Nanoscale Three-Dimensional Rock Imaging Using Focussed Ion Beam

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RESEARCH OBJECTIVES

Computation of effective flow properties of fluids in porous media based on three-dimensional (3-D) pore-structure information has become more successful in the last few years, due to improvements in both input data and network models. Computed x-ray microtomography has been successful in 3-D pore imaging at micron scale, which is adequate for many sandstones. For other rocks of economic interest, such as chalk and diatomite, nanoscale resolution is needed to resolve the 3-D-pore structure. To achieve such resolution, a new method of pore imaging using Focussed Ion Beam (FIB) technology is being developed.

APPROACH

To directly access the pore structure at nanoscale scale, the FIB is used to mill successive layers of the rock material. FIB mills layers as wide as 50 micrometers and as thin as 10 nanometers by sputtering of atoms from the sample surface. The FIB, 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 caused by ion milling is apparent. After each milling step, as a new surface is exposed, a 2-D image of the surface is obtained using either the ion beam or the electron beam. While epoxy impregnation improves the contrast in the images, unimpregnated samples can yield excellent images (Figure 1). The high pore-matrix contrast is used next to binarize the 2-D images and, by stacking them, to reconstruct the 3-D structures of the pore space.

ACCOMPLISHMENTS

The FIB method was used to generate, for the first time, 3-D images of the pore spaces of Belridge diatomite and North Sea Chalk. Both these rocks have pores and pore throats in the tens to hundreds of nanometer range.

SIGNIFICANCE OF FINDINGS

Large oil reserves are present in both diatomite and chalk formations. By obtaining the 3-D pore structure of these rocks, we accomplished the first step in generating the input data needed for pore-network simulators. The 3-D pore images of complex fragile rocks, such as diatomite and chalk, are essential for the construction of idealized pore networks and the subsequent simulations of flow experiments for various displacement processes (drainage, imbibition, water-alternating gas, secondary drainage, etc.). Such experiments would be extremely difficult, if not impossible, to perform in the laboratory. The calculations provide scientific understanding of the pore-scale flow phenomena of mixed-wet rocks, which control large-scale hydrocarbon or carbon dioxide flow.

RELATED PUBLICATION

Tomutsa, L., and V. Radmilovic, Focussed ion beam assisted three-dimensional rock imaging at submicron scale. Proceedings of the 2003 International Symposium of the Society of Core Analysts, Pau, France, September 21–24, 2003.

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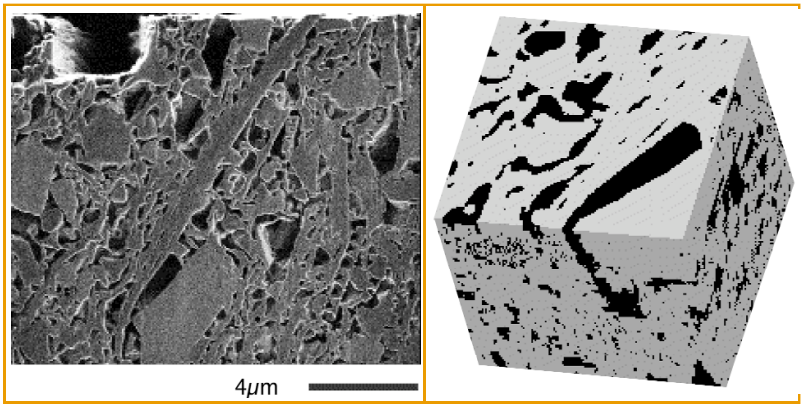

Figure 1. (a) Ion-beam image of diatomite at 20,000x magnification, (b) Diatomite volume reconstructed from binarized successive images spaced at 0.2 micron intervals. The pore space is dark .