International Journal of Coal Geology xxx (xxxx) xxx–xxx

Contents lists available at ScienceDirect

International Journal of Coal Geology

journal homepage: www.elsevier.com/locate/coal

# A dry polishing technique for the petrographic examination of mudrocks

Olga Gorbanenko

*University of Oxford, Department of Earth Sciences, South Parks Road, OX1 3AN Oxford, UK*

ARTICLE INFO

*Keywords:*
Dry polishing
Mudrocks
SEM
Optical microscopy
Clay minerals

ABSTRACT

A method of high-quality dry polishing is presented here, in which lubricating agents such as water, isopropyl alcohol, oils and water-based diamond suspensions are eliminated. This method has been developed to avoid various problems that arise through wet-polishing techniques, and to meet the quality requirements for high-resolution microscopy and SEM analysis. The technique has been tested on two epoxy embedded blocks using abrasive silicon carbide papers ranging from 240 (avg. grain size 58.5 μm, Struers) to 2500 grit (avg. grain size 8 μm, Struers), but with different final polishing stages. Both methods show comparable high-quality results, with no optical relief or thermal alteration of embedded organic matter. Such methods could be applied to other rock-types that might be sensitive to the use of any kind of lubricant. The dry-polishing technique enables the high-resolution study of clay minerals and fine-grained sediments using optical and scanning electron microscopy (SEM).

## 1. Introduction

Recent advances in microscopic analysis enable the study of sediment morphology, texture and the sedimentological fabric of clay minerals in great detail. Increasing the resolution of clay mineral analyses, however, also imposes a high requirement on polishing techniques. Standard wet-polishing techniques can typically produce samples with an irregular concave surface by washing away clay minerals. This method naturally affects the results of quantitative analysis of clay mineral types and complicates optical investigation. Use of the dry polishing method described here helps produce a finely polished surface in mudrocks and increases the quality of obtained results.

The dry polishing method was briefly described by Simpson (1979), who compared the polishing quality produced by dry and wet methods. The description of this method, however, appears to be insufficient for reaching the finest polishing quality and only involved a manual use of 240 grit (avg. grain size 58.5 μm, Struers), 600 grit (avg. grain size 26 μm, Struers) silicon carbide papers and 1 μm aluminium oxide powder (Struers) at a final polishing stage. In this paper, a new detailed protocol of dry polishing for organic-rich mudstones is introduced, which has been adapted for use with an automatic polishing machine with a sample holder attachment. In addition, two methods of polishing are presented, using aluminium oxide powder and a diamond spray as abrasive media. The first method is suitable for optical microscopy, whereas the second is appropriate for scanning electron microscopy (SEM) use since diamonds do not contribute to determination of clay mineral composition. These methods produce a completely flat surface on block samples of mudrocks and can also be applied to other rock-types such as organic-rich black shales that are sensitive to lubricants and which require high-quality polishing.

## 2. Experimental

Two mudrock samples used to test the new dry polishing technique were prepared as described in the following sections.

### 2.1. Sample mounting and preparation for grinding/polishing

Whole-rock samples (3 × 3 cm) were embedded in resin in an orientation perpendicular to the bedding plane (Littke et al., 2012). Subsequently, samples were embedded in a mixture of Epofix$^{tm}$ epoxy resin and Epofix$^{tm}$ hardener mixed at the ratio of 15:2 and allowed to harden at room temperature (~21 °C) for approximately 9 h. For embedding, standard round-shaped mould forms were used, which usually have a thickness (~2 cm) of about half its diameter (~4 cm). The edges of each embedded sample block were rounded using a coarse grinding disc in order to minimize the damage to grinding and polishing papers (Simpson, 1979). In addition, the surfaces of the block were aligned parallel to each other in order to obtain an accurate high-quality polish. The samples were then polished dry with consecutively finer abrasive papers until the surface was flat and showed no optical relief.

*E-mail address:* olga.gorbanenko@earth.ox.ac.uk.

http://dx.doi.org/10.1016/j.coal.2017.03.013
Received 23 January 2017; Received in revised form 10 March 2017; Accepted 21 March 2017

### 2.2. Grinding protocol

To obtain a high-quality polished finish on each sample block, an automated Struers Lapo Pol 35 polishing machine was used. Prior to each polishing stage, the sample block was cleaned using a combination of compressed air applied laterally (to avoid generating additional surface scratches) and tissue papers. In addition, the surface of each sample block was inspected with a microscope between the steps to make sure that scratches made during preceding steps were removed (Taggart, 1977).

After sample mounting, the surface of the sample was exposed by manually grinding away the resin on a diamond wheel. To avoid an increase in heat and, as a consequence, possible maturation of the sample, the contact time of sample and grinding disc did not exceed 5 s (Simpson, 1979).

The occurrence of microscopic surface scratches was progressively reduced by polishing with 240 grit (avg. grain size 58.5 μm, Struers) silicon carbide paper for 3 min, under an applied pressure of 2.5 N and a speed of 200 rpm. This step was repeated 3–4 times until the surface became homogeneous, and was subsequently followed by grinding with 400 (avg. grain size 35 μm, Struers), 800 (avg. grain size 22 μm, Struers), 1200 (avg. grain size 15 μm, Struers) and 2500 grit (avg. grain size 8 μm, Struers) abrasive silicon carbide papers on the same polishing machine, using identical speeds and applied pressures (see Table 1, Fig. 1–2).

The 1200-grit (avg. grain size 15 μm, Struers) and 2500-grit (avg. grain size 8 μm, Struers) abrasive papers required more polishing time, with variable levels of applied pressure depending on the sample, across a range of 2.5–5 N. The highest pressures were applied to the harder samples, and the lowest to those with the highest proportion of clays, those containing significant amounts of pyrite crystals and framboids, and those containing oil, for which viscosity decreases under increased temperature. When no more scratches were visible under the microscope at 100 × magnification, the sample was polished using a Chem-MD polishing cloth with two different abrasives (see Table 1, Fig. 1–2).

### 2.3. Polishing protocol

When polishing either using alumina or diamond spray as an abrasive medium, the pressure applied to the sample during the polishing stage varied depending on rock hardness (see Table 1). Polishing time ranged from 30 s to 2 min, in all cases avoiding overheating of the sample. This polishing procedure was repeated as many times as necessary until the treated surface showed no optical relief under 500 × magnification.

When polishing the samples with alumina (0.3 μm), it was only necessary to use a few grams of the abrasive, which, however, had to be evenly distributed over the lap at the beginning of the exercise. In addition, it was also necessary to wipe the alumina off the surface and the sides of the sample, to prevent the formation of a stubborn mineral and aluminium oxide coating that would be difficult to remove without resorting to a previous, coarser grinding stage (Simpson, 1979). The final stage was a brief polish with a clean Chem-MD polishing cloth, which helped to remove most of the alumina and produced a fine glossy polished surface (Fig. 1F). This polishing method worked well for samples intended for optical microscopy, but the use of alumina could potentially affect the results of elemental analysis of clays by SEM. In this case, a diamond spray (1 μm, Struers) is recommended, which should be first distributed over the polishing lap and left for a few minutes to dry. When polishing, minimum pressure (0.5 N) and time interval were required to obtain a high-gloss surface and to prevent the sample from overheating. This method also worked well for samples that contained a high proportion of bitumen (Fig. 2 F). The final polished surface of the sample, using both alumina and diamond spray, remained flat and polished with no trace of grain-related relief (Figs. 1F–2F).

**Table 1**
Schematic protocol describing the dry polishing technique.

| Stage[a] | Materials [grit] | | Speed [rpm] | Pressure [N] | The time limit[b] [min] | An average total time[c] [min] |
|---|---|---|---|---|---|---|
| Grinding | Silicon carbide papers | 240 | 200 | 2.5 | 3 | 5 |
| | | 400 | | 2.5 | 3 | 15 |
| | | 800 | | 2.5 | 3 | 20 |
| | | 1200 | | 2.5–5 | 3–2 | 45–60 |
| | | 2500 | | 2.5 | 3–2 | 60 |
| Polishing | Chem-MD | Alumina (9416) | 200 | 2.5–0.5 | 2–30 s | 60 |
| | | Diamond spray (9545) | | | 1–30s | 30 |

Note: the time may vary depending on the mineral composition of the sample.
[a] Cleaning: after every grinding/polishing session, gently, with the soft paper tissue. More attention should be paid to the borders of the sample.
[b] A maximum countinous polishing time, during which the sample is on the lap.
[c] An approximate total time spent at the grinding/polishing stage prior moving on to the next stage.

## 3. Discussion

Although the dry polishing technique outlined here is ideally suited to circumvent the washing away of clay minerals, two problems might be associated with this method of mudrock preparation for petrographic and SEM analyses. Firstly, the generation of heat during sample grinding may increase oil mobility and affect determination of organic-matter maturity. The polishing cloth (Chem-MD) used at the final stage of the sample preparation can further exacerbate this problem. To avoid this difficulty, it is necessary to rest the sample for at least 1 min between stages to allow it to cool fully. In addition, the presence of trapped oil in the samples, whose mobility increases with temperature, may act as a lubricant and affect the final polish quality by altering the optical relief.

A second problem that may influence the polishing quality and complicate the polishing procedure of the sample relates to a high proportion of pyrite, which is not uncommon in organic-rich mudrocks. Pyrite crystals disintegrate under the pressure applied during the procedure, and can create deep scratches which are difficult to remove (Fig. 1E). These effects can be reduced by polishing under low (about 0.5 N) pressure, at a speed of 250 rpm for 30 s and repeated until the scratches are eliminated.

Although it might be suspected that dry polishing would produce local heating and affect the maturity of the sample, Simpson's (1979) results indicate that this is not the case. His comparison of both dry and wet polishing techniques on a shale sample, with known vitrinite reflectance, showed no increase in values in the former case. In addition to that, the melting point of the Epofix resin is 40 °C/104 °F, which is lower than the temperature of onset of the oil window (VRo = 0.5%, 65 °C). Here, the considerable vertical pressure, under which the sedimentary rock had been formed, is not taken into consideration. Although there is no temperature alteration of the organic matter in mature samples, when suggested maximum continuous polishing time is not exceeded, the effect of the temperature on the immature samples, when polished dry, requires further investigation.

## 4. Conclusions

Wet polishing can adversely affect the optical relief of rock samples intended for optical microscopy or SEM analysis, by washing away clay minerals. The dry preparation method outlined in this paper helps to circumvent these issues. Mudrock samples prepared using these tech-