

A DEVICE FOR IN SITU SEM STUDY OF ROCK TEXTURES AT HIGH TEMPERATURE AND PRESSURE ($T < 600^{\circ}\text{C}$, $P < 100\text{ MPa}$)

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The difficulty in interpreting experimental results on rock permeability at high temperatures and pressures fostered scientific works aiming at observing the behavior of the samples in-situ in the SEM. In the present study, we present designs of our experimental arrangements (cell), and describe the principles of permeability calculations from the distribution of crack width and length with size.

The cell reported herein has been designed to observe the sample surface in the vacuum a “Tesla” SEM at pressures to 100 MPa and temperatures to 600°C. A screw-chamber and nut are made from stainless steel and sealed with a lens, thus constituting a high-pressure vessel. A cylindrical, 4.6 mm-diameter by 5.2 mm-long sample is enclosed in a gold capsule edges being curled. The cell uses water to exert pressure on the gold insulating shell of the sample; it is supplied through a capillary. The cell materials are all nonmagnetic.

The heating coil used in the cell is made of a 0.3 – mm-diameter Ni-Cr wire and has a resistance of around 60 ohm. The cell thus assembled is mounted on a displacing support and fixed by screw. To input wires and impose pressure, a special lock is made, with 6 electrical vacuum entries and capillaries. The 4-mm-diameter capillaries of the cell and lock are connected by a thin capillary (1 and 0.2 mm in outside and inside diameter, respectively) twisted as a loop. This permits displacement of the cell inside the SEM. Pressure in the cell is generated by a 1 cm³ - volume micropress and measured with a pressure transducer with an accuracy of $\pm 0.5\text{ MPa}$. Outer terminals of the lock are joined to universal voltage source and connect the thermocouple and pressure transducer to a microvoltmeter. Power consumption of the electric furnace is not greater than 100 watts.

If conditions of the run call for, one of the sample ends was polished and a graphite coating was or was not applied. Prior to the run, the sample surface was analyzed for mineral composition with an electron microprobe.

After careful checking, the cell was mounted in the SEM. The temperature of the sample was then raised by turning on the power to the furnace. To avoid cracking due to the effect of thermal stresses, the temperature was raised at a rate of 2-3°C/min. The sample surface was photographed at constant temperature. In order to photograph a sample area of about 0.7-0.8 cm², 30-35 exposures were required at a magnification of 200. The portions of interest were photographed separately at higher magnification.

Treatment of photographic material consisted in laying out microphotographs of the sample field, mapping a network of cracks and measuring their widths and lengths. There are a few approaches to estimation of rock permeability using a network model. The approaches all use an analogy between Ohm's and Kirchhoff's law on the one hand and Darcy's law and general laws of hydrodynamics on the other. Such a procedure allows determination of fluid flow parameters from their electrical analogues.

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