SEDIMENTS, SEC 2 • PHYSICAL AND BIOGEOCHEMICAL PROCESSES • RESEARCH ARTICLE

There's no such thing as 'undisturbed' soil and sediment sampling: sampler-induced deformation of salt-marsh sediments revealed by 3D X-Ray computed tomography

Simon J. Carr[1,2] • Lucy M. Diggens[2] • Kate L. Spencer[2]

[1]Science, Natural Resources and Outdoor Studies, University of Cumbria, Rydal Road, Ambleside, Cumbria LA22 9BB, United Kingdom

[2]School of Geography, Queen Mary University of London, Mile End Road, London, E1 4NS, United Kingdom

✉ Simon J. Carr

simon.carr@cumbria.ac.uk

Abstract

Purpose: Within most environmental contexts, the collection of 'undisturbed' samples is widely relied-upon in studies of soil and sediment properties and structure. However, the impact of sampler-induced disturbance is rarely acknowledged, despite the potential significance of modification to sediment structure for the robustness of data interpretation. In this study, 3D-computed X-ray microtomography (µCT) is used to evaluate and compare the disturbance imparted by four commonly-used sediment sampling methods within a coastal salt-marsh.

Materials and methods: Paired sediment core samples from a restored salt-marsh at Orplands Farm, Essex, UK were collected using four common sampling methods (push, cut, hammer and gouge methods). Sampling using two different area-ratio cores resulted in a total of 16 cores that were scanned using 3D X-Ray computed tomography, to identify and evaluate sediment structural properties of samples that can be attributed to sampling method.

Results and discussion: 3D qualitative analysis identifies a suite of sampling-disturbance structures including gross-scale changes to sediment integrity and substantial modification of pore-space, structure and distribution, independent of sediment strength and stiffness. Quantitative assessment of changes to pore-space and sediment density arising from the four sampling methods offer a means of direct comparison between the impact of depth-sampling methods. Considerable disturbance to samples result from use of push, hammer and auguring samplers, whilst least disturbance is found in samples recovered by cutting and advanced trimming approaches.

Conclusions: It is evident that with the small-bore tubes and samplers commonly used in environmental studies, all techniques result in disturbance to sediment structure to a far greater extent than previously reported, revealed by µCT. This work identifies and evaluates for the first time the full nature, extent and significance of internal sediment disturbance arising from common sampling methods.

Keywords Deformation • Disturbance • Saltmarsh • Sediments

1 Introduction

The analysis of the chemical, physical and biological attributes of surface soils and sediments is easy to achieve through either in situ examination or the collection of surface sediments. However, examination of the sub-surface environment can be more challenging and is usually achieved through the collection of core samples. Once cores are extracted and/or returned to the laboratory sediments can be subjected to a range of ex situ analytical techniques. Consequently, soil and unconsolidated sediment cores are frequently collected from a wide range of environments for multiple applications in the earth and environmental sciences. For example, sediment and soil cores may be required for the simple classification of constituent elements (e.g. particle size), for the observation and assessment of key state variables (e.g. fabric, porosity, moisture content) and to evaluate geotechnical, mechanical and engineering properties such as shear strength, compressibility and permeability (Viana da Fonseca and Pineda 2017). Vertical changes in physical, chemical, and biological sediment properties provide proxy records of environmental change (e.g. Kemp 1985; Nuttle and Hemond 1988; Jahnke and Knight 1997; Spencer et al. 2003; Allaire et al. 2009; Menzies et al. 2010; Palmer et al. 2012; Lowe and Walker 2015). Sediment cores may also be used as laboratory mesocosms to quantify environmental processes such as biogeochemical cycling or hydrological behaviour (Allaire et al. 2009; Rezanezhad et al. 2016; Corzo et al. 2018). Such structures, properties and processes, and how they vary spatially within the sub-surface environment are inherently 3-dimensional. Therefore, there is a requirement, and frequently an assumption, that recovered sediment core samples are 'undisturbed', i.e., that the physical characteristics of the sediment in- and ex situ are identical.

A range of stresses are exerted on soils and sediments during core sampling (core insertion and extraction, and extrusion of sediment from the core tube) transport and storage. Such stresses can result in significant alteration of the physical sediment properties – ‘disturbance’ (Hvorslev 1949; Buller and McManus 1979; Bullock et al. 1985; Clayton 1986; Gilbert 1992; Clayton et al. 1995; Glew et al. 2002; Glew and Smol 2016; Viana da Fonseca and Pineda 2017). The style and magnitude of these stresses depends upon the coring technique and equipment deployed (Hvorslev 1949; Baligh 1985; Gilbert 1992; Lotter et al. 1997; Clayton et al. 1995; Frew 2014; Spencer 2017; Viana da Fonseca and Pineda 2017). Some of these stresses act upon all samples to different degrees (Fig. 1), irrespective of sampling method. Normal stresses are generated as coring devices are inserted and extracted from the substrate, and also when sediment is extruded from the core tube. These will result in various strain responses within the sediment, including expansion (tensile stress), compression (compressive stress), brittle failure as samples are detached from the substrate, and dilation through pressure release. Whilst shear stresses typically occur through frictional drag and rotation, for example at the contact between sediment and core tube, they are particularly prevalent where cores are rotated as they are inserted, e.g. gouge or Russian corers, or where root material is abundant (Hvorslev 1949; Baligh 1985; Hight 1986; Gilbert 1992; Clayton et al. 1995; Ladd and DeGroot 2004). Other stresses reflect particular methods of sampling, for example through the use of extendable coring rods which, by their nature, are not perfectly rigid, resulting in potential deformation during both insertion and retraction (Glew and Smol 2016). Subsequent vibration or knocking during transport may result in settling, or if pore-water pressures are sufficient, even liquefaction of the sample. On return to the laboratory, further disturbance may occur if samples are stored below 4°C, particularly if they are stored at a different orientation to that of their original state (Hvorslev 1949; Environment Canada 1994; Clayton et al. 1995) or if they are allowed to dessicate.

Whilst it is known that sampling may cause disturbance to the sediment structure, the characteristic features of sampling disturbance are often poorly recognized and systematic studies of disturbance relating to sediment sampling methods have received little attention since the mid-1990s (e.g. Hvorslev 1949; Blomqvist 1991; Wright 1991; 1993; Gilbert 1992; Clayton et al. 1995). Many studies simply note that a sample is 'undisturbed', 'intact', or that subjective actions (such as carefully or slowly collecting the sample) have been taken to minimise disturbance (e.g. Lane and Taffs 2002). Disturbance caused by friction, compressive and tensile stress has been qualitatively observed as core shortening or ‘smearing’ along the core edge (Blomqvist 1991; Lane and Taffs 2002) or through the visual assessment of cut sample sections (e.g. Hvorslev 1949). Quantitative assessments have also been made such as estimations of percentage compaction through gross-scale volumetric changes (Spencer et al., 2003) or geotechnical modelling (Brain et al. 2017). However, these approaches only provide a snapshot of disturbance in a single plane (typically horizontal or vertical), and themselves are an integration of the impacts of disturbance along the entire core. This makes it difficult to recognise the impact of the complex three-dimensional stress patterns and structures imparted during sampling (Fig. 1). Equally, the process of creating of a face or thin section for such evaluation is also likely to induce disturbance, and often precludes further analysis of the samples collected (Bendle et al. 2015).

Failure to acknowledge, observe or quantify disturbance in soil and sediment cores may lead to the misinterpretation of structural features or the over/under estimation of environmental processes. For example, core shortening or lengthening can lead to the over- or underestimation of vertical sediment accretion rates misrepresenting rates of environmental change (Turner et al. 2006) and friction at sediment edges can lead to the loss of fine laminations and cross contamination between sediments of varying chemical properties. In studies of sediment structure, it may be impossible to distinguish between primary deformation (e.g. in sediments which have been emplaced by deformation such as subglacial traction tills, or have been deformed through seismic or tsunami events) and sampling artefacts (Carr 2004; Araújo-Gomes and Ramos-Pereira 2014). Finally, some geotechnical and engineering properties, e.g. shear strength and hydraulic conductivity, are a function of physical sediment characteristics such as porosity, and quantification of these properties ex situ may not be representative of the in situ environment. Given the importance attached to many studies of microstructure, porosity and hydraulic or biogeochemical profiles, and the development of ever more sensitive laboratory tests for soils and sediment (Viana da Fonseca and Pineda 2017), this issue clearly warrants renewed consideration.

3D-computed X-ray microtomography (µCT) is a non-destructive imaging technique that allows samples to be reconstructed, visualised and analysed in three-dimensions, with minimum preparation required prior to scanning, at spatial resolutions down to <10 microns (Ketcham and Carlson 2001; Taina et al. 2008; Cnudde and Boone 2013). Therefore, this method is ideally-suited for evaluating disturbance, but has previously only been used to examine disturbance in the production of sediment thin sections (Bendle et al. 2015). This study applies innovative µCT methods to evaluate and quantify the disturbance associated with four different but commonly-used sediment depth-sampling methods on the structure and properties of fine-grained unconsolidated sediment cores.

2 Methodology

2.1 Field site and sampling

Samples were collected from Orplands Farm Managed Realignment (MR) Site (Fig. 2), a restored salt-marsh located within the Blackwater Estuary in Essex (Emmerson et al. 1997; Spencer et al. 2008; Tempest et al. 2015; Spencer et al. 2017). As a result of antecedent land use and the restoration process, the site has developed a sediment stratigraphy whereby a low-density, readily deformable, saturated upper facies of uniform fine-grained estuarine mud (sandy-silt), typically 60-80 mm in thickness, overlies a lower facies of stiffer, drier and slightly coarser muds and sands reflecting pre-restoration agricultural land-use (Spencer et al. 2008; 2017). Consequently, each sediment facies has contrasting geotechnical and rheological properties and the boundary between facies, which is consistently sharp and sub-horizontal (as observed in exposures on the edges of saltmarsh creeks), offers an ideal opportunity to assess sampling disturbance.

Sediment sampling methods chosen for investigation (Table 1) reflect examples of the broad range of push, hammer, rotation and cutting approaches used within environmental contexts. Hvorslev (1949) suggests that the area ratio of a sampler, defined as the area of the annulus of the sampling tube divided by the area of the sediment core, offers a useful evaluation of the potential for sampling disturbance. Area ratios <0.1 (or <10 %) are considered optimal, and that samplers with higher ratios are more likely to induce disturbance (Clayton et al. 1995). Two sets of sampling tubes with area ratios of 0.291 and 0.099 were selected for use in core push, hammering and cutting methods, with the smaller-bore tubes selected as equivalent in size and area ratio to the gouge auger employed. These smaller-bore tubes have an increased likelihood of generating structures characteristic of disturbance during sampling (Hvorslev 1949; Clayton et al. 1995), and are also typical of many applications in soil, peat and sediment sampling. Whilst area ratio is a key design consideration in geotechnical sampling contexts, they are rarely noted in environmental studies.

All samples were inserted to a depth of 150 mm below salt-marsh surface in order to ensure that both sediment facies were sampled and the contact between facies was captured. The tubs used in this study were 100 mm deep, and thus only sampled to this depth. Samples were collected within a 1 m^2 area to restrict the impact of spatial variability of sediment, and for each method adjacent paired samples were recovered from within 100 mm of each other. The depth of sampling was determined by markers on the sampling chambers to provide a depth reference to the ground surface, enabling assessment of the relative compression or expansion of tube samples caused by each technique (e.g. Doran and Mielke 1984; Burt 2009). Sampling chambers and tubes were left in place for approximately 5 minutes to allow for *in situ* cohesive forces to act on the inside of the tube; this reduces the risk of sample loss during extraction (Hvorslev 1949; Clayton et al. 1995; Ladd and DeGroot 2004). Sample chambers and tubes were carefully retrieved using a shovel or trowel to lever the sample up, following the guidelines of Hvorslev (1949). All samples were immediately (<1 minute) sealed with parafilm and plastic ziploc bags, which were secured in place with tape to prevent loss of moisture and air ingress (Hvorslev 1949; Environment Canada 1994). Samples were tightly packed and supported with bubble wrap and transported back to the laboratory in the same orientation as when sampled avoiding physical movement and/or shaking where possible. Following the guidance of Clayton et al. (1995) and Environment Canada (1994), samples were stored at 4°C before scanning.

2.2 Evaluation and analysis: X-ray computed microtomography

Samples were scanned within two days of collection using a Nikon XT H 225 X-ray tomograph (Nikon, Tokyo, Japan) using Nikon InspectX software (Quiggin 2011), using identical scan parameters to permit comparison between samples, and reconstructed using CTPro (Ray 2011). Resulting volumetric models have a voxel size of 76.0 μm, with the exception of the larger Tub samples which have a lower resolution of 111.4 μm.

Qualitative description of each reconstructed volume was undertaken to identify sediment structures indicative of sampling deformation using Drishti 2.6.3 open-source volume-rendering software (Limaye 2012), supplemented with information extracted using FIJI open-source image-analysis software (Schindelin et al. 2012). Binary segmentations of macro-pore space were derived by a combination of grayscale thresholding and curvature mapping within FIJI and quantitatively analysed using BoneJ (Doube et al. 2010), complementing visualisation and qualitative description of macropore space using Drishti. Finally, depth profiles of grayscale values of sediment matrix were generated at 100-slice (= 7.6 mm) intervals, with the Tub datasets normalised for the same depth increments. Given that all the sediments sampled in this study come from the same source, variations in X-ray energy attenuation (recorded as grayscale values) most likely represent differences in material bulk density, and thus infers the relative degree of compaction through changes in microporosity and bulk density arising from each sampling method (Jones and Thomasson 1976; Ketcham and Carlson 2001; Viana da Fonseca and Pineda 2017).

3 Results

3.1 Gross vertical length changes of tube samples

Figure 3 illustrates the differences in length of sediment samples recovered relative to the 150 mm sampler insertion depth for both types of tube for cut, push and hammer methods. In all small-bore (area ratio 0.291) samples, vertical compression is identified at the edges of samples, whereby frictional drag against the sampling tubes during insertion results in 6-22% shortening. Within the large-bore tubes (area ratio 0.099) the impact is smaller in most samples (typically 6-11% shortening) but with notable variations, with both cut samples demonstrating vertical extension (1-9%) at the sample edges, and one push sample experiencing 24% shortening on one edge.

Length change recorded in the centres of both small- and large-bore sampling tubes is more variable, both in the direction and magnitude of length change, and offers insight into the gross deformation of the entire sample resulting from each sampling method. Within the small-bore tubes (Fig. 3a), there is -9 to 15% shortening in samples, with length changes consistent within each sampling method applied. The Cut samples demonstrate the least net change in length, and are within the likely ±5% (7.5 mm) potential measurement error arising from the small-scale irregularity of the current saltmarsh surface, suggesting minimal gross disturbance of the centre of samples recovered by this method. By contrast, in both Push and Hammer samples, changes in length are typically >5%, but with different trends. Hammer samples demonstrate overall compression, with 4 to 12% vertical shortening, whilst Push samples demonstrate significant vertical lengthening of 7 to 9%. The compression occurring in the Hammer samples was observed during the insertion of the sample tube, and that subsequent recovery occurred during the five minute ‘rest’ period before sampler removal, and is reported in more detail below. No similar compression was observed during the Push sampling, but subsequent ‘rest’-stage vertical lengthening was also observed in these samples, which is interpreted as sediment response to vertical pressure-release at the end of sampler insertion.

In the large-bore sampling tubes (Fig. 3b), the measured range in centre length change in samples is smaller (-9 to 4%), but is also less consistent, with four samples experiencing net lengthening, and two experiencing net compression. Both cut samples display identical vertical extension of 9%, whilst push and hammer samples demonstrate smaller, but variable degrees of both shortening and lengthening, but all within the ±5% error margin noted above. Slightly different sampler shape, diameter and area ratio preclude direct comparison of the gouge with the tube samples, but observation of gouge-auguring in the field suggests a similar pattern of sample length change observed to that of the push samples, with vertical edge-shortening and central lengthening.

All sampling methods utilised in this study have generated changes in sample length through combinations of shortening through vertical compression and lengthening vertical pressure release, as previously described as a strain path by Baligh (1985), but that different methods elicit different sediment responses. These responses are further conditioned by the size of sampler adopted, as seen in the contrasts between similar sampling methods with tubes of differing area ratio. How these bulk changes in the sediment samples impact on resulting sediment structure is outlined below.

3.2 Identification and qualitative description of disturbance

Features indicative of sample deformation have been identified and described from reconstructed µCT volumes of all small-bore samples (Table 2), based on the approach of Kemp (1985) and Carr (2004).

3.2.1 Deformation structures evident at facies contact and sample edges

The stratified nature of the sediments at Orplands Farm MR site facilitates identifying sampling-induced deformation through distortion of the sharp sub-horizontal contact between the upper and lower facies. All samples display deformation due to frictional drag at the edges of sampling chambers (‘edge’ deformation), with displacements extending up- and down-sample, although dominated by the latter, suggesting that frictional drag during sampler recovery is mainly responsible for the deformation observed. This data complements the measurement of vertical sample compression presented in 3:1. The extent of edge deformation varies considerably between sampling methods, with samplers involving pushing resulting in substantial vertical drag and displacement extending in excess of 30% of the width of the samples recovered (Fig. 4a). By contrast, edge deformation is limited in samples that were cut, trimmed or hammered to a narrow zone <10 % of the sample width, although vertical displacement in this zone tends to be considerable in hammer samples.

Evidence for edge-deformation of samples is further supported by volume rendering of the edges of the sediments in contact with the sample tubes, which identifies distinctive gross-scale structures (Fig. 4b). Furrows are found on the edges of Push and Hammer samples in particular and represent the gouging or ploughing of particles undergoing frictional drag against the sampling chamber, whilst the smearing of sediment along sample edges is also apparent in the hammer samples (Fig. 4c). In both instances, the structures are consistent with disturbance during sampler insertion, as the sampling chamber is being forced through a static sediment pile.

In addition to edge deformation, almost all samples display evidence of distortion of the sediment facies contact right through the interior of samples (‘centre’ deformation), primarily as undulation and low amplitude open folding of the sub-horizontal facies contact. However, in the push samples, centre deformation is substantial, forming highly contorted diapirs (Fig. 4a) resulting from movement due to contrasting geotechnical and rheological characteristics between facies. Finally, whilst the observed contact between upper and lower sediment facies at Orplands Farm is sharp when viewed in exposures within saltmarsh gullies, within most samples there is some evidence of sediment mixing between facies, reflecting remobilising of sediment at the facies contact during sampling. This is particularly visible in push and gouge samples (Fig. 4a,b), and is often associated with the larger scale distortion of the facies contact noted above.

The value of undertaking 3D analysis of the facies boundary is demonstrated in Fig. 4d, which shows that imaging a Push sample in different 2D planes identifies widely differing degrees of deformation observed at the facies contact. Given the widespread use of thin sectioning, with samples often recovered across facies boundaries, this observation of highly variable deformation has obvious implications for the potential integrity of such samples, particularly within sediments with low or contrasting structural competence.

3.2.2 Disturbance evident in macropore space

The nature of porosity within sediments is critical in determining both their hydrological and rheological behaviour (Beven and Germann 1982; 2013; Twiss and Moores 1997; Allaire et al. 2009; Quinton et al. 2009; Knappett and Craig 2012; Rezanezhad et al. 2016). However, existing methods of describing sediment porosity are typically limited to bulk measurements or description from 2D thin sections. Unlike other methods of investigation, µCT permits direct observation and analysis of in situ pore spaces (Spencer et al. 2017). Natural, *in situ* macropores in saltmarsh sediments are typically channels resulting from micro-invertebrate burrowing, root penetration, or degassing of methane and carbon dioxide generating vesicles. Consequently, macropores in these sediments act as strain markers, with macropores having different structural forms to vesicles and channels representing disturbance to the sediment structure during the sampling, transport and storage process.

Table 2 summarises the nature of macropores within all samples. Within the low area-ratio Tub samples (Fig. 5a), macropores are sporadic, and mainly concentrated at the boundary between the two sediment facies. Visible pores within these samples are vesicles, with smooth, rounded surfaces, displaying no evidence of distortion. Within the higher area ratio Cut samples (Table 2), vesicles are significantly more common, concentrated in the less stiff upper sediment facies, but showing little evidence of distortion. Within the Push, (Fig. 5b) and Hammer (Fig. 5c) samples, pore abundance, size type and geometry are heavily modified, and whilst vesicles remain common in these samples, these are generally much larger and many have been distorted into more complex, irregular pores better described as as vughs. Additional vughs and linear fissures are associated with edge dragging both at the sample surface and at depth within push and hammer samples. The effect of percussive impact is clearly visible in the hammer samples, where curvilinear fissures define discrete planes of fracture within the sample. Finally, the Gouge samples display almost total reworking of macropore space, with an absence of compact vesicles and the development of a connected, complex network of fissures associated with craze-planes of sediment undergoing extensive brittle failure (Fig. 5d).

3.3 Quantification of sampling disturbance

One of the key advantages of µCT datasets is that their digital nature allows interrogation and quantification of identified bulk phases. Segmenting the original reconstructed volume through a combination of grayscale thresholding and curvature mapping into a binary image of macropores (white) and everything else (black) permits quantified analysis of the nature of pore-space within each sample. Beyond the bulk analysis presented in this study, Spencer et al. (2017) demonstrate that sediment porosity can be quantified as a topological network from such binary segmentations, allowing assessment of the effectiveness of pore-spaces to conduct water and solutes. In this study, differences in pore-space, either directly imaged macroporosity or inferred microporosity offer insight with regard the impact of different sediment sampling methods.

3.3.1 Macroporosity

Macroporosity (pores with diameters >80 µm; Beven and Germann 2013) is primarily defined as a bulk measure of pore-space by volume, but the µCT datasets also permit the quantification of the size, shape and volume of every identified pore and also an assessment of the density of spacing of macropores within each sample (Table 3). Bulk macro-porosity ranges from 1.0 - 5.8% by volume, and whilst some of this variability may be accounted for by the properties of the pre-restoration salt-marsh sediments at Orplands Farm (Spencer et al. 2017), there remain systematic differences in macroporosity that can be attributed to the different sampling methods employed.

The Tub samples, demonstrating the least qualitative evidence of sampling disturbance, provide a useful basis for comparison; in both samples macroporosity is low, with sparsely distributed, large macropores present. These are suggested to represent the closest approximation to the in situ, natural porosity of the sediments at Orplands Farm. Cut samples have similar bulk macroporosity to the Tub samples, but pore volumes are smaller and the density of spacing increases by an order of magnitude, suggesting there has been substantial modification to the macropore system as a result of sampling, albeit with a consistent effect in both samples. By contrast, Push, Hammer and Gouge samples all display considerable intra- and inter-sample variation in the three parameters presented in Table 2, suggesting that these methods of sampling generate dramatic, but inconsistent modification to macropore space. Within this highly variable dataset, it is however evident that all of these sampling methods result in fragmentation of pre-existing macropores into smaller, more densely-spaced pores, as well as introducing many new macropores, typically increasing macroporosity, irrespective of the sampling method used. Thus, the quantified datasets presented in Table 2 support the qualitative evidence described in Fig. 5b-d.

3.3.2 Microporosity and sediment matrix compaction

The use of identical X-Ray µCT scanning and reconstruction parameters and an assumed similarity in sediment mineralogy between samples means that differences in greyscale values in the samples likely reflect variations in bulk density of the sediments (Turburg et al. 2014; Viana da Fonseca and Pineda 2017). Partial-volume effects (Cnudde & Boone 2013), reflecting sub-voxel scale changes in compaction therefore provide an indirect measure of the relative proportions of micropore space within the matrix of the sediment phases (Ketcham and Carlson 2001; Turburg et al. 2014). Within this study, differences in matrix grayscale values and thus compaction and microporosity between samples are interpreted to primarily result from the different sampling methods employed.

Figure 6 presents aggregated matrix grayscale data from the lower and upper sediment facies recorded from each sampling method. Pre-restoration tillage and agricultural practice confer significant variation in the bulk density and inferred microporosity of the lower facies (see Spencer et al. 2017), but it is notable that variation is lowest in the Tub and Cut samples, and significantly greater in Gouge, Hammer and Push samples (Fig. 6a). The upper, post-restoration sediment facies can be assumed to be far more uniform in bulk density, but is also less stiff, and thus offers a more sensitive indicator of the impact of sampling on microporosity (Fig. 6b). Whilst the Tub samples display similar and consistent ranges of grayscale values, and thus bulk density and microporosity, all other sampling methods result in considerable inter and intra-sample variability, implying substantial disturbance to this weaker sediment during the sampling process. There are also differential responses between sediment facies when exposed to a particular sampling method. Within the Hammer samples for example, the stiffer lower facies has increased grayscale values inferring sediment compaction, whilst lower grayscales in the upper facies imply sediment dilation. Whether these represent the impact of different stages of insertion and removal of the sampler, or differential responses of rheologically-different sediments to the same stress is discussed below, but serves to demonstrate that the sediment response to sampling can vary considerably within one sample.

When mean matrix grayscale data is plotted at depth-intervals (Fig. 7) a distinct stepped profile marking the boundary between the two facies facies emerges, as would be expected from the contrasts in facies noted above. However, the depth of the boundary between facies varies from ~11 to ~42 mm beneath the sediment surface in the samples, and represents the combined effects of gross distortion of the sediment facies during sampling noted above (Fig. 3, 4), as well as some limited spatial variation in what in exposed sections is a very sharp, regular boundary (±5 mm). What is particularly notable is the considerable variability of mean grayscale values within the weaker upper facies between each sampling method. The combination of data presented in Figures 6 and 7 strongly suggest that, particularly in weaker, less stiff sediment, the process of sampling confers significant disturbance to sediment bulk density and microporosity.

4 Discussion

The key outcome of this study is to identify that none of the sediment sampling methods employed recover a truly undisturbed sample. Although sampling-induced disturbance has been previously considered (e.g. Hvorslev 1949; Gilbert 1992; Clayton et al. 1995), this issue, and the implications for soil and sediment structural analyses particularly at microscopic scales, remains an over-looked, yet potentially significant problem. Whilst it is unsurprising to record disturbance associated with all sampling methods, the degree to which sampling disturbance has been identified using X-Ray μCT and the impact on fundamental sediment structural properties throughout the samples is perhaps unexpected.

4.1 Assessment of sampling disturbance to sediment structure

Whilst the classic work of Hvorslev (1949) remains the definitive reference for industry guidelines for soil and sediment sampling, with occasional updates (e.g. Gilbert 1992; Clayton et al. 1995; Ladd and DeGroot 2004), the advent of more advanced laboratory methods of analysing soils and sediments mean that the potential for field-sampling disturbance of such materials can no longer be ignored (Viana da Fonseca and Pineda 2017). Many studies make reference to the collection of 'undisturbed' samples from cores, boreholes and exposures (Lanesky et al. 1979; Black et al. 2002; Carr 2004; Palmer et al. 2008; Araujo-Gomes and Ramos Pereira 2014; Glew and Smol 2016), typically for analysis of high-resolution environmental proxy records. However, it is clear from the data presented in this study that disturbance resulting from the sampling approach is common, and is not, as is often assumed, restricted just to the edges of the sample where it interacts with the sampling chamber. Table 3 summarises and compares the impact of sampling method in terms of disturbance features imparted upon the sediments investigated in this study. It is clear that all methods that require application of significant force during insertion (pushing, rotation or percussive hammering: Fig. 1) generate significant structural modifications, and that disturbance is limited where passive methods of block cutting are employed. Whilst this is perhaps to be expected, the extent of disturbance of sediment during sampling by commonly-used coring and depth-sampling methods is surprising.

Cutting of sample blocks, referred to as the advanced trimming method by Hvorslev (1949) results in the least disturbance to sediment structure (Table 4), but this method is not suited to sampling sediments at depth, except when artificial or natural exposures are available. Even when this method is used, variations in the geotechnical properties of different facies held within a sampling chamber can result in fracture and gross-scale disturbance of the sample (Fig. 8), compromising subsequent analysis of key state and mechanical properties.

Continuous pushing of samples has been previously considered to be an appropriate means of recovering a relatively undisturbed sample (Hvorslev 1949; Clayton et al. 1995; Knappett and Craig 2012), as opposed to methods where intermittent or incremental stresses are applied. However, in the sediments at Orplands Farm, such continuous push results in dramatic gross distortion of the sediments, both in terms of the length of samples and the contact between facies, as well as substantial modification to both macro- and micro-structure as evidenced by pore-space (Table 3). In particular, the extreme intra-facies variations in bulk density and microporosity demonstrated in the lower, pre-restoration facies (Fig. 6) attests to the development of force chains (Peters et al. 2005; Fonseca et al. 2013) resulting in dramatic heterogeneity and partitioning of the stress-field through the sample. This essentially renders the Push samples worthless for most subsequent ex situ laboratory sedimentological and geotechnical tests.

Percussion, piston or hammer coring is a very common form of depth sampling in terrestrial, aquatic and marine contexts (Gardner et al. 2009; Knappett and Craig 2012; Xu et al. 2011; Montagna et al. 2017), but it is evident that such methods impart considerable modification to sediment samples (Table 3). The effect of percussive hammering is particularly evident in the less-stiff upper facies at Orplands Farm, where both macro- and microporosity has been dramatically increased (Fig. 5d, Fig. 6b), interpreted to result from de-watering of the lower sediment facies during percussive compression. During field sampling, the lengthening of the Hammer samples after vertical compression was observed during the five-minute relaxation period before sample recovery. In geotechnical terms, the resulting soil state and mechanical properties of each facies have been modified, but in contrasting ways. The already significant overall net shortening of the small-bore hammer samples (Fig. 3) is therefore partly masked by the dilational lengthening during relaxation of the upper facies prior to sample removal. As with the Push sampler, it is evident that the structural properties of the Hammer samples have been significantly compromised by the sampling method.

Rotary drilling, gouge and auguring methods are equally common depth sampling approaches as the push and hammer methods noted above (Knappett and Craig 2012). It is apparent however (Table 2, 3) that sediment structural integrity is heavily compromised by the rotary sampling process either during insertion, or recovery (as is the case in this study). The stiffer, more competent lower facies has been sheared during the rotation required to set the sediment in the sample chamber, resulting in complex fracturing (Fig. 5c), fundamentally altering porosity and pore structure, and compressing the matrix of the sediment (Fig. 6a). The impact on the less stiff upper facies is more pervasive with highly variable changes to bulk density and microporosity and complete loss of the vesicles present in all other samples (Fig. 6b, 7) suggesting complete re-working of sediment structure. As such, it would appear that sampling methods incorporating an element of torque rotation are highly likely to substantially modify sediment or soil structure.

Once sampled, irrespective of the sampler used, the potential for additional disturbance during recovery and transport back to the laboratory can be significant (Viana da Fonseca and Pineda 2017). Hypothetical stress paths (Baligh 1985; Baligh et al. 1987) demonstrate the temporal changes in stress field applied to sediments during sampling and recovery. Whilst it is not possible to directly evaluate the post-recovery modification of the samples in this study, the variations in structural characteristics explored above suggest that the primary disturbance in this instance is through the sampling method. The precautions taken in sealing, wrapping and transporting samples noted in Section 2:1 seem to have avoided further visible sample disturbance.

Pore water content and sediment shear strength are critical factors in determining the susceptibility of sediments to disturbance during the sampling process (Knappett and Craig 2012), but this study demonstrates that these are of secondary importance compared to the actual method of sampling employed. The presence of significant deformation structures and changes to bulk density/microporosity throughout both the weak, saturated upper facies and the drier, stiffer lower facies from Push, Hammer and Gouge samples illustrates that whilst the style of disturbance is partly controlled by sediment state, disturbance is recognised in the entire sample.

As outlined by Hvorslev (1949), larger sampler area ratios increase the likelihood of disturbance to sediment structure during sampling and recovery (Fig. 3). The samples reported in this study reflect a deliberate choice to mainly use sample tubes with a high area ratio in order to better describe and characterise the nature of disturbance, but that even when larger diameter (and thus much smaller area ratio) chambers were deployed, disturbance of the sediments was still observed from all methods. In addition, the smaller-bore sample tubes used in this study are very typical of the chamber sizes of Russian, Livingstone and push corers (Yang and Flower 2009; Lowe and Walker 2015; Glew and Smol 2016), and have similar area ratios to larger corers with thicker, thermally insulated chambers (e.g. Jahnke and Knight 1997). As such, the evidence of significant disturbance reported in this study is likely to be considerably more widespread than previously reported.

4.2 Implications for sediment structural analysis

In recent decades, there have been considerable advances in the microstructural analysis of sediments and soils through thin section (e.g. Kemp 1985; van der Meer 1993; Stoops 2009; Menzies et al. 2010; Phillips et al. 2011; van der Meer and Menzies 2011) and more recently µCT (e.g. Quinton et al. 2009; Luo et al. 2010a; 2010b; Tarplee et al. 2011; Rezanezhad et al. 2016; Spencer et al. 2017; Rabot et al. 2018; Tseng et al. 2018). Few of these studies evaluate the potential impact of sampling disturbance on structural properties, but some note that discrepancies seen between field and laboratory analyses are likely to be influenced by sampling disturbance (Nuttle and Hemond 1988). For sediments where deformation mechanisms are critical in their emplacement, such as subglacial traction tills (Kilfeather and van der Meer 2008; Menzies et al. 2010; Tarplee et al. 2011), differentiating between synsedimentary evidence of deformation and structures arising from the sampling process becomes even more problematic.

This study demonstrates that structural properties (porosity, bulk density, sediment structural fabric) have all been modified through the sampling process, and that, as such, caution must be expressed over the interpretation of visible macroporosity and structure. By consistently adopting the block-cutting/advanced trimming method to minimise disturbance, informed by the work reported here, Spencer et al. (2017) demonstrate significant contrasts in porosity, structural pore network efficiency and complexity between natural and restored salt-marsh sediments that explain subdued hydrological response to tidal forcing in restored salt-marsh at Orplands Farm, accounting for sub-optimal restoration outcomes. Had other sampling methods been employed, it is clear that such interpretations as provided by Spencer et al. (2017) relating sediment structure and functional behaviour would have been fundamentally compromised.

Porosity of sediment and soil is a fundamental structural property, influencing hydrological function, gas and solute transport and global biogeochemical fluxes (Beven and Germann 1982; 2013; Nuttle and Hemond 1988; Alley et al. 2002; Kilfeather and van der Meer 2008; Deurer et al. 2009; Quinton et al. 2009; Kettridge and Binley 2010; Kumar et al. 2010; Luo et al. 2010a; 2010b; Alaoui et al. 2011; Munkholm et al. 2013; Rab et al. 2014; Turburg et al. 2014; Naveed et al. 2016; Spencer et al. 2017, Müller et al. 2018). For example, soil structure quality assessments, heavily based on assessment of porosity, are a key tool in tillage and land management decision making, but visual field assessments are often criticised due to poor correlation with laboratory analysis (Ball et al. 2007; Johannes et al. 2017; Rabot et al. 2018). Whilst limitations in visual methods and variable field conditions can partly explain such poor correlations (Johannes et al. 2017), the impact of sampling method on laboratory analysis of pore-space such as bulk density used to check such visual assessment is not considered. Given the modifications to macropore (Fig. 5) and micropore (Fig. 6) space demonstrated in this study, this is potentially a significant oversight, and demonstrates how compromised this key soil and sediment structural property can be as a result of sampling method.

The complex sediment response to sampler type, exemplified by the hammer samples discussed in section 4:1 demonstrates other implications for studies based on depth sampling and core and borehole investigations. The differential response of the two sediment facies in terms of vertical compression (lower facies) and vertical extension (upper facies) to the sampler fundamentally change the geometry and form of the sediment stratigraphy and structure at Orplands Farm. Such changes in other sediment sequences with intra-facies and geotechnical contrasts are largely unrecognised, but differential compression and extension introduces another area of uncertainty in the interpretation of high-resolution environmental proxies like varves (c.f. Palmer et al. 2008; 2012; Bendle et al. 2015). Whilst the geotechnical contrasts between varves are typically lower than those within this study, the potential for alteration of varve thickness datasets through the sampling method is still significant.

With the application of increasingly sophisticated laboratory methods for the examination of sediments (Viana da Fonseca and Pineda 2017), and the importance of characterising state and mechanical properties of sediments and soils, it is clear that gaining better understanding of sampler disturbance is critical in evaluating the quality of such analyses.

4.3 Value of X-Ray computed tomography

The expanding use of X-Ray μCT to image the 3D properties of sediments and soils is revolutionising understanding of the properties and function of environmental materials in a wide range of contexts (Taina et al. 2008; Cnudde and Boone 2013). The non-destructive study of the spatial configuration of soils and sediments offered by μCT has enabled significant advances in understanding the processes, interactions and interrelations between soil and sediment components. Yet, despite making frequent reference to undisturbed samples, very few studies explicitly apply sampling methods that restrict disturbance or evaluate the extent to which samples have been disturbed by the sampling process. Viana da Fonseca and Pineda (2017) demonstrate the value of indirect inference of changes in bulk density of a silty-clay deposit as an indicator of sampling disturbance, similar to this study, and Bendle et al. (2015) show the extensive modification of sediment structure of samples being processed for thin section manufacture. Otherwise, such evaluations are notably absent from the literature.

In identifying numerous structural characteristics that can be attributed to disturbance through the different sampling processes (Table 3), this study demonstrates the value of μCT as a tool in assessing the degree and nature of sampling disturbance. Such non-destructive analysis can be performed prior to other laboratory analysis, informing sub-sampling and to provide quality assurance in subsequent analyses. In particular, pore-space is a structural characteristic that appears particularly vulnerable to sampling modification, and as such can be used to recognise and evaluate the nature of sampling disturbance in a sediment or soil sample (Luo et al. 2010; 2010b).

An opportunity afforded by μCT is that due to the non-destructive nature of the scanning, it is possible to scan and re-scan the same sample as it experiences manipulation. One of the more tantalizing outcomes of this study is to note that the disturbance of samples, particularly the Hammer and Gouge samples, represents evolution of the sediment under a changing strain path during progressive driving, sampling or recovery of the material. Experiments to reproduce and image the impacts of such mechanisms allow the hypothesised pathways proposed by Baligh (1985) and Baligh et al. (1987) to be assessed in a far more sophisticated manner than the bulk strain responses to triaxial compression currently used (Viana da Fonseca and Pineda 2017). Such analysis to explore the spatial component of sampling disturbance, in terms of the partitioning of strain, offers the potential for far greater understanding of the likely impact of use of different depth-sampling methods in unlithified sediments.

5 Conclusions

This study demonstrates that in the majority of environmental studies involving sediment recovery through coring or other depth sampling, there is no such thing as an undisturbed sediment sample. By undertaking the analysis of closely co-located samples recovered from restored salt-marsh sediments at Orplands Farm, Essex, UK, sediment structural differences between samples can be confidently attributed to different sampling methods employed. The novel use of X-ray μCT scanning of sealed sediment cores has enabled the identification and evaluation of the nature and extent of sample disturbance resulting from four common types of sediment recovery methods. Differences in gross sample length, distortion of sediment contacts and changes to macro- and microporosity and bulk density arise from the use of specific sampling method employed.

Whilst block cutting methods limit sediment disturbance, continuous push, hammer and rotary gouge sampling introduce considerable changes to sediment structure, most notably to bulk porosity and pore network characteristics, such that much of the primary sediment structure appears to have been overprinted during the sampling process, even within stiff, competent sediments. This has particular relevance for the subsequent use of coring and depth sampling of sediments for analysing state variables (e.g. porosity, sediment fabric) and mechanical properties (permeability, sediment strength). Estimates and modelling of gas and fluid fluxes as elements of key biogeochemical cycles, or of sediment behaviour and response to stress-field or pore-water pressure changes rely on robust data of such state variables and mechanics. Core shortening/lengthening occurs, particularly where normal stress is applied, and this will result in over/under estimations of rates of change in both paleo and modern environmental change studies. When quality assurance for such datasets is critical, analysis of X-ray μCT scans of sediment samples offers the opportunity to evaluate and quantify the extent to which the sampling process has compromised sediment structure.

Acknowledgements **T**he authors would like to thank Kurt Kjaer for permission to use the image visualization shown in Fig. 8.

References

Alaoui A, Lipiec J, Gerke HH (2011) A review of the changes in the soil pore system due to soil deformation: A hydrodynamic perspective. *Soil Till Res* 115-116: 1-15.

Allaire SE, Roulier S, Cessna AJ (2009) Quantifying preferential flow in soils: a review of different techniques. *J Hydrol* 378: 179-204.

Alley WM (2002) Flow and Storage in Groundwater Systems. *Science* 296(5575): 1985–1990.

Araújo-Gomes J, Ramos-Pereira A (2014) The new CutSprof sampling tool and method for micromorphological and microfacies analyses of subsurface salt marsh sediments, Algarve, Portugal. *Geo-Mar Lett* 35: 69–75.

Baligh MM (1985) Strain path method. *J Geotech Eng, ASCE* 36: 1108-1136.

Baligh, MM, Azzouz AS, Chin CT (1987) Disturbances due to "ideal' tube sampling. *J Geotech Eng ASCE* 113: 739–757.

Ball BC, Batey T, Munkholm LJ (2007) Field assessment of soil structural quality- a development of the Peerlkamp test. *Soil Use Mgmt* 23: 329-337.

Bendle JM, Palmer AP, Carr SJ (2015) A comparison of micro-CT and thin section analysis of Lateglacial glaciolacustrine varves from Glen Roy, Scotland. *Quatern Sci Rev* 114: 61-77.

Beven K, Germann P (1982) Macropores and water flow in soils. *Water Resour Res* 18: 1311-1325.

Beven, K, Germann P (2013) Macropores and water flow in soils revisited. *Water Resour Res* 49: 3017-3092.

Black HJ, Dainat M, Köster M, Meyer-Rei L-A (2002) A Multiple Corer for Taking Virtually Undisturbed Samples from Shallow Water Sediments. *Estuar. Coast Mar Sci* 54: 45–50.

Blomqvist S (1991). Quantitative sampling of soft-bottom sediments – problems and solutions. *Mar Ecol Prog Ser* 72: 295-304.

Brain MJ, Kemp AC, Hawkes AD, Engelhart SE, Vane CH, Cahill N, Hill TD, Donnelly JP, Horton BP (2017). Exploring mechanisms of compaction in salt-marsh sediments using Common Era relative sea-level reconstructions. *Quatern Sci Rev 167*: 96–111.

Buller AT, McManus J (1979). Sediment sampling and analysis. In: Dyer KR (ed) *Estuarine Hydrography and Sedimentation: A Handbook.* Cambridge University Press, Cambridge pp 87-130.

Bullock P, Federoff N, Jongerious A, Stoops G, Tursina T (1985) *Handbook for Soil thin Section Description.* Waine Research, Wolverhampton..

Burt, R (ed) (2009) Soil survey field and laboratory methods manual. *Soil Survey Investigations Report No. 51, Version 1.0*. US Department of Agriculture, Natural Resources Conservation Service, Nebraska, US. Available at ftp://ftp-fc.sc.egov.usda.gov/NSSC/Lab_References/SSIR_51.pdf accessed 29 September 2018

Carr SJ (2004) Micro-scale features and structures. In: Evans DJA, Benn DI (eds) *A Practical Guide to the Study of Glacial Sediments*. Arnold, London pp115-144

Clayton CRI (1986) Sample disturbance and BS5930. In: Hawkins AB (ed) *Site Investigation: Assessing BS5930*. Geological Society, Engineering Geology Special Publication No. 2. Geological Society, London pp 33-41.

Clayton CRI, Matthews MC, Simons NE (1995) *Site Investigation*. Blackwell Science, Oxford.

Cnudde V, Boone MN (2013) High-resolution X-ray computed tomography in geosciences: A review of the current technology and applications. *Earth-Sci Rev* 123: 1–17.

Corzo A, Jimenez-Arias JL, Torres E, Garcia-Robledo E, Lara M, Papaspyrou S (2018) Biogeochemical changes at the sediment – water interface during redox transitions in an acidic reservoir : exchange of protons , acidity and electron donors and acceptors. *Biogeochemistry*, *139*: 241–260.

Deurer M, Grinev D, Young I, Clothier BE, Müller K (2009) The impact of soil carbon management on soil macropore structure: a comparison of two apple orchard systems in New Zealand. *Eur J Soil Sci* 60: 945-955.

Doran JW, Mielke LN (1984) A rapid, low-cost method for determination of soil bulk density. *Soil Sci Soc Am J* 48: 717-719.

Doube M, Kłosowski MM, Arganda-Carreras I, Cordelières FP, Dougherty RP, Jackson JS, Schmid, B., Hutchinson, J.R., Shefelbine, SJ (2010) BoneJ: Free and extensible bone image analysis in ImageJ. *Bone* 47(6): 1076–1079.

Emmerson RHC, Manatunge JMA, MacLeod CL, Lester JN (1997) Tidal exchanges between Orplands Managed Retreat Site and the Blackwater Estuary, Essex. *J Chart Ins Wat Environ Manage* 11: 363-372.

Environment Canada (1994) *Environmental Protection Series: Guidance document on collection and preparation of sediments for physicochemical characterisation and biological testing*. Report EPS 1/RM/29. Environment Canada, Ottawa, Canada.

Fonseca J, O'Sullivan C, Coop MR, Lee PD (2013). Quantifying the evolution of soil fabric during shearing using scalar parameters. *Géotechnique*, 63(10): 818–829.

Frew C (2014) Coring Methods. In: *Geomorphological Techniques, British Society of Geomorphology*. 1, 1–10. Available at: http://geomorphology.org.uk/sites/default/files/geom_tech_chapters/4.1.1_Coring.pdf. Accessed 02 March 2018.

Gardner WS, Mccarthy MJ, Carini SA, Souza AC, Lijun H, Mcneal KS, Puckett MK, Pennington J (2009) Collection of intact sediment cores with overlying water to study nitrogen- and oxygen-dynamics in regions with seasonal hypoxia. *Cont Shelf Res* 29: 2207–2213.

Gilbert PA (1992) Effect of sampling disturbance on laboratory-measured soil properties. *US Army Corps of Engineers Miscellaneous Paper* GL-92-35.

Glew JR, Smol JP (2016) A push corer developed for retrieving high-resolution sediment cores from shallow waters. *J Paleolimn* 56: 67–71.

Glew JR, Smol JP, Last WM, (2002) Sediment Core Collection and Extrusion. In: Last WM, Smol JP (eds) *Tracking Environmental Change Using Lake Sediments, Basin Analysis, Coring, and Chronological Techniques Developments in Paleoenvironmental Research*. Kluwer Academic Publishers, London pp 73-105

Hight DW (1986) Laboratory testing: Assessing BS5930. In: Hawkins AB (ed) *Site Investigation: Assessing BS5930*. Geological Society, Engineering Geology Special Publication No. 2. Geological Society, London pp 43-58.

Hvorslev MJ (1949) *Subsurface Exploration and Sampling of Soils for Civil Engineering Purposes*. Waterways Experiment Station, Vicksburg, Mississippi.

Jahnke RA, Knight LH (1997) A gravity-driven, hydraulically-damped multiple piston corer for sampling fine-grained sediments. *Deep-Sea Res Pt I* 44: 713–718.

Johannes A, Weisskopf P, Schulin R, Boivin P. (2017) To what extent do physical measurements match with visual evaluation of soil structure? *Soil Till Res* 173: 24–32.

Jones FGW, Thomasson AJ (1976) Bulk density as an indicator of pore space in soils usable by nematodes. *Nematologica* 2:, 133-137.

Kemp RA (1985) *Soil Micromorphology and the Quaternary*. QRA Technical Guide No 2. Cambridge.

Ketcham RA, Carlson WD (2001) Acquisition, optimisation and interpretation of x-ray tomographic imagery: applications to the geosciences. *Comput Geosc*i 27: 381-400.

Kettridge N, Binley A (2010) Evaluating the effect of using artificial pore water on the quality of laboratory hydraulic conductivity measurements of peat. *Hydrol Process* 24: 2629-2640.

Kilfeather AA, van der Meer JJM (2008) Pore size, shape and connectivity in tills and their relationship to deformation processes. *Quatern Sci Rev* 27: 250-266.

Knappett JA, Craig RF (2012). *Craig's Soil Mechanics (8th Edition)*. Spon Press, London,

Kumar S, Anderson SH, Udawatta RP (2010) Agroforestry and grass buffer influences on macropores measured by computed tomography under grazed pasture systems. *Soil Sci Soc Am J* 74: 203-212.

Ladd CC, DeGroot DJ (2004). *Recommended practice for soft ground site characterisation: Arthur Casagrande Lecture*. 12th Pan-American Conference on Soil Mechanics and Geotechnical Engineering, Massachusetts Institute of Technology, Cambridge, MA, USA.

Lane CM, Taffs KH (2002). The LOG corer - a new device for obtaining short cores in soft lacustrine sediments. *J Paleolimn* 27: 145-150.

Lanesky DD, Logan BW, Brown RG, Hine AC (1979) A new approach to portable vibracoring underwater and on land. *J Sed Petrol* 49: 654–657.

Limaye A (2012) Drishti: a volume exploration and presentation tool. *SPIE Proceedings, 8506 (Developments in X-Ray Tomography VIII)*, 85060X.

Lotter AF, Merkt J, Sturm M (1997) Differential sedimentation versus coring artifacts: A comparison of two widely used piston-coring methods. *J Paleolimn* 18: 75-85.

Lowe JJ, Walker MJC (2015) *Reconstructing Quaternary Environments (3rd Edition)*. Routledge, London,

Luo L, Lin H, Schmidt J (2010a) Quantitative relationships between soil macropore characteristics and preferential flow and transport. *Soil Sci Soc Am J* 74: 1929-1937.

Luo L, Lin H, Li S. (2010b). Quantification of 3-D soil macropore networks in different soil types and land uses using computed tomography. *J Hydrol*, 393: 53-64.

Menzies J, van der Meer JJM, Domack E, Wellner JS (2010) Micromorphology: As a tool in the detection, analyses and interpretation of (glacial) sediments and man-made materials. *Proc Geol Assoc* 121: 281–292.

Montagna PA, Baguley JG, Hsiang C, Reuscher MG (2017) Comparison of sampling methods for deep-sea infauna. *Limnol Oceanogr Methods* 15: 166–183.

Munkholm LJ, Heck RJ, Deen B (2013) Long-term rotation and tillage effects on soil structure and crop yield. *Soil Till Res* 127: 85–91.

Naveed M, Schjønning P, Keller T, de Jonge LW, Moldrup P, Lamandé M (2016) Quantifying vertical stress transmission and compaction-induced soil structure using sensor mat and X-ray computed tomography. *Soil Till Res* 158: 110–122.

Nuttle WK, Hemond HF (1988) Salt marsh hydrology: implications for biogeochemical fluxes to the atmosphere and estuaries. *Global Biogeochem Cycles* 2: 91-114.

Palmer AP, Rose J, Lowe JJ, Walker MJC (2008) Annually laminated Late Pleistocene sediments from Llangorse Lake, South Wales, UK: a chronology for the pattern of ice wastage. *Proc Geol Assoc* 119: 245-258.

Palmer AP, Rose J, Rasmussen SO (2012) Evidence for phase-locked changes in climate between Scotland and Greenland during GS-1 (Younger Dryas) using micromorphology of glaciolacustrine varves from Glen Roy. *Quatern Sci Rev* 36: 114–123.

Peters JF, Muthuswamy M, Wibowo J, Tordesillas A (2005) Characterization of force chains in granular material. *Phys Rev Lett* 72: 041307.

Phillips E, van der Meer JJM, Ferguson A (2011) A new “microstructural mapping” methodology for the identification, analysis and interpretation of polyphase deformation within subglacial sediments. *Quatern Sci Rev*, 30: 2570–2596.

Quiggin N (2011) *Inspect-X User Manual*. Nikon Metrology, Hertfordshire, England..

Quinton WL, Elliot T, Price JS, Rezanezhad F, Heck R (2009) Measuring physical and hydraulic properties of peat from x-ray tomography. *Geoderma*, 153: 269-277.

Rab MA, Haling RE, Aarons SR, Hannah M, Young IM, Gibson D (2014) Evaluation of X-ray computed tomography for quantifying macroporosity of loamy pasture soils. *Geoderma* 213: 460–470.

Rabot E, Wiesmeier M, Schlüter S, Vogel HJ (2018) Soil structure as an indicator of soil functions: A review. *Geoderma*, 314: 122–137.

Ray A (2011) *CT Pro user manual*. Nikon Metrology, Hertfordshire, England.

Rezanezhad F, Price JS, Quinton WL, Lennartz B, Milojevic T, Van Cappellen P (2016) Structure of peat soils and implications for water storage, flow and solute transport: A review update for geochemists. *Chem Geol*, 429: 75–84.

Schindelin J, Arganda-Carreras I, Frise E, Kaynig V, Longair M, Pietzsch T, Preibisch S, Rueden C, Saalfeld S, Schmid B, Tinevez J, White DJ, Hartenstein V, Eliceira K, Tomancak P, Cardona A (2012) Fiji: an open-source platform for biological-image analysis. *Nature Methods* 9: 676-682.

Spencer KL (2017) Sediment sampling and analysis. In: ECSA practical handbooks on survey and analysis methods: In: Mitchell, S, Uncles, R (eds) *Estuarine and coastal hydrography and sedimentology, 2nd Edition*. Cambridge University Press, Cambridge, pp 153-170.

Spencer KL, Cundy AB, Croudace IW (2003) Heavy metal distribution and early- diagenesis in salt marsh sediments from the Medway Estuary, Kent, UK. Estuarine, *Coast Shelf Sci* 57: 43–54.

Spencer KL, Cundy AB, Davies-Hearn S, Hughes R, Turner S, MacLeod CL (2008) Physicochemical changes in sediments at Orplands Farm, Essex, UK following 8 years of managed realignment. *Estuar Coast Shelf Sci* 76: 608-619.

Spencer KL, Carr SJ, Diggens LM, Tempest JA, Morris MA, Harvey GL (2017) The impact of pre-restoration land-use and disturbance on sediment structure, hydrology and the sediment geochemical environment in restored saltmarshes. *Sci Tot Environ* 587–588: 47-58.

Stoops G (2009) Evaluation of Kubiëna's contribution to micropedology. At the occasion of the seventieth anniversary of his book "Micropedology." *Eurasian Soil Sci* 42: 693–698.

Taina IA, Heck RJ, Elliot TR (2008) Application of X-ray computed tomography to soil science: A literature review. *Can J Soil Sci* 88: 1–19.

Tarplee, MFV, van der Meer JJM, Davis GR (2011) The 3D microscopic 'signature' of strain within glacial sediments revealed using x-ray computed tomography. *Quatern Sci Rev* 30: 3501-3532.

Tempest JA, Harvey GL, Spencer KL (2015) Modified sediments and subsurface hydrology in natural and recreated salt marshes and implications for delivery of ecosystem services. *Hydrol Process* 29(10): 2346–2357.

Tseng CL, Alves MC, Crestana S. (2018) Quantifying physical and structural soil properties using X-ray microtomography. *Geoderma*, 318: 78–87.

Turberg P, Zeimetz F, Grondin Y, Elandoy C, Buttler A (2014) Characterization of structural disturbances in peats by X-ray CT-based density determinations. *Eur J Soil Sci*, 65: 613–624.

Turner RE, Milan CS, Swenson EM, (2006) Recent volumetric changes in salt marsh soils. *Estuar Coast Shelf Sci* 69: 352-359.

Twiss RJ, Moores EM (1997) *Structural Geology*. W.H. Freeman and Company, New York, USA.

van der Meer JJM (1993) Microscopic evidence of subglacial deformation. *Quatern Sci Rev* 12: 553-587.

van der Meer JJM, Menzies J (2011) The micromorphology of unconsolidated sediments. *Sed Geol* 238: 213–232.

Viana da Fonseca A, Pineda J (2017) Getting high-quality samples in "sensitive" soils for advanced laboratory tests. *Innov Infrastruct Solut* 2: 34.

Wright HE (1991) Coring Tips. *J Paleolimn* 6: 37-50.

Wright HE (1993) Core Compression. *Limnol Oceanogr* 38: 699-701.

Xu J, Wang Y, Yin J, Lin J (2011) New series of corers for taking undisturbed vertical samples of soft bottom sediments. *Mar Environ Res* 71: 312–316.

Yang H, Flower RJ (2009) A portable hand-operated sampler for shallow-water surface sediments with special reference to epipelic communities, *J Palaeolimn* 42: 317–324.

List of Tables

Table 1: Sampling techniques investigated. Techniques are broadly based on Hvorslev (1949) and Clayton et al. (1995) unless otherwise indicated.

Table 2: Summary of qualitative description and quantitative analysis of samples from Orplands Farm. Macropopore abundance/degree of deformation: - none evident, ● low, ●● moderate ●●● high. Macropore type (after Kemp, 1985): C channels and chambers, Ve vesicles, V vughs.

Table 3: Comparison of sampling disturbance from the methods investigated in this study.

List of Figures

Fig. 1 Theoretical stresses imposed on a sample during common forms of depth sampling. (A) Continuous tube push or advanced trimming; (B) Rotary methods; and (C) Mechanical methods, including percussion coring and vibrocoring. σ1 is the stress with the highest magnitude and likelihood of imparting structure on the sediment being sampled, σ4 the lowest. After Hvorslev (1949), Twiss and Moores (1997).

Fig. 2 Location of Orplands Farm Managed Realignment site. The cores for this study were extracted from Site B, which was simply allowed to inundate after the sea defences were deliberately breached in 1995. Site A experienced surface ploughing immediately prior to inundation, resulting in a less pronounced facies boundary (Emmerson et al., 1997; Spencer et al., 2008).

Fig. 3 Changes in sample core length of tube samples, indicating gross-scale deformation of samples as a function of the sampling method. (A) small-bore sampling tubes. (B) Large-bore sampling tubes.

Fig. 4 Sampler-related disturbance to sample edges and the boundary between the two sediment facies at Orplands Farm. (A) Significant edge furrowing and distortion of facies boundary, Push 2. (B) Deep edge furrows, dragging and distortion of facies boundary, Push 1. (C) Mixing of sediment facies, Hammer 1, including distortion of facies boundary, smearing of upper facies down the edges of the lower facies and edge furrows. (D) 2D vertical slices of Push 1, demonstrating different levels of disturbance in different planes, showing the importance of looking at samples in 3D. (E) Minimally-disturbed sample, Cut 1.

Fig. 5 Macropore space conditioned by sampler type. The right-hand image of each pair shows the extent of the lower, pre-breach facies for reference. (A) Assumed undisturbed sample, Tub 2. Note that macroporosity is low, with sporadic vesicles found only at the boundary between sediment facies. (B) Significantly higher macroporosity, found in both facies of the sample, Push 2. Pore types are mainly vesicles, but a number of these are squashed and deformed into vughs, with fissures forming associated with edge furrows and dragging. (C) Large-scale reworking of macropore space, Gouge 2. In situ vesicles largely absent, replaced by a complex fracture/fissure complex associated with the twisting of the sampling chamber. (D) Complete reworking if macropore space, Hammer 1. Large cluster of vesicles in upper facies attests to water escape and large-scale remobilisation of sediments in the upper facies. Fractures and distortion to surface pore-space demonstrates plastic bulk deformation of the entire sample.

Fig. 6 Box-and-whisker plots of greyscale values from the matrix of upper and lower sediment facies at Orplands Farm, as a function of sampler type. Central line is the median, box delimits interquartile range and the whiskers denote the 10th and 90th percentile range.

Fig. 7 Mean grayscale values of sediment matrix plotted against depth. Note the approximate location of the sediment facies boundary. There is close inter-sample similarity between the paired Cut and Tub samples, which contrasts with considerable variation between the paired Push, Hammer and Gouge samples, suggesting that the latter have been substantially disturbed.

Fig. 8 X-Ray μCT volume of a mammoth tin box sample (loess - tephra interbeds, Eldvatn, Iceland) which despite careful sampling by advanced trimming has developed substantial macropore space introduced through fracturing during sampling disturbance. Whilst it is possible to remove the sampling disturbance features from the sample, it is inevitable that key ‘signal’ is lost as well as the unwanted ‘noise’ of pores that are bisected by or touch these fissures.

Table 1: Sampling techniques investigated. Techniques are broadly based on Hvorslev (1949) and Clayton et al. (1995) unless otherwise indicated.

Method	Label	Description
Gouge Augering	Gouge1, Gouge2	Gouge pushed into sediments vertically in a single push to required depth; barrel rotated to secure sediment in sample chamber and recovered. Sample transferred to plastic drainpipe and wrapped in parafilm and ends sealed with ziploc bags and tape to prevent loss of moisture and entry of air.
Continuous tube push	Push1, Push2	Tube pushed in by placing block of wood over the top to more evenly distribute pressure and leaning on block to push tube into substrate with a single, continuous push.
Percussive tube push	Hammer1, Hammer2	Tube hammered vertically into sediment; block of wood placed over top of tube to prevent tube shattering and to distribute force more evenly. Repetitive percussive hits from 1.2kg mallet progressively drives tube into substrate (Hammer 1 = 21 repetitions, Hammer 2 = 29 repetitions)
Advanced trimming	Cut1, Cut2 Tub 1, Tub 2	Tube placed on surface, knife used to roughly cut around tube and sever vegetation. Sharp knife used to progressively excavate sediment around sampler and tube gently pushed down vertically into sediment; motion stopped as soon as resistance felt and cutting restarted. Plastic tub placed on surface and cut in, as would be done with a Kubiena tin (Kemp, 1985; Carr, 2004; Stoops, 2009), and progressively cut into sediment pile.

Table 2: Summary of qualitative description and quantitative analysis of samples from Orplands Farm. Macropopore abundance/degree of deformation: - none evident, ● low, ●● moderate ●●● high. Macropore type (after Kemp, 1985): C channels and chambers, Ve vesicles, V vughs. Macropore deformation:

Sample	Qualitative Description			Quantitative Analysis		
	Macropore abundance	Macropore type	Macropore deformation	% Macroporosity	Macropores per mm^3	Mean volume (mm^3)
Tub 1	●	Ve	-	1.3	0.028	0.081
Tub 2	●	Ve	-	1.0	0.019	0.083
Cut 1	●●	Ve	●	1.5	0.264	0.053
Cut 2	●	Ve	●	1.2	0.368	0.030
Gouge 1	●●●	F/V	●●●	4.4	0.533	0.077
Gouge 2	●●●	F/V	●●●	5.8	0.176	0.063
Hammer 1	●●	V/Ve/F	●●	3.5	0.643	0.052
Hammer 2	●	V/Ve/F	●●	1.6	1.059	0.012
Push 1	●●	V/F	●●	3.0	0.386	0.076
Push 2	●●	Ve/F	●●●	4.3	0.378	0.111

Table 3: Comparison of sampling disturbance from the methods investigated in this study.

Sampler Type	Anticipated Principal Stress Field (Figure 1)	Gross Distortion (Figure 3), 4	Macrostructural Modification (Table 2, Figure 5)	Microstructural Modification (Figure 6, 7)	
Cutting/Advanced Trimming (Tub 1,2; Cut 1,2)	Frictional drag on edges	Edge shortening, central legthening;	Minimal. Some distortion to existing pore samples (Cut samples only)	Some differential compression/extension in Cut samples;	LEAST DISTURBANCE ↑
Continuous Push (Push 1,2)	Vertical compression, frictional drag on edges	Edge shortening, central legthening; Distortion to sediment boundary	Substantial, increase in pore-space and distortion to sample.	Dramatic , inconsistent changes to bulk density and microporosity. Some sediment mixing	
Percussion/Hammer (Hammer 1,2)	Percussive vibration, frictional drag on edges	Significant shortening, particularly on edges; Distortion to sediment boundary.	Substantial changes in pore-space and generation of fissures. Distortion to existing pore structures.	Differential compression/extension to sediment facies. Considerable sediment mixing.	↓
Rotary (Gouge 1,2)	Torque rotation, vertical compression	Substantial distortion of sediment boundary.	Complete re-working of pore-space. Increase in bulk porosity.	Wide variability of bulk density and microporosity in both facies	MOST DISTURBANCE

Figure 1

Click here to access/download;Figure;Figure 1.tiff

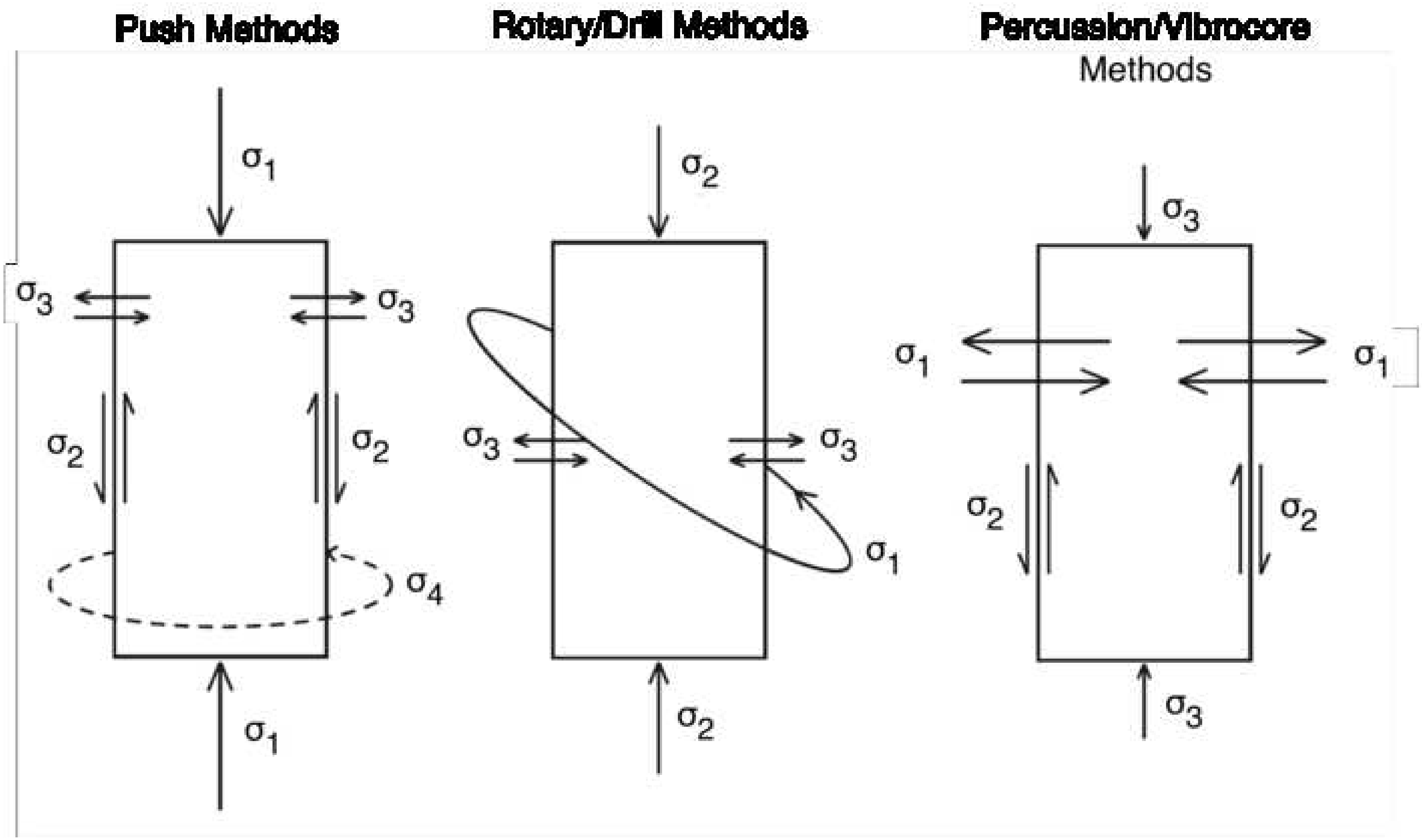

Figure 2

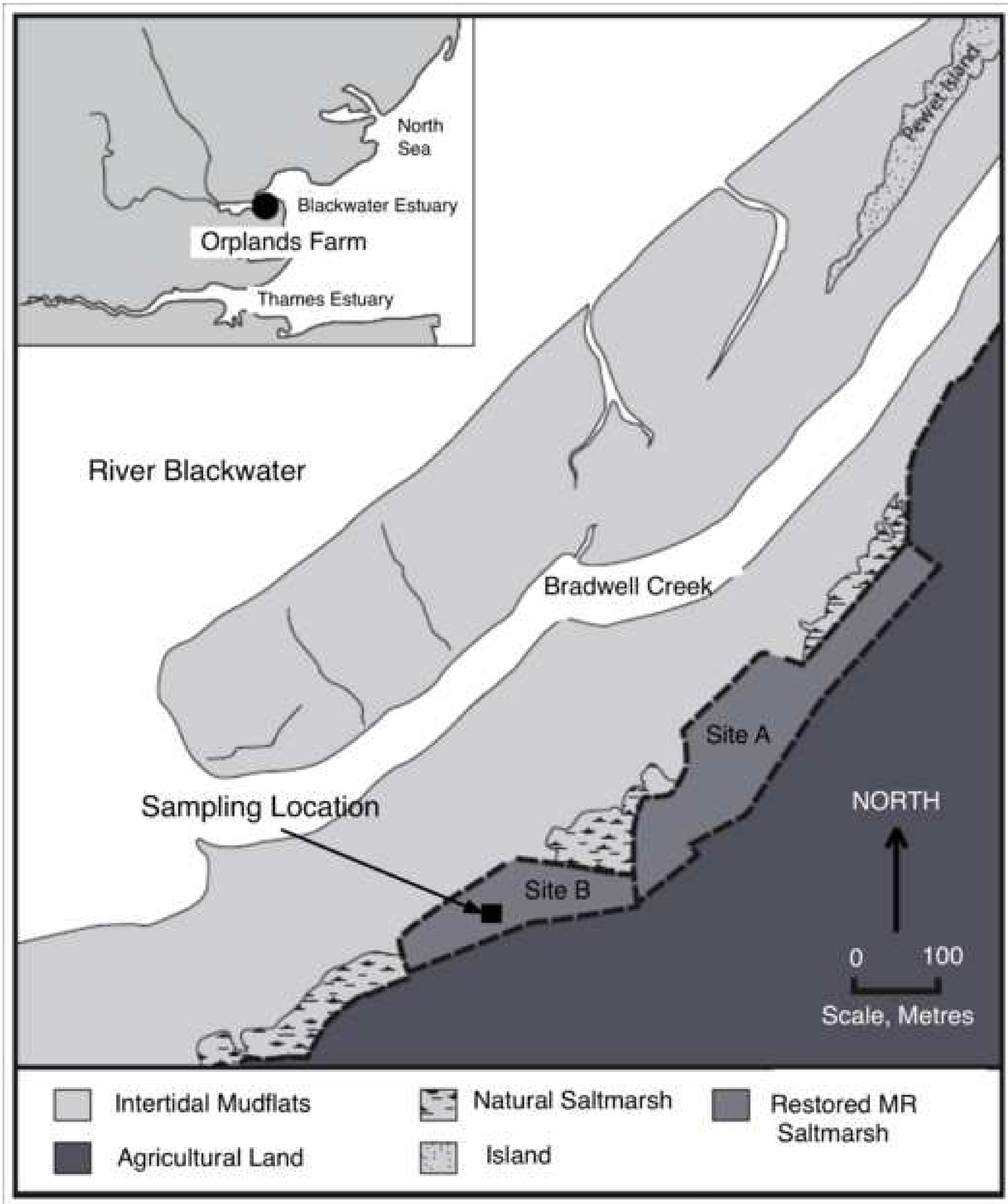
North Sea
Blackwater Estuary
Orplands Farm
Thames Estuary
River Blackwater
Pewet Island
Bradwell Creek
Site A
Site B
Sampling Location
NORTH
0
100
Scale, Metres
Intertidal Mudflats
Natural Saltmarsh
Restored MR Saltmarsh
Agricultural Land
Island

Figure 3

Click here to access/download;Figure;Figure 3.tiff

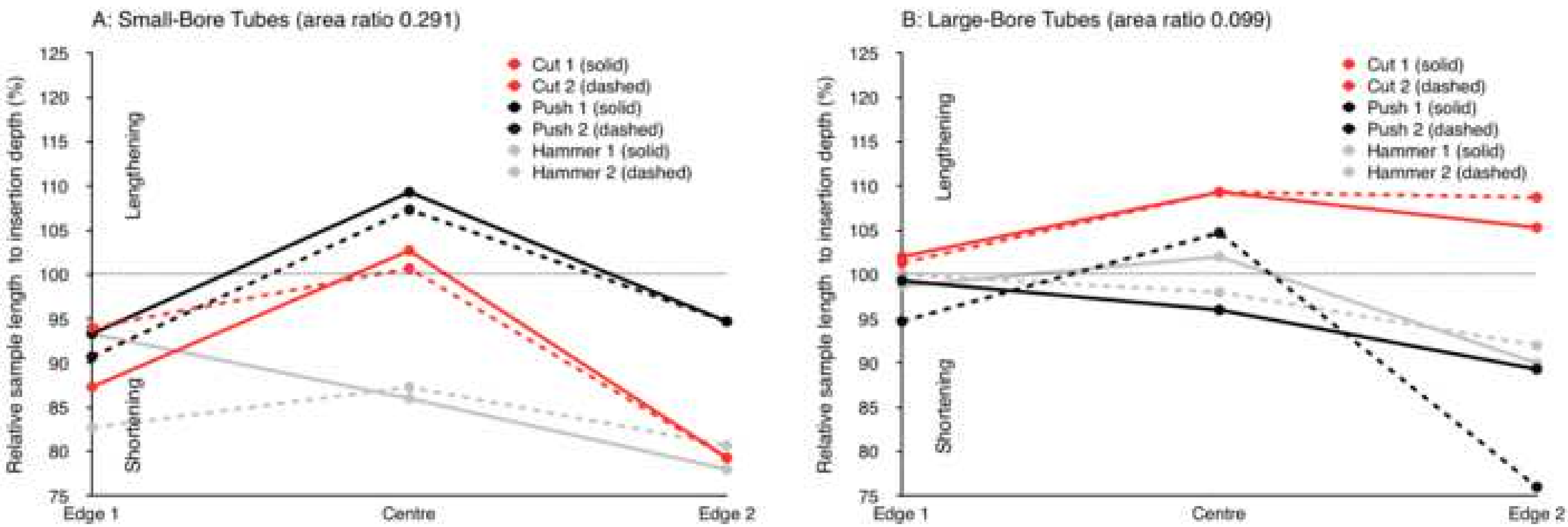

Figure 4

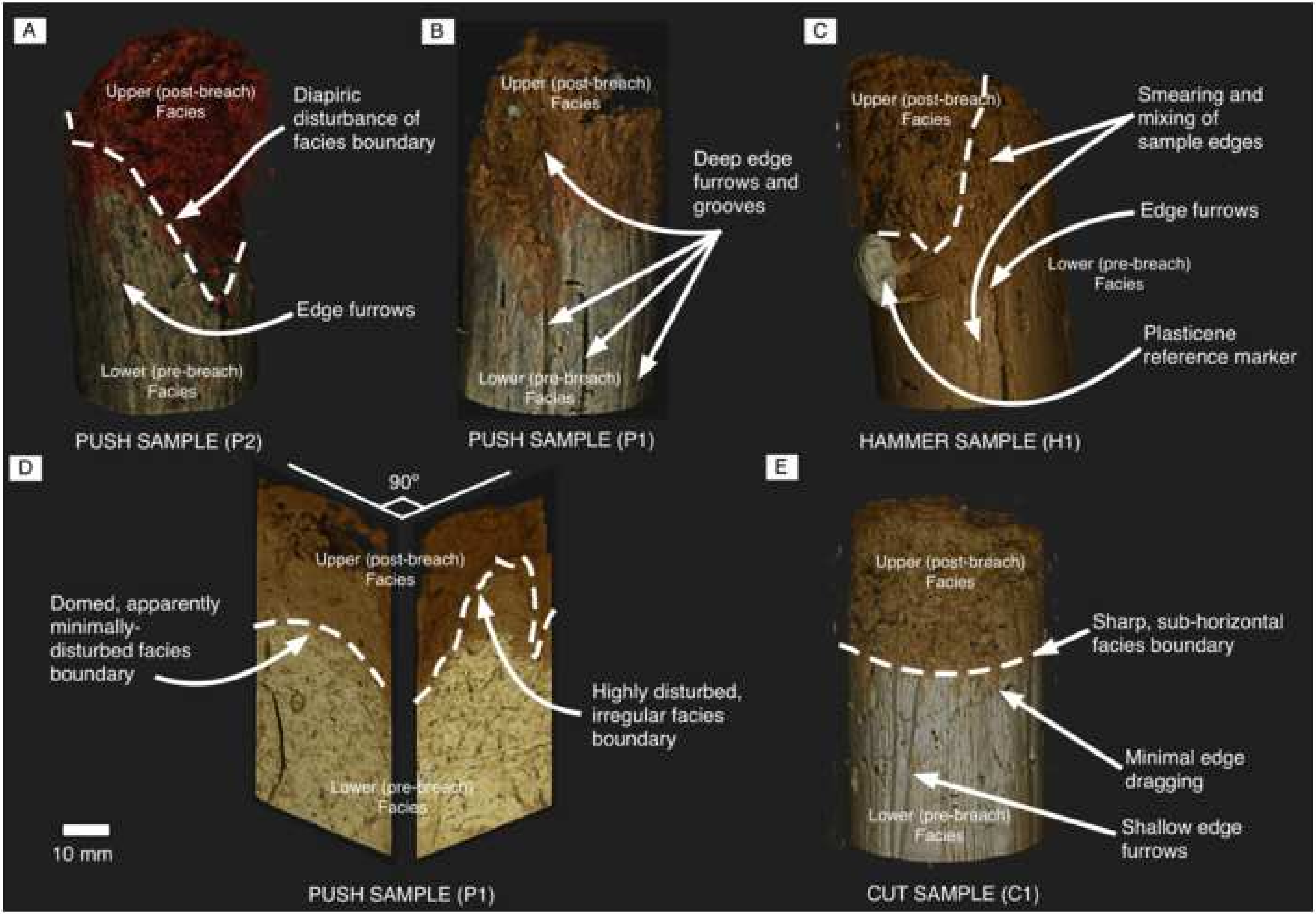
A
Upper (post-breach) Facies
Diapiric disturbance of facies boundary
Edge furrows
Lower (pre-breach) Facies
PUSH SAMPLE (P2)
B
Upper (post-breach) Facies
Deep edge furrows and grooves
Lower (pre-breach) Facies
PUSH SAMPLE (P1)
C
Upper (post-breach) Facies
Smearing and mixing of sample edges
Edge furrows
Lower (pre-breach) Facies
Plasticene reference marker
HAMMER SAMPLE (H1)
D
90°
Upper (post-breach) Facies
Domed, apparently minimally-disturbed facies boundary
Highly disturbed, irregular facies boundary
Lower (pre-breach) Facies
10 mm
PUSH SAMPLE (P1)
E
Upper (post-breach) Facies
Sharp, sub-horizontal facies boundary
Minimal edge dragging
Lower (pre-breach) Facies
Shallow edge furrows
CUT SAMPLE (C1)

Figure 5

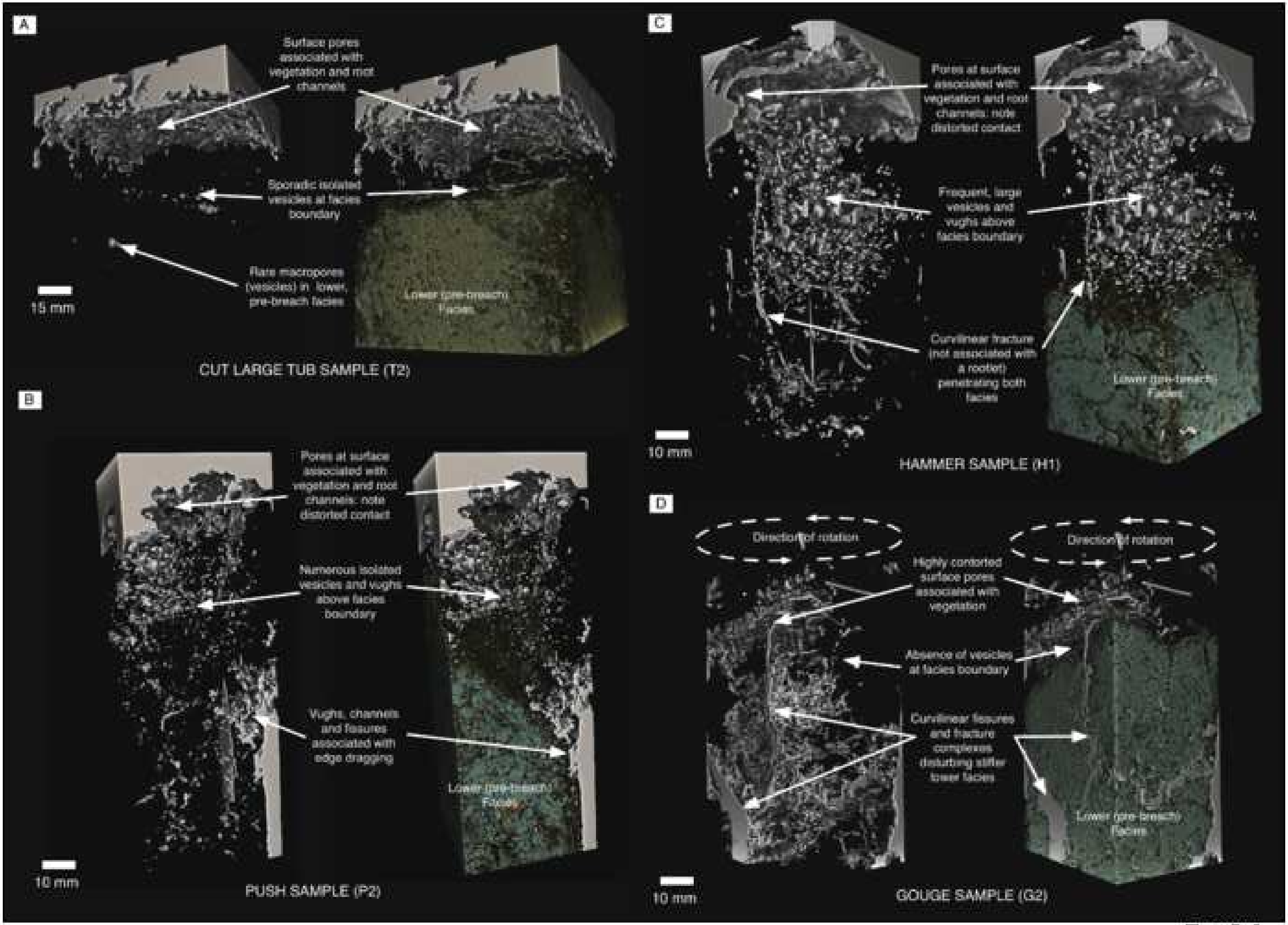

Figure 5a,b

Figure 5c,d

Figure 6

Click here to access/download;Figure;Figure 6.tiff

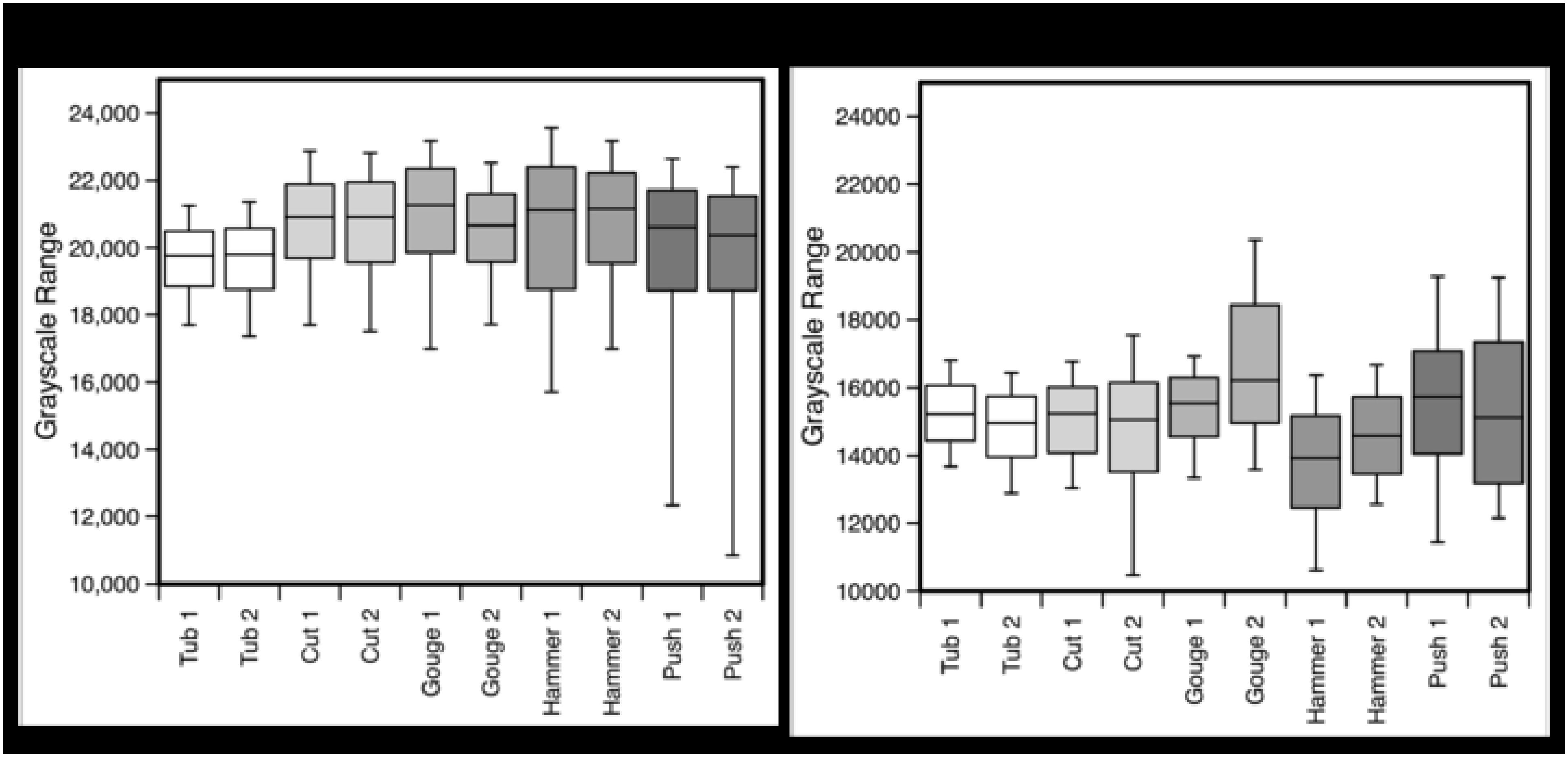

Figure 7

Click here to access/download;Figure;Figure 7.tiff

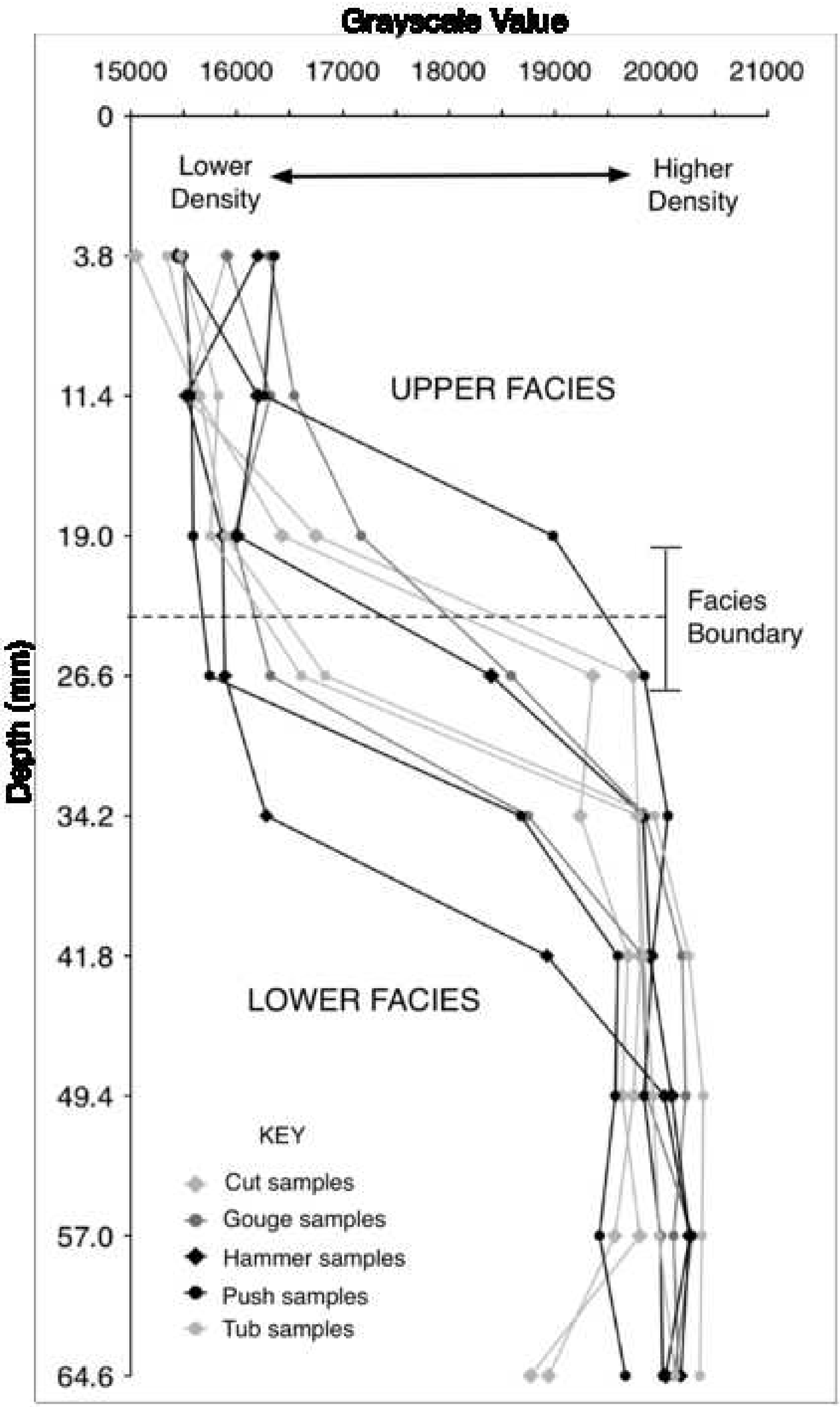

Figure 8