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1 Sampling and preparation of c.200 mm 2 diameter cylindrical rock samples for 3 geomechanical experiments

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15 machining

16

17 Highlights

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

22 Abstract

23 Experimental investigation of rock mechanical properties of real and artificial samples often
24 requires much care and attention to detail during sample preparation. This especially applies to high
25 fidelity state of the art complex experimental apparatuses where sample tolerance is low due to the
26 complexity of the measuring and stress control devices as well as the nature of the experiments to
27 be conducted. Although sometimes mundane, the sample preparation methodology is as equally
28 important as the experimental apparatus itself, and can require several new technological

29 developments. The methodology and technical developments required to prepare realistic
30 heterogeneous, fractured and natural reservoir analogue rock samples for coupled thermo-hydro-
31 mechanical-chemical process experimental investigation is described here. We present the sample
32 recovery and preparation procedures for large (c.200 mm diameter), cylindrical samples of 200 mm
33 +/- 5 mm length, with variable composition and mechanical properties e.g. rock strength, existing
34 fractures/fracture networks, macro-porosity, or lithic fragments. Although the technology
35 demonstrated is for a specific application, the procedures developed, equipment and methodology
36 are applicable to multiple sizes of sample requirements.

37 1 Introduction

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

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

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

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

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

75 The majority of true-triaxial testing apparatuses require cubic or prismatic samples due to their
76 choice of loading by orthogonally orientated pistons (or the equivalent, flat-jacks) in the three
77 principal directions i.e. σ_1 , σ_2 , and σ_3 . Sample preparation for these apparatuses involves excavation
78 of blocks of rock, or generation of synthetic rock analogues in specific moulds, that can then be
79 trimmed with saws and finished on grinding wheels to create perfect cubes or rectangular prisms.
80 Opposing faces must be as close to parallel as possible and orthogonal with respect to other planes
81 in the cuboid to ensure the loads are applied in a true triaxial manner. Consequently, many true
82 triaxial testing apparatuses use samples less than 100 mm x 100 mm x 100 mm in size, which makes
83 the sample preparation process more manageable with standard rock preparation techniques e.g.
84 ^{7,13-20}.

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

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

96 +/- 5 mm length) to representative temperatures, fluid pressures, and stresses in subsurface geo-
97 energy applications. It is categorised as a Type-II/flexible medium type true triaxial cell and applies
98 loads to the sample through a combination of steel platens (axial load), and axially aligned fluid-filled
99 bladders known as Pressure Exerting Elements (PEEs). The PEEs apply their individual pressures to
100 segments of the cylindrical surface of the specimen, and they are located in an annulus between the
101 outer confining cell steel ring and the rock sample ⁸. The PEEs are longer than the sample length to
102 ensure the pressure is applied to the whole length of the sample. Combined with a 2 mm Viton
103 sheath between the sample and the PEEs, this ensures a hydraulic seal is created with the top and
104 bottom platens.

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

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

119 2 Sample Construction or Collection

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

123 2.1 Synthetic sample construction

124 Experimental investigations of hydraulic fracture propagation and fracture flow in low
125 permeability rocks, e.g. ^{8,23,24}, can benefit from the repeatability and controllability of constructing
126 synthetic samples in the laboratory.

127 The synthetic samples constructed for the GREAT cell experiments ⁸ are made from a water-
128 clear polyester casting resin cured with an MEKP catalyst at 1% concentration. Each sample is built

129 up in a series of individual pours that are allowed to cure individually to prevent the sample from
130 reaching too high a temperature during the curing process. Once the catalyst is mixed into the resin
131 as evenly as possible, this is poured into a specifically designed mould made of High Density
132 Polyethylene (HDPE) that has non-stick properties for ease of removal of the sample once it is cured.
133 The mould is then placed in a vacuum degassing chamber and a vacuum applied to remove any air
134 bubbles in the mixture. The vacuum pump can evacuate the chamber to conditions < 1 mbar (

135



136

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

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

146 2.2 Analogue sample collection

147 Rock samples from the field are extracted in one of two methods; coring *in situ* with a
148 portable coring device, or excavating a large block or rock mass, followed by later coring at the
149 facilities at the University of Edinburgh. In each case, coring is performed with a 200 mm outer
150 diameter core barrel used in conjunction with a Hilti 220 portable drill. Coring is performed wet, with
151 water supplied to the core barrel from within the drill. The pressure of the water is kept minimal: a

152 head of approximately 1 m is sufficient to maintain lubrication of the core barrel and to remove fine
153 material and cuttings. The drill is fixed to a stand that allows us to control the cut angle depending
154 on the desired orientation of the core with respect to bedding planes and/or fracture geometry.

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

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

167 3 Sample Preparation

168 In apparatuses requiring an exact core diameter, such as the GREAT cell, the samples need to be
169 trimmed to that diameter before they can be used. The cylindrical surfaces need to be within a
170 tolerance of 0.3mm²⁵ and the top and bottom surfaces of the sample must be parallel to within
171 0.001 x diameter, with a squareness of ends to within 0.001 radians²⁶.

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

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

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

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

193 Following trimming, the sample remains in the jig and is transferred to a specially designed
194 grinder to machine the ends flat and parallel (Figure 4). This new grinder comprises a leadscrew-
195 driven table running, via precision linear bearings, on a pair of parallel steel rods. The inherent
196 accuracy of the design is achieved by clamping the two endplates of the grinder together during
197 fabrication, and machining as a single piece on a mill, the same method being used for the sample
198 table bearing support bars. This ensures the parts are exact duplicates, with all similar edges parallel,
199 and the table base exactly at a right angle to the grinding wheel face. Parallelism of sample ends
200 now simply depends on rotating the sample around the vertical axis by exactly 180 degrees. Using a
201 230mm diameter diamond cup grinding wheel, each sample end is trued and flattened by multiple
202 passes across the wheel face with small incremental movements into the wheel. The tolerance on
203 samples prepared in this way is approximately 0.06-0.1 mm across the 196 mm pre-turned diameter
204 (ISRM standards require this to be within 0.196 mm).

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

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

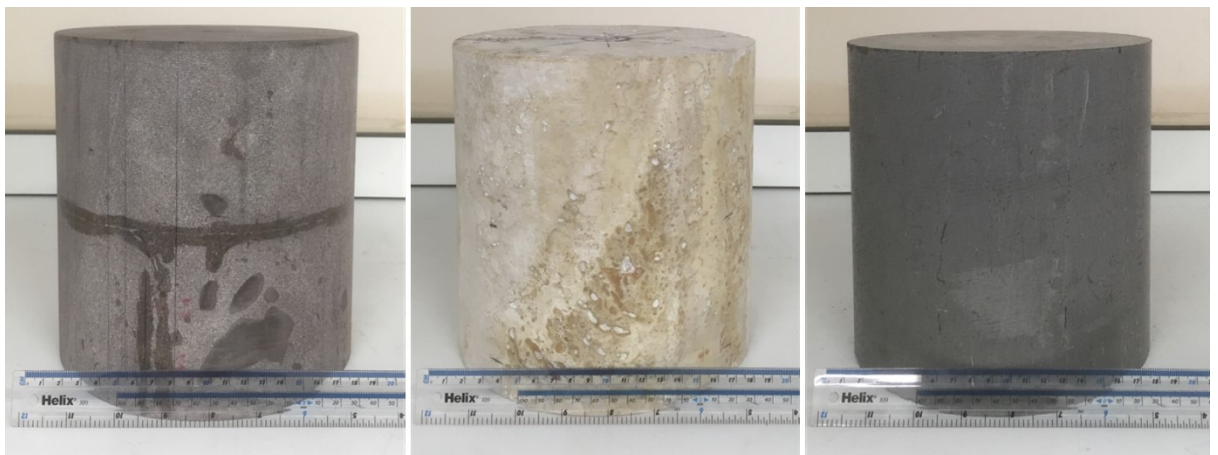
218 The RTR is essentially a 3-axis machine where one of the axes is a fixed rotational axis and
219 the other two axes are under Computer Numerical Control (CNC). The fixed rotational axis consists
220 of a 1500 RPM single phase TEC 0.75HP electric motor housed beneath the main structure that is
221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

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

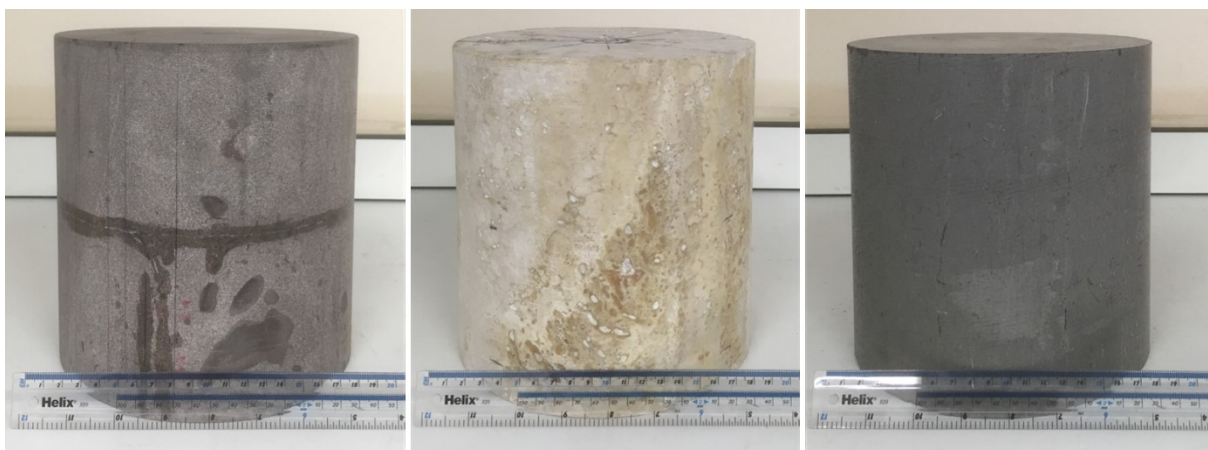
238 Both sides of the machine come together in an aluminium v-slot frame that sits on the
239 10mm thick aluminium base plate. This also provides the structure from which the linear actuators
240 are fixed and kept perpendicular to the platen. The movement of the cutting tool is computer
241 controlled and programmable. The CNC system controls the X and Z axes with two stepper motors
242 turning the lead screw on each of the axes. The code is sent from a Raspberry Pi with a 7" touch
243 screen running Universal G code Sender to an Arduino controller, which, in turn, sends the power to
244 the two stepper motors. The stepper motors will then run in either direction and at a defined speed
245 for fine control of the cutter. When desired, a command sequence is entered as a macro so the
246 machine can do multiple passes without further input from the operator. The operational RTR is
247 shown in Figure 6.

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

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



261
262 Figure 7 shows three completed samples of different structures and strengths prepared to the
263 requirements of the GREAT cell. The lengths vary slightly due to the length tolerance of the GREAT
264 cell and the desire, from an experimental point of view, to maximise sample size where possible.

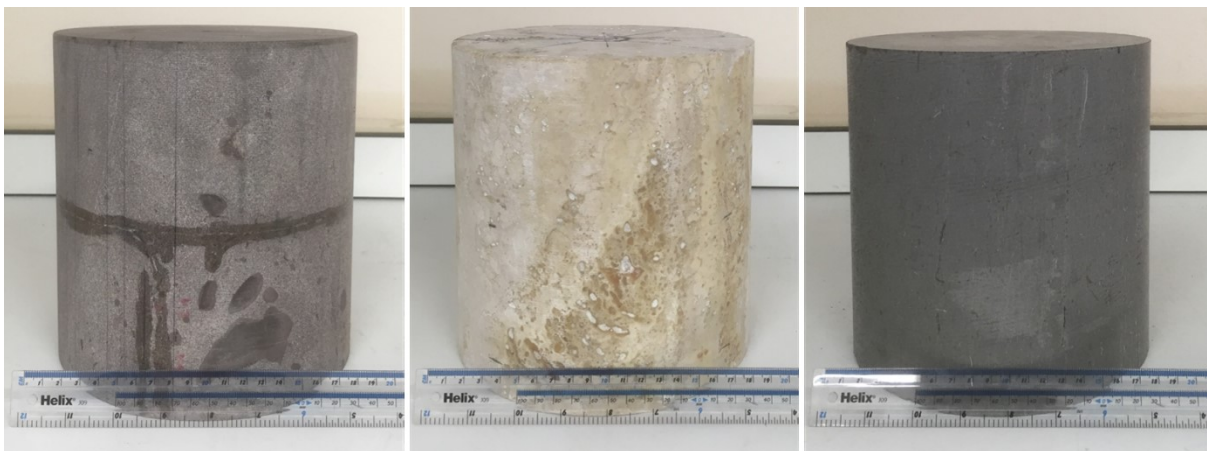


265
266 Figure 7a is a greywacke with variable material stiffness caused by the inclusion of clasts of different
267 rock type,



268

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



270

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

273 4 Conclusions

274 Methods for sample construction of synthetic samples and collection techniques for large (c.
 275 200 mm diameter) reservoir-analogue rock samples are described. Synthetic samples are generated
 276 in the laboratory using water-clear polyester resin in specially designed moulds of High Density
 277 Polyethylene (HDPE), and reservoir-analogue rocks are cored *in situ* or from excavated blocks. These
 278 unfinished cores require further preparation for use within experimental equipment designed to
 279 simulate subsurface conditions on large samples.

280 Synthetic samples are machined using a workshop lathe to adhere to ISRM standards of end
 281 parallelism and squareness. However, rock samples require the development of new apparatuses to
 282 machine the natural material to very tight tolerances. These include a large grinding wheel for facing
 283 the ends of the samples, and a new Arduino controlled Rock Turning Rig (RTR) – a vertically
 284 orientated rock-cutting lathe – to machine the cylindrical samples to the exact diameter required for

285 the experimental equipment. A clear step-by-step approach for sample preparation is presented and
286 demonstrated for multiple rock types with different structural and mechanical properties.

287

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302 6 Competing interests statement

303 The authors have no competing interests to declare.

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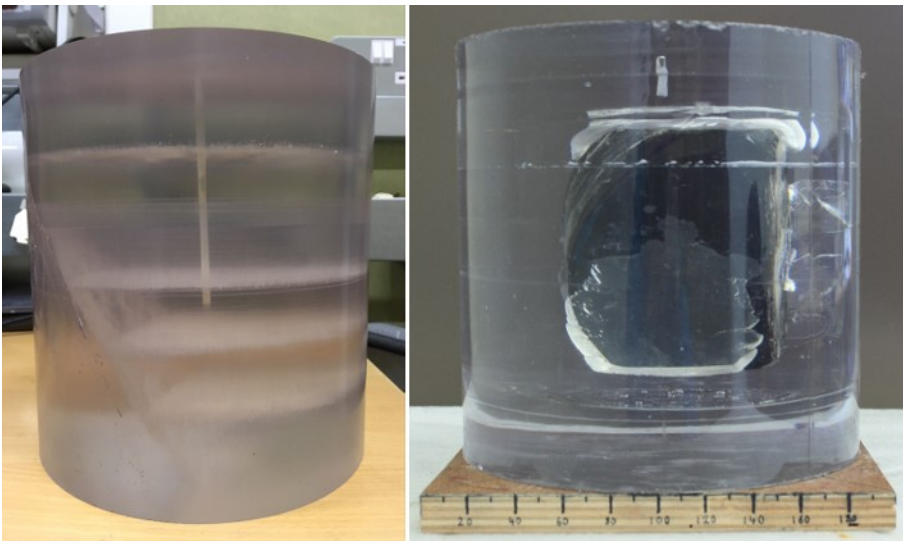
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379 **Figure 1:** Degassing set-up for the resin pours. The vacuum pump is on the left, the degassing
380 chamber with pressure gauge in the centre, and the second mould on the right

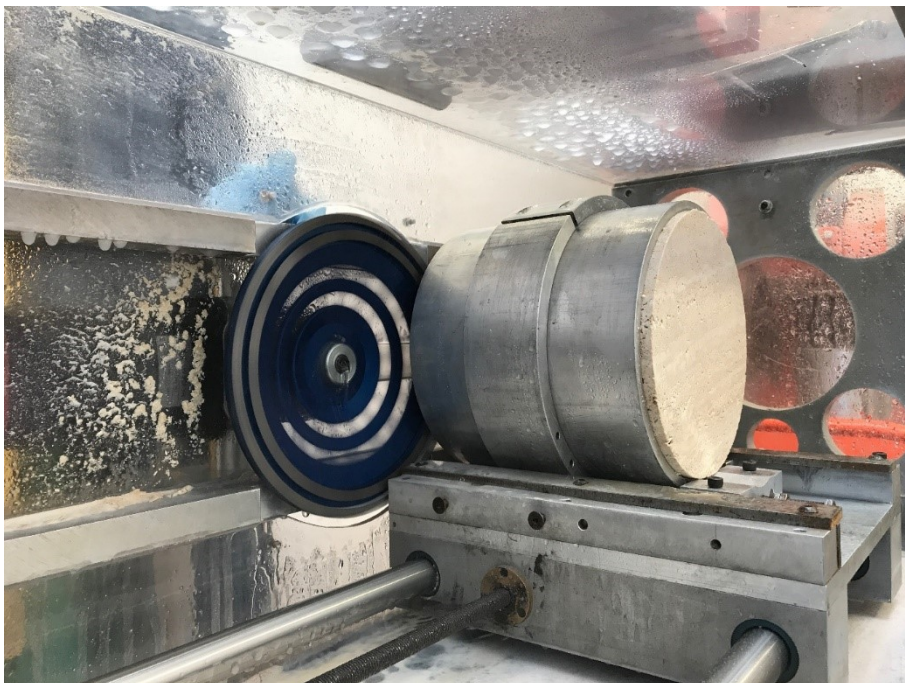


381
382 **Figure 2:** Polyester samples showing included heterogeneity (left) and encasing irregular shaped
383 samples (right)



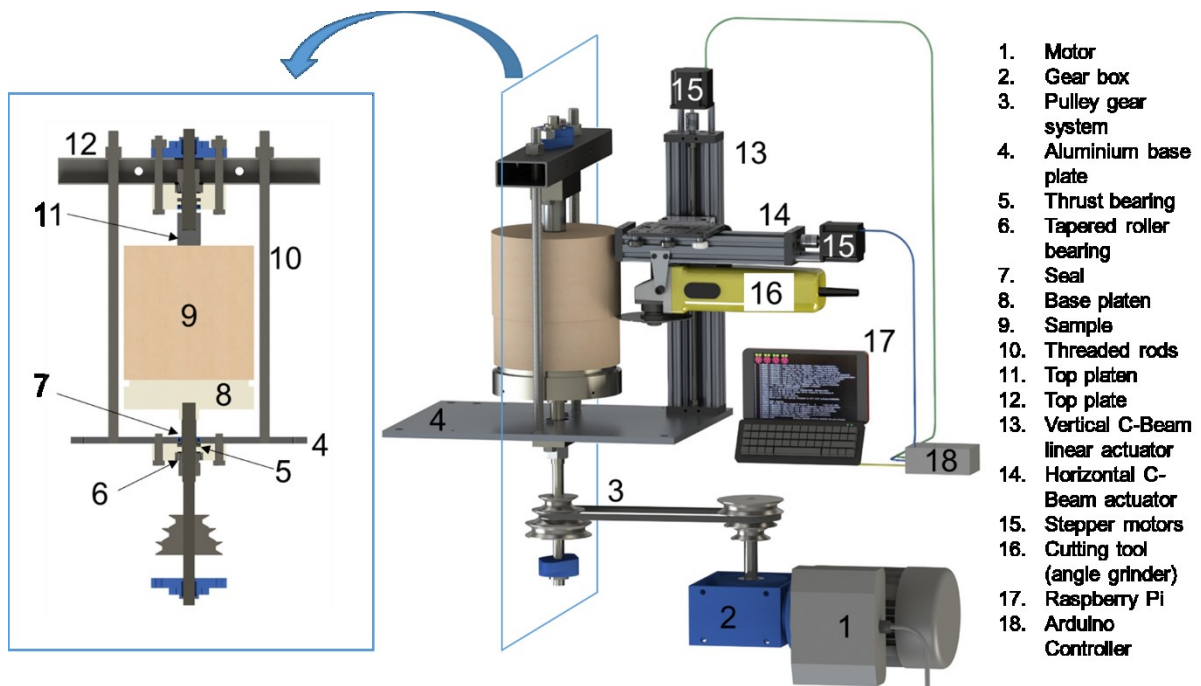
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385 **Figure 3:** An over-sized claystone sample (208 mm diameter) secured in the steel ring ready for
386 trimming to length with the clipper saw.



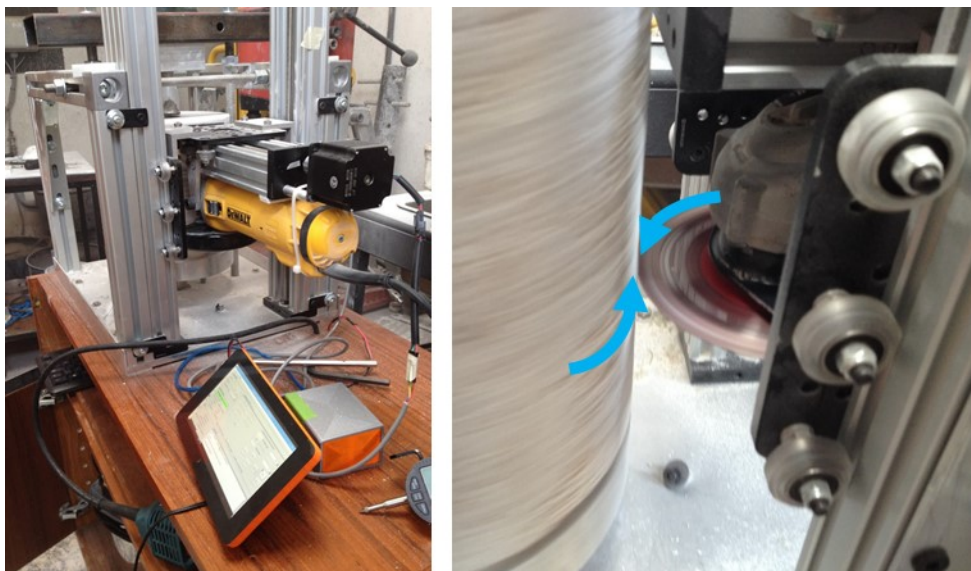
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388 **Figure 4:** Facing samples using mounted grinder. The sample is clamped to the carriage on the right
389 and moves past the 9" diamond grinding wheel on the left



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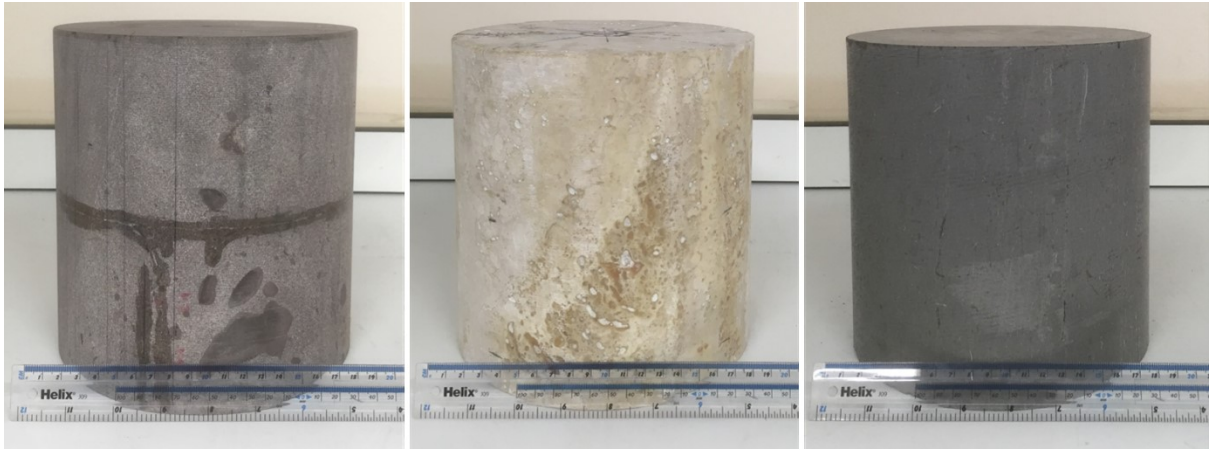
391 **Figure 5:** Annotated rendered diagram of the RTR design showing the key components. The inset is a
 392 cross-section through the platen-sample stack showing the bearings, platens, and spindle
 393 configuration. The aluminium v-slot frame, second vertical C-beam linear actuator, and the
 394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



395

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

399



400
401 **Figure 7:** Examples of samples prepared in the methods described in this paper, **a)** greywacke, **b)**
402 heterogeneous carbonate, and **c)** Opalinus Clay. Each present different challenges based on different
403 material strengths and compositions (e.g. clasts in the greywacke), large voids (carbonate), and
404 existing weaknesses (Opalinus Clay).

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