



McDONALD INSTITUTE CONVERSATIONS

Inspired geoarchaeologies: past landscapes and social change

Essays in honour of Professor Charles A. I. French

Edited by Federica Sulas, Helen Lewis & Manuel Arroyo-Kalin



Inspired geoarchaeologies



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Edited by Federica Sulas, Helen Lewis
& Manuel Arroyo-Kalin

with contributions from

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Courtesy of Kasia Gdaniec.

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Chapter 7

Making thin sections for geoarchaeology

Tonko Rajkovaca

Archaeological soil micromorphology is an established, constantly evolving, and versatile technique with a long tradition of application in academic research and increasingly important in the commercial sector. As applications widen in context and scope, the processing of micromorphology blocks into thin sections has also been evolving and being refined to allow for sampling and analysis of different deposits and materials. Building on the experience and expertise in developing protocols for thin section making at the McBurney Laboratory for Geoarchaeology, headed by Charly French, this chapter offers a guide to sampling and processing of micromorphological samples for academic research and commercial work.

Soil micromorphology is the analysis of soils/sediments in thin section using a polarizing microscope. In archaeology, soil micromorphology is an established, constantly evolving and versatile technique with a long tradition of application in academic research and increasingly important in the commercial sector too, generating a vast literature and reference sources across different environments, cultures and periods (French 2003; 2015; Macphail & Goldberg 2018a,b; Nicosia & Stoops 2017). It is particularly useful when applied to buried soils (or palaeosols) where it can be used to investigate sequences of past land use and landscape change (e.g. French & Pryor 2005; French *et al.* 2007; 2020; Lewis 2012), and to floors and occupation surfaces associated with possible structures, where it can elucidate construction materials and techniques, trampling/compaction, micro-settlement refuse and use-in-life of a floor/room/structure (e.g. Matthews *et al.* 1997a,c; Macphail & Cruise 2001; Karkanis & Efstratiou 2009; Milek 2012; Nicosia *et al.* 2012; Banerjee *et al.* 2015).

As archaeological soil micromorphology grows in depth and reach, the processing of micromorphology blocks into thin sections has also been evolving and is being refined to allow for sampling and analysis of

different contexts and materials (Jongerijs & Heintzberger 1975; Fitzpatrick 1984; Murphy 1986). Since the early 1990s, the Charles McBurney Laboratory for Geoarchaeology, under the leadership of Professor Charly French, has been developing and refining protocols for the processing of micromorphology blocks. In the early 1990s, Professor French established a thin sectioning facility in a shed at the Department's storage facility (Shorts), where he made his first slides and trained his first geoarchaeology technician, Julie Boreham, as well as his first post-doctoral researcher and PhD students. In those early days, practices and tools that would become distinctive features of thin section manufacturing at Cambridge were established: the basic laboratory equipment, experimenting and adapting impregnation mixtures to the characteristics of diverse soils and sediments, the production of 'mammoth'-sized slides. The first thin section facility had a large diamond saw, a vertical Brot grinding machine, a large fume cupboard, a few vacuum chambers, chemical storage cupboard, and an oven. Over time, the lab acquired grinding plates for the Brot to hold slides of different sizes. A major turning point was the completion of the McDonald Institute buildings, which included a designated basement laboratory for thin sectioning, along with a variety of storage spaces for sample storage, bringing all the micromorphology-oriented operations of the McBurney Lab into one building, along with the microscope lab and Professor French's office. Over the years, it became possible for him to leave much of the thin section production training to his technicians, initially Julie and subsequently the author of this chapter.

Without welcoming me into the Cambridge department, with his poise, erudition and vast knowledge of archaeology, I would not have achieved half of what I have over the last decade. The support of

Professor Charly French for my ambition to maintain a full-time position while embarking on PhD study abroad made a world of difference, helping me realize that anything is possible. We made the McBurney Lab our ‘home from home’ and a second home for our students and scholars. Here’s to many more fruitful endeavours!

This chapter brings together the experience and practice of experimenting, refining and advancing protocols for sampling and processing of thin sections made of thousands of samples brought to the McBurney Laboratory since I joined it in 2008. Updating our first guidelines for thin section making (French & Rajkovaca 2015), this chapter aims to provide a practical and versatile step-by-step guide to the sampling, transport, and processing of micromorphology samples for both university researchers and professional archaeologists.

Soils and micromorphology in archaeology

Soils are important in archaeology in two main ways. Firstly, they shape the nature and processes of preservation of archaeological materials. Different soil types have different chemical and physical properties, such as pH, texture, moisture, and iron content to name a few, which will differentially affect preservation of archaeological materials. Some soils will readily preserve organic material, whereas others will completely remove nearly all traces of it. In such cases, artefact patterning is not driven by human activities but is rather a factor of soil preservation potential that only a detailed understanding of the soil matrix can elucidate. Secondly, soils themselves can be a valuable part of the archaeological record. Buried soils are evidence of past land surfaces because soils need a relatively stable land surface on which to form. They provide important environmental and land use information, and complement other forms of environmental archaeology. As soils are also products of climate and organisms, their properties can be used to make inferences about the conditions (e.g. humid or arid, forest or grassland) under which they formed, and by extension, the conditions under which the associated archaeological materials were deposited.

Soil science approaches to archaeological sites involve accurately describing the stratigraphic sequence on- and off-site and identifying and sampling old land surfaces, buried soils, possible occupation surfaces and/or floors. If the site or landscape preserves a buried soil under either later deposits (such as peat or flood silts and clays) and/or upstanding monuments (like banks and barrows), extensive sampling is called for. In contrast, where no sign of an old land surface or

buried soil is found, for example, when the depth of archaeological deposits is within reach of the modern plough, geoarchaeological applications are more challenging.

Sampling soils and sediments

Sampling of buried soils and archaeological sediments takes different forms depending on the context and questions to be addressed, the available equipment, and key resources such as time and funding. Common practices for sampling for soil physical and chemical analyses include:

- Systematic gridded sampling of both surface and subsurface deposits for prospecting for and characterizing buried soils and archaeological contexts. Sampling intervals also depend on a number of factors and often range between 1/2/5/10 m, with closer intervals for a possible living surface and wider intervals for a field system.
- Excavation of one-metre-square test pits through the buried soil profile at regular intervals (e.g. 5, 10 or 20 m) for artefact retrieval, bulk sampling for macrobotanical analysis, stratigraphic recording and soil sampling.
- Soil sampling from either these test pits and/or sections through the buried soil and/or upstanding monuments or cut features should comprise taking a continuous set of block samples for micromorphological analysis (see below) and associated small bulk samples (or two handfuls of soil) taken either from each horizon or every 10 cm, depending on the stratigraphy.
- Where a site has been damaged by ploughing and is associated with a thin modern soil cover (usually less than 50–70 cm), sampling might focus on the fills of cut features. These contexts are generally disturbed by pre- and post-depositional mixing, but they can also preserve features of interest, such as possible pit linings, organic standstill horizons in ditches, or redeposited soil material, with primary fills usually being the most reflective of ‘use in life’ of the feature and its immediate surroundings.

The samples collected can then be used for a range of physical analyses (organic content, particle size, magnetic susceptibility) and chemical analyses (phosphates and multi-element) that will provide information about preservation conditions and inputs from various human activities (French 2015). For example, soil chemical analyses can detect change of chemical

properties and enrichment of chemical elements resulting from manuring, middening or ash deposition; soil magnetic properties can be altered by burning, the presence of hearths, or metal working.

Sampling for soil micromorphological analysis

Sampling for micromorphological analysis requires taking intact soil/sediment blocks, transporting them to a laboratory undamaged, and then a slow processing over 4–8 weeks to obtain slides or thin sections. Briefly, this entails impregnating the block with a clear casting resin under vacuum, curing for one month, cutting a thin slice off the impregnated block, polishing the slice, and mounting it on a polished glass microscope slide, and grinding it to a thickness of 25–30 microns (μm), and covering the slide with a glass coverslip (see detailed description below).

Taking block samples is done by using common tools, but the process itself requires an expert eye and skilful hands. Blocks are taken using the following tools and materials (Fig. 7.1):

- Trowel and/or strong knife.
- Containers to hold sample blocks: aluminium Kubiena tins or c. 5–8 cm square sectioned plastic downpipe cut into c. 10–14 cm lengths with one long side cut off, or clean empty cardboard juice cartons, or foil take-away/frozen food containers.
- Cling film, paper towel and parcel tape for wrapping.
- Water-proof marker for labelling.

Sampling follows detailed recording of the section, profile, or context under investigation. At its simplest, recording entails cleaning to clearly identify horizons, features, and boundaries in between them. The section will need to be described, drawn and photographed before sampling takes place. Having completed the recording, the sampling procedure in the field might proceed as follows:

1. Sample either as a series of continuous sets of blocks or as discrete blocks, with each block about 10–14 cm in length and 5–6 cm thick.
2. If possible, sample across the main layer/horizon/context boundaries.
3. With a knife or trowel, cut vertical slots along the sides of the prospective soil block in order to fit the container from the section face; start wide and work your way in; place the container over the prospective sample block; cut out from well behind the section face and block. Blocks can also be carved out by free hand.
4. Carefully remove the block from the section and take note of the top (up), trim the sample block as necessary. For sandy, rubbly or poorly consolidated material, several attempts might have to be made to cut out and release an intact block.
5. Gently wrap the sample block in paper towel and then cling film. Use parcel tape to seal the wrapping.
6. Using an indelible marker, mark the top on each side by adding an arrow showing the way up and



Figure 7.1. Professor Charly French taking soil micromorphology samples using a knife, cling film, paper tape and a tinfoil container. Image: Tonko Rajkovaca.

add the label. The latter usually contains the site name, context or unit with depths, and a unique identifier number for the sample.

7. For transport to the laboratory, wrapped blocks need to be carefully packed in a hard case such as a suitcase or a wooden/cardboard box, ideally cushioned with soft material (e.g. newspapers, sponges, clothes) so as to fill out any empty space between blocks. At this stage, the blocks might contain dry or slightly moist material and it is important to keep them as undisturbed as possible until they can be treated in the laboratory.
8. It is common practice and a valuable addition to take at least one small (two handfuls) sample of loose soil from the same horizon/spot as each block sample, for physical and/or chemical analyses.

Thin section making

Collection and drying of samples

In the laboratory, the sample blocks will first be inspected to ascertain their conditions, then logged in and prepared for drying as follows:

- Samples are set in open trays or plastic boxes with appropriate field markings, and cut open with scissors or a razor blade to reveal enough surface to allow air drying. Special care should be taken not to disturb the original composition and structure (Fig. 7.2).
- Sample labels, including site designations and section orientation/profile, are checked and written on the outside of the containers. Arrows can be added as an indication of the top of the profile, and where the block should be cut for thin sectioning.
- The samples are then placed on the shelves in the lab for a period of up to one month in order to remove moisture before impregnation. If samples arrive from the field wet, water will have to be removed by acetone replacement (see separate protocol).
- After a month, the samples are taken from the shelves and placed in the oven for a final drying period, starting at 25 degrees C and increasing to 40 degrees C for 24–48 hours, depending on the material.

Impregnation and vacuum treatment

Once the blocks are sufficiently dry, impregnation can take place. This process consists of impregnating the blocks with a mixture of a (polymeric) resin, acetone (to increase its viscosity), and a catalyst to speed up

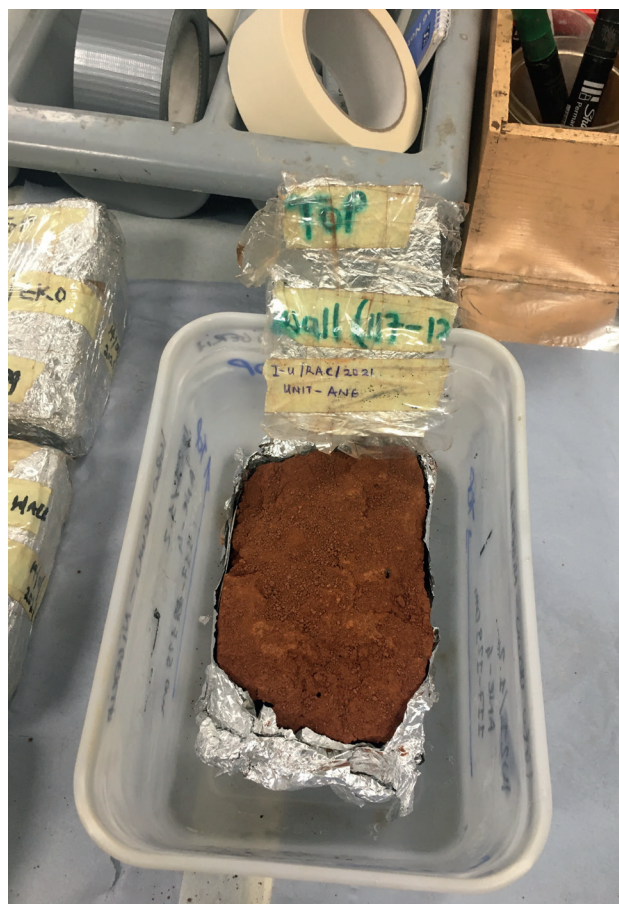


Figure 7.2. Example of micromorphology block unpacked and placed in a plastic container for laboratory processing (see Fig. 7.3). Image: Federica Sulas.

the hardening of the resin (Fig. 7.3). Recipes and products vary between laboratories, and also depending on the nature of sample material and conditions. The McBurney Laboratory has been developing recipes and experimenting on different materials for three decades. The standard recipe used on most samples is the following:

1. Following oven-drying overnight (25–40 degrees C), the samples are taken to the fume cupboard for impregnation while still warm. A standard fume cupboard can fit in no more than eight plastic containers at each impregnation session
2. Working inside the fume cupboard and wearing appropriate protective gear, pour 1800 ml of resin into a graduated plastic decanter. The colour of the resin is a clear light blue, and special attention should be given to the expiry date. All resin must be used within the shelf life of the product, or the impregnation and cure will be poor.

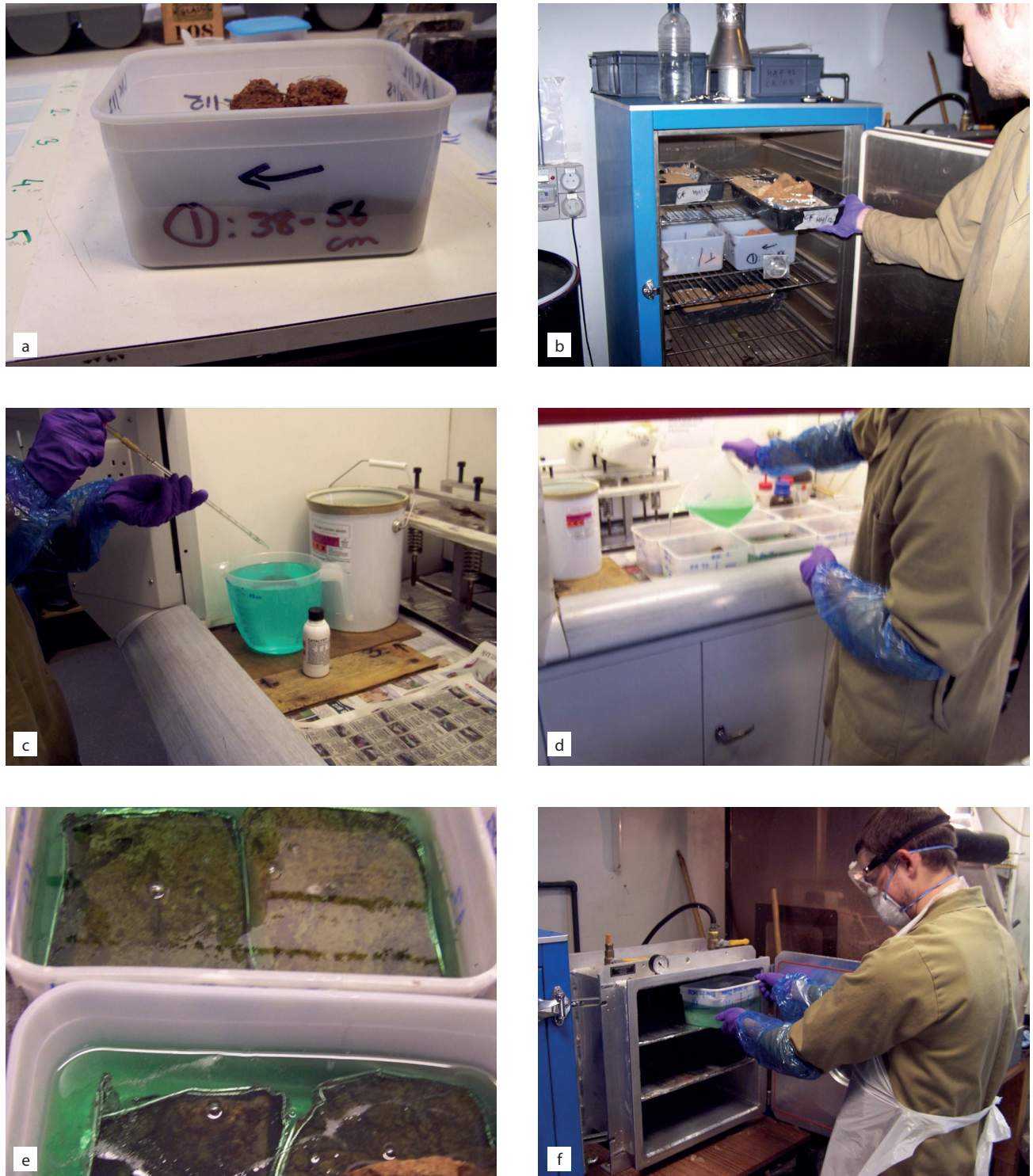


Figure 7.3. *Impregnation: (a) plastic containers labelled to record the orientation (top) and ID of the block; (b) following a period of drying at room temperature on a shelf, the samples undergo a final drying in the oven; (c) impregnation using resin mixed with acetone (large plastic beaker), to which MEKP is added by means of a Pasteur pipette; (d) the impregnation mix is then poured slowly and very carefully into the sample container; (e) the samples are then left to allow for full impregnation of the mix by capillary rise; (f) the container is then placed under vacuum to gently remove any remaining air in the blocks. Images: Tonko Rajkovaca.*



Figure 7.4. Curing of impregnated blocks: left, cabinet for curing impregnated blocks; right, impregnated block ready for sawing. Images: Tonko Rajkovaca.

3. Add 200 ml of acetone to the resin by gently and slowly folding it into the resin, then stirring it until fully amalgamated into the resin.
 4. Use a pipette to add 1.0 ml of methyl ethyl ketone peroxide (MEKP) into the mix, and gently stir until thoroughly mixed. This should take 3–5 minutes, and an immediate colour change from blue to green takes place. Any gloves that become contaminated with MEKP must be discarded into a sealed plastic bag, and new gloves put on to protect against potential chemical burn.
 5. Check that all the air bubbles in the resin from mixing have settled out, and then slowly pour around and down the inside of the sample container to prevent disturbance, fully immersing the samples.
 6. The samples are left to infiltrate with resin by capillary rise for up to an hour within the fume cupboard. The resin level should be monitored, and further topped up if it should drop below the original immersion mark. Place the date of impregnation on the outside of the container. This will act as an aide in monitoring the length of the curing time.
 7. After capillary rise, the samples are placed within the vacuum chamber, and slowly brought to 12–28 mercury vacuum, or until bubbles can be seen to gently evacuate from the samples. The samples are left under pressure for an initial 24-hour period.
 8. After 24 hours, a second resin mixture is prepared. After releasing the vacuum, the samples are taken from the chamber and placed in the fume cupboard. They are 'topped up', and completely re-immersed in the resin, and then placed back into the vacuum chamber for a further 24-hour period, using the same vacuum pressure as before.
 9. After the final vacuum, the samples are taken from the chamber and placed in a ventilated curing storage cupboard.
 10. Curing takes place over two weeks to a month or until the blocks are completely hardened. Periodic checks should be made on the resin level in the containers, and 'topping up' carried out if needed.
 11. After the samples have hardened, they are subjected to a final curing in the oven at 50 degrees C for a 24- to 48-hour period.
- The resin and MEKP used for impregnation must be stored in a ventilated curing storage cupboard after use. Beakers and associated impregnation equipment should be wiped clean with acetone and tissue. The MEKP graduated cylinder must be rinsed with at least 30 ml of acetone, and all rinsed liquid stored in a toxic waste bin. Any tissue or contaminated gloves used in the cleaning of MEKP must be disposed of separately and placed in sealed plastic bags. All the waste from impregnation is disposed of in the fire-bin and removed by the end of the working day to a separate waste store facility.

Sawing of samples

Once fully cured and hard dry, the impregnated blocks can be cut to obtain slices that will be mounted on microscopy slides. The cutting is performed by means of an electric saw as follows (Fig. 7.5):

1. The main switches of the saw and extractor fan are turned on, and the silt-box is topped up with fresh water. Extra care is taken to hose out the drainage tray, so that all wastewater from sawing will run away smoothly to the silt-box. Any adjustments to the water spray feeding the saw blade must be made before beginning to saw the samples. No person should ever attempt to saw samples without the saw blade being fed with water.
2. The hardened resin blocks are brought to the sawing sled, and the plastic container is cut to separate the samples. Slow, even pressure is used to push the sawing sled with the container to the blade. The saw should be allowed to pull the block through at an even pace; extra force will cause friction problems.
3. Before making an actual cut to the block, note which face of the block is wanted for thin sectioning. Decide the best way to cut down the block to obtain the needed sample slice, then proceed cutting the block with a plan.
4. Cut slices should be at least 4–5 mm thick; it is better to cut a thicker slice, than to cut a slice too thin to run on the thin section machine. Ideally, two sample slices should be cut from every block for backup in the thin-sectioning process.

5. A small notch should be cut on the top of each sample slice to indicate which side of the slice is up in section.
6. The cut block and its sample slices are laid upright on newspaper within the fume cupboard to dry. When completely dry, the sample slice needed for thin sectioning is selected, and the face crossed and labelled with a permanent marker. The block is re-marked if needed and put in a sealed bag with site designations.
7. When sawing is completed, care is taken to hose out residual soil and resin. The sawing sled and Perspex guards are washed and wiped down, and the main switches for the saw are turned off.
8. All protective clothing worn while sawing should be carefully cleaned. If the rubber gloves and plastic sleeves used are in good condition, they can be rinsed with water and hung to dry for next use. Cotton masks and plastic aprons should be thrown away and new ones should be used each time. Ear guards and eye goggles or glasses for sawing must be safely stored away for next use in the laboratory.

Thin section grinding of soil samples

The slices obtained from the cutting are then mounted on microscopy slides and ground to the required thickness using a grinding machine (Fig. 7.6). The number of samples that can be processed at any one time depends on the size of thin sections being made, the grinding equipment, and the quality of impregnation. This description illustrates the key steps of processing large (or mammoth) size slides (c. 13 × 5 cm) using a Brot grinding machine.

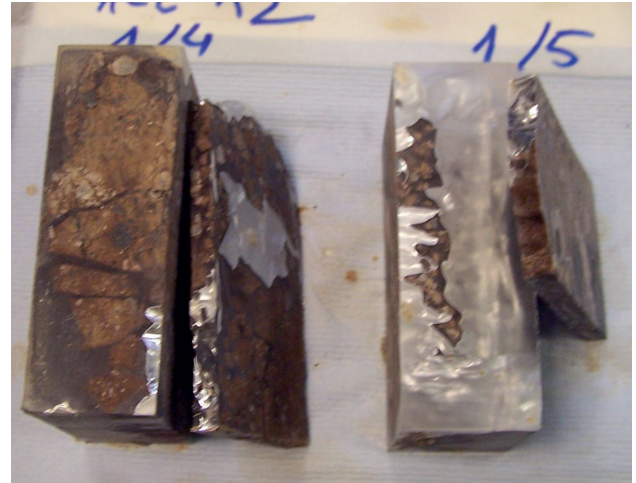


Figure 7.5. Sawing: left, a. the electric saw used to cut impregnated blocks; right, slides of 4–5mm thickness cut, the top side is marked with a notch. Images: Tonko Rajkovaca.

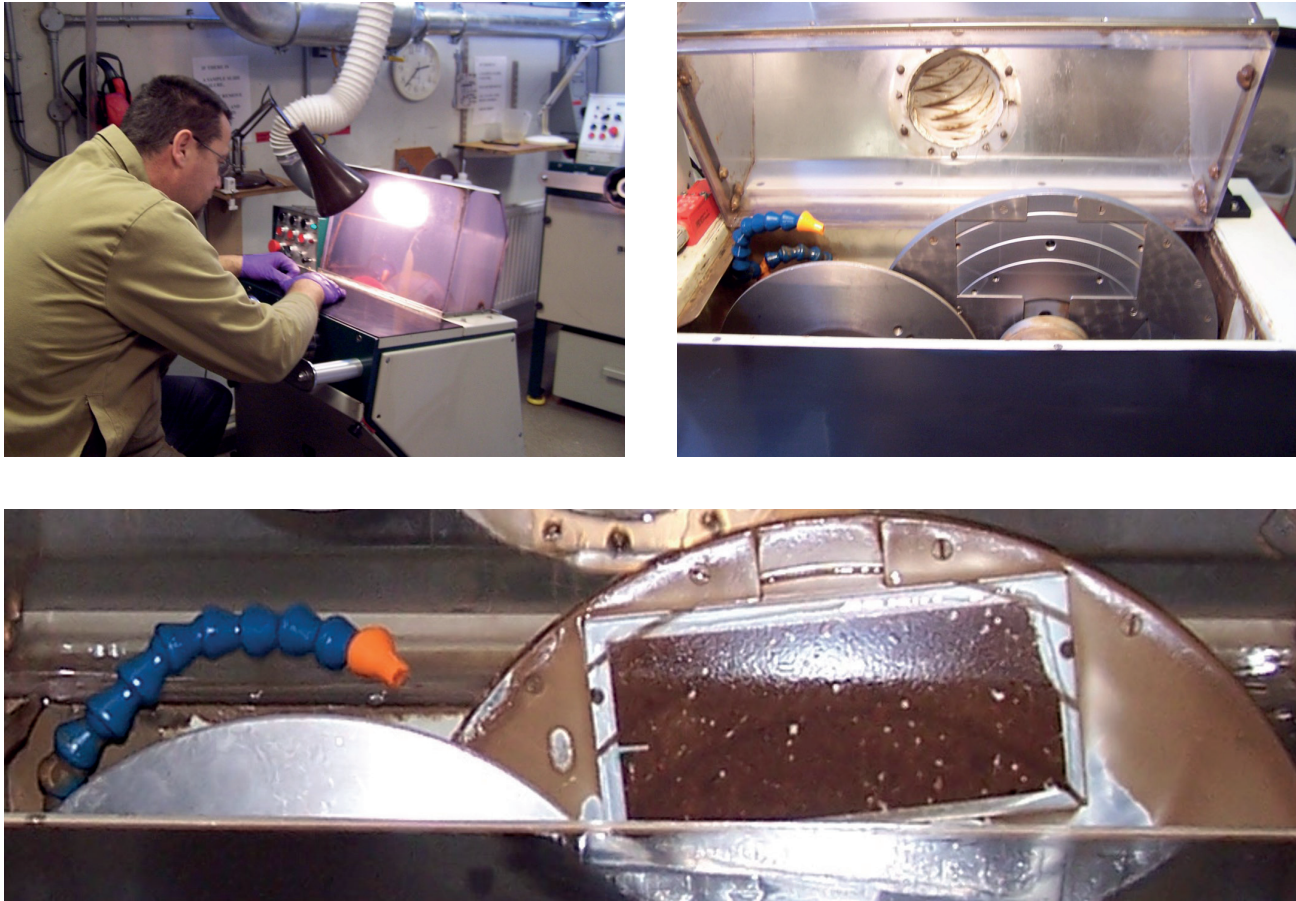


Figure 7.6. Thin sectioning using a Brot machine: top left, the author monitoring the grinding and, right, the wheel with slots to hold glass slides; bottom, slide mounted on the slot. Images: Tonko Rajkovaca.

1. Sample slices are selected in groups of three (the number of slots on the mounting head of the thin section machine), usually of the same size and width.
2. The slices are fixed to face outwards on coarse-ground glass slides with 'super-glue' gel. A small drop of 'super glue' in each corner is sufficient to hold the sample through the temporary grinding/polishing of the face.
3. The machine is turned on at the wall and set to manual. In this setting, the mounting head should be rotated and wiped clean. Each slot should be squirted down with clean oil to remove dust and fine particles. The glued slices/slides are then placed on the mounting head, with the aid of oil squirted on the back of the glass slides. Each slice/slide should be pressed firmly into the slot and pushed up and down to check that it is held fast to the plate by the capillary vacuum of the oil.
4. The machine is set to automatic mode to perform a coarse wheel grinding process. As soon as all outward markings have been ground away on the slice faces, the machine is stopped. The grinding wheel is changed to a finer grade, and the machine is set to proceed grinding until a fine finish/polish is accomplished on all the slices.
5. The machine is set back to manual, and the slices/slides are taken off the machine and set face down on a tissue to dry. They are then removed from the glass slide with a palette knife or Stanley blade, and acetone if necessary to soften the 'super glue'.
6. All oil from machining is wiped from the slices with lab paper towel, and they are re-labelled on the rough face. They are then placed on a drying rack, and a cool/warm hairdryer is used to blow remnant oil out of the slices. Periodic cleaning of the slices with acetone helps lift the oil during the drying-out process.
7. As soon as most oil has been removed from the slices, they can be permanently mounted to previously prepared, finely polished glass slides. The mounting mixture is 20 ml of polyester crystic

resin, with 0.7 ml of MEKP. Special care must be taken to wipe the thin section face clean with acetone, in order to remove any remnant oil or dust before applying the resin. Resin is poured in small amounts onto the slice face and spread thinly and evenly with a wooden mixing stick. A clean glass slide is then placed, polished face to sample, on top of the resin slice laid on the press. Pressure is applied on the press, and the slices are allowed to set and cure on the glass slides over a 24-hour period.

8. The slices/slides are then taken from the press, and remnant resin cleaned off the glass back and sides with a Stanley blade, acetone and paper towels.
9. The permanently mounted slices/slides are put in their original order back onto the sectioning machine and the same process of coarse and fine

grinding takes place to achieve finished 25–30 micron-width thin sections.

10. The thin sections are then taken from the machine and all oil is wiped away. Hand-finishing may be needed to achieve the right overall micron thickness for microscope analysis. Silicon carbon sandpaper of assorted grades can be used with some oil to obtain a finished section.
11. The finished thin sections are thoroughly cleaned with acetone, and a glass cover slip is applied with the same resin mix used for permanent mounting. A spray cover might also be applied to seal the finished thin sections.
12. Special care should be taken to store thin sections properly, either in sealed plastic containers, or stored in a laboratory reference drawer in foam slots. Periodically, slides should be maintained and cleaned with acetone.

Inspired geoarchaeologies

Geoarchaeological research captures dimensions of the past at an unprecedented level of detail and multiple spatial and temporal scales. The record of the past held by soils and sediments is an archive for past environments, climate change, resource use, settlement lifeways, and societal development and resilience over time. When the McDonald Institute was established at Cambridge, geoarchaeology was one of the priority fields for a new research and teaching environment. An opportunity to develop the legacy of Charles McBurney was bestowed upon Charles French, whose 'geoarchaeology in action' approach has had an enormous impact in advancing knowledge, principles and practices across academic, teaching and professional sectors. Many journeys that began at Cambridge have since proliferated into dozens of inspired geoarchaeologies worldwide. This volume presents research and reflection from across the globe by colleagues in tribute to Charly, under whose leadership the Charles McBurney Laboratory became a beacon of geoarchaeology.

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