Grant agreement No. 640979

ShaleXenvironmentT

Maximizing the EU shale gas potential by minimizing its environmental footprint

H2020-LCE-2014-1

Competitive low-carbon energy

D3.5

Relations between tomographic and geomechanical observations, implications for reservoir conditions and sweet spot identification

WP 3 – Advanced Imaging and Geomechanical Characterisation

Due date of deliverable 31/08/2018 (Month 36)

Actual submission date 31/08/2018 (Month 36)

Start date of project 1st September 2015

Duration 36 months

Lead beneficiary UoM

Last editor Lin Ma

Contributors UoM, GFZ

Dissemination level Public (PU)

This Project has received funding from the European Union’s Horizon 2020 research and innovation programme under grant agreement no. 640979.
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<td>06-08-2018</td>
<td>Mike Chandler/ Johannes Herrmann/ Erik Rybacki/Lin Ma/ Peter Lee / Kevin Taylor</td>
<td>First draft</td>
</tr>
<tr>
<td>1.1</td>
<td>23-08-2018</td>
<td>Lin Ma</td>
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**Key word list**

Tomography, strength, Young’s modulus, effective medium theories, creep, proppant embedment

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**Definitions and acronyms**

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</tr>
<tr>
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</tr>
<tr>
<td>TOC</td>
<td>Total Organic Carbon</td>
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1. Introduction

1.1 General context

To fulfil the demand of an energy transition from conventional resources (e.g., coal, oil) towards renewable energies such as wind or solar, energy extraction from so-called bridge technologies is necessary. Energy recovered from unconventional resources, in particular shale gas, is often considered as an optional main contributor to these bridge energy technologies. A profound understanding of the geochemistry, petrology, mineralogy and geomechanical properties of shale is critical for an economical and sustainable energy recovery from these reservoirs.

Shales have been exploited directly as natural gas reservoirs utilising hydraulic fracturing (frac’ing) to increase the effective permeability of the naturally tight shale formations (Rutter and Mecklenburgh, 2017). Fracture growth and sealing properties in shales are also of interest in the fields of CO₂ sequestration and radioactive waste disposal (Fauchille et al., 2016). Mechanical data for shale rocks is, therefore, of significant interest, especially those relating to the propagation and sealing of fractures. Fracture growth and sealing in the subsurface is often complex with tortuous pathways and significant horizontal and vertical extent (Fisher and Warpinski, 2012; Thiercelin et al., 1987, Backeberg et al. 2017). Based on geochemistry, petrology, mineralogy and geomechanical properties, sweet spots within a particular reservoir are defined as regions that represent the highest potential for an economic production of hydrocarbons. To increase the production rate by creating a larger inflow area, artificial fractures are generated by hydraulic stimulation, which are believed to connect with pre-existing natural fractures. To keep these fractures open, proppants are typically added to the stimulation fluid. However, the production rate often decreases non-linearly over time, probably induced by fracture closure due to proppant embedment into the shale matrix. The fracture closure behaviour within shale rocks strongly depends on their mechanical properties, which are affected by pressure, temperature and stress conditions, and on petrophysical properties (e.g., mineralogy, porosity). The knowledge of these properties is crucial to estimate the closure rate of fractures within shale formations.

In the last decade, synchrotron-based time-resolved X-ray tomography (4D XCT) has become a powerful tool in many engineering fields for the analysis of in situ deformation of materials (Cai et al., 2014; Karagadde et al., 2015; Mostafavi et al., 2015). The application of synchrotron 4D XCT to shale is in its early stages, but it can be used for very rapid imaging and qualitative study of fracture development in shales (Figueroa Pilz et al., 2017). This technique allows to improve the understanding of the local conditions around a fracture as it develops and seals. The relations between tomographic and geomechanical observations will, therefore, improve the understanding of shale mechanical properties significantly and have strong implications for reservoir conditions and sweet spot identification.
1.2 Deliverable objectives

The specific objectives are:

1. To measure the mechanical properties (e.g., strength and static elastic constants) of selected shale samples, allowing estimates of proppant embedment-induced fracture closure to be made.

2. To conduct advanced in-situ experiments using a synchrotron beamline (e.g., real time 4D X-ray tomography), allowing the observation of fractures induced by hydraulic pressure and the sealing.

3. To seek correlations between mechanical properties and tomographic images.

2. Methodological approach

2.1 Deformation of shales at reservoir conditions

To measure the mechanical properties of shale rocks recovered from different shale formations, two types of deformations experiments were performed at varying confining pressures, $p_c$, and temperatures, $T$. First, intact samples were deformed at constant strain rate, $\dot{\varepsilon}$, allowing to determine the triaxial compressive strength, $\sigma_{TCS}$, and static Young’s modulus, $E$, from measured stress-strain curves. Secondly, sample deformation was performed at constant stress, $\sigma$, to unravel the sample’s creep properties from observed creep curves. To investigate the indentation behavior of proppants into the shale matrix, additional creep tests were performed on split samples containing a monolayer of proppants. We used proppants composed of $> 99$ wt% quartz.

Samples examined include Carboniferous Upper and Lower Bowland (BOS) shale (UK) recovered from boreholes (Preese Hall1, Marl Hill Moor) and an outcrop located in Lancaster, which is believed to be England’s most prospective shale play. In addition, we studied Cambrian Alum (ALM) shale (DK), Lower Jurassic Posidonia (HAR, HAD, DOT) shale (GER), Haynesville (HVS) shale (US) and Marcellus (MAS) shale (US). The experiments were focussed on the comparison between mature, clay – rich Posidonia (HAR) shale and mature, quartz – rich Upper Bowland shale, recovered from an outcrop (BOS_OC). The latter was selected due to insufficient availability of borehole core material required to perform deformation tests. Detailed petrophysical data including porosity measured by He-pycnometry of all investigated samples are given in Table 1.

All experiments were performed on cylindrical samples (length = 20 mm, diameter = 10 mm), prepared perpendicular to bedding, using a Paterson type deformation apparatus (Paterson 1970). The samples were jacketed by copper sleeves to prevent intrusion of the
gas-confining pressure medium. Recorded axial force were converted to axial stress assuming constant volume deformation and corrected for the strength of jackets. Axial strain was determined from measured axial displacement and corrected for system compliance.

In what follows we briefly describe the procedures implemented for (i) constant strain rate tests, (ii) constant stress tests, and (iii) constant proppant indentation tests.
Table 1: Mineralogical composition, density and organic content of investigated samples.

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Sample abbreviation: HAD = Haddessen, HAR = Harderode, DOT = Dotternhausen (all Posidonia formation); MAS = Marcellus; HSV = Haynesville (North American shales); BOS1 – 7 = Upper Bowland formation from Preese Hall 1; BOS8 – 10 = Lower Bowland formation from Preese Hall 1; BOS11 – 14 = Upper Bowland formation from Marl Hill Moor; BOS_OC = Upper Bowland formation from outcrop). VRr (vitrinite reflectance) is maturity, ρ is bulk density, φ is total connected porosity, TOC is total organic matter content. CTMP (= Cly + TOC + Mica + φ), Cb and QFP (= Qtz + Fsp + Py) display weak, intermediate and strong phases in vol%.
i. **Constant strain rate tests**
Samples were deformed at $\dot{\varepsilon} = 5 \times 10^{-6}$, $5 \times 10^{-5}$ and $5 \times 10^{-4}$ s$^{-1}$, $p_c = 50$, 75 and 100 MPa and $T = 75$, 100 and 125 °C. We conducted three different test series, for which two of the three parameters ($\dot{\varepsilon}$, $p_c$, T) were held constant while the remaining parameter was changed. $\sigma_{TCS}$ is represented by the peak stress value in the measured stress – strain curve and $E$ was calculated from the slope of the tangent drawn at 50 % of the peak strength value. Most experiments were terminated after shear failure of the specimen. Few other samples showed a barrel shape after high strain and were finished after < 20% strain.

ii. **Constant stress tests**
Creep tests were performed at $\sigma = 160 – 453$ MPa, $p_c = 65 – 115$ MPa and $T = 75 – 150$ °C. Similar to constant strain rate experiments, we also performed three test series to characterize the influence of $\sigma$, $p_c$ and T on the time-dependant deformation behaviour. Depending on the applied conditions, tests were either stopped after sample failure or after certain strain if no failure occurred. Constant stress tests were restricted to Posidonia (HAR) and Bowland (BOS, BOS_OC) shales.

iii. **Proppant indentation tests**
Indentation tests were performed at constant stress ($\sigma = 37$-93 MPa), $p_c = 75$ MPa and $T = 90$ °C. A monolayer of quartz proppants was positioned between two cylinders (length = 9,85 mm, diameter = 9 mm) of outcrop Bowland (BOS_OC) and Posidonia (DOT) samples. The applied load was stepwise increased up to 93 MPa for BOS_OC and 71 MPa for DOT, respectively. For each step, the deformation was recorded and the load increased if the inelastic strain rate was < 7 x $10^{-9}$ s$^{-1}$. High resolution scanning electron microscopy (SEM) performed on thin sections prepared from deformed sample – proppants assemblies was used to inspect the indentation behavior of quartz grains into the shale rock matrix.

### 2.2 In-situ 4D tomographic imaging- hydraulic fracturing test using a newly designed triaxial apparatus

Fluid injection experiments were conducted using the newly developed High Energy X-Ray Imaging of FRACturing (HEXIFRAC) pressure vessel shown in Figure 1a and Figure 1b. This vessel, with the associated pressurisation system were mounted on the rotation stage on the I12 beamline at Diamond Light Source, Harwell, UK. Experiments were conducted between 31/01/18 and 05/02/18. Imaging was conducted using the Miro camera and consisted of 800 projections with $1280 \times 800$ pixels over a 20 mm field of view, for a $\approx 16 \mu$m resolution.
Figure 1: (a) Diagram of the HEXIFRAC pressure vessel. The vessel is made from T6 7075 Aluminium alloy which is a good compromise between strength and x-ray transparency. (b) Photograph of the vessel and pressurisation system installed on the I12 beamline. (c) Example injection pressure record.

Samples of 20 mm diameter and ~40mm height were confined at pressures up to 60MPa, with a low viscosity synthetic ester (Di-ethylhexyl sebacate) confining fluid. Each sample contained a 1.2mm diameter borehole from the base of the sample reaching 20mm through the sample height. The confining pressure was applied gradually, with regular pauses to record tomographs of the sample material under increasing confinement. Tomographs were recorded by rotating the stage through 180° over 15 seconds. Once the desired confining pressure was reached, the pressurisation system was reconnected and used to increase the borehole pressure, simulating a rise in fluid injection pressure. Injection pressure was raised gradually, with pauses for tomographic imaging. Once the breakdown pressure was reached, the fracture growth (and resultant pressure drop) was found to be rapid and uncontrolled. Figure 1c shows an example plot of injection pressure as a function of the volume of fluid injected. After failure, the injection pressure was lowered to zero, and the confining pressure was raised, in order to image the pressure-driven closure of fractures.

3. Summary of activities and research findings

3.1 Influence of rock composition and loading conditions on mechanical properties of shale rocks

In total, 82 deformation experiments were performed to investigate the influence of composition, pressure, temperature and differential stress on the mechanical behavior of various European and North American shale rocks.
3.1.1 Strength and Young’s modulus

Constant strain rate experiments were performed on all shales at $\dot{\varepsilon} = 5 \times 10^{-4} \text{ s}^{-1}$, $p_c = 50 \text{ MPa}$ and $T = 100^\circ \text{C}$, which may roughly simulate the short-term deformation under in-situ conditions. The results reveal that quartz – rich and carbonate – rich Bowland shales recovered from borehole (PH1) are stronger than quartz – rich outcrop material. Bowland and Marcellus shales are noticeably stronger than clay – rich Posidonia or Alum shales as well as Haynesville shale (Figure 2a). Bowland and Marcellus shale deform brittle, characterized by pronounced elastic and only minor plastic deformation before sample failure. Posidonia and Alum shales deform semibrittle, expressed by very little elastic and marked plastic deformation. In general, the triaxial compressive strength increases almost linearly with increasing static Young’s modulus (Figure 2b).

![Figure 2: (a) Representative stress – strain curves of shales deformed under triaxial conditions and (b) empirical correlation between triaxial compressive strength, $\sigma_{TCS}$, and static Young's modulus, $E$. U – BOS = Upper Bowland shale, L – BOS = Lower Bowland shale, HSV = Haynesville shale, MAS = Marcellus shale, ALS = Alum shale, POS = Posidonia shale, HAR = Harderode, DOT = Dotternhausen, HAD = Haddessen, OC = outcrop, PH1 = Preese Hall 1, MHM = Marl Hill Moor. Deformation conditions are indicated.](image)

The influence of sample mineralogy on $\sigma_{TCS}$ can be visualized in a ternary diagram (Figure 3). Here, we separated the sample composition into 1) strong constituents (quartz, feldspar, pyrite), 2) intermediate strong constituents (carbonates) and 3) weak constituents (clay, total organic carbon, mica) including porosity. At the applied deformation conditions, no influence of volumetric fractions of strong or intermediate strong minerals on $\sigma_{TCS}$ is evident, whereas a strong correlation between $\sigma_{TCS}$ and the fraction of weak components is obvious. The same correlation holds for the influence of sample mineralogy on $E$, as suggested by Figure 2b.
Figure 3: Ternary plot displaying mineral composition of investigated samples with superimposed values of triaxial compressive strength, $\sigma_{TCS}$, in [MPa]. Composition is separated into mechanically strong (QFP = Qtz + Fsp + Py), intermediate strong (Cb) and weak (Cly + TOC + Mca + Poro) fractions. Qtz = quartz, Fsp = feldspar, Py = pyrite, Cb = carbonate, Cly = clay, TOC = total organic carbon, Mca = mica, Poro = porosity.

For shales containing less than $\approx 25$-$30$ vol% weak materials, $\sigma_{TCS}$ appears to be strongly dependent on the volumetric fraction of weak components, whereas above this threshold the strength is almost unaffected by the fraction of weak phases (Figure 4). We interpret this behavior of shales by a gradual change of deformation that is mainly accommodated by a load-bearing framework of strong constituents towards deformation adapted by interconnected weak layers.

A generalized mixture rule as supposed by (Ji, 2004) was used to predict upper (V) and lower (R) strength bounds of two phase materials. Almost all Bowland shales plot close to the lower bound (Figure 5), which is expected for bedding-perpendicular deformation. $\sigma_{TCS}$ of Posidonia shales including literature data (Rybacki et al., 2015) locate between the lower bound and the average of the upper and lower bound ($V - R - H$).

Figure 4: Influence of volumetric fraction of clay + TOC + mica + porosity on triaxial compressive strength, $\sigma_{TCS}$. Deformation conditions are indicated.
Figure 5: Triaxial compressive strength, $\sigma_{TCS}$, versus volumetric fraction of weak components (clay + TOC + mica + porosity). Grey symbols display mechanical data of black shales measured by (Rybacki et al., 2015) at similar deformation conditions. V and R indicate upper and lower bounds, respectively. V – R – H line represents the average value of upper and lower bounds. Deformation conditions are indicated.

Another commonly used mechanical parameter to describe the characteristics of shale rocks with respect to successful hydraulic stimulation is the so-called brittleness, B. We estimated the brittleness calculated from the elastic modulus (Rybacki et al., 2016). Comparable to the observed trends between $\sigma_{TCS}$ and composition (Figure 3), B remains almost unaffected by the volumetric amount of strong or intermediate strong components, but clearly decreases with increasing fraction of weak constituents (Figure 6).

Figure 6: Ternary plot displaying mineral composition of investigated samples with superimposed values of brittleness determined from static Young's modulus. Brittleness of 1 and 0 indicate brittle and ductile behavior, respectively. Composition is separated into mechanically strong (QFP = Qtz + Fsp + Py), intermediate strong (Cb) and weak (Cly + TOC + Mca + Poro) fractions. Qtz = quartz, Fsp = feldspar, Py = pyrite, Cly = clay, TOC = total organic carbon, Mca = mica, Poro = porosity.
Beside composition, the confining pressure and temperature also affect the mechanical behavior of shales. For example, with increasing confining pressure the mechanical stress-strain behavior shifts from brittle towards more semibrittle deformation of the investigated Bowland (outcrop) and Posidonia shales (Figure 7a). \( \sigma_{\text{TCS}} \) and the maximum axial strain before failure, \( \varepsilon_{\text{max}} \), increases as the confining pressure increases, indicating sample strengthening. This effect is more pronounced for clay–rich Posidonia shale than for Upper Bowland shale with less weak phases. Increasing temperature yields little sample weakening, expressed by slightly decreasing \( \sigma_{\text{TCS}} \) of Posidonia and Bowland shales and minor to absent increase of \( \varepsilon_{\text{max}} \) (Figure 7b).

![Figure 7: (a) Influence of confining pressure, \( p_c \), and (b) temperature, \( T \), on stress – strain behaviour of Posidonia (HAR) and Upper Bowland (OC) shales. Deformation conditions are indicated.](image)

In contrast to \( \sigma_{\text{TCS}} \), the Young’s modulus is almost unaffected by a change of confining pressure and temperature within the investigated ranges. Experiments performed at \( T = 90 \) °C and \( p_c = 75 \) MPa showed negligible impact of strain rate in the range between \( 5 \times 10^{-6} \) and \( 5 \times 10^{-4} \) s\(^{-1} \) on the mechanical properties of Posidonia and Bowland shales.

### 3.1.2 Creep behaviour

To investigate the time dependent creep behavior of Upper Bowland and Posidonia shales, constant stress experiments were performed at \( \sigma = 206 \pm 4 \) MPa, \( p_c = 75 \) MPa and \( T = 90 \) °C (Figure 8a). The weak Posidonia shale displays all 3 characteristic parts of a creep curve after the initial elastic deformation: 1) primary (decelerating) creep, where the strain rate continuously decreases, 2) secondary (apparent steady state) creep, characterized by constant strain rate and 3) tertiary (accelerating) creep, defined by a constantly increasing...
strain rate until sample failure. In contrast, the quartz–rich Bowland (OC) shale displays only elastic deformation and subsequently a primary creep phase. At the simulated reservoir T-p_c conditions (depth ≈ 3 km) the cumulative elastic and inelastic axial creep strain of weak Posidonia shale is ≈ 2.5 times higher than the measured axial strain of strong Bowland shale.

Core-derived Bowland shales show at σ = 442 ± 11 MPa and p_c = 75 MPa, T = 90 °C. Solely primary creep was displayed in quartz-rich Bowland samples whereas carbonate-rich Bowland sample exhibited either only primary or in addition to only primary also secondary and eventually tertiary creep. (Figure 8b).

![Creep curves of Posidonia (HAR = Harderode) and Upper Bowland (BOS) shales showing the influence of sample composition on creep behavior of shale rocks recovered from different formations (a) and measured on different Bowland core samples (b). Deformation conditions are indicated. Elevated confining pressure, p_c, and temperature, T, simulate in-situ conditions of approximately 3 km depth.](image)

Figure 8: Creep curves of Posidonia (HAR = Harderode) and Upper Bowland (BOS) shales showing the influence of sample composition on creep behavior of shale rocks recovered from different formations (a) and measured on different Bowland core samples (b). Deformation conditions are indicated. Elevated confining pressure, p_c, and temperature, T, simulate in-situ conditions of approximately 3 km depth.

The influence of confining pressure, temperature and differential stress on the creep response of Posidonia and Bowland shales is exemplarily shown in Figure 9. Increasing confining pressure (black → green line) results in sample hardening expressed by decreasing creep strain. As expected, the effect is less pronounced for strong Bowland than for weak Posidonia shale. With increasing temperature (black → blue line) the axial creep strain of both shales increases as a result of sample weakening. Posidonia shale is more sensitive to an increase of temperature than Bowland shale, quite similar to the results obtained in constant strain rate tests. A slight increase of differential stress (black → orange line) is sufficient to enhance axial creep strain substantially and to induce a change of creep behavior from only primary to additionally secondary and tertiary creep. Note that Figure 9 displays only selected creep curves to show the overall trend of varying loading conditions on the deformation behavior.

In summary, constant strain rate and constant stress experiments revealed that the mechanical properties of the investigated black shales strongly depend on their composition. Within the investigated confining -temperature range, the strength and creep
behavior are also influenced by the magnitude of confining pressure and to a small extent by temperature. Bowland shale with a high fraction of strong minerals is distinctly stronger, more brittle and creeps more slowly than Posidonia shale, which contains a high proportion of weak phases. Accordingly, the mechanical behavior of Bowland shale is also less sensitive to a variation in confining pressure and temperature than Posidonia shale.

![Figure 9](image)

**Figure 9:** Influence of confining pressure, $p_c$, temperature, $T$, and stress, $\sigma$, on the creep properties of Posidonia (HAR)\(\text{(a)}\) and outcrop Upper Bowland (OC) \(\text{(b)}\) shale. Deformation conditions are indicated.

### 3.2 Fracture initiation and healing at simulated in-situ conditions

#### 3.2.1 Correlation between fracture orientation, breakdown pressure and the anisotropic fracture toughness

Fluid injection experiments were conducted on samples manufactured in two orientations with respect to the bedding planes (Figure 10). In all tested shale materials, the mode I fracture toughness, $K_{Ic}$, was found to be 1.3-6 times lower when the fracture propagates in the plane of the bedding than when extending perpendicular to bedding.

During fluid injection experiments, most fracture geometries fell into one of two geometries: either lying parallel to the axis of the borehole (Figure 11), or perpendicular to the borehole axis (Figure 11\(\text{b}\), a “pancake” fracture). Table 2 lists the bedding orientations and failure orientations for each sample tested during the beamtime experiments described in Section 2.2.
Figure 10: (a) Sample geometry and dimensions used in fluid injection experiments. (b) sample geometry for cores perpendicular to the bedding planes. (c) sample geometry for cores parallel to the bedding planes.

Figure 11: Major fracture orientations during fluid injection experiments. (a) Borehole-parallel fracture. (b) Borehole-perpendicular “pancake” fracture.

Table 2: Fracture orientations (as shown in Figure 11) generated from samples of each material cored in the orientations shown in Figure 10. II refers to borehole-parallel fractures (Figure 11a). X refers to borehole-perpendicular fractures (Figure 11b).

<table>
<thead>
<tr>
<th>Material</th>
<th>Layering on the sample scale?</th>
<th>Fracture Orientation</th>
<th>Core oriented perpendicular to bedding (Figure 10b)</th>
<th>Core oriented parallel to bedding (Figure 10c)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mancos shale</td>
<td>laminated</td>
<td>X &amp; II</td>
<td>II</td>
<td></td>
</tr>
<tr>
<td>Bowland shale</td>
<td>unlaminated</td>
<td>II</td>
<td>II</td>
<td></td>
</tr>
<tr>
<td>Haynesville shale</td>
<td>laminated</td>
<td>X</td>
<td>II</td>
<td></td>
</tr>
<tr>
<td>Kimmeridge clay</td>
<td>laminated</td>
<td>X</td>
<td>N/A</td>
<td></td>
</tr>
<tr>
<td>Whitby mudrock</td>
<td>laminated</td>
<td>X</td>
<td>II</td>
<td></td>
</tr>
<tr>
<td>Solnhofen limestone</td>
<td>unlaminated</td>
<td>II</td>
<td>II</td>
<td></td>
</tr>
</tbody>
</table>
Notably, fractures occur in the borehole-parallel orientation in bedding-parallel samples of all materials. In bedding-perpendicular samples, fractures only develop in the borehole-parallel orientation in the unlaminated materials. In materials where layering is present, the fractures were commonly seen to grow in a borehole-perpendicular orientation, so as to follow the layering in the material.

Breakdown pressure during these experiments was found to increase with confining pressure, but was seen to be isotropic between the two sample orientations, and not strongly sensitive to the different materials. This is likely due to the closure of existing microcracks, which predominantly lie parallel to bedding planes (as will be discussed in Section 3.2.2.). At ambient conditions these microcracks have a strong effect on the propagation of fractures, but this is suppressed by the microfracture closure at elevated $P_{\text{conf}}$.

### 3.2.2 Crack healing: Crack closure in relation to the applied confining pressure

During the fluid injection experiments described in Section 2.2, existing fractures were observed in some of the initial sample materials. Pre-existing fractures were generally more numerous in the more laminated materials. The Haynesville shale material was particularly dense in pre-existing fractures, almost all of which were oriented parallel to the bedding. Pre-existing fractures were also observed in the Mancos shale and Whitby mudrock, with orientations mostly parallel to bedding, but some deviations and tortuous forms. Kimmeridge clay displayed a small number of pre-existing fractures. Pre-existing fractures were observed in the Bowland shale and Solnhofen limestone, but were very scarce. Neither of these two materials displays laminations within the sample scale.

**Figure 12** shows a series of tomographic cross-sections through a sample of Haynesville shale oriented parallel to the bedding. An initial population of fractures exists parallel to the bedding, and is seen to close up entirely by $P_{\text{conf}} = 23$MPa. As $P_{\text{inj}}$ is raised to $23$MPa, some of the pre-existing fractures that intersected the borehole are observed to reopen slightly. As soon as $P_{\text{inj}}$ is raised above $23$MPa, the main connected fractures are seen to open widely, as well as some fractures that appear not to be connected to the borehole. This suggests that there are small connecting cracks (either pre-existing or generated during breakdown) that are below the resolution of our images. After the sample breakdown, $P_{\text{inj}}$ was lowered to zero, and $P_{\text{conf}}$ gradually raised until the fractures were seen to close up. $P_{\text{conf}}$ had to be raised to $\simeq 60$MPa for complete closure to be observed after the fluid injection, despite the fractures having initially closed up by $P_{\text{conf}} = 23$MPa.
3.2.3 Closure of propped fractures under differential stress

We examined the indentation of proppant (quartz) grains into the shale rock matrix at simulated reservoir conditions (depth = 3km, $p_c = 75$ MPa, $T = 90$ °C, $\sigma \approx 35 - 95$ MPa). SEM micrographs of deformed shales containing a single proppant layer between samples with planar interfaces reveal that for dry (outcrop) Bowland shale the indentation is minor and restricted to few areas with intact proppants (Figure 13). Many other proppants are broken and fragmented, likely due to the high stress applied at the final load steps that induce damage by the resulting high Herzian contact stresses. In case of indentation, a localized ‘damage zone’ appears, characterized by compaction of the shale matrix due to pore closure and the formation of inter- (bold black arrow in Figure 14) and intracrystalline microfractures (open black arrow in Figure 14) within dolomite (Dol) and quartz (Qtz) grains.
The strength and creep properties of rocks usually depend on their water content, in particular for rocks with a high fraction of weak phases. A high water content lowers the strength and enhances the creep rate. We added water to one Bowland shale – proppant assembly to study the influence of water on the indentation behavior. The observed indentation is quite similar to the behaviour obtained under dry conditions (Figure 15).

Figure 13: SEM micrograph of a deformed dry Upper Bowland shale – proppant assembly. Quartz grains (proppants). Loading direction is perpendicular to proppant layer.

![Figure 13](image1.png)

Figure 14: SEM photograph of damage zone generated due to indentation of a quartz grain into the matrix of dry Upper Bowland shale. Py = pyrite, Dol = dolomite, Qtz = quartz.

Figure 15: SEM micrograph of a deformed wet Upper Bowland shale – proppant assembly. Loading direction is perpendicular to proppant layer.

Figure 15: SEM micrograph of a deformed wet Upper Bowland shale – proppant assembly. Loading direction is perpendicular to proppant layer.

Since Posidonia shale contains a high volumetric fraction of weak phases and the shale is weaker and creeps faster than Bowland shale, we expect a stronger indentation of
proppants into the matrix of Posidonia than of Bowland shale. This was confirmed by an experiment performed at similar deformation conditions, revealing that indentation of proppants into the matrix of dry Posidonia (DOT) shale is significantly more pronounced than for dry and wet Bowland shale (Figure 16). Crushing of quartz proppants is also substantially less, resulting in only minor fines production. Most of the matrix deformation in the ‘damage zone’ close to the proppant grains was accommodated by compaction and bending of weak phyllosilicates (Figure 17).

Note that the final axial load steps do not represent in-situ loading conditions and were just applied to accelerate indentation within experimental time scale. Applying only low axial stresses presumably would result in less fines generation.

Figure 16: SEM micrograph of a deformed dry Posidonia (DOT) shale – proppant assembly Loading direction is perpendicular to proppant layer.

Figure 17: SEM micrograph of a damage zone generated by indentation of a quartz grain into the matrix of dry Posidonia (DOT) shale. Cal = calcite, Rut = rutile, Py = pyrite, Qtz = quartz, Phy = phyllosilicate.
4. Conclusions and future steps

At the imposed confining pressures and temperatures, the studied black shales revealed brittle to semibrittle deformation behaviour, depending on mineralogy and p_c–T conditions. The triaxial compressive strength and static Young’s modulus were strongly dependent on confining pressure and less on temperature. Below about 30 vol% of weak constituents these parameters strongly decrease with increasing fraction of weak phases, presumably reflecting deformation of shales accommodated by a load bearing framework. At high fraction of weak constituents, deformation is likely accommodated by interconnected weak layers and both strength and Young’s modulus do not depend on the total amount of weak constituents. In most cases, these mechanical properties plot between the lower bound and the average of the upper and lower bound calculated from empirical effective medium theory approaches.

Shales loaded under constant stress and simulated in-situ p_c – T conditions of about 3 km depth deform only in the primary creep regime if they consist mainly of strong minerals (e.g., quartz – rich Boland shales). For shales with a high proportion of weak phases (e.g., Posidonia shale) we observed in addition secondary and tertiary creep regime. The creep rate increases with increasing stress and temperature, but decreases with increasing confining pressure. In that sense, the mechanical behavior of shales varies with mineralogy and loading conditions in long-term creep tests in the same way as in short-term constant strain rate tests.

In line with the mechanical strength and creep properties, indentation of proppants composed of fine quartz grains into the shale matrix is significant only for shales composed of a high fraction of weak phases, like Posidonia shale. Conversely, at high differential stress, proppant crushing and fines generation is more pronounced in shales consisting mainly of strong minerals as for example Bowland shale.

The in-situ and ex-situ X-ray tomography experiments have been proved to be powerful tools for mechanical behaviour observations. Samples with different rock composition, microstructures and orientations have different behaviour with loading conditions. During the in-situ fluid injection experiments, most fracture geometries fell into one of two geometries: either lying parallel to the axis of the borehole, or perpendicular to the borehole axis. In all tested shale materials, K_{1c} was found to be 1.3-6 times lower when travelling in the plane of the bedding than when in one of the perpendicular orientations. Also, some existing fractures were observed in some of the initial sample materials in the in-situ experiments. Pre-existing fractures were more numerous in the more laminated materials.
Our results suggest a substantially lower in-situ fracture closure rate in brittle Bowland shales compared to semibrittle Posidonia shales, which seems to suggest a possibly economical and sustainable extraction of hydrocarbons. However, the relatively low porosity and hydrocarbon content of the Upper Bowland shale may limit the prospectivity. For a better quantitative understanding of fracture closure and conductivity, propped fracture permeability experiments at simulated in-situ conditions will be performed in future. To avoid grain crushing induced by unrealistically high differential stress, long-term embedment tests at low stress are required, which limits the number of experiments.

This is the last deliverable in this work package, and the observations have integrated the previous results of microstructure characterisation, tomographic imaging of systems, fluid and high-pressure behaviour. Relations between these characterisations and results obtained within other work packages will be discussed in the final report to provide a comprehensive understanding of shale properties.
5. **Publications resulting from the work described**

1. “Deformation experiments on Bowland and Posidonia shale – Part I: Strength and Young’s modulus at ambient and in-situ $p_\alpha$, $T$-conditions” – Herrmann et al. 2018 (Rock Mechanics and Rock Engineering, accepted)


6. **Bibliographical references**


