

British **Geological Survey** NATURAL ENVIRONMENT RESEARCH COUNCIL



Exploring the micro-scale controls on fracturing in a Carboniferous limestone, and their implications for carbon capture and storage RACHAEL ELLEN *¹, STEPHANIE ZIHMS², SUSANA GARCIA³ NAZIA MUBEEN FAROOQUI³, M. MERCEDES MAROTO-VALER³, HELEN LEWIS² AND GRAHAM LESLIE¹

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Introduction

Carboniferous rocks have long been recognised as some of the most economically viable strata in the UK, with a rich history of coal, iron, limestone and shale extraction since the 18th century. Today, both onshore and offshore Carboniferous strata are becoming increasingly economically important for future energy resources and solutions. Carboniferous strata in the UK are typically heterolithic, comprising sequences of mudstone, limestone and coal. With renewed interest in these rocks and the potential they could provide for the energy market, it is important to understand their mechanical response to natural brittle deformation, and the implications that may have on their future use as energy resources. This study explores how natural fractures, and fractures potentially induced during drilling/injection, in a prominent limestone unit in central Scotland would respond to CO2 storage at conditions similar to that under the North Sea. We approach this using a combined field, geomechanical and geochemical approach.



Geological Setting and Methodology

The Spireslack surface coal mine in East Ayrshire, Scotland, exposes faulted and fractured Carboniferous strata. The mine is bounded on one edge by the McDonald (Hosie) Limestone, a fossiliferous biomicrite of marine origin. This limestone pavement is abundant in fossils, including Planolites, Rhizocorallium and Chondrites, as well as brachiopods, trilobites and shark spines. The overall structure of the strata at Spireslack is disposed across the north-east trending Muirkirk Syncline, apparently generated during sinistrally transpressive deformation during the mid- to late- Carboniferous Era. The strata are displaced by two sets of left-lateral, oblique-slip curvilinear faults with a NNE and NW orientation and a range of displacements, and are particularly well exposed in the McDonald Limestone pavement (photo above). To study fractures in this limestone, a sample was taken from the hanging wall of a 20 cm displacement fault cutting the McDonald limestone (sample 1NF). An unfaulted sample, the protolith (sample PL), was also collected to deform in the lab in order to study drilling/injection induced fractures in limestone. The samples were cored, and sent for XRT scanning prior and post CO2-brine experiments. Two cores were taken from PL so that one could be deformed in the lab (PLa).

PL: Protolith



		3 cm	<mark>250 μm</mark>	<u>_500µm_</u>		$ \begin{array}{c} 40 - & \\ - & \\ - & \\ 20 - & \\ 0 - & \\ 0 - & \\ 0 - & \\ 0 - & \\ 0 - & \\ 0 - & \\ 5 - & \\ 10 - & \\ 5 - & \\ 10 - & \\ 10 - & \\ 15 - & \\ 10 - & \\ 10 - & $	50 microns	Beddi	3.8 cm
Core f	rom the protolith sam-	Thin section of protolith, cut	BSE-SEM image of PL. Gastropods and	Ca and Fe element maps for PL. Ca is		Ca element map for matrix of 1NF, and	BSE-SEM image of 1NF. ?Siderite minerals	Thin section of 1NF. Two sets of	Core taken from naturally
ple, pe	rpendicular to bedding.	perpendicular to bedding. Note	brachiopods form the matrix of this fossil-	predominant in calcite veins and shell		element spectra of ?siderite minerals	forming the matrix of the rock have been	calcite/siderite/Fe filled fractures,	fractured sample, sampled
Top sur	rface of the limestone is	one set of bedding parallel	iferous limestone, along with calcite and	fragments, whereas Fe is more		(Fe-Mg carbonate signature).	sheared and comminuted, forming discrete	formed sub-parallel to fault &	perpendicular to the fault to
att	he top of the photo.	fractures, mineralised by calcite.	Fe-Mg carbonate minerals (?siderite).	distributed throughout the matrix.	\checkmark		shear bands within the matrix.	bedding (see PL). No fault rock.	capture fault-related damage.

PLa: Induced-fractured Protolith

The impact of newly formed fractures (i.e. formed due to drilling, CO2 injection, or other changes to subsurface conditions) on rock-fluid interactions was investigated by inducing fractures through triaxial compression (Pc: 25MPa) on the protolith sample. Understanding the fracture pattern also aids prediction of rock responses in the subsurface. One dominant fracture formed following deformation, with an aperture of c. 0.5 mm, along with a network of subsidiary and lower apertures fractures. Future SEM and thin section analysis will study any host rock control on fracture development



PLa sample plug (Ø 38mm) main fracture after triaxial compression: a through-going planar fracture developed at c. 70 degrees to σ 1. Subsidiary fractures also formed, with a smaller aperture.

High Pressure-High Temperature (HP-HT) experiments: CO2-brine-rock interactions



- Samples 1NF, PL and PLa were used for rock-fluid interaction experiments, to study and compare the effect of protolith, naturally fractured, and freshly fractured rock on CO2-brine interactions, under conditions representative of a CO2 storage reservoir in the North Sea. - These limestone cores were used in (HP-HT) batch experiments.

- The limestone-CO₂-brine experiments were carried out for **7.2** weeks under conditions likely to be found in a relevant downhole environment in the North Sea (*T=80°C, P= 25MPa*). - Brine chemical composition was analysed before and after the reactions by ICP-OES (inductively coupled plasma optical emission spectrometry).



Results (a work in progress)



Picture A = PLPicture B = 1NFPicture C = PLa

Conclusions and Future Work

Fault related fractures from the McDonald Limestone mostly failed in extension. Some evidence for shear bands (and decreased porosity) along carbonate mud.

PLa: now-open fractures formed in direction of loading accommodating dilation in σ^3 direction. Induced fractures appear to be connected. The overall orientation is steeper compared to natural fractures (1NF) - this could be due to the sample length.

Heriot Watt FLEDGE r r SEM analysis ماابر rese Hamilton for coring and Thanks to SMRT for rille all defor access to Spireslack **0**0 the

Batch experimental tests were conducted to understand the impacts of fracture age and limestone reactivity upon CO₂ injection.

Both, precipitation and dissolution of mineral phases, are inferred from changes in the chemical composition of reacted brines.

Further analyses are on-going in order to link brine compositional changes with solids mineralogies.

Future Work

Strain analysis of PLa

Thin section and SEM analysis of all post-treatment samples to understand impact on mineralogy and compare with lab-induced fractures.

Deform more samples under different conditions

The mineralogy of the starting materials and reaction products will be determined by X-ray diffraction (XRD) analysis.

XRD measurements will be linked to brine compositional and pH changes for the three samples.

Geochemical reactions responsible for chemical changes in the system could then be inferred and ultimately, a relationship between geochemistry and observed geomechanical changes, could be established.



CO2-brine experiment results

■Na+ ■K+ ■Mg2+ ■Ca2

[a+] = 104015ppm (out of scale)

Sample 1NF and PLa (i.e. both fractured) obtained a strong red colour following batch experiments, and precipitation of new minerals along the base of the sample. SEM and thin section analysis will identify the cause of this reddening, and identify the minerals. However, PL showed no colour difference, inferring the presence of fault-related or 'fresh' fractures significantly enhances rock-fluid interactions in the McDonald limestone.

A large decrease was observed in Na concentration in the CO2 brine solution following the 7.2 weeks experiment, which is probably due to precipitation of Na-bearing minerals as a consequence of the limestone-CO₂brine interaction.

Mineral dissolution of limestone in the CO₂ saturated brines is inferred from increased concentrations of Ca, K and Mg in the reacted brines. Concentrations of Fe, S and Mn were also observed in the reacted brine (not originally present).

XRT results

Jnreacted brin

XRT measures the density of the sample and a darker colour correlates to a smaller density. No obvious overall differences in XRT results after CO2 treatment. Likely that mineralogical changes are below the resolution (52µm)of XRT but will be visible in thin section SEM analysis. The lab-induced fractures (Fig YB) are visible which can be used to measure strain.



Figure 1 Pre-treatment XRT reconstruction (52µm voxel resolution) A: 1NF B: PLa C: PL

