



High temperature triaxial tests on Rochester shale

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Phyllosilicates are one of the major components of the crust, responsible for strength weakening during deformation. High pressure and temperature experiments of natural samples rich in phyllosilicates are needed to test the relevance of proposed weakening mechanisms induced by phyllosilicates, derived from lab experiments on single phase and synthetic polyphase rocks and single crystals.

Here, we present the preliminary results of a series of high temperature triaxial tests performed on the illite-rich Rochester Shale (USA - New York) using a Paterson type gas-medium HPT testing machine. Cylindrical samples with homogeneous microstructure and 12-14% porosity were fabricated by cold and hot-isostatically pressing, <150 μm crushed Rochester Shale particles. Experiments were conducted at 500- 700 $^{\circ}\text{C}$ temperature and varying strain rate from 5×10^{-6} to 7×10^{-4} s^{-1} , while keeping confining pressure constant at 300 MPa. Synthetic hot-pressed samples were deformed up to a total shortening of 7.5 to 13%. To study the significance of mica dehydration, iron or copper jackets were used in combination with non-porous or porous spacers. Water content was measured before and after experiments using Karl Fischer Titration (KFT).

All experiments show, after yielding at 0.6% strain, rapid hardening in nearly linear fashion until about 4-5% strain, from where stress increases at reducing rates to values at 10% strain, between 400 and 675 MPa, depending on experimental conditions. Neither failure nor steady state however, is achieved within the maximum strain of 13%. Experiments performed under 500 $^{\circ}\text{C}$ and 300 MPa confining pressure show weak strain rate dependence. In addition, iron-jacketed samples appear harder than copper-jacketed ones. At 700 $^{\circ}\text{C}$ samples are 17 to 37% weaker and more sensitive to strain rate than during 500 $^{\circ}\text{C}$ experiments. Although, iron-jacketed samples behave stronger than copper-jacketed ones. By visual inspection, samples appear homogeneously shortened. Preliminary analysis suggests that deformation is mostly accommodated by pore collapse. Although, with finite strain, pore collapse becomes less significant.

A temperature, strain rate and jacket material dependent elastic deformation was inferred from significant differences between total and finite strain. For 500 $^{\circ}\text{C}$ experiments using copper jackets, elastic strain component of total strain was 49-57%, but for iron jacket experiments, the elastic strain component ranged between 58 and 70%. At 700 $^{\circ}\text{C}$, iron- and copper-jacketed samples experienced elastic strain component ranging between 6 and 28% and around 47%, respectively. In any case, elastic strain is larger than strain at the yield point, suggesting additional mechanisms play a role here.

KFT analysis reveals that water loss is dependent on jacket material and presence of deformation. In deformation experiments, the iron jacket and non-porous spacers trap water completely. Iron jackets are slightly permeable during static experiments (0.02 wt% H₂O/hr). With temperature, copper jackets become more permeable for water (0.06 wt% H₂O/hr at 500 $^{\circ}\text{C}$ and 1.0 wt% H₂O/hr at 700 $^{\circ}\text{C}$).

A temperature dependent linear relationship exists between permanent strain and porosity decrease. During 500 $^{\circ}\text{C}$ and 700 $^{\circ}\text{C}$ experiments, by approximation, porosity reduces at a rate of respectively 0.45% and 0.56% per strain percent. Density increases linearly with permanent strain as well (slope is 0.03 g cm⁻³ per strain percent).