability of mineral pore surfaces to change magnetization of the spins due to their collision or interaction. It is common to use a single value of relaxivity value for a single core assuming that pore surface properties of a small sample are homogeneous enough. The transformation from relaxation times to geometrical sizes requires a calibration by direct methods. The results of such calibration based on X-ray microCT images study of core mini-plugs are presented below.

Modern **SEM** provide much higher resolution (Figure 1) than X-ray microCT (down to \sim 1 nm) and, unlike NMR T_1 – T_2 spectroscopy, it is a direct imaging method. Thus, SEM images reflect the geometry of rock internal structures in much more detail; therefore, it can be used for

accurate numerical simulations of physical processes in porous rock. However, it is worth bearing in mind that SEM is a 2D imaging technique. An attractive and promising idea is to register a high-resolution 2D image with 3D images micro-CT image for mapping the subvoxel characteristics. For example (see below), effective subvoxel porosity can be assigned to each voxel of 3D X-ray microCT image, i.e. some voxels are classified as neither fully solid, nor fully void, but partially void with some porosity value.

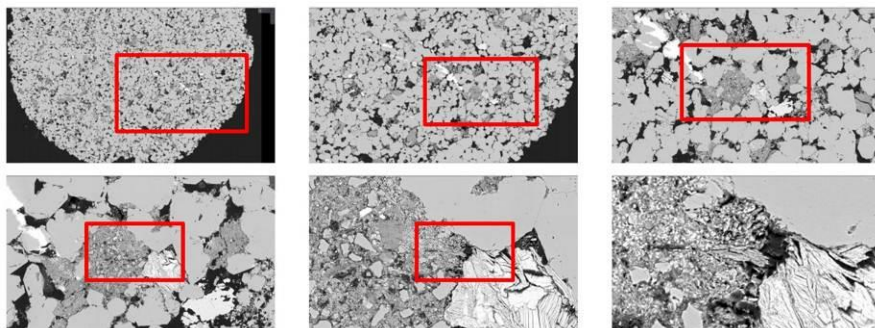


Figure 1: High-resolution SEM image of a sandstone Ø8 mm mini-plug, consecutive zooming in.

Discussed SEM results were obtained by QEMSCAN 650F (FEI) microscope, which is a special modification of Quanta 650F (FEI). For the aforementioned 8-mm mini-plugs, the tile-by-tile scanning of a full slice with 1-nm resolution would produce a huge image of size 8 million \times 8 million pixels (>50 TB). Thereby, the acquisition parameters should be modified by decreasing the region of interest and/or the resolution. High-resolution maps were collected from the full slice of the mini-plug with resolution approximately 100 nm (image size: \sim 80,000 \times 80,000; file size: \sim 8 GB).

Energy Dispersive X-Ray Analysis. EDX is one of the most popular elemental analysis techniques among various options and modes of the SEM method. The current generation of EDX detectors was presented in the late 1990s. However, even a decade ago these detectors were still too slow to map a distribution of elements along the surface of a typical rock sample, as described above. Instead, EDX was used to analyze individual points. Recent advances in EDX technology decreased the time required to analyze 4,000 \times 4,000 points of the sample surface to about 24 h, which is similar to the X-ray microCT imaging duration. Existing software allows automatic determination of mineralogy in each pixel, based on its EDX spectrum [4]. An example of such classification, i.e. a 2D mineral map, is demonstrated in Figure 2.

As in a previous case, the complementation of the 2D mineral map and the 3D X-ray microCT allows construction of a 3D mineral map of the sample. We note at once that in some cases it is possible to construct 3D mineral map based only on tomographical data because of good enough X-ray attenuation contrast between minerals constituting the sample [5].

Mineralogical information is very important by itself. Knowledge of reservoir rock mineralogy makes an impact on understanding and predicting potential of hydrocarbon reservoir and, as a result, improving of oil/gas field development. Moreover, 3D mineral distribution allows performing numerical simulations on 3D digital rock model concerning the interior mineral distribution.

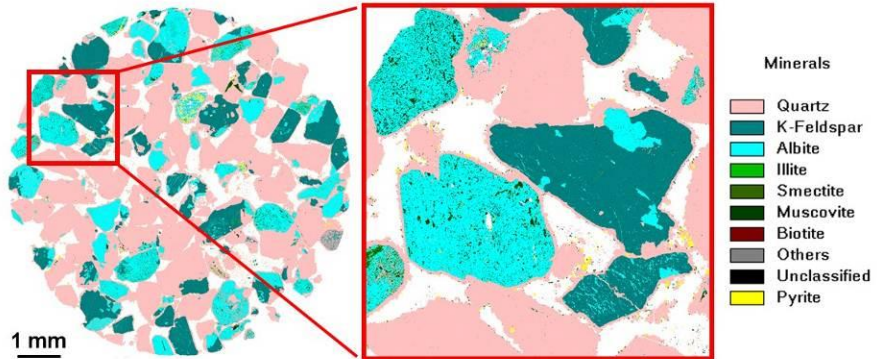


Figure 2: 2D mineral map of a sandstone core mini-plug (Ø8 mm) derived from SEM EDX analysis.

All utilized methods and related questions are described in general. Corresponding results are presented in the next section.

Results

Calibration of NMR by X-ray MicroCT. In contrast to NMR, X-ray microCT allows direct measurement of pore space geometry in 3D. For this purpose, the reconstructed 3D image of a core sample should be segmented into two classes of objects: pores and minerals. Further calculation of the pore size distribution can be performed in various ways.

Individual pore analysis. The most intuitively obvious approach consists of the separation of a whole pore space on a set of individual pores and subsequent quantitative analysis of each. The general algorithms for separation are based on watershed technique [6]. Equivalent spherical diameter is one of the most commonly used quantities for estimating the effective size of the object body. Finally, the histogram of the size values can be constructed.

Sphere-fitting technique. An alternative method for calculating the object size distribution was proposed previously [7]. This technique is based on a sphere fitting inside the 3D structure. In that case, the local thickness of the object at point A is defined as the diameter of the largest possible sphere that fully inscribes into the structure and covers point A. Thus, point A may not be a center of the covering sphere. The final distribution of the pore sizes may be obtained by calculating the histogram of the 3D local thickness distribution.

In our view, the second algorithm more adequately describes the pore space and more correctly fits with physical phenomena underlying the NMR method.

Calculated pore size distribution from 3D X-ray microCT image should be superposed with NMR T_1 and T_2 spectra. A note is to be made that, due to limitations of X-ray microCT in spatial resolution, the calibration between relaxation times and pore sizes should rely mostly on the right part of the larger peak in size distribution, as shown in Figure 3. Figure 3 demonstrates the application of the described approach for a sandstone core mini-plug.

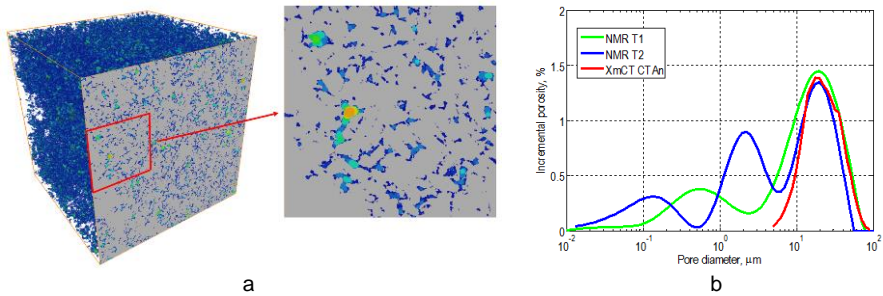


Figure 3: Sandstone core mini-plug ($\varnothing 8$ mm) with composition: silica 62%, feldspar 20%, clays 18%; porosity 27.8%, permeability by air 407 mD. a) 3D local thickness distribution of pore space. Grey color stands for solid phase. Pore space has colors from blue to red in dependence on local thickness value. b) Pore size distribution from X-ray microCT and calibrated NMR.

Obtained relaxivity value (a coefficient of the proportionality between NMR time and pore size) is in satisfactory agreement with the literature. It should be noted that incremental porosity (axis Y on Figure 3b) derived from NMR and X-ray microCT is approximately the same in the range above X-ray microCT resolution without any manual fitting. Moreover, as one can see, NMR has detected more porosity (27.6%) for the core sample than X-ray microCT (15.7%) and this value perfectly fits with laboratory-measured porosity on the mini-plug (27.8%).

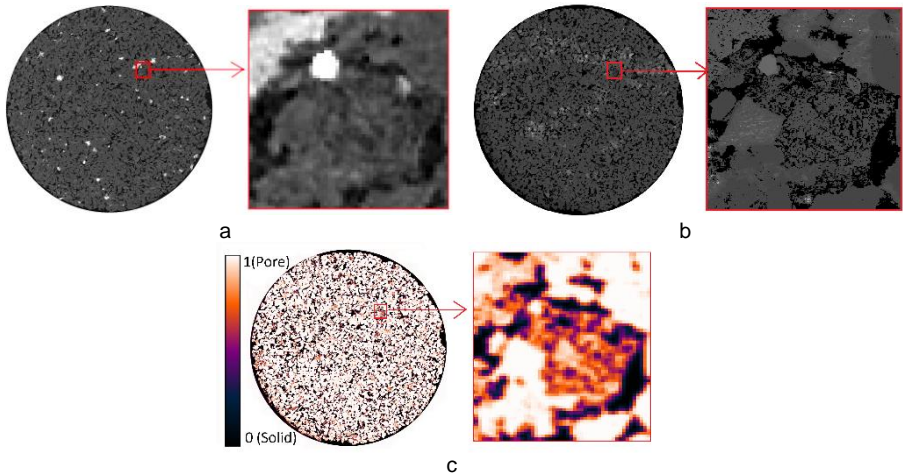


Figure 4: 2D images of a sandstone core mini-plug ($\varnothing 8$ mm). a) Slice from 3D micro-CT image; b) high-resolution SEM image; and c) porosity distribution with subvoxel accuracy.

3D Porosity Distribution with Subvoxel Accuracy is an estimation of real porosity distribution (Figure 4). Two ideas have been implemented to make this estimation. First, the porosity of a 3D X-ray microCT image may be fitted to the laboratory-measured porosity. During this procedure, voxels in some range of ambiguous colors are considered as mixture of solid and void phases with the ratio corresponding to the local X-ray attenuation. The proportionality coefficient should satisfy the equality of image and laboratory-measured

porosity. Second, high-resolution SEM imaging may support X-ray microCT interpretation. Proper registration of the SEM 2D image with virtual slice of 3D X-ray microCT image allows adjusting parameters of correspondence between greyscale levels and subvoxel porosity values. Besides, having registered 2D SEM with 3D X-ray microCT images of the same sample, it is possible to use machine learning techniques for further classification of the whole 3D image.

It should be mentioned that correct and efficient registration of rather large 2D and 3D images is a complicated task by itself. Generally, mechanical cutting of the sample for SEM imaging could not be done exactly parallel to X-ray microCT slices. Thereby, some mismatching in coordinates and orientation angles occurs between both images. To solve the problem, we have applied contour registration approach reducing the task from 2D–3D to 1D–2D. The approach is based on the assumption that porous core mini-plugs have a rather individual 2D side surface and each slice would have a 1D contour uniquely matched with the whole side surface.

The obtained distribution is suitable and can potentially increase the quality of numerical simulation of physical processes in porous rock. As an example, the calculation of electrical resistivity can result in significant overestimation if only large (resolved by X-ray microCT) pores are taken into account. While subvoxel porosity distribution preserves information about pore space connectivity and provides much more adequate results.

3D Mineralogy Mapping. The essence of the method consists in finding correspondence between the various characteristics of X-ray microCT image voxels and their mineral content, defined from SEM-EDX image [8]. As in previous case, this technique involves spatial registration of 2D elemental or mineral distribution image with 3D X-ray microCT image. A number of local features is considered to characterize each voxel of a 3D X-ray microCT image: reconstructed X-ray attenuation coefficient value, textural, and morphological properties. Unfortunately, greyscale value (attenuation) itself is not enough for distinguishing different minerals in a general case (Figure 5). Finally, the full 3D microCT image is classified according to its mineral type (Figure 6).

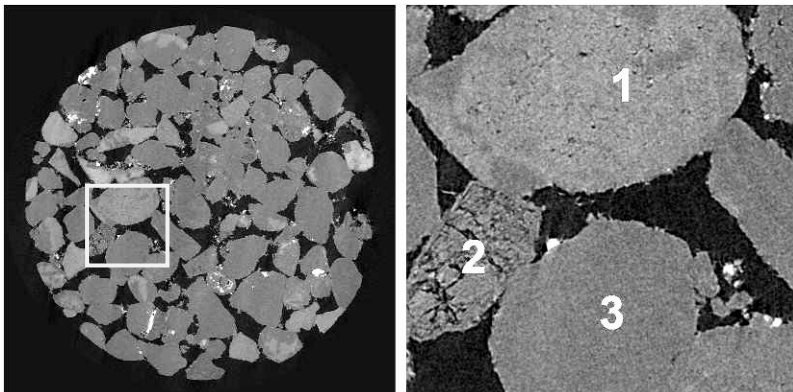


Figure 5: Sandstone core mini-plug ($\varnothing 8$ mm) cross-section (left) and its fragment (right). 1, 2, and 3 are different mineral grains with similar X-ray attenuation values.

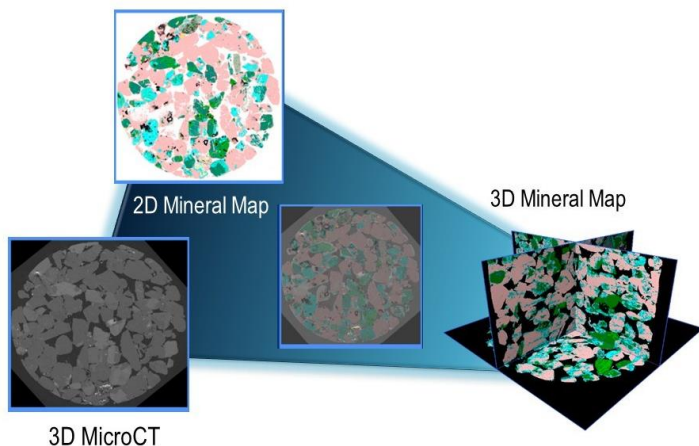


Figure 6: Construction of 3D Mineral Map for sandstone core (Ø8 mm) mini-plug.

Conclusion

X-ray microCT as well as any experimental method has its advantages and drawbacks. In this study, we have demonstrated how the combination of X-ray microCT with other techniques breaks through some limitations of these methods alone. As a result, NMR T_1 – T_2 spectroscopy complemented with X-ray microCT allows studying of the pore size distributions in more robust way. Together with high-resolution SEM imaging, X-ray microCT can produce evaluated 3D maps of core microporosity. Finally, X-ray microCT supported by SEM-EDX is a perspective approach for constructing 3D Mineral Models of the geological specimens.

References:

1. <http://www.skyscan.be/products/1172.htm>.
2. Kenyon, W.E., "Nuclear Magnetic Resonance as a Petrophysical Measurement," *Nuclear Geophysics*, vol. 6, no. 2, pp. 153–171, 1992.
3. Denisenko, A., and Yakimchuk, I. "X-Ray Microtomography and NMR: complementary tools for evaluation of pore structure within a core." SCA 2014. http://www.scaweb.org/assets/papers/2014_papers/SCA2014-038.pdf.
4. Butcher, A.R., Helms, T.A., Gottlieb, P. et al., "Advances in the quantification of gold deportment by QemSCAN." Seventh Mill Operators' Conference, Australasian Institute of Mining and Metallurgy, pp. 267–271, 2000.
5. Koroteev, D., Mutina, A., and Sasov, A. "Using X-ray microCT for 3D mineral mapping," Book of Abstracts of MicroCT User Meeting, pp. 40–45, 2011.
6. Meyer, F., "Topographic distance and watershed lines," *Signal Processing*, vol. 38, pp. 113–125, 1994.
7. Hildebrand, T., and Ruegsegger, P., "A new method for the model independent assessment of thickness in three dimensional images," *Journal of Microscopy*, vol. 185, pp. 67–75, 1997a.
8. Yakimchuk, I.V., Varfolomeev, I.A., and Korobkov, D.A. "Use of X-Ray MicroCT and Scanning Electron Microscopy for Constructing 3D Mineral Maps of Rock Samples," 18th International Microscopy Congress, Prague, Czech Republic, 7–12 September 2014. <http://www.microscopy.cz/abstracts/2759.pdf>