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Investigations on the Microstructure and Microanalysis of the Gas Shale Sample Prepared by SEM Ion Mill by Off-Centering the Ion Beams

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Recently there has been a lot of interest on sedimentary rocks, which is driven by its economic potential of the environmentally attractive energy resource they contain. Gas Shale is among one of the potential sedimentary rock to extract energy. Shales present unique challenges in measurement and description. Shale can be defined as an organic-rich fine-grained sedimentary rock containing kerogen (a solid mixture of organic chemical compounds) from which liquid hydrocarbons called shale oil can be produced. Shale is defined by grain size (< 39 μm); clays are commonly associated with this small grain size, thus linking the association of clays and shales. Other minerals present in Shales are quartz and calcite. Organics in the form of kerogen is very common in gas shales. The organics add new dimensions to the shale, e.g. they lower density, increase porosity, provide the source of the gas, impart anisotropy, alter wettability and introduce adsorption. Their distribution, habit and concentration become important in any economic assessment. In order to get information about the various phases present in the shale sample and also porosity, it is very important to get a smooth wide area of the shale sample, suitable for scanning electron microscopy and EDAX measurement. The present investigations on gas shale sample is centered to get qualitative and quantitative information from the gas shale sample by preparing the sample in an SEM ion mill.

A broad smooth area of the gas shale sample for microstructural investigation and microanalysis by scanning electron microscopy (SEM) and EDAX can be prepared by displacing the ion sources to an off-center position in the SEM ion mill. The standard (default) position of the ion sources causes the ion beams to cross each other. If the stage rotation is set to continuous 360° rotation, the resulting milled area is the size of the beam crossover (as shown in the schematic Fig. 1). But, if the ion beam ellipses are set in the configuration (as shown in the schematic, Fig. 2) and the shale sample was rotated continuously, we get a broad smooth milled area of the sample. The ion beams can be brought in this configuration by displacing the ion gun manually through the side screws located in the knob of the ion source. With the above configuration of the beams touching each other, we could mill a sample of size 20mm x18mm. A carbonate shale sample of size 20x20x10mm was polished to a 9um finish. It was ion milled at 5kV (left beam), 5kV (right beam) Beam angle (2 degrees) Time (2.5 hrs) rotation 360.

The resulting wide milled area of the sample was very suitable for microstructural investigations and to get qualitative and quantitative information from the gas shale sample regarding the grain morphology, gain size, organic phases present and porosity of the sample.

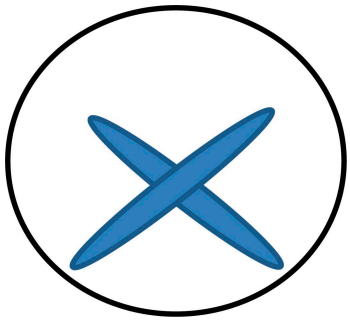

Fig. 1: Schematic showing the ion beams crossing each other

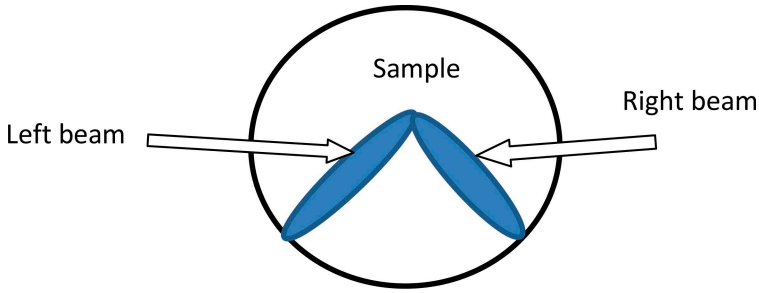

Fig. 2: Schematic showing the ion beams touching each other

Following ion milling operations, shale samples were examined by scanning electron microscopy (SEM).

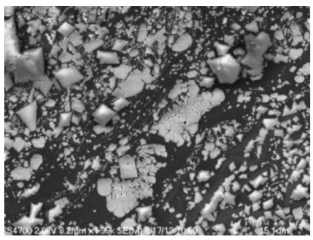

Fig. a

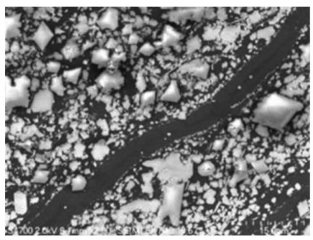

Fig. b

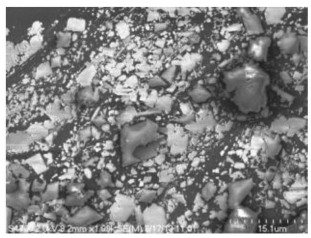

Fig. c

Carbonate shale at the center position of the sample holder (Fig. b), 5 mm away in the + X direction (Fig. a), 5 mm away in the + Y direction (Fig. c),

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