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Sampling and preparation of *c.* 200 mm diameter cylindrical rock samples for geomechanical experiments

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Highlights

- Large (*c.* 200 mm diameter) synthetic rock-analogue sample construction and rock sample collection techniques described
- New sample preparation apparatuses described for large natural rock samples
- Step-by-step sample preparation methodology presented

Abstract

Experimental investigation of rock mechanical properties of real and artificial samples often requires much care and attention to detail during sample preparation. This especially applies to high fidelity state of the art complex experimental apparatuses where sample tolerance is low due to the complexity of the measuring and stress control devices as well as the nature of the experiments to be conducted. Although sometimes mundane, the sample preparation methodology is as equally important as the experimental apparatus itself, and can require several new technological developments. The methodology and technical developments required to prepare realistic heterogeneous, fractured and natural reservoir analogue rock samples for coupled thermo-hydro-mechanical-chemical process experimental investigation is described here. We present the sample recovery and preparation procedures for large (*c.*200 mm diameter), cylindrical samples of 200 mm +/- 5 mm length, with variable composition and mechanical properties e.g. rock strength, existing fractures/fracture networks, macro-porosity, or lithic fragments. Although the technology demonstrated is for a specific application, the procedures developed, equipment and methodology are applicable to multiple sizes of sample requirements.

1 Introduction

For the investigation of subsurface processes and their interactions, as relevant to industrial applications and geoenergy technologies, specialised experimental equipment is required (e.g. True Triaxial Testing of Rocks [1]). Representing representative subsurface conditions in the laboratory is a prerequisite for conducting realistic experiments under controlled conditions.

Rock mechanics and geoenergy experimentation can be broadly divided by sample shape – cylindrical samples are traditionally used in conventional triaxial (axisymmetric) testing, while cuboid samples are used for true triaxial equipment. Each sample shape demands its specific sample preparation considerations. For example, cylindrical conventional triaxial cells are commonly designed to accommodate standard core sized samples e.g. 38 mm or 100 mm cores, and only require the end faces to be parallel to each other, and perpendicular to the sample axis, in order to meet the established suitability requirements. Circumferential loading in the axisymmetric case is achieved by the imposition of a uniform fluid confining pressure that is separated from the sample (which can have some surface irregularities) by a membrane. The preparation of the sample is typically performed by careful coring, followed by preparation of the ends via a grinding process that employs a specific jig to ensure the parallelism and perpendicularity of the ends.

In the field of rock mechanics experimentation, the control of subsurface stress is essential since it is a primary factor that governs rock deformation processes that may range from shear slip events associated with earthquakes, to hydraulic fracture propagation, and to fluid flow characteristics in fractured reservoirs [2–4]. Because the subsurface stress conditions are almost always of a true-triaxial nature [5], true-triaxial apparatuses have been developed to improve the understanding of coupled thermo-hydro-mechanical-chemical processes and properties under these conditions. Recently, these apparatuses have been developed for the investigation of coupled thermo-hydro-mechanical-chemical processes relevant to geoenergy applications [6–8]. These apparatuses are often impressive feats of engineering, involving many years of development.

However, as impressive as these technologies are, the matter of sample collection/manufacture, and preparation for installation into the apparatus, remain an integral and important part of the scientific process.

The roles of heterogeneous material-parameter distributions and/or the presence of pre-existing discontinuities, and the identification and quantification of coupled process parameters, have been highlighted as a key area for research into future subsurface geoenergy applications [9]. Larger sample sizes enable the investigation of spatially-variable materials, such as studies of the impacts of an array of pre-existing fractures (e.g. [6,10]), because in large samples, individual features such as fractures may have spatial arrangements such that they do not dominate the respective process to the large extent as happens with a single through-going feature. Large samples do, however, pose their own challenges with respect to collection and preparation [11,12]. Nevertheless, the scientific gains to be made by increasing sample size provide a motivation to overcome the potential challenges associated with large sample sizes.

The majority of true-triaxial testing apparatuses require cubic or prismatic samples due to their choice of loading by orthogonally orientated pistons (or the equivalent, flat-jacks) in the three principal directions i.e. σ_1, σ_2, and σ_3. Sample preparation for these apparatuses involves excavation of blocks of rock, or generation of synthetic rock analogues in specific moulds, that can then be trimmed with saws and finished on grinding wheels to create perfect cubes or rectangular prisms. Opposing faces must be as close to parallel as possible and orthogonal with respect to other planes in the cuboid to ensure the loads are applied in a true triaxial manner. Consequently, many true triaxial testing apparatuses use samples less than 100 mm x 100 mm x 100 mm in size, which makes the sample preparation process more manageable with standard rock preparation techniques e.g. [7,13–20].

There are two notable exceptions to the sample shape rule – the SMART cell [21,22], and the newly commissioned Geo-Reservoir Experimental Analogue Technology (GREAT) cell [8]. These designs both employ cylindrical sample shapes and apply radially-variable circumferential loads via fluid filled cushions. While the SMART cell can accommodate up to 100 mm cores, the GREAT cell is designed for 193.75 mm diameter samples and therefore has more-challenging sample preparation requirements, whose solution we report herein.

This manuscript first describes the methodology and required technological developments to prepare large artificial samples and rock samples for experiments within the newly commissioned GREAT cell [8]. Although we describe specifically the requirements for this apparatus, the methodology, new tools and techniques are widely applicable. The GREAT cell is a novel true triaxial apparatus capable of subjecting large bench-scale cylindrical samples (193.75 mm diameter, 200 mm

+/- 5 mm length) to representative temperatures, fluid pressures, and stresses in subsurface geo-energy applications. It is categorised as a Type-II/flexible medium type true triaxial cell and applies loads to the sample through a combination of steel platens (axial load), and axially aligned fluid-filled bladders known as Pressure Exerting Elements (PEEs). The PEEs apply their individual pressures to segments of the cylindrical surface of the specimen, and they are located in an annulus between the outer confining cell steel ring and the rock sample [8]. The PEEs are longer than the sample length to ensure the pressure is applied to the whole length of the sample. Combined with a 2 mm Viton sheath between the sample and the PEEs, this ensures a hydraulic seal is created with the top and bottom platens.

Currently, sample strain is measured with a high-resolution fibre optic strain sensor that is wrapped around the circumference of the sample. To connect to the light source, the fibre must exit the cell between the sample-platen stack and the PEEs i.e. running up the sample, across the join between the sample and the platen, and out the top of the cell. This has resulted in the necessary design requirement that the sample diameter is the same as the platen diameter because pressure applied to the fibre across a small bend radius at non-matching platen and sample diameters would result in fibre damage. Recording strain in this way yields significant volumes of high quality data that can show how sample heterogeneity can influence deformation in response to applied loads (including in fractured materials) [8]. Similar strain-acquisition methods could be successfully applied in existing axisymmetric cylindrical apparatuses, which would require similar preparation techniques to those described in this paper.

First the methodology for artificial sample construction is described, then, the procedure for obtaining natural rock samples. Following this, the development of new equipment designed to machine large diameter rock samples to low tolerance is described.

2 Sample Construction or Collection

Due to the large size of the GREAT cell, cores at *c.*200 mm diameter recovered from deep boreholes are not generally available, so representative samples need to be sourced from readily-accessible locations, or artificial rock analogues need to be constructed.

2.1 Synthetic sample construction

Experimental investigations of hydraulic fracture propagation and fracture flow in low permeability rocks, e.g. [8,23,24], can benefit from the repeatability and controllability of constructing synthetic samples in the laboratory.

The synthetic samples constructed for the GREAT cell experiments [8] are made from a water-clear polyester casting resin cured with an MEKP catalyst at 1% concentration. Each sample is built up in a series of individual pours that are allowed to cure individually to prevent the sample from reaching too high a temperature during the curing process. Once the catalyst is mixed into the resin as evenly as possible, this is poured into a specifically designed mould made of High Density Polyethylene (HDPE) that has non-stick properties for ease of removal of the sample once it is cured. The mould is then placed in a vacuum degassing chamber and a vacuum applied to remove any air bubbles in the mixture. The vacuum pump can evacuate the chamber to conditions < 1 mbar (

Figure *1*). Following degassing, the mould is removed from the chamber and allowed to continue curing in the fume cupboard.

By changing the orientation of the mould for each pour, different orientations of heterogeneities can be created within the polyester samples (Figure 2, left). Heterogeneity could be caused by variations in resin properties between pours, or, as here, by adding thin films of sand grains on the interfaces between individual pours. These methods create inclined weaknesses that act like sealed faults. The polyester resin can also be used to enable tests with rock samples that are too small for the GREAT cell (e.g. 100 mm cores from the field) by casting them into a resin sheath (Figure 2, right).

2.2 Analogue sample collection

Rock samples from the field are extracted in one of two methods; coring *in situ* with a portable coring device, or excavating a large block or rock mass, followed by later coring at the facilities at the University of Edinburgh. In each case, coring is performed with a 200 mm outer diameter core barrel used in conjunction with a Hilti 220 portable drill. Coring is performed wet, with water supplied to the core barrel from within the drill. The pressure of the water is kept minimal: a head of approximately 1 m is sufficient to maintain lubrication of the core barrel and to remove fine material and cuttings. The drill is fixed to a stand that allows us to control the cut angle depending on the desired orientation of the core with respect to bedding planes and/or fracture geometry.

When coring *in situ*, the stand is fixed to the substrate with a single mechanical or resin anchor and then cored to the desired depth. It is usually necessary to apply an extra axial load to ensure the initial cut of the barrel is smooth and does not catch and move the drill. To extract the sample, surrounding material is removed to allow access the base of the core. Care must be taken not to damage the sample or cause movement on any existing fractures that may be present. The sample can then be broken from the substrate and lifted out of the ground. Samples excavated from the field are wrapped in cling film and tin foil to minimise moisture loss, and then wrapped in a protective plastic sheet, similar to the method proposed by McDermott et al.[12].

In some cases, it may be possible to recover large block samples of the material (e.g. in a quarry), in which case it is easier to drill in a laboratory, where the drill and stand are fixed to a permanent frame. At the University of Edinburgh, this frame incorporates two clamping arms holding the blocks firmly ensuring a straight cut.

3 Sample Preparation

In apparatuses requiring an exact core diameter, such as the GREAT cell, the samples need to be trimmed to that diameter before they can be used. The cylindrical surfaces need to be within a tolerance of 0.3mm [25] and the top and bottom surfaces of the sample must be parallel to within 0.001 x diameter, with a squareness of ends to within 0.001 radians [26].

The sample preparation for the synthetic samples is relatively straight-forward: once cured, the samples are faced off at both ends to ensure parallel and flat ends, before being turned to the required diameter on a high-precision machining lathe. The nature of the polyester resin requires a slow turning speed of 30 RPM and small cuts to be made with each pass.

The sample preparation for excavated rocks, however, requires the following procedure:

1. Trim to length using a clipper saw – cutting to approximately 5 mm longer than final target length
2. Top and bottom surfaces are faced off to ensure they are parallel using an in-house designed grinder
3. Sample centre is located and a shallow-depth 3.2 mm hole drilled into the ends, to centre the sample on the turning equipment
4. Turning of the rock to a predefined diameter (193.75 mm for the GREAT cell) on in-house designed equipment

To trim the sample to an approximate length whilst ensuring that the cut is reasonably accurate, the sample is placed in a jig specifically designed to hold the sample in place for both the trimming and facing. This consists of a split steel tube within an adjustable steel ring that can be fixed to a plate and held in place on the saw bench. After coring, the sample is placed in the steel tube and held in place by tightening the steel ring. Over-sized samples may be secured in the steel ring directly (Figure 3) and require an extra iteration of steps 1, 3, and 4 to bring the sample down to a suitable size for the split steel tube. The ring is then fixed to the base plate with two rods, and the plate secured in place on the bench of a clipper saw.

Following trimming, the sample remains in the jig and is transferred to a specially designed grinder to machine the ends flat and parallel (Figure 4). This new grinder comprises a leadscrew-driven table running, via precision linear bearings, on a pair of parallel steel rods. The inherent accuracy of the design is achieved by clamping the two endplates of the grinder together during fabrication, and machining as a single piece on a mill, the same method being used for the sample table bearing support bars. This ensures the parts are exact duplicates, with all similar edges parallel, and the table base exactly at a right angle to the grinding wheel face. Parallelism of sample ends now simply depends on rotating the sample around the vertical axis by exactly 180 degrees. Using a 230mm diameter diamond cup grinding wheel, each sample end is trued and flattened by multiple passes across the wheel face with small incremental movements into the wheel. The tolerance on samples prepared in this way is approximately 0.06-0.1 mm across the 196 mm pre-turned diameter (ISRM standards require this to be within 0.196 mm).

Once faced at both ends, the diameter is reduced to 193.75 mm to fit the GREAT cell. This allows for a 0.125 mm radial tolerance between the platen and retaining ring, necessary to prevent the sheath and PEEs from extruding at pressures up to 100 MPa. To achieve this precision in complex geological materials, we developed a sample preparation tool known as the Rock Turning Rig (RTR) (Figure 5). The RTR design is based on a vertically orientated rock-cutting lathe, with the rock held in place by a combination of gravity and a light axial load applied by a small adjustable top plate. The advantages of this approach are that it is fast and simple to set up, uses the sample weight as an advantage, keeps debris off a precision lathe bed, and can be confined for operator safety.

In preparation for turning in the RTR, the sample centre must be found and a small locating hole drilled. This ensures the sample rotates around the central axis as the locating hole fits onto a locating pin on the RTR. Due to the small amounts of material being removed in this turning operation (from 196 to 193.75 mm), it is critical that this locating hole is central to ensure a cylinder can be turned.

The RTR is essentially a 3-axis machine where one of the axes is a fixed rotational axis and the other two axes are under Computer Numerical Control (CNC). The fixed rotational axis consists of a 1500 RPM single phase TEC 0.75HP electric motor housed beneath the main structure that is fixed to the table above. The motor is geared down with a 30:1 worm gear box to 50 RPM then reduced again to its final speed of 30 RPM by means of a pulley on a second shaft. This second shaft has a shoulder, which sits in a bearing housing block on a large aluminium base plate (Figure 5). The block contains a thrust bearing for axial load and a tapered roller bearing for radial load. The shaft also has a seal to prevent lubricant or dust entering the bearings. The bottom platen is attached to this second shaft and has a small 3.2mm diameter locating pin in the centre. Two threaded M16 rods either side of the bottom platen locate a top plate that houses a small top platen held vertically by an identical bearing housing block. The sample is located on the bottom platen and the top plate tightened down to apply a small axial force and keep the sample in place. A digital level is used to ensure the load is applied vertically between the two threaded rods.

The CNC side of the RTR is comprised of three C-Beam Linear actuators housed in aluminium v-slot extrusion to provide extra rigidity and to prevent tool kick back (Figure 5 No.13 and No.14) (the second vertical actuator and the frame are not included in Figure 5 for clarity). For the Z-axis control of the cutting tool, two 500 mm in height actuators move a third actuator that holds the interchangeable cutting tool in a perpendicular orientation, up and down the sample. This third actuator (250mm long) controls the position of the cutting tool in the X-axis by moving the tool closer or further away from the sample.

Both sides of the machine come together in an aluminium v-slot frame that sits on the 10mm thick aluminium base plate. This also provides the structure from which the linear actuators are fixed and kept perpendicular to the platen. The movement of the cutting tool is computer controlled and programmable. The CNC system controls the X and Z axes with two stepper motors turning the lead screw on each of the axes. The code is sent from a Raspberry Pi with a 7” touch screen running Universal G code Sender to an Arduino controller, which, in turn, sends the power to the two stepper motors. The stepper motors will then run in either direction and at a defined speed for fine control of the cutter. When desired, a command sequence is entered as a macro so the machine can do multiple passes without further input from the operator. The operational RTR is shown in Figure 6.

The RTR is a versatile machine because different cutting tools can be placed on the central linear actuator. For the rock turning phase of sample preparation, a Dewalt DWE4206 angle grinder with 4 ½” diamond blade is used. This rotates in the same direction as the rotation of the sample so the direction of movement of the sample and blade are opposing at the cutting surface (Figure 6).

The high rpm of the angle grinder ensures a clean cut. The blade is brought forward to the edge of the sample carefully using the manual step control on the computer program. Once in place, a series of passes are programmed in so the blade cuts from top to bottom at a rate of 13 mm/min to ensure the entire circumference is cut before the blade advances down the sample. Just before the sample is brought to its final diameter, the machine is switched off and a dimension is taken of the outside diameter to confirm the exact measurement the blade has still to advance. The cutter is then stepped forward in small increments of around 50-100 microns and the final passes completed until the desired diameter is achieved. The manual control over the location of the blade with respect to the sample enables the sample to be turned to very fine tolerances e.g. +/- 0.01 mm.

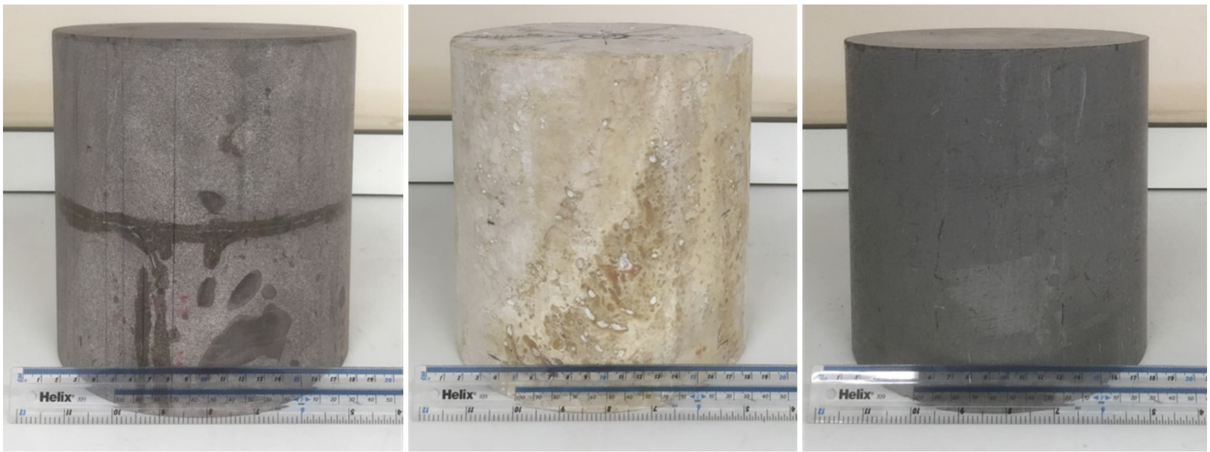

Figure ***7*** shows three completed samples of different structures and strengths prepared to the requirements of the GREAT cell. The lengths vary slightly due to the length tolerance of the GREAT cell and the desire, from an experimental point of view, to maximise sample size where possible.

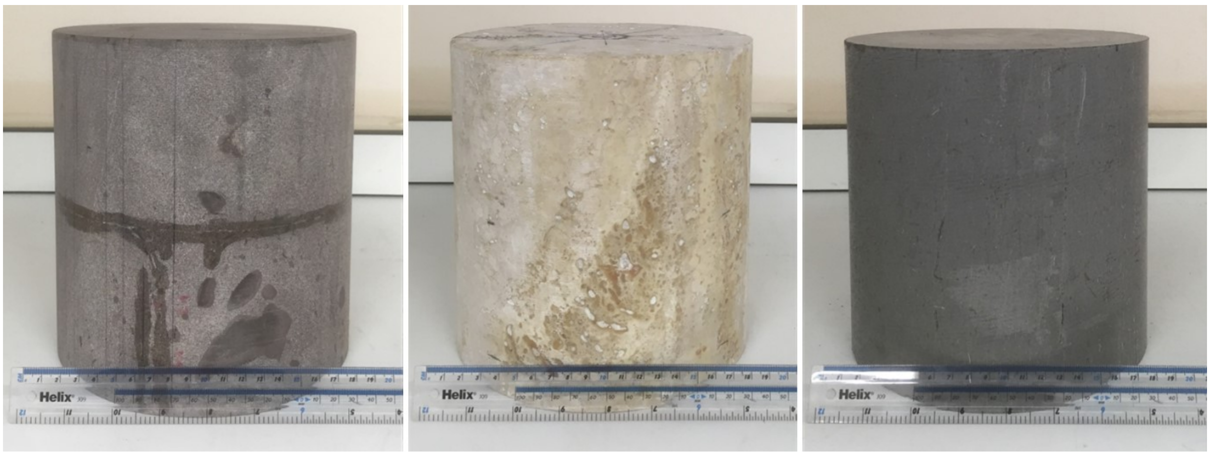

Figure ***7***a is a greywacke with variable material stiffness caused by the inclusion of clasts of different rock type,

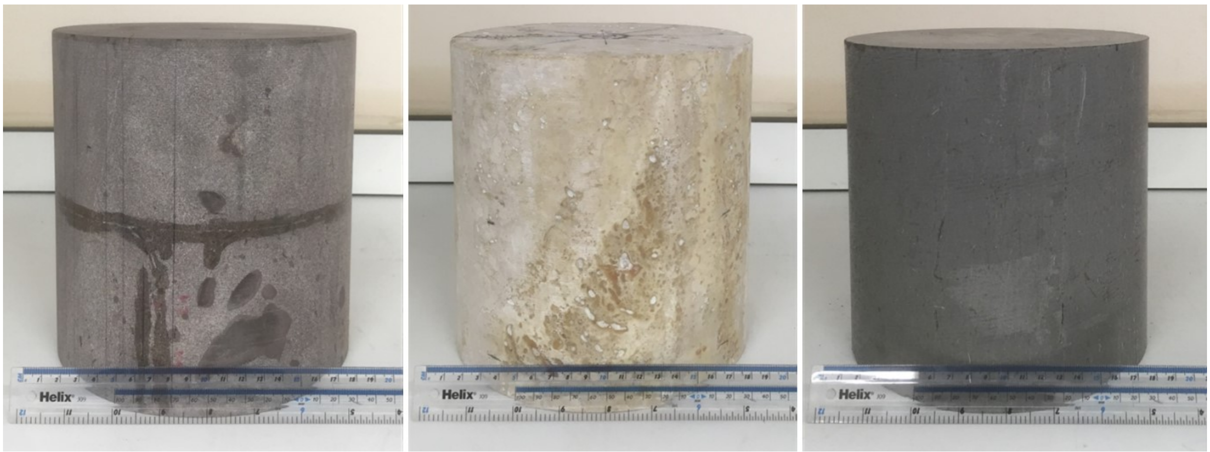

Figure ***7***c shows an extremely heterogeneous, hard carbonate with large pore spaces and

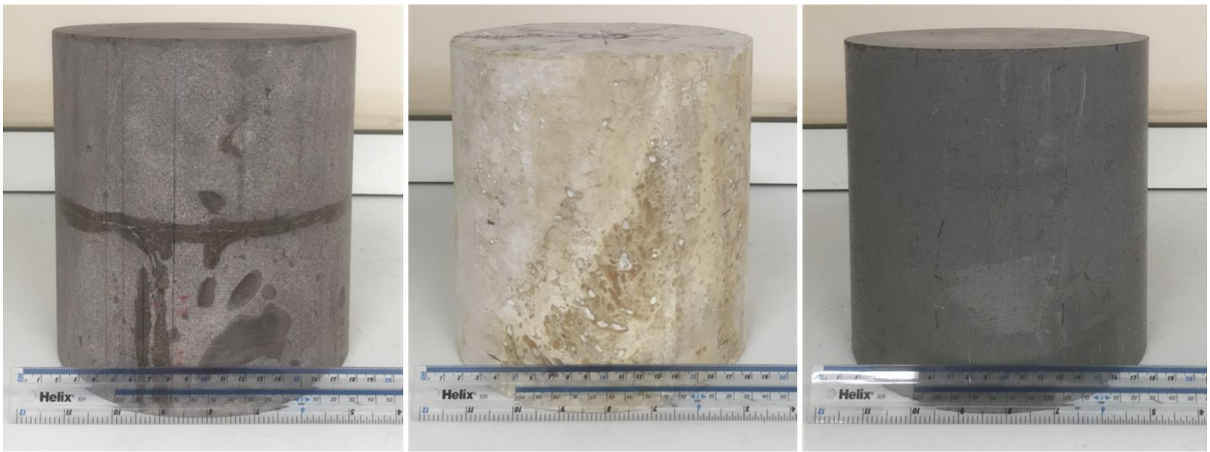

Figure ***7***b shows an Opalinus clay sample with pre-existing weaknesses. These samples demonstrate the capability of the RTR to prepare samples with varied mechanical and structural properties.

4 Conclusions

Methods for sample construction of synthetic samples and collection techniques for large (*c.* 200 mm diameter) reservoir-analogue rock samples are described. Synthetic samples are generated in the laboratory using water-clear polyester resin in specially designed moulds of High Density Polyethylene (HDPE), and reservoir-analogue rocks are cored *in situ* or from excavated blocks. These unfinished cores require further preparation for use within experimental equipment designed to simulate subsurface conditions on large samples.

Synthetic samples are machined using a workshop lathe to adhere to ISRM standards of end parallelism and squareness. However, rock samples require the development of new apparatuses to machine the natural material to very tight tolerances. These include a large grinding wheel for facing the ends of the samples, and a new Arduino controlled Rock Turning Rig (RTR) – a vertically orientated rock-cutting lathe – to machine the cylindrical samples to the exact diameter required for the experimental equipment. A clear step-by-step approach for sample preparation is presented and demonstrated for multiple rock types with different structural and mechanical properties.

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6 Competing interests statement

The authors have no competing interests to declare.

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Figure 1: Degassing set-up for the resin pours. The vacuum pump is on the left, the degassing chamber with pressure gauge in the centre, and the second mould on the right

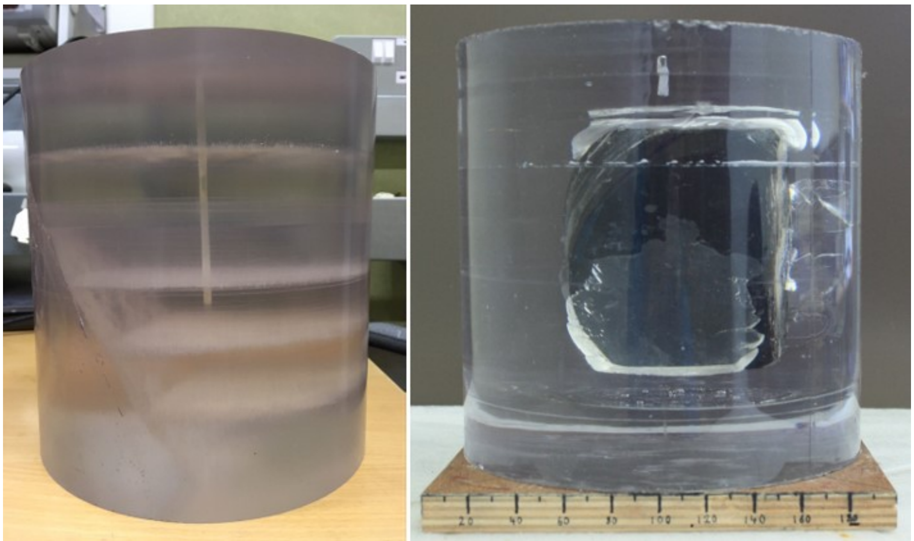

Figure 2: Polyester samples showing included heterogeneity (left) and encasing irregular shaped samples (right)

Figure 3: An over-sized claystone sample (208 mm diameter) secured in the steel ring ready for trimming to length with the clipper saw.

Figure 4: Facing samples using mounted grinder. The sample is clamped to the carriage on the right and moves past the 9” diamond grinding wheel on the left

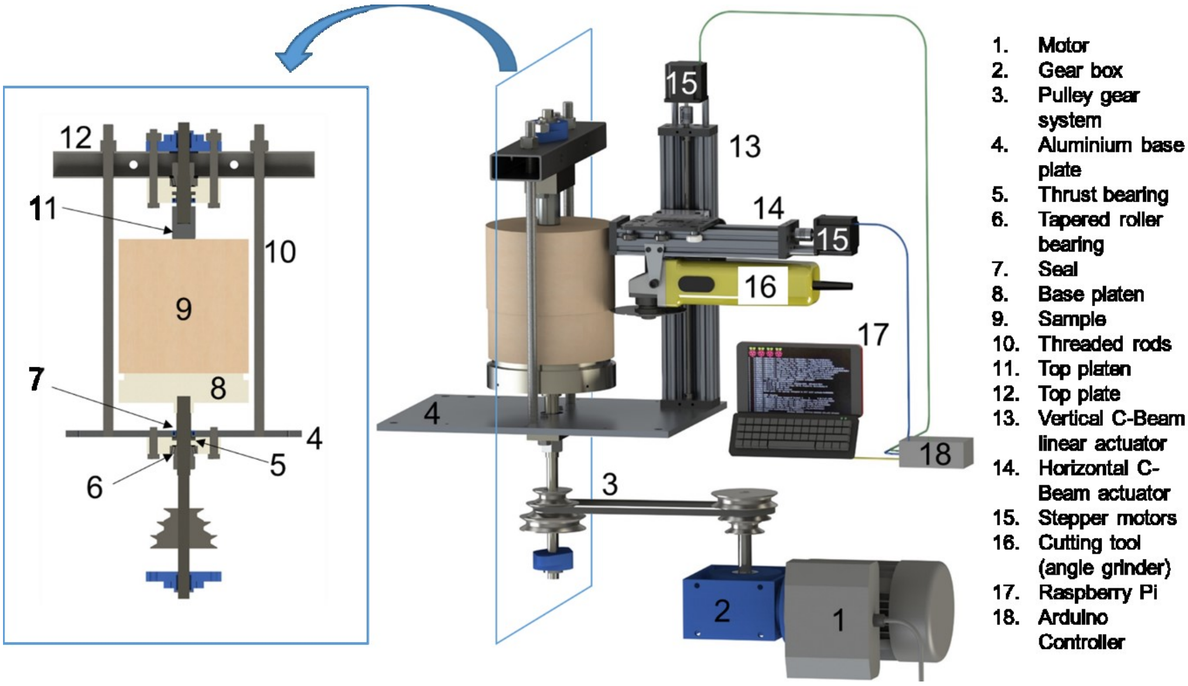

Figure 5: Annotated rendered diagram of the RTR design showing the key components. The inset is a cross-section through the platen-sample stack showing the bearings, platens, and spindle configuration. The aluminium v-slot frame, second vertical C-beam linear actuator, and the supporting stand are removed from this image to show the internal components more clearly.

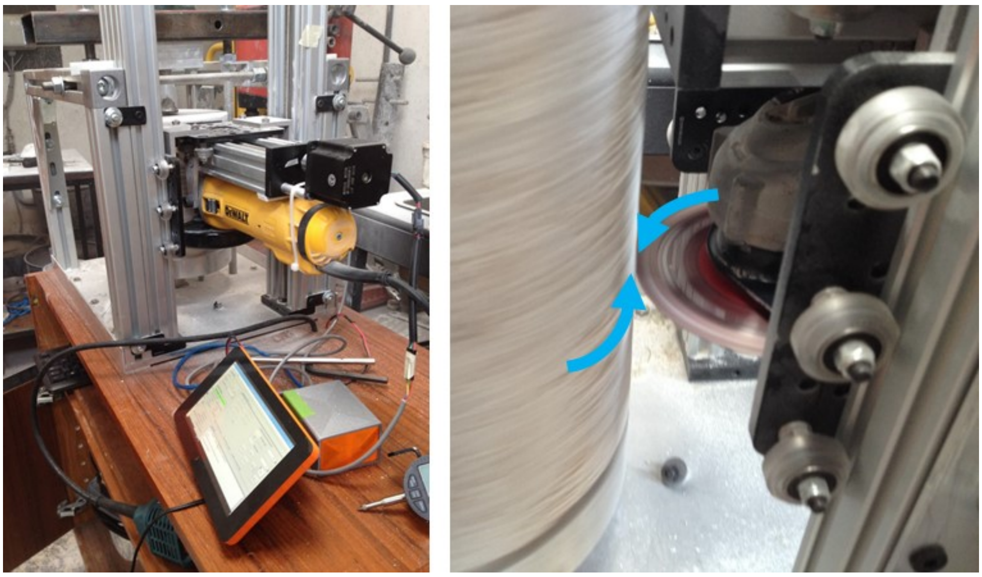

Figure 6: Image of the RTR set-up and a close up of the cutting tool reducing the sample diameter. The cutting blade rotates in the same direction (anti-clockwise, arrows) ensuring that the cutting blade and sample are spinning in opposite directions at the cutting surface.

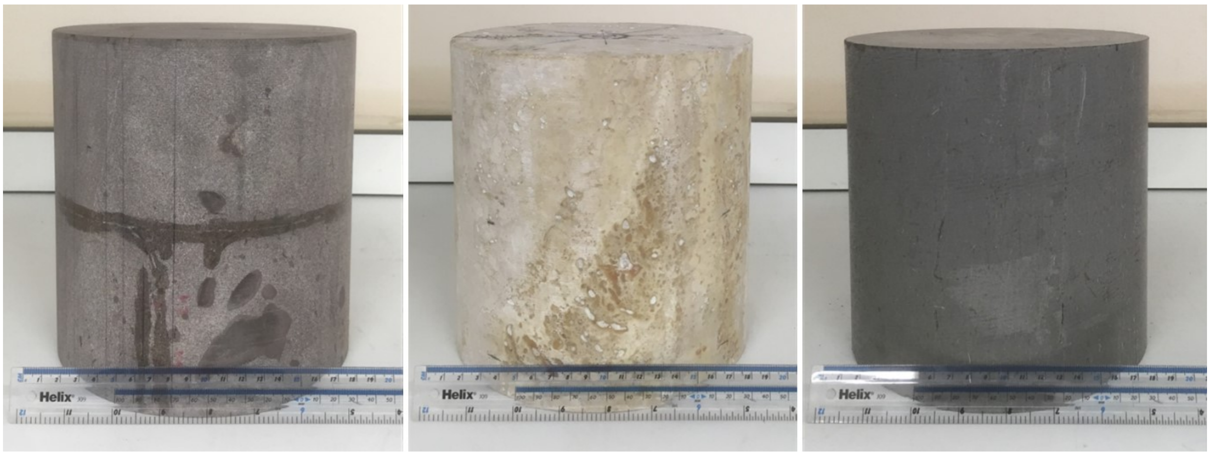

Figure 7: Examples of samples prepared in the methods described in this paper, **a**) greywacke, **b**) heterogeneous carbonate, and **c**) Opalinus Clay. Each present different challenges based on different material strengths and compositions (e.g. clasts in the greywacke), large voids (carbonate), and existing weaknesses (Opalinus Clay).