SCA2003-47: FOCUSSED ION BEAM ASSISTED THREE-DIMENSIONAL ROCK IMAGING AT SUBMICRON SCALE

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ABSTRACT

Computation of effective flow properties of fluids in porous media based on three dimensional (3D) pore structure information has become more successful in the last few years, due to both improvements in the input data and the network models. Computed X-ray microtomography has been successful in 3D pore imaging at micron scale, which is adequate for many sandstones. For other rocks of economic interest, such as chalk and diatomite, submicron resolution is needed in order to resolve the 3D-pore structure.

To achieve submicron resolution, a new method of sample serial sectioning and imaging using Focused Ion Beam (FIB) technology has been developed and 3D pore images of the pore system for diatomite and chalk have been obtained. FIB was used in the milling of layers as wide as 50 micrometers and as thin as 100 nanometers by sputtering of atoms from the sample surface. The focused ion beam, consisting of gallium ions (Ga+) accelerated by potentials of up to 30 kV and currents up to 20,000 pA, yields very clean, flat surfaces in which the pore-grain boundaries appear in high contrast. No distortion of the pore boundaries due to the ion milling is apparent. After each milling step, as a new surface is exposed, an image of the surface is generated. Using secondary electrons or ions, resolutions as high as 10 nm can be obtained. Afterwards, the 2D images are stacked in the computer and the 3D pore structure is reconstructed.

INTRODUCTION

Knowledge of the 3D pore structure is essential in computation of effective flow properties of fluids in porous media. Advances in the resolution of 3D pore structure at micron scale [1] and the sophistication of the network models [2] have yielded highly accurate predictions for petrophysical properties of certain rocks of interest to petroleum industry such as sandstones. While X-ray computed microtomography has been successful in 3D pore imaging of sandstones at micron scale resolutions, for submicron pore characterization, indirect methods, such as mercury injection, low field NMR or neutron scattering [3], or statistical data from 2D micrographs [4,5] have been used to construct synthetic 3D pore structures. Until now, direct 3D imaging at submicron scale, needed for other rocks of economic interest, such as chalk and diatomite, was not possible. To directly access the pore structure at submicron scale, we present a new approach, which uses FIB to mill successive layers of the rock material. The FIB

technology has been used extensively in microelectronics to access individual components with nanoscale accuracy to modify the circuitry at the prototype stage during the IC design. It also has been used in material sciences to prepare thin samples for use in Transmission Electron Microscopy (TEM). In Earth Sciences its previous use has been in preparing samples for TEM or to access inner regions for performing microanalysis [6].

EQUIPMENT

The FIB apparatus used is model FEI DB235, which has both electron and ion columns. It operates similarly to the scanning electron microscope (SEM), plus it also can focus unto the sample gallium ions (Ga+), accelerated by potentials up to 30kV. Thus, both secondary electrons and secondary ions can be generated at the point of impact on the sample. These charged particles are collected by appropriate electron (SED, CDM-E) or ion (CDM-I) detectors for image formation. The digitized images are stored in 1024 x 954, 8 bit, TIF format. Because the morphology of the sample is reflected in the amount of secondary electrons or ions generated, a contrast is possible between the high and low regions of the sample and a 3D image of the surface is produced. The contrast can also be due to difference in atomic numbers of the materials present at the sample surface. Because the gallium ions are orders of magnitude more massive than the electrons, the FIB mills the surface as it images it. To reduce the milling effect while imaging, the ion beam current is kept in the 10-100pA range, and the imaging time is reduced to seconds. On the other hand, if milling is the desired effect, the ion beam current can be increased by orders of magnitude (up to 20,000pA) and the exposure time can be increased up to hours. Another important feature of the instrument is a 5-axis computer controlled stage (x, y, z, rotation and tilt), which allows repeatable access to the same location on the sample surface. The stage allows orienting the sample either normal to the ion beam for drilling submicron scale patterns, or quasi parallel for milling successive layers.

PROCEDURES

To perform sample serial sectioning and imaging using FIB technology, samples have to be prepared by cleaning, cutting, polishing, mounting and carbon coating. Good sample cleaning is important to minimize hydrocarbon contamination of the instrument. Because the FIB mills submicron layers, the sample surface has to be flat at the micron scale. High roughness of the sample surface can significantly increase the time required for the FIB to mill the surface to the flatness required for serial sectioning. For soft materials, such as diatomite, a microtom can be used to prepare rectangular smooth samples. For samples impregnated with epoxy, diamond saw cutting and standard thin section procedures for polishing can be followed. To facilitate the sample orientation and milling, the sample has to be rectangular, presenting for milling a sharp edge with two surfaces at 90degree angle. The samples are mounted on 45degree SEM aluminum stubs using conducting carbon adhesive. The 45degree angle is necessary for the cutting to take place at the 90degree edge of the sample given the relative position of the stage, the ion and electron beam columns and the CDM detector. The last step is the standard SEM carbon coating procedure, to prevent build up of charges on the sample surface

during the imaging. The milling time required to mill areas 50x50 µm with a depth of 0.1µm is of the order of minutes, for ion currents of few thousands pA, and depends strongly on the current and the sample material. While higher currents (7000-20,000 pA) cut faster, they can yield rougher surfaces, which will negatively affect the image quality. The ion beam imaging gives better quality images than the electron beam imaging, due to less charging of the surface, but it is significantly more time consuming to implement because it requires to change the sample position and to refocus between the milling and imaging steps. If a good contrast exists between the pore space and the matrix, and the charge build up on the sample is not too rapid, electron imaging (SED detector) could be tried by using short scan times. For nonconducting materials, although the electric charge is removed from the surface by the milling process, it builds up rapidly afterwards if one uses continuous electron scanning mode, as the fresh surface exposed by milling is not carbon coated. After each milling episode, images at the appropriate magnifications are acquired. To reconstruct the 3D image, the individual images are first aligned using markers milled in the sample before the serial sectioning takes place and, second, binarized by selecting the appropriate threshold. The series of 2D binary images are stacked and, using interpolation and surface rendering algorithms, the 3D pore or grain structure is reconstructed. To process the data, the software package Adobe Photoshop can be used for image alignment and contrast equalizing. Image-J from National Institute of Health can be used for batch image manipulation and individual thresholding.

RESULTS

The FIB method was tested on cleaned Belridge diatomite and on epoxy impregnated North Sea Chalk. The ion beam milling yields very clean, flat surfaces in which the pore-grain boundaries appear in high contrast. No distortion of the pore boundaries due to the ion milling is apparent, although the pore/matrix contrast is more pronounced in the epoxy-impregnated sample.

The 2.7x1.8x1.4mm diatomite sample was imaged by the CDM-E detector using a 15 pA ion beam and a 22.6s scan with the sections imaged 0.2 µm apart. As the sample was not impregnated with epoxy, features within the pores could be observed, similarly to the more traditional SEM approach (Fig.1). On the other hand, the presence of areas of variable brightness within pores can hinder the thresholding stage in which a binary image is generated for the pore/matrix system. These images required a more involved image processing, before generating the binary pore images used for the 3D pore reconstruction (Fig. 2).

The 6 x 4 x 2mm chalk sample was first vacuum impregnated with epoxy, followed by up to 1400 psi pressure and then polished by diamond discs, in succession from 30µm to 1µm size. The chalk sample presented a good contrast between the epoxy filled pore space and the matrix, which allowed secondary electron imaging (SED detector) at 5kV electron energy by using a 12s scan, with minimal charging taking place (Fig. 3). The sections imaged were spaced 0.1 µm apart. The good contrast allowed for direct

thresholding of the images to generate the binary image. The 3D reconstruction using a subset of the binary data is shown in Figure 4.

CONCLUSIONS

A new method of sample serial sectioning and imaging using FIB technology has been used to image with submicron resolution the 3D pore structure of diatomite and chalk. FIB was used to mill successive sample layers as wide as 50 μm and as thin as 0.1μm by sputtering of atoms from the sample surface. Very clean, flat surfaces in which the pore-grain boundaries appear in high contrast have been generated with no apparent distortion of the pore boundaries for both the epoxy impregnated and nonimpregnated samples.

ACKNOWLEDGMENTS

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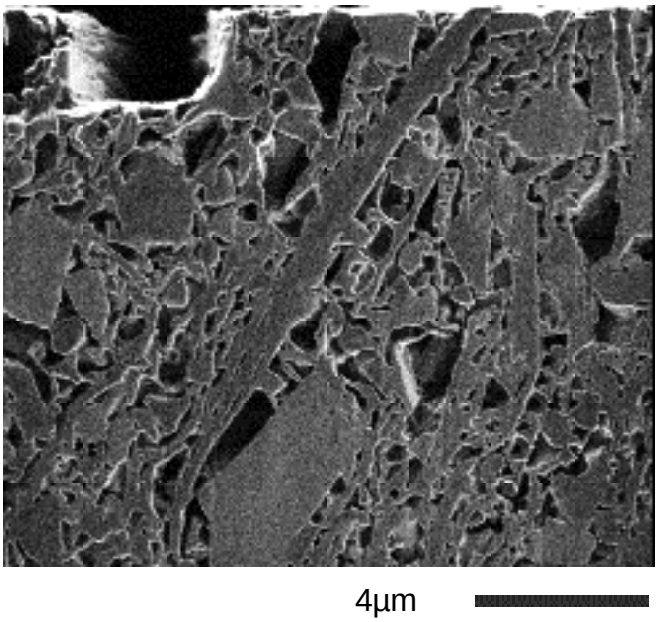

Figure 1. Ion beam image of diatomite at 20,000x magnification. Visible at top left, is a feature cut for alignment purposes.

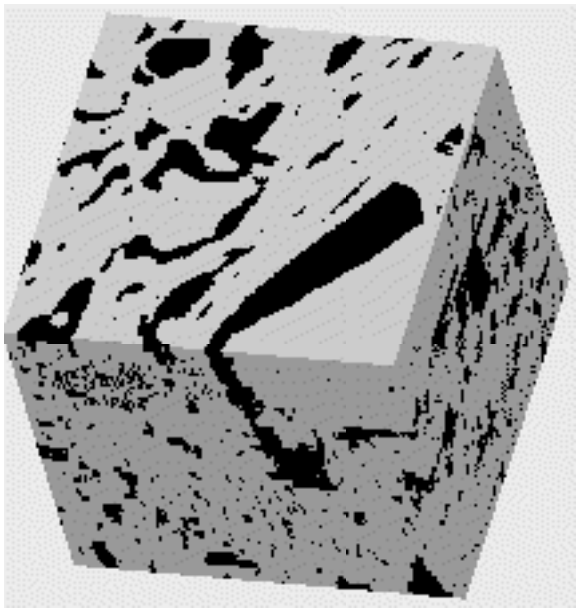

Figure 2. Diatomite volume reconstructed from binarized successive images spaced at 0.2 micron intervals. The pore space is dark.

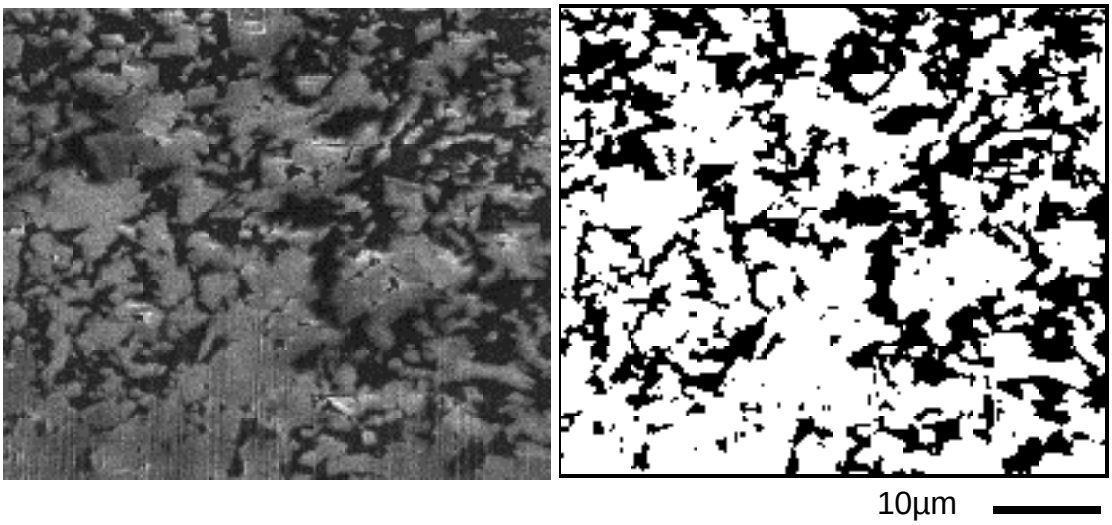

Figure 3 Electron beam image of chalk at 6,500x magnification (left) and binarized (right)

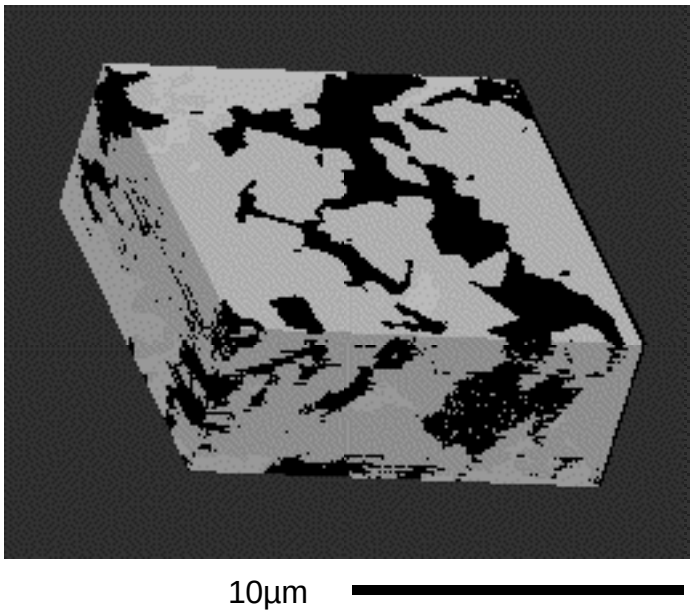

Figure 4. Chalk volume reconstructed from successive binarized images spaced at 0.1 micron intervals. The pore space is dark.