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Neutron scattering study of strain behaviour of porous rocks subjected to heating and unconfined uniaxial compression

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Abstract. Neutron scattering (at ENGIN-X) was used to examine strain distributions in two hydrocarbon reservoir rock types (i.e. sandstone and chalk) under uniaxial stress (up to 35 MPa) at temperature as high as 70 °C to utilise the advantages of neutron scattering to draw a detailed picture of the structural evolution in the porous rock core plugs subjected to heating. Each sample was cylindrical in shape (i.e. 38.1 mm diameter and 48 mm length). The corresponding strain free lattice parameter (d_0) for each sample at each measurement point at 5 MPa compressive stress condition were obtained by testing at 25°C. Various microstructural and material characterisation were carried out using X-ray diffraction, energy dispersive spectroscopy and scanning electron microscopy. Comparison of Rietveld refinement for multiple peak of crystalline phases in both samples were made. Results are discussed in terms of the influence of temperature and compressive stress on the residual strain profile along radial direction of cylindrical rock sample. It was found that the sandstone sample has significantly high strain bearing capability when compared with the chalk, however, the overall strain profile from the central axis in the radial direction looked very similar.

1. Introduction

Quantifying the relationship between stress-strain state and rock failure (i.e. fracture initiation and propagation) is important in characterisation of fracture and design of a hydraulic fracturing job as a means of well stimulation. The development of predictive fracture growth monitoring tools (currently a challenge and industry interest) is critical in oil and gas, carbon sequestration, shale gas and the geothermal power industry particularly with regards to the high costs associated with field trials in extreme conditions (e.g. North Sea, Gulf of Mexico or Arctic hydrocarbon fields). There is numerous data associated with porous rock fracture investigations at macro-scale, but the value of this information is limited by inability to experimentally measure micro-scale stress-strain concentrations (leading to failure) within the sample mass near critical potential fracture planes.

Understanding and quantifying the micro-scale stress-strain relationship and porous rock material failure behaviour and having the ability to predict these quantities for identified load conditions, is crucial to all geo-mechanical studies and flow characterisation applications [1-3]. The constitutive

equations governing the deformation of various rock types are typically adequate for bulk or large-scale deformation and stress analyses. However, they fail to make accurate predictions in physical and mechanical processes where highly localised heterogeneity exists or where the presence of geometric irregularities such as nano/micro-cracks are prevalent. Therefore, fracture initiation and propagation models at micro-scale are needed to understand hydraulic behaviour of naturally fractured reservoirs and design efficient hydraulic fracturing treatments particularly in presence of localised in-situ stress concentrations. Conventional rock mechanical tests subject samples to controlled load conditions and measure bulk deformations of the sample or more localized deformations only on exposed surfaces of the sample. They are currently not able to map the deformation state within the sample at high resolution.

In this research we used a neutron scattering facility (ENGIN-X) which gives high resolution and deep penetration to study localised micro-scale strain behaviour of porous rock materials. For stimulation treatments such as hydraulic fracturing, the in-situ stress profiles of a region within the rock mass can significantly influence the overall failure behaviour [4]. Therefore, the investigators believe that developing a means to measure stress-strains within samples subjected to fracture loading conditions (at high temperatures) will provide a useful tool for understanding the localised effects not captured by conventional techniques and may serve as a method for improving fracture evolution models and fracturing operations design. Neutron diffraction, unlike other measurement techniques, offers the opportunity to non-destructively measure the properties of polycrystalline materials, including porous geological materials [e.g. 1-3, 5-14]. Such properties include microstructure (grain size, grain shape, defect densities), texture (crystallographic preferred orientation) and stress.

The overall goal of this study is to better understand the micro-scale localized strain behavior of rocks and relate it to the residual stress distribution. It is important to note that time-of-flight (TOF) technique have been particularly important because they record continuous spectra that can be analysed for peak positions, peak intensities and peak shapes with the Rietveld method or multiple-peak analysis [2]. The test methodologies developed through this work can have a significant effect on developing future strategies in execution of hydraulic fracturing jobs and exploitation of naturally fractured reservoirs.

2. Methodology

The present study examines the strain distribution in two main different hydrocarbon reservoir rock types (i.e. sandstone/Clashach: SiO_2; chalk/limestone: $CaCO_3$). Neutron scattering facility (ENGIN-X) was utilised to exploit the advantages of neutron scattering to measure strain in the porous rock samples subjected to unconfined uniaxial stress at moderate to high temperature. The rock samples tested were cylindrical in shape and had 38.1 mm diameter and 48 mm length. As listed in **Table 1**, the sandstone samples were subjected to uniaxial compression stress of 20 MPa and 35 MPa (both at 70 °C), whereas, the chalk samples were subjected to uniaxial compression stress of 8 MPa (at 25 °C) and 12 MPa (at 25 °C and 50 °C), example shown in **Fig. 1b**. The compressive stress applied to the samples are different (i.e. chalk samples subjected to lower applied stress because of the lower ultimate strength). It is important to note that as per the original test plan, the chalk samples were also subjected to uniaxial compression stress of 20 MPa (at 70 °C), however, the samples failed while loading.

One set of three measurement points was evaluated across the radial direction per stress level (**Fig. 1b**). To apply temperature, the samples were mounted in a furnace and were heated at 2°C/min rate to achieve the final test temperature. Strain measurements were performed using a gauge volume of 4 × 4 × 4 mm^3 (fully submerged). Neutron diffraction data was collected at each measurement points (*a:* sample center, *b:* 10 mm and *c:* 15 mm away from the central axis in the radial direction) in the sample at each stress level, as shown in **Fig. 1b**. The corresponding strain free lattice parameter (d_0) for each sample at each measurement point at 5 MPa compressive stress condition were obtained by testing at 25 °C. The direct elastic strain in the material in the measured direction was calculated from

the following expression: $\varepsilon = (d\text{-}d_0)/d_0$, where d and d_0 are the lattice parameter for the sample under stress and unstressed sample (at 5 MPa compressive loading and 25 °C in this analysis), respectively.

Sample	Pressure	Temperature	Measure
Sandstone	5 MPa	25 °C	d_0
	20 MPa	70 °C	d
	35 MPa	70 °C	d
Chalk	5 MPa	25 °C	d_0
	8 MPa	25 °C	d
	12 MPa	25 °C	d
	12 MPa	50 °C	d

Table 1. Uniaxial compression test matrix.

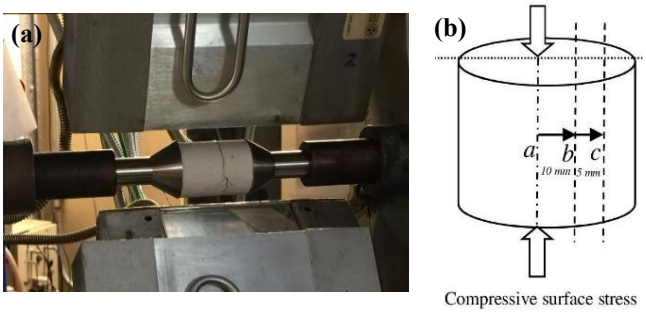

Fig. 1. (a) Uniaxial compression test (at ENGIN-X) of chalk at 5 MPa, and (b) measurement scheme.

Various microstructural and material characterisation were carried out using X-ray diffraction (XRD), energy dispersive spectroscopy (EDS) and scanning electron microscopy (SEM). Rietveld refinement for multiple peak of crystalline phases in both samples were made. The sandstone sample (Clashach) investigated previously [15] for its micro-scale fractured poro-elastic flow dynamics had the typical petrophysical characteristics as follows: porosity: 0.154, permeability: 315 mD, Young's modulus: 40 GPa, Poisson's ratio: 0.14, and compressibility: 3e-10 Pa^{-1}.

3. Results

3.1. Microstructure and X-ray diffraction

The determination of pore size distribution of a rock is often complicated, however, as shown in **Fig. 2**, the surface morphology of fractured surface reveals granular structure (average size about 100 µm to 250 µm in sandstone and 1 µm to 5 µm in chalk). The pore size ranges in nanometric-to-micrometric scale and all the spatial distribution of pores is roughly uniform.

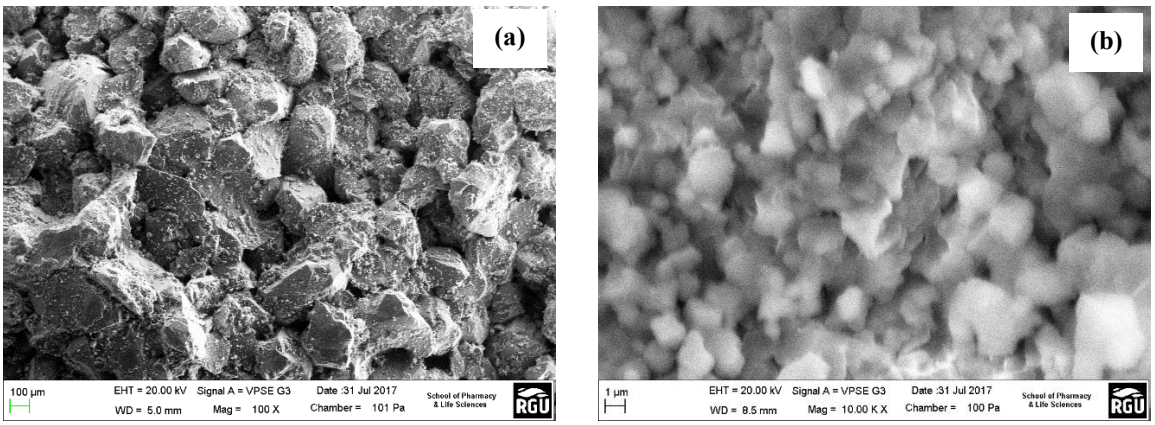

Fig. 2. Scanning electron microscopy: (a) sandstone, and (b) chalk.

X-ray powder diffraction patterns were collected at room temperature on a PANalytical X'Pert Powder diffractometer with Cu K_α radiation. Data were recorded in the range $5° < 2\theta < 80°$, with a step size of 0.0131°. From the X-ray diffraction pattern (**Fig. 3a**), sandstone was mainly indexed as SiO_2 (quartz) with hexagonal crystal system. There was no evidence of any other polymorphs of quartz. There are some other small peaks that would tentatively be index as potassium aluminium silicate ($KAlSi_3O_8$). From the X-ray diffraction spectrum (**Fig. 3b**), chalk was indexed as calcium

carbonate ($CaCO_3$) only, and there was no evidence of any un-indexed peaks. **Figure 3** also displays the crystallographic planes identified through X-ray diffraction analysis.

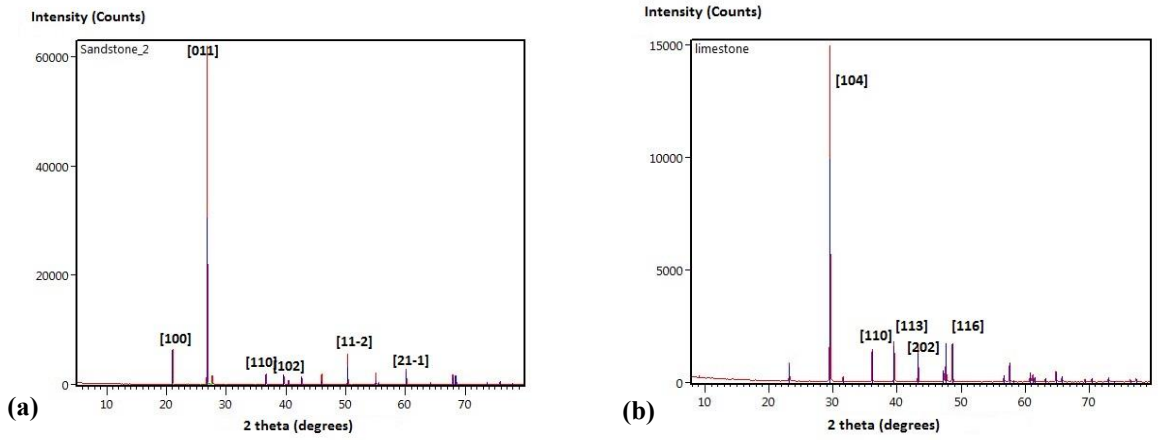

Fig. 3. X-ray diffraction spectrum: (a) sandstone, and (b) chalk.

3.2. Neutron scattering residual strain analysis

Figure 4 shows the *d*-spacing and crystallographic planes (example shown for 5 MPa compressive loading) identified through neutron diffraction analysis of the sandstone and chalk samples. From the neutron diffraction pattern (**Fig. 4**), 25 and 28 individual peaks (or crystallographic planes) were identified in sandstone and chalk, respectively. The obtained neutron diffraction pattern (**Fig. 4**) confirms the phase composition, as determined by quantitative X-ray analysis (**Fig. 3**).

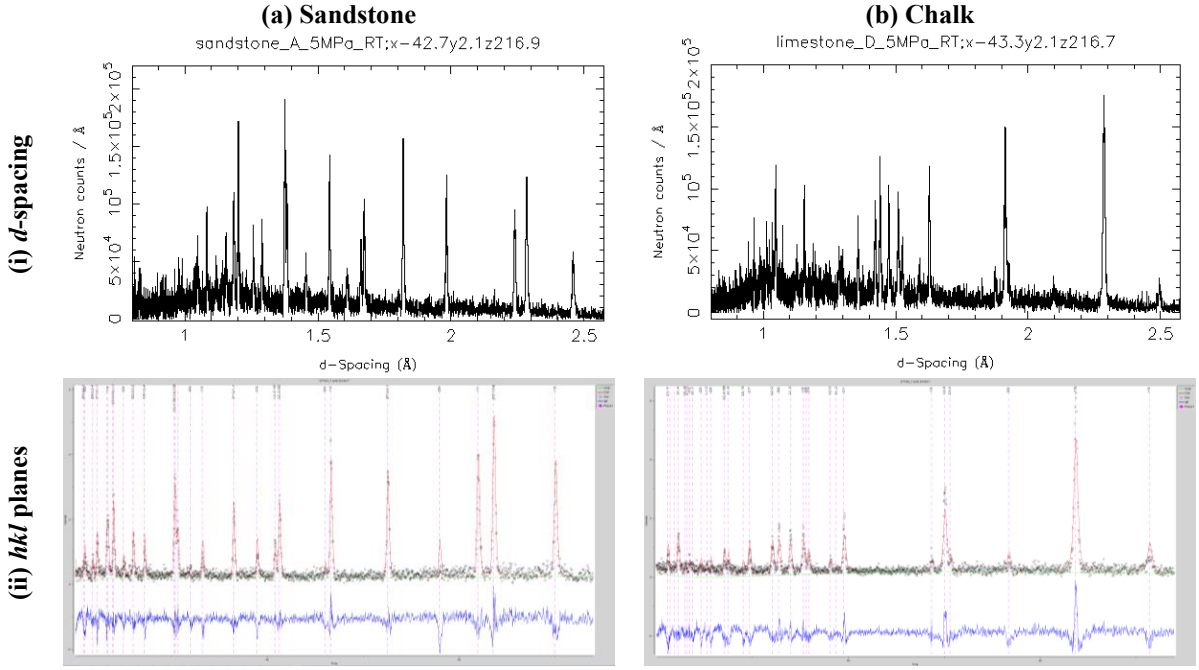

Fig. 4. Neutron diffraction at the center of the specimens (compressive loading under 5 MPa and room temperature conditions): (a) sandstone (*hkl* planes in (ii) R to L: [110], [102]/[10$\bar{2}$], [111], [200], [201]/[20$\bar{1}$], [112], [003], [202]/[20$\bar{2}$], [103]/[10$\bar{3}$], [210], [211]/[21$\bar{1}$], [113], [300], [301]/[30$\bar{1}$], [203]/[20$\bar{3}$], [104]/[10$\bar{4}$], [302]/[30$\bar{2}$], [220], [221], [213]/[21$\bar{3}$], [114], [311]/[31$\bar{1}$], [204]/[20$\bar{4}$], [222],

[303]/[30$\bar{3}$]), and (b) chalk (*hkl* planes in (ii) R to L: [110], [113], [202], [20$\bar{4}$], [10-8], [116], [211], [21-2], [1010], [214], [208], [119], [21$\bar{5}$], [300], [0012], [217], [201$\bar{0}$], [218], [30$\bar{6}$]/[306], [220], [1112], [223], [31$\bar{1}$], [312], [2110], [101$\bar{4}$], [314], [211$\bar{1}$]).

Comparison of Rietveld refinement (using GSAS code for ENGIN-X) for multiple peak of crystalline phases in both samples were made. Results shown in **Fig. 5** are discussed in terms of the influence of temperature and compressive stress on the residual strain profile along radial direction of cylindrical rock sample. As mentioned above and shown in **Fig. 6**, the chalk sample subjected to uniaxial compression stress of 20 MPa (at 70 °C) failed or crumbled while loading, whereas, the sandstone sample survived (e.g. **Fig. 5a**). Therefore, it can be estimated that the sandstone sample has significantly high strain bearing capability when compared with chalk sample, however, the overall strain profile from the central axis in the radial direction looked very similar.

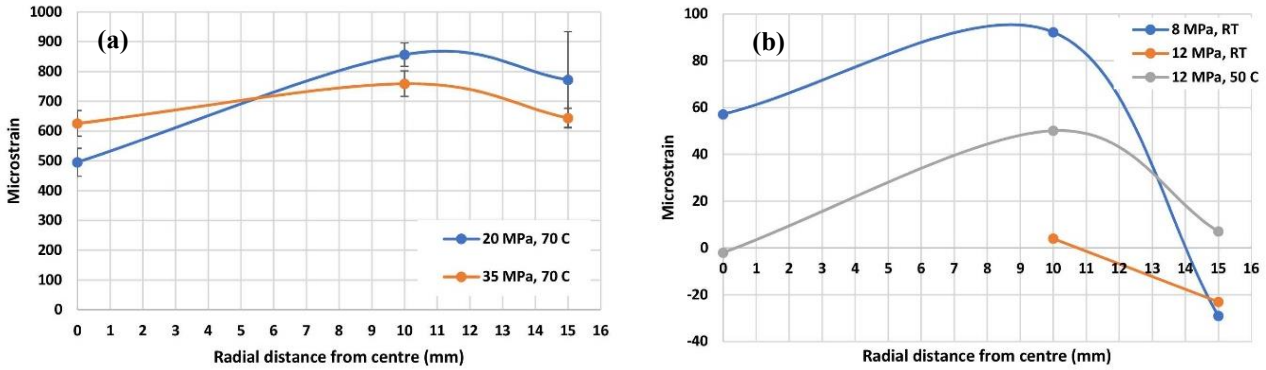

Fig. 5. Neutron diffraction residual strain (based on Rietveld analysis) comparison during uniaxial compression stress, showing strain variation along the radial direction from the centre of the sample: (a) sandstone, and (b) chalk.

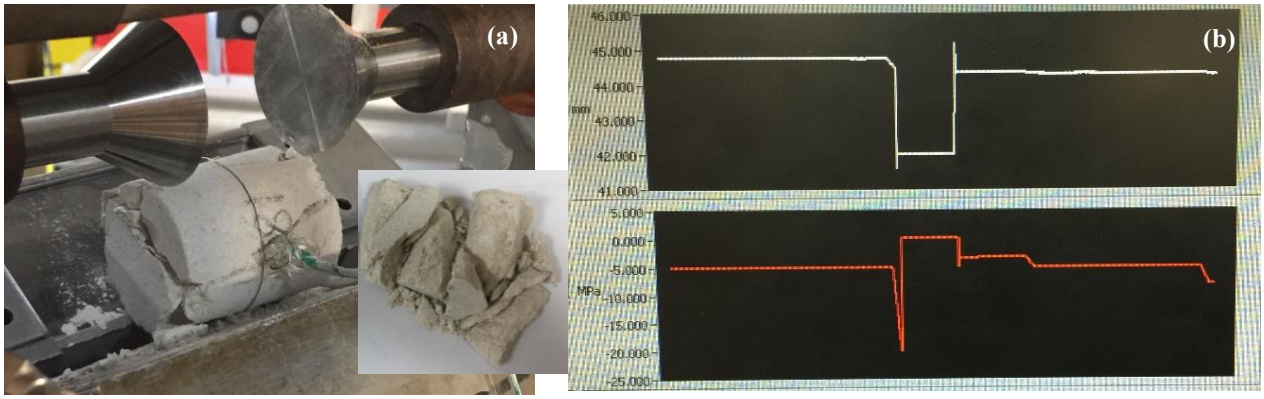

Fig. 6. Uniaxial compression stress of 20 MPa (at 70°C) of chalk sample fracture: (a) multiple failure during compression which includes diagonal shear planes, vertical fractures, vertical splitting, shear, conical and spalling (see inbox), and (b) displacement and stress jump leading to failure.

4. Discussion

Through the enhanced strain analysis of rock core samples both in this study and in previous and future investigations, it can be better understood how rocks may behave (i.e. deformation and fracture) under the different pressures induced through drilling and other production operations.

4.1. Deformation

Laboratory based uniaxial compression tests are generally conducted to determine the unconfined compressive strength of a rock (i.e. measuring whole-rock properties [5]). However, the difference between lattice strain data using neutron scattering and mechanically measured values during compression loading (e.g. **Fig. 5**) can reflect the influence of specific changes within the sample such as changes in the orientation of grains as well as the reconstruction of the pore volume or the anisotropy of the elastic constants within the involved crystals [8], but these can be part of further detailed analysis. The most precise way to analyse this phenomenon would be to track the distribution of stresses and strains inside the rocks (e.g. **Fig. 1b**) as they undergo mechanical loading but the scientific community in not near to succeeding in this task as of yet [18], however, *in-situ* neutron diffraction strain analysis may be useful in characterising deformation in rock samples (e.g. **Fig. 5**). To check the discrepancy of macro- and micro-strain for rocks of different structural characteristics, additional investigations of the microstructural change after deformation are needed [8].

4.2. Fracture

Fundamental understanding of the strength and fracture process of rocks such as sandstone which are very commonly found in reservoir formations and in mining requires realistic modelling and analysis of characteristics from both the single-grain and bulk scales [16]. Fracture takes place in locations where there are weaker stress planes in the rock; displacement of the grains usually appears to be in directions perpendicular to the lowest principal stress under external loading. Also shown through an example (in **Fig. 6a**), the common failure mechanisms for this kind of test are; axial splitting, spalling, single shear fracture or multiple fractures [17]. Although these failure modes represent a range where by a certain rock failure may be categorized, predicting the failure mode exactly is still a very difficult and complex procedure. The standard (ATSM D7012-04 [18]) for uniaxial compression tests generally results in axial splitting in a direction parallel to the maximum principal stress because of wing cracks that cannot be tolerated. These wing cracks are generated from tensile stresses exceeding the maximum tensile strength at the tip of any defect present in the rock.

Crack propagation is largely down to the relative displacement of the individual grains that make up the rock however methods are not yet well established on how to account for these ‘micro-effects’ in predicting bulk strength, and neutron diffraction could be beneficial. Methods like the finite element method (FEM) and the discrete element method (DEM) are advanced computational approaches that require realistic inputs to more easily accurately predict rock characteristics. To further investigate the influence of temperature on the residual strain a mathematical model approach can be applied. This approach can be based on contact mechanics (confined or unconfined) of cylindrical sample to predict the average strain due to the compressive loading and temperature. Computational numerical simulations as part of further work can allow the visual assessment of stress acting inside a rock which can provide insight into where and when the rock is likely to fatigue and fail.

Future application of ENGIN-X could be used to link pore orientation, pore volume, and mineral orientation for systematic studies on flow and mechanical properties of the microstructure, including testing at cryogenic conditions (currently under investigation by the authors [19]).

5. Concluding remarks

Compressive stress behaviour and general understanding of rock mechanics are essential to predict the wellbore stability, choose the appropriate drilling fluid and the type of hydraulic fracture. Rock failure is dependent on the ultimate strength of the material; a factor that is practically hard to determine when preparing to drill into a formation beneath the surface or assessing the wellbore stability. We have therefore presented a preliminary description of porous geological samples in micro-scale (sandstone and chalk), where the analysis gives new insights into the strain (deformation) distribution in radial direction of cylindrical sample during uniaxial compression loading, with following key conclusions:

a. From comparison of Rietveld refinement for multiple peak of crystalline phases in both samples, it was found that the sandstone has significantly high strain bearing capability when

compared with chalk, however, the overall strain profile from the central axis in the radial direction looked very similar.

b. Comparison of the residual strain profile between the sandstone and chalk indicate that the average residual strain in both samples are largely tensile with some compressive residual strain in the chalk (near the outer periphery of the cylindrical sample).
c. This application of neutron scattering also makes it possible to target microscopic volumes and at the same time collect statistically relevant data for large sample volumes.

References

[1] Voigtlaender A, Leith K, Krautblatter M, Walter J M and Scheffzük C 2015 *Geophysical Research Abstracts* **17** EGU2015-11269-1
[2] Wenk H R 2005 *Geophysical Research Abstracts* **7** 03911
[3] Darling T W, TenCate J A, Brown D W, Clausen B and Vogel S C 2004 *Geophysical Research Letters* **31** L16604
[4] Sanaee R, Shadizadeh S R and Riahi M A 2010 *International Journal of Rock Mechanics and Mining Sciences*, **47(4)** p 599-605
[5] Schofield P F, Covey-Crump S J, Stretton I C, Daymond M R, Knight K S and Holloway R F 2003 *Mineralogical Magazine* **67(5)** p 967-987
[6] Wenk H R 2006 *Reviews in Mineralogy and Geochemistry* **63(1)** p 399-426
[7] Wenk H R, Lutterotti L and Vogel S C 2010 *Powder Diffraction* **25(3)** p 283-296
[8] Frischbutter A, Neov D, Scheffzuk Ch, Vrana M and Walther K 2000 *Journal of Structural Geology* **22** p 1587-1600
[9] Wenk H R, Lutterotti L and Vogel S C 2003 *Nuclear Instruments and Methods in Physics Research A* **515** p 575-588
[10] Drobek T, Strobel J, Park S, Phuong K L, Altermann W, Lemmel H, Lindner P and Stark R W 2011 http://jgmaas.com/SCA/2011/SCA2011-48.pdf [SCA2011-48] p 1-6
[11] Radlinski A, AGSO Research Newsletter 1999 http://www.agso.gov.au/information/publicationslresnews/ p 29-30
[12] Radlinski A P, Ioannidis M A, Hinde A L, Hainbuchner M, Baron M, Rauch H and Kline S R 2002 www.jgmaas.com/SCA/2002/SCA2002-35.pdf [SCA2002-35] p 1-12
[13] Kahlel A, Winkler B, Radulescu A and Schreuer J 2004 *European Journal of Mineralogy* **16** p 407-417
[14] Wenk H R 2012 *JOM* **64(1)** p 127-137
[15] Sanaee R, Oluyemi, G F, Hossain M and Oyeneyin M B 2013 In: *Proceedings of the 47th U.S. rock mechanics/geomechanics ARMA symposium*, 23 - 26 June 2013, San Francisco, US. Alexandira, VA: ARMA, document ID 13-442
[16] Antony S, Olubenga A and Ozerkan N 2017 *Rock Mechanics and Rock Engineering* **51(2)**, p 451-464
[17] Mardalizad A, Scazzosi R, Manes A, Giglio M 2018 *Journal of Rock Mechnaics and Geotechnical Engineering* **10** p 197-211
[18] ASTM D7012-04. Standard test method for compressive strength and elastic moduli of intact rock core specimens under varying states of stress and temperatures. West Conshohocken, USA: ASTM International; 2004
[19] Faisal N H, Sanaee R, Singh R M, Mclaughlin A, Lee T-L, Kelleher J 2018 Neutron scattering of porous rocks subjected to uniaxial compression in cryogenic conditions for offshore drilling operations in Arctic, Neutron beamtime by STFC/Engin-X (RB1810235); https://data.isis.stfc.ac.uk/doi/INVESTIGATION/92924528/

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