Application Note

Coal Thin & Ultra Thin Section Preparation

1. Introduction

High quality thin and 'ultra thin' sections provide an increasingly significant tool for sample investigation where the quality of the thin section is crucial to the results achieved during analysis. Analytical techniques have significantly improved in the last decade and this data sheet provides an overview of high quality thin and ultra-thin section preparatio, producing results similar to the analytical techniques used in today's modern laboratory.

2. Processing

A. Impregnation

When processing a thin section of coal, it is important to protect its structure from potential damage during all process stages. To achieve this, the coal is initially dried at 40°C for a 24hr period.

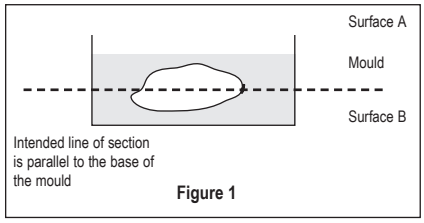

Figure 1

The sample is then placed in an impregnation mould and using an appropriate fixative such as double-sided adhesive, the coal is positioned on the mould base in an orientation appropriate to the line of sectioning needed. Fig 1 illustrates this process.

The sample is then impregnated using a Logitech IU30 Vacuum Impregnation Unit and Epoxy Pack 301 resin.


Logitech IU30 Impregnation Unit

B. Mounting

Following impregnation and curing, the coal is removed from the mould and the bottom face is temporarily bonded to a glass substrate using Quartz Wax.

Quartz Wax is a high shear strength, solvent soluble wax ideally suited to 'temporary bonding' of samples to microscope slides.

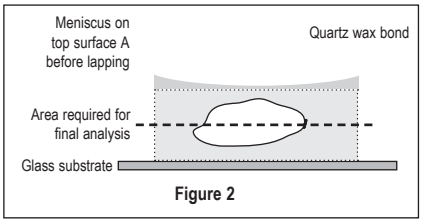

Figure 2

At this stage, geometric control is not of prime importance so this initial bond may be created by hand.

C. Meniscus Lapping

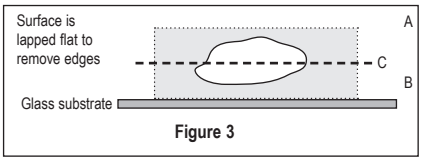

Figure 3

To ensure adequate thickness uniformity in the final sample it is necessary to flat lap the first face of the impregnated block, to remove the meniscus created after impregnation. This is carried out on a PP5GT Precision Polishing Jig, the glass substrate being retained on the vacuum chuckface of the jig. The sample is then lapped on a cast iron lapping plate with a slurry of calcined aluminium oxide lapping powder.


Logitech PM5 Lapping & Polishing System

The time taken to produce a suitable quality, flat surface will depend upon the volume of impregnating resin to be removed and the particle size of abrasive used. Thirty minutes is an average duration of this process.

D. Cutting the sample

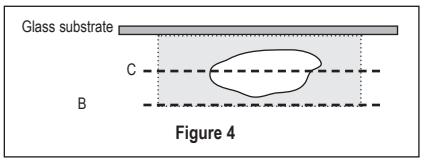

Figure 4

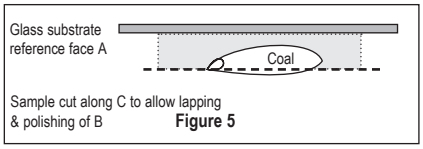

Figure 5

To retain the flatness and rigidity of the block a further glass slide is then bonded to the lapped face of the block and the original glass slide removed by either cutting with an Annular Saw, dissolving in a suitable solvent (Ecoclear) or by heating to melt the wax (Fig 4).

When removing by heating the wax, care has to be taken that excessive heat is not employed which will create expansion problems between the coal and resin, resulting in a weakened bond at a later stage.

An annular saw such as the APD1 or APD2 is now used to cut through the impregnated sample along C, so exposing the area required for first face lapping (Fig 5).

E. Sample First Face Lapping & Polishing

The newly-exposed surface of the sample is then lapped and, particularly if an ultra-thin section is to be prduced, polished (Fig 6). This allows the sample to be accurately mounted for the remaining lapping and polishing process.

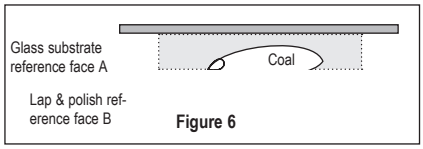

Figure 6

F. "Pip" Bonding of Coal

When using a "zero-bond" technique to prepare a thin section, the lapping & polishing machines use the back of the (glass) substrate as its reference plane. To be successful, this method requires that the sample bond be of known dimensions and that the back surface of the substrate be parallel to the back surface of the sample. However, impregnated specimens often exhibit substantial differences in relief and distortion between the specimen material (low relief) and the impregnating medium (high relief). The stages of this process are detailed below.

1. Firstly, select a microscope slide and roughly gauge its thickness with a micrometer.

2. Take the lapped and polished sample, place it face up, and position the microscope slide on top of it (Fig 7).

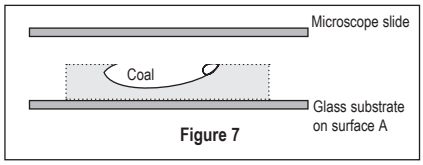

Figure 7

3. Mark the required alignment on the reverse of the substrate (Fig 8).

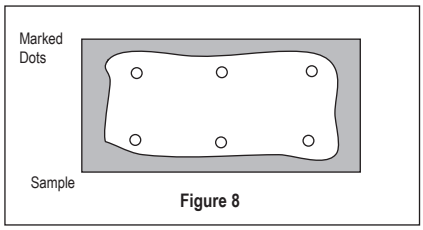

Figure 8

4. Apply UV resin over the marked dots. The created pips will cure in 1min under UV light.

5. Lap the pips to between 50 and 80 microns in height to create an even raised surface. This also ensures that the sample does not sit directly on the glass substrate.

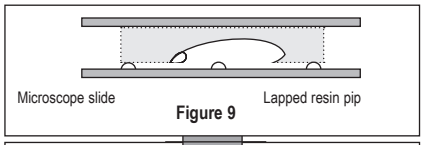

Figure 9

6. Apply a small quantity of adhesive between the pips on the microscope slide. Place the sample on top of the slide, taking care to eliminate bubbles (Fig 9).

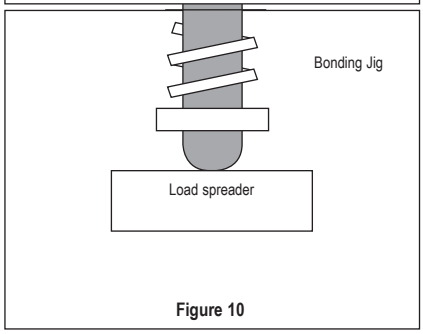

Figure 10

The adhesive selected must not be a type which contracts during curing.

7. Epoxy resin, such as Epoxypack 301,is very effective for permanent bonding. Bonding should take place as soon as possible after polishing.

8. Apply sufficient pressure so that the specimen fully contacts all the pips simultaneously. *Note that excessive pressure should be avoided to prevent distortion of the specimen.*

9. The sample is then placed in a light load bonding jig to set (Fig 10).

10. Allow the adhesive layer to cure.

G. Final Trimming, Lapping & Polishing

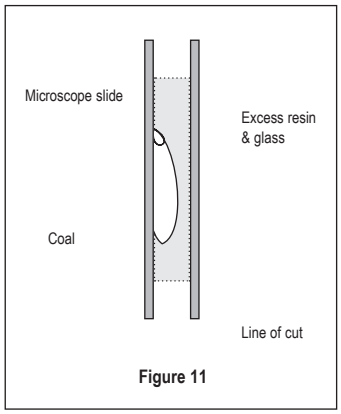

Figure 11

The excess resin and glass is trimmed off using an Annular Saw, such as the APD1 or APD2 (Fig 11).

The final stage in the preparation of thin coal sections is the thinning down of the sample to the thickness required. This is achieved by a repetition of the process used for the first face, as described in "Cutting the Sample" and "First Face Lapping and Polishing" above.The sample should be lapped to a thickness of 30 microns and polishing to 10 microns or less (Fig 12).

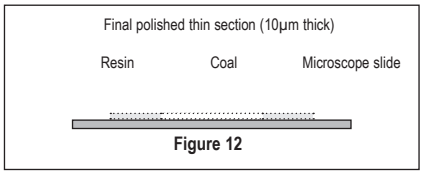

Figure 12

Certain applications, particularly those involving ultra-thin sections, will subsequently require a polishing process to be applied to the sample benefitting those customers requiring particularly high clarity for microscopic analysis. The PM5 machine provides the ideal means to achieve optimal lapping and polishing results. A polishing pad and a suitable particle size of diamond polishing slurry support the polishing process, whilst lapping is carried out on a cast iron plate, using a slurry of Aluminium Oxide powder in Ethane Diol carrier fluid.

3. System Specification

The system is based on a PM5 Precision Lapping and Polishing Machine. The PP5GT Precision Polishing Jig is the main sample holding fixture, and a APD Precision Saw is used for the cutting operations. Sample Impregnation is carried out by the IU30 Vacuum Impregnation Unit.


LogitechAPD1 Annular & Peripheral Saw

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