

Digital Multiphysics Interferometry: A new approach to study chemo-thermo-hydro-mechanical interactions in geomaterials

H Roshan, MAQ Siddiqui, K. Regenauer-Lieb, and A. Lv

School of Petroleum Engineering, UNSW Australia, Sydney, NSW 2052, Australia

A. Hedayat

Department of Civil and Environmental Engineering, Colorado School of Mines, USA

M. Serati

School of Civil Engineering, University of Queensland, QLD, Australia

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ABSTRACT: Investigation of multiphysics processes including chemical, thermal, hydraulic and mechanical interactions in geomaterials in particular shales has considerably expanded in recent years. The core scale multiphysics experimentation is often very complex to conduct and more importantly involves significant uncertainty. In addition, the micro-scale mechanisms deriving the macro-scale phenomena are yet to be explored. The missing links between micro-macro scale processes have led to mostly modelling approaches with uncertain input parameters. We therefore, for the first time, design a novel shear device coupled with interferometry technique to investigate the multiphysics phenomena from μm to cm scale in real time. In the new setup, the special shearing cell accommodates a $1\text{cm} \times 1\text{cm} \times 4\text{mm}$ cubic sample resembling the plane strain condition. The cubic sample can undergo hydrostatic or shear stresses while being continuously exposed to high pressure working fluids circulating in the cell. An imbedded sapphire window allows the observation of the micro-fracture development and pore structure alteration in real time using green light interferometry technique. The technique can be readily coupled with static image scanning such 3D micro-ct scanning for further insight. In an example application, a shale sample was placed in the cell and was exposed to deionized (DI) water at hydrostatic and shear stress states and images were taken from the sample using interferometer. The obtained images were then registered and Digital Image Correlation (DIC) technique was used to analyze the developed strains during the experiment. The results showed for instance that hydration induced stresses are unlikely to cause any micro-fracture initiation under hydrostatic stresses however micro-fractures appeared under shear stress. This behavior seems to attribute to loss of sample's strength when exposed to water rather than a phenomenon caused by increase in internal hydration stresses. The obtained results have a wide applications such as the design of hydraulic fracturing and completion fluids, the gas flow in micro- fractures/micropores and characterization of the hydration induced micro-crack development at μm - to cm -scale at different stress states.

1. INTRODUCTION

Rock containing clays including shales form more than 40% of all sedimentary rocks on earth crust (Al-Ani and Sarapaa, 2008). Rocks containing high amount of clay minerals often represent complex multiphysics behaviour (Roshan and Oeser, 2011). Clay structures are hydrated phyllosilicates (sheets of silica (Si)) with aluminium (Al), magnesium (Mg), or iron (Fe) as vital constituents. The sheets can be tetrahedral (T) or octahedral (O) in shape which are linked together consisting of stacked planes of silicon, oxygen or hydroxyl groups (Grim, 1962). Linking of T and O (e.g. 1:1 TO or 2:1 TOT) sheets results in exposed basal surfaces of oxygen atoms and hydroxyl groups (Hoffmann and Lipscomb, 1962) which causes most clay minerals to generally adsorb water. Smectite group (e.g. montmorillonite) adsorb the most amount of water and lead to swelling where the clay layers are forced apart on contact with water (Roshan and Fahad, 2012). Illite also

exhibits swelling although less than montmorillonite, however, kaolinite is classified as non-swelling but dispersive clay (Krueger, 1988). Interestingly such volumetric strain (swelling) can be induced by the fabric of the rock and its pore structure even in presence of non-swelling clay minerals. This is due to the development of diffuse double layer at nano-scale pores (Roshan et al., 2015).

Impact of clay swelling hinders the ability of fluids to flow (permeability) even in conventional (high porosity, high permeability) hydrocarbon reservoirs such as shaly sandstones (Aksu et al., 2015). Problems caused by presence of clay minerals are extended to many areas including drilling (Roshan and Aghighi, 2011; van Oort et al., 1996). Another major cause of not only economic but technical and great environmental concern is the high fracturing fluid loss occurring in shales (Dehghanpour et al., 2012). Several mechanisms have been proposed to identify the reasons for such high fluid

lost (e.g. clay hydration and osmosis, capillary imbibition, micro-fracture creation, and entrapment in close fractures) (Dehghanpour et al., 2013; Engelder et al., 2014; Ezulike and Dehghanpour, 2014; Makhanov et al., 2014; Roshan et al., 2016b; Xu and Dehghanpour, 2014; Zolfaghari et al., 2015). However, none of these phenomena can solely explain the kinetics and amount of the water uptake into the shale. These phenomena are in fact all tightly coupled i.e. it seems that the wettability (affinity of rock surface to a particular fluid) thus capillary force is of dominant nature (Dutta et al., 2014). It was strongly postulated that the overall wettability of shale rocks is somehow related to the individual wettabilities of their comprising clay minerals (Roshan et al., 2016a) which controls the water uptake.

Interestingly enough, recent studies show that the imbibition tests used for petrophysical characterization and water uptake of shale rocks can pose severe bias due to micro-structural changes with exposed fluids (Cagnola et al., 2017; Peng and Xiao, 2017; Zhang and Sheng, 2018). Such microstructural (micro-fractures and pores) alterations by exposed fluid under stress are quite complex. This complexity is further extended due to multiphysics responses (mechanical, thermal, hydraulic and chemical interactions) of these rocks. We have therefore proposed to use the interferometry technique to monitor such changes. In order to achieve this goal, we have designed a new shear cell equipped with micro-hydraulic rams and high pressure cell with a sapphire window to monitor and record the changes live with micron resolution at the cm scale. Such introduced technique combined with the designed shear cell has wide applications beyond what is shown in the study such as characterization of purely mechanical damage, gas-liquid swelling, fracture opening-closing, thermal expansion, chemical weakening, acid fracturing and etc.

2. EXPERIMENTAL METHODOLOGY

2.1. Experimental setup

The experimental setup consists of a newly designed shear cell along with glass-compensated green light interferometer (Fig. 1a and b). The cell has an internal area of $10 \times 10 \times 5$ mm which hosts a cubic sample i.e. thin cube is chosen to resemble the plane stress problem. Two micro-rams provide the force on the sample from each direction i.e. hydrostatic or shear state of stress can be therefore applied. Two other sides of the cell contain the ports for injection and drainage of the fluid i.e. fluid circulation. One of the connections is linked to an ISCO pump and the other connection is attached to a back pressure regulator to maintain the pressure while circulating the fluid (DI water here). The top of the sample is covered with a 3 mm thick sapphire window sealed with a flat ring to give access to interferometer for monitoring and recording the sample changes. A full scan of $\sim 2 \text{ mm} \times 2.5 \text{ mm}$ 3D surface of the sample will take less than 30 sec in which $1.0 \mu\text{m}$ lateral

and 50 nm vertical resolutions are attainable (with the current objective lenses). In addition, the cell (thus sample) temperature can be controlled and maintained with the rubber heat sheet placed at the bottom of the cell.

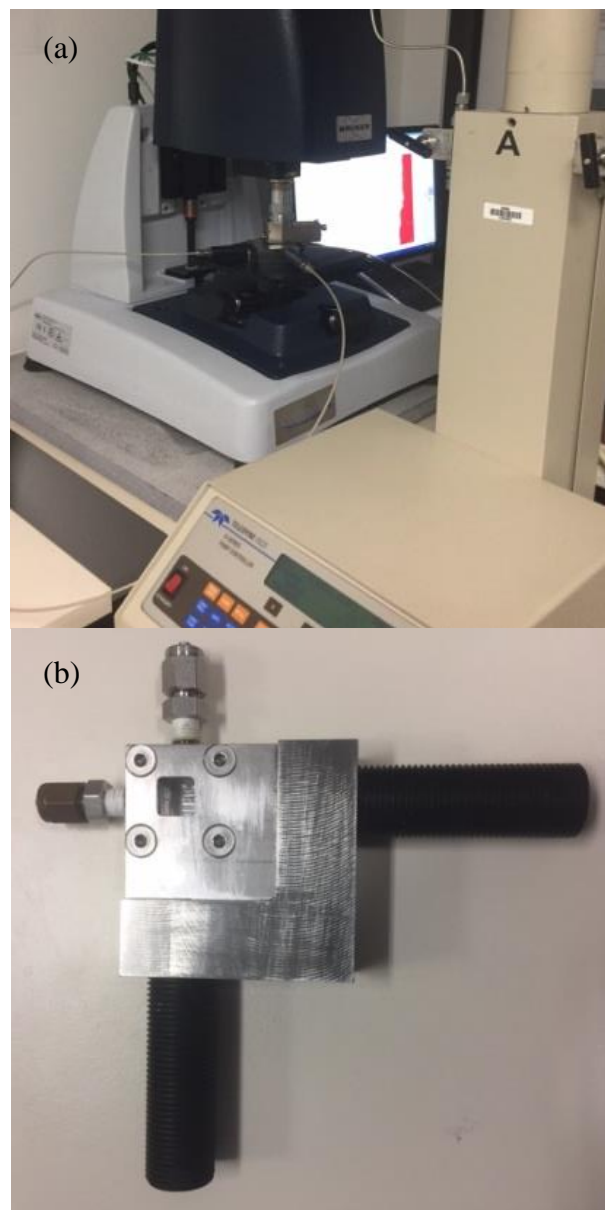


Fig. 1. A) Compensated glass interferometer and b) newly designed shear cell.

2.2. Sample preparation and experimental procedure

The organic-rich shale sample extracted from 3012 m depth from a gas bearing formation in on-shore Perth Basin, Western Australia was used in the study. The organic carbon content was 2.85 wt. % as measured by LECO CN analysis and the major mineral phases of the sample were obtained through XRD analysis. The measured mineral phases were quartz (27.1 wt.%), muscovite (14.3 wt.%), kaolinite (9.6 wt.%), chlorite (9.6 wt.%), illite (28.2 wt.%), albite (9.3 wt.%), and pyrite (1.9 wt.%). Other properties including the pores size distribution (PSD) measured by mercury intrusion

and nitrogen adsorption techniques for this sample have been reported previously (Roshan et al., 2016c). A cubic sample of 10×10×4 mm was air-cut out of the core using Apollo saw i.e. sample was trimmed to exact dimensions using a holder made by 3D printing and polished down to 0.01 mm on edge sides. It was then vacuum-dried at 105 °C for 24 hrs before moving to desiccator under vacuum to reach room temperature. The sample was next moved to the shear cell. An initial hydrostatic and then shearing stresses were applied on the sample (sample was held at compression (600 psi) and shear (600×900 psi) and images were taken using interferometer after approximately 3 hours at dry condition. Then the sample at compression (600 psi) was exposed to DI water circulating in the cell for 6 hours and images were taken. In a final step, the sample was undergone shearing (600×900 psi) while exposed to DI water and images were taken after 6 hrs.

2.3. Boundary effect

In order to quantify the effect of stress-boundaries on overall displacement response, a sample was placed in the cell and hydrostatically compressed while a high-definition Basler camera connected to a computer through designated Pylon software was used to capture and store the images. Digital Image Correlation analysis was then performed on the sample to investigate the possible deviation from stress scenario due to boundary effect (Fig. 2). Acceptable strain field was observed under pure compression i.e. no significant shearing on the platen edges was observed i.e. all shear strains center around zero.

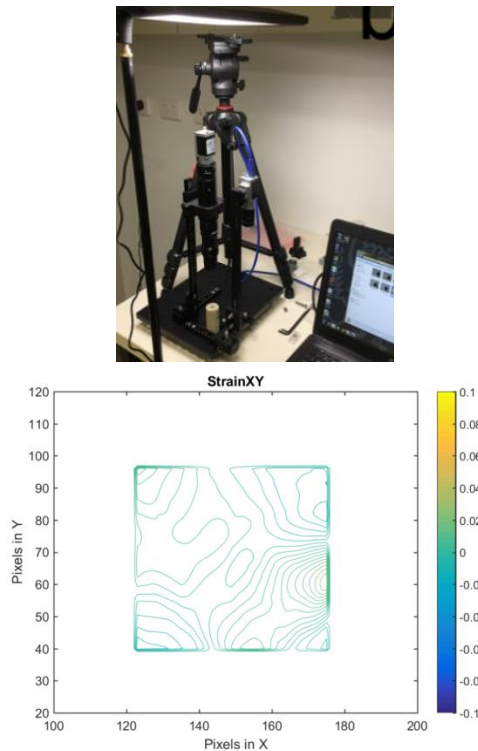


Fig. 2. The setup used to identify the boundary effect of the tiny platen on the sample (top) and DIC analysis performed under compression (no significant shear stress exists thus confirming that the stress scenario is followed and boundary effect is minimal) (bottom).

3. RESULTS AND DISCUSSION

The results of the interferometry show that the sample under compression or shearing at dry condition was not affected by applied stresses and no damage was observed even after 3 hours of stress maintenance (representative area of 1.8 by 2.4 mm was scanned). When sample was exposed to DI water under compression (Fig. 3), no damage was still observed after 6 hrs of exposure emphasizing that the micro-fractures are not developed under compression even when exposed to DI water.

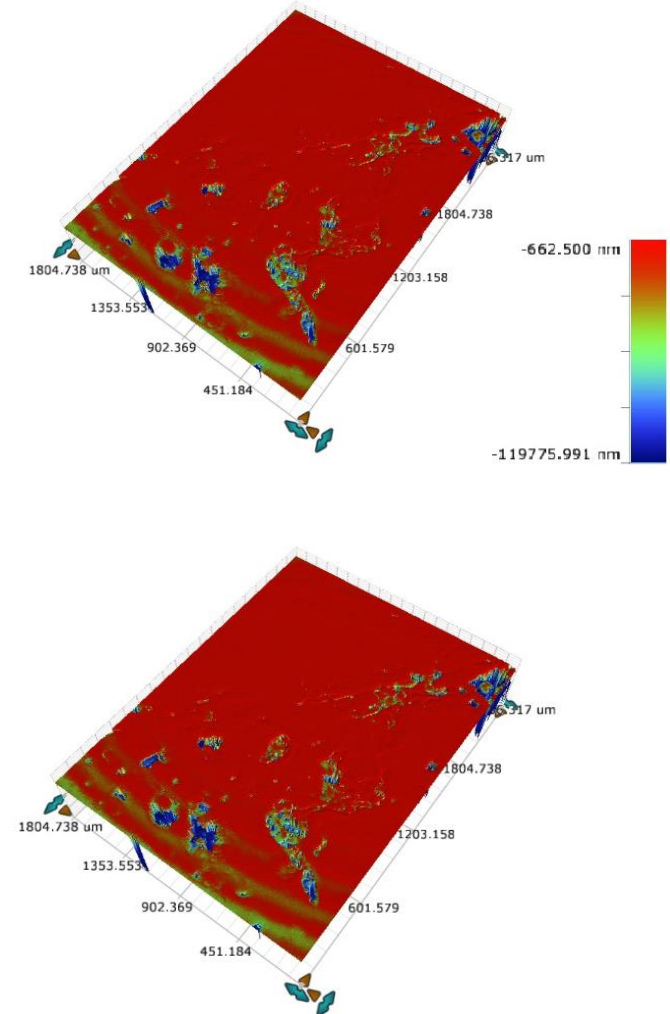


Fig.3. 3D images of the sample under compression (600 psi) exposed to DI water at initial time (top) and after 6 hrs of exposure (bottom). The legend shows the 3D surface structure in nm.

Interestingly, when the sample was subjected to shearing (Fig. 4 and 5), clear microstructural damage and considerable micro-fracture development were identifiable. In addition, the opening of some pore spaces is seen from the images showing the possible detachment of clay minerals from the sample surface thus leading to pore opening. DIC analysis conducted on 2D images confirmed the micro-fracture development in the sample exposed to DI water undergone shearing for 6 hrs.

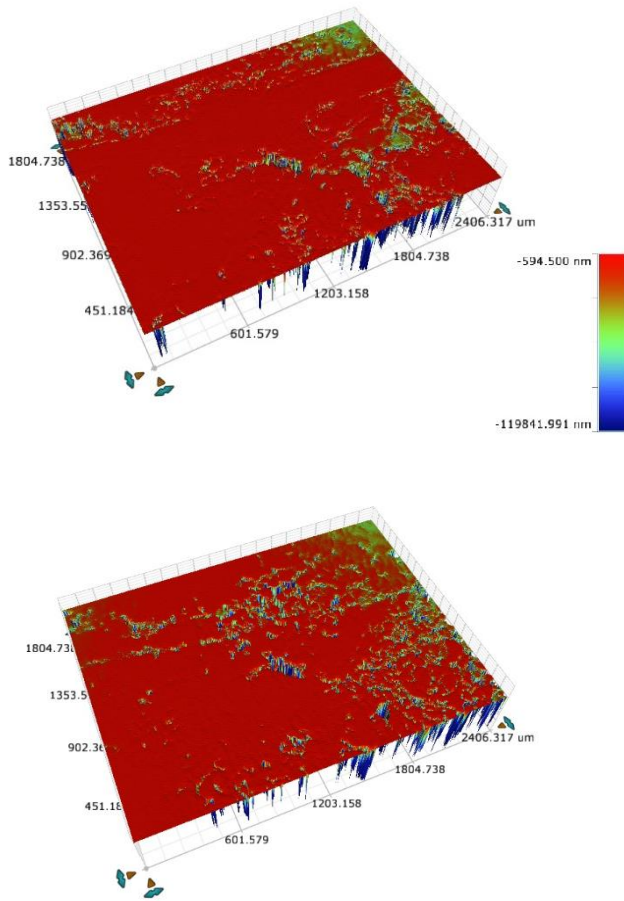


Fig.4. 3D images of the sample under shear (600×900 psi) exposed to DI water at the initial time (top) and after 6 hrs of exposure (bottom)

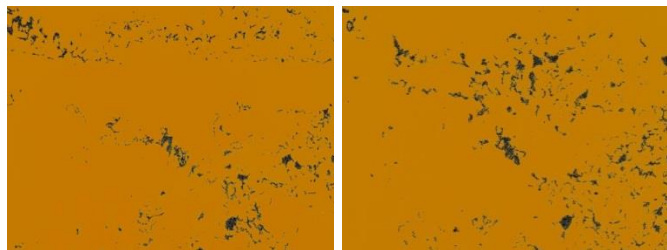


Fig.5. 2D images of the sample under shear (600×900 psi) exposed to DI water at the initial time (right) and after 6 hrs of exposure (left) which were used for DIC analysis.

The hydration induced micro-fracture development under compression was therefore limited if not possible at all. Under shearing through, the micro-structural alteration including the micro-fracture initiation and associated damage is observed. It is known that the clay mineral hydration in shale rocks causes the interval stress build up and resultant bulk swelling. However such hydration induced stresses is unlikely to cause any internal damage when exposed to in situ stresses. Interestingly though the clay hydration dose not only increase the internal stress but reduce the rock overall strength (mathematically shifting the yield surface closer to failure). Latter is therefore more likely to be responsible for the damage observed under shearing. Such effect under compression is obviously very limited strongly supported by experimental observation where no change was observed in the sample structure under compression. This is especially pronounced for sample containing high amount of clays i.e. the sample used in this study contained more than 47 wt.% clay minerals.

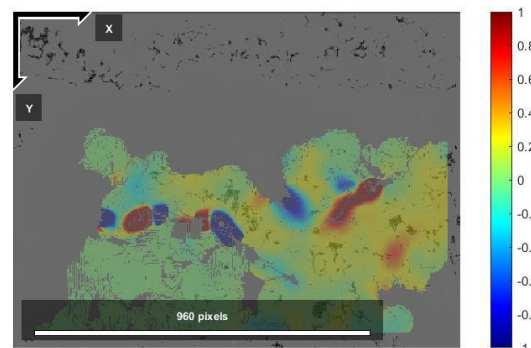
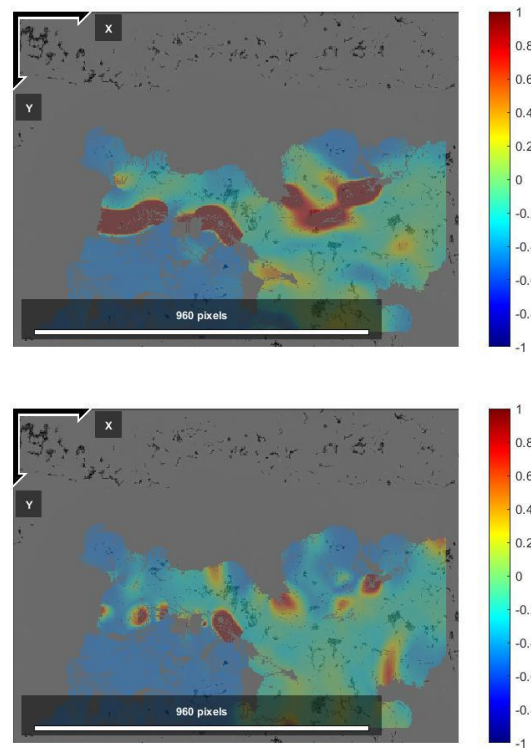


Fig.6. Strain development (xx, yy and xy on top, middle and bottom) from DIC analysis on 2D images for shearing test confirming the initiation of micro-fracture.

4. CONCLUSION

A new technique coupled with novel experimental apparatus is introduced in this study to investigate the multiphysics phenomena in clay-rich materials. The dynamic observation of micro-structural damage of a shale sample when exposed to fluid (DI water) under both compression and shear stresses are investigated as an example application. In this example, it was shown that the sample does not experience any structural damage when exposed to DI water under hydrostatic stress. Interestingly the damage was induced when shearing state of stress exists or in other words when the water exposure assisted in induction of damage through shearing by weakening the rock and reducing its overall strength. This is therefore evident that hydration induced micro-fractures (damage) are not developed when sample is under hydrostatic compression but rather appears when sample is under shearing. Whether the internal hydration stresses or an overall reduction in strength of the rock is the main driving force for such damage is still an open question for future work.

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