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British Geological Survey, Keyworth, Nottingham NG12 5GG — CLAIRE R. HALLSWORTH

Royal Museum of Scotland, Chambers Street, Edinburgh EH1 1JF, UK — ALEC LIVINGSTONE

British Geological Survey, Keyworth, Nottingham NG12 5GG — ANDREW C. MORTON

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The preparation of double-polished fluid inclusion wafers from friable, water-sensitive material

IN a study of sediment diagenesis, polished wafers for fluid inclusion analysis must: 1, have a high optical quality; 2, be very thin (i.e. less than 100 μm); 3, be processed under low-temperature conditions to prevent inclusion stretching and leakage; 4, maintain coherency in order to differentiate between cements and detrital grains.

The tried and tested methods of wafer preparation (Shepherd *et al.*, 1985; Roedder, 1984, p. 155) are in general not suitable for the preparation of polished wafers from clastic sedimentary rocks, especially those with a friable and/or water soluble nature. In the case of sandstones where fluid-inclusion data are required on the diagenetic cements, very thin wafers (50 μm and less) are required to overcome the problems of darkening and 'clouding' caused by sample grain size and diagenetic crystal size. This is illustrated in Fig. 1, where it can be seen that very small grains and crystals have not come into contact with the wafer edges and have therefore not been polished. The surface characteristics of the grains are preserved, which makes the detection of all other features, including any cement and fluid inclusions, difficult.

In polishing to an extremely thin wafer size, it has been discovered that many sedimentary samples, even those with a diagenetic cement, are too friable and tend to disintegrate. The problem can be traced to the cement/wax that bonds the sample to the glass plate for polishing. Several cements were tested and a brand of 'superglue' (Bostik[tm] Superglue 4) gave the desired product, i.e. thin, coherent wafers approximately 1–3 centimetres in size. Under magnification, some improvement in cement (and inclusion) observation was clearly achieved with increased thinning of the wafer (Fig. 2). The whole procedure, used at Imperial College for samples of Rotliegend Sandstone from the Southern North Sea, is described below. In these rocks some of the cements, e.g. halite and to a lesser extent, anhydrite, are regarded as water-soluble.

Impregnation and encapsulation

The required section is broken from the sample, or cut using an oil-lubricated diamond saw. A cut section must be cleaned thoroughly with

water-reduced alcohol to remove all traces of oil. To help maintain sample coherency, the dry section is vacuum impregnated using Araldite[tm] Epoxy Resin AY.105 and Hardener HY.932 mixed in a ratio 100:27 by weight. The mixture can be thinned with acetone or dyed if required by the addition of Sudan Blue (0.5%). The vacuum oven is pumped down and the resin is drawn into the pores during air evacuation. After approximately 4 hours, the vacuum is released and the sample removed from its container with any residual resin. It is then placed on aluminium foil to cure for 24 hours at 30–55 °C. The cured and impregnated section is labelled and placed in a Struers[tm] 30 millimetre polyethylene mould and encapsulated using a cold-setting clear polyester casting resin (Bonda[tm] Clear Casting Resin).

Preparing wafer face A

The polyester cast is removed from the mould and ground on both sides using an oil-lubricated diamond grinder to expose the sample. The purpose of this is to smooth the faces of the polyester cast, which are uneven and irregular, before further processing. The use of oil as opposed to water is necessary to prevent the dissolution of some minerals.

One exposed sample face is then flattened by hand, using a glass plate, silicon carbide and thin oil. Initially, 3F carbide grit is used for this process followed by 1000F grit. The polyester cast is cleaned thoroughly with alcohol after each stage in an ultrasonic cleaning bath.

The sample is then polished using an Engis[tm] polishing machine with a met cloth and 1/5 μm diamond paste.

Preparing wafer face B

The clean, oil-free polished face A is mounted onto a frosted glass slide using a cyanoacrylate bonding medium (Bostik[tm] Superglue 4). This glue is ideal as it prevents wafer disintegration in the final polishing stages and it is similar to some conventional mounting waxes, in that it can be set at low temperatures (less than 50 °C) to prevent inclusions stretching during processing.

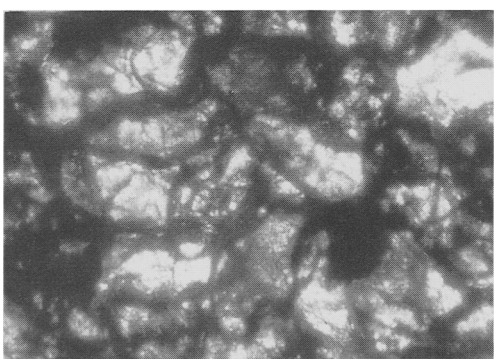
The mounted block is trimmed on an oil-lubricated diamond saw to leave a section 2–3 millimetres thick, and then ground to 100 μm on an oil-lubricated diamond grinder. The section is then taken down to the required thickness (50 μm or less) using a glass plate, 3F–1000F silicon carbide and thin oil, and polished as before.

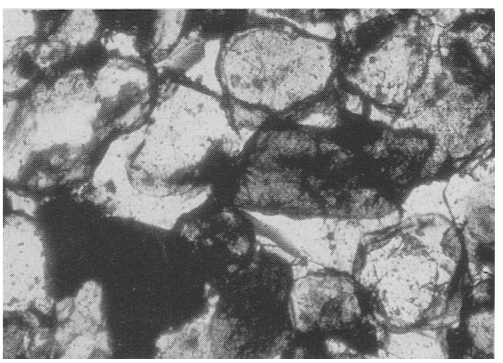
Removal and cleaning of the wafer

The section is soaked overnight in acetone in order to remove the cyanoacrylate bonding medium (superglue). The acetone does not significantly affect the impregnating resin used to help hold sample grains together; only a slight etching occurs but the effects can be severe if the acetone is in contact with the resin for a long period of time. An ultrasonic vibrator can be used to speed up this separation process, but as the wafers are less than 50 μm thick, they can break up with little effort. When the wafer is free the acetone is poured off gently. The wafer is washed in alcohol and then carefully removed and dried.

Conclusions

The above procedure fulfils the wafer requirements outlined at the beginning of this discussion, but optimum results are achieved in samples with





FIGS. 1 and 2. FIG. 1. (*left*). Double-polished wafer (>50 μm thick) produced using the original preparation method. FIG. 2 (*right*). Double-polished wafer (<50 μm thick) produced using the improved method (1 cm = 30 μm).

the grain sizes greater than 30 μm. Samples with grain sizes less than 30 μm are difficult to enhance even if very thin wafers are produced (warping is a serious problem with thin wafers). These samples will be of little importance to fluid inclusion studies as the cements, if present, will contain very few inclusions with a size (greater than 5 μm) that enables accurate microthermometric analyses to be made. It should also be remembered that when polishing to a wafer thickness of less than 50 μm, the cement in the sample will be clear but there is a greater chance that some large inclusions will be polished away (Roedder, 1984).

Safety procedures

Established laboratory safety procedures should be adhered to when using all of the materials mentioned especially when handling the resins. Araldite[tm] resin and hardeners in particular are irritants and should not be used without a fume cupboard due to inhalation restrictions. Araldite[tm] is also a carcinogen when heated to decomposition.

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Department of Geology, Imperial College, London SW7 2BP

B. MCNEIL
E. MORRIS