

A NOVEL METHOD OF MOUNTING MICRO SAMPLES FOR MANIPULATION AND ANALYSIS D. Johnson¹, P. Landsberg¹ and M. M. Grady^{1,2} ¹School of Physical Sciences, The Open University, Milton Keynes, MK7 6AA, UK, Diane.Johnson@open.ac.uk, ²Natural History Museum, Cromwell Road, London, SW7 5BD, UK.

Introduction: The micromanipulation and mounting of micron-sized grains is a specialist skill that requires training and patience. Over the years, as new instrumentation has enabled analysis of ever smaller particles, it has been recognized that there is a need to develop new and different methods for the manipulation of individual grains, methods that are keyed specifically to the types of analyses that are to be carried out.

Methods frequently used to manipulate micron-sized grains include use of electrostatic forces to keep the sample in position, mounting in epoxy resin or glue or crushing of the sample into a soft metallic substrate, but all have their drawbacks. Electrostatic forces are useful to transfer very small grains for short times, for example from one glass slide to another within a clean bench. Electrostatic forces lack precision and stability, and cannot be used as a stand-alone technique, for example if sample transfer to other facilities is needed. Samples > 30 μm in diameter are also too heavy to rely on electrostatic attraction to ‘stick’ to a needle.

Epoxy resin or glue is a universally-recognised and ideal technique for mounting of samples that require polishing for quantitative analysis. Unfortunately, though, three dimensional context is lost, as is information about any material adhering to the outside of the grain. Even more problematic, though, are the levels of contamination, especially organic contamination, associated with resin. It is practically impossible to remove all the resin from a mounted sample, so once mounted, the grain is not really suitable for trace-level studies of organic material.

If a sample is crushed into a substrate, such as indium, three-dimensional information is again lost and organic contamination levels increased. Of course, these techniques are essential for, e.g., SIMS analysis, and for spectroscopy studies if indigenous organic molecules are not the focus of the work. All of these traditional methods will invariably physically damage or contaminate small samples; if the sample is to be studied by others at a later date, then these issues are significant and undesirable.

Few, if any, modern studies have been performed specifically to develop and assess success of mounting and manipulation methods of these very small but precious materials. Yet with the forthcoming return of samples from the Hayabusa 2 and Osiris-Rex missions, as well as from missions to the Moon and Mars, a new grain handling technique may be required to enable the most science-rich analyses of the returned materials to

be performed. Our study aims to mount a silicate mineral test grain of 50 – 80 μm diameter to enable the sample to be analysed by several methods, such as SEM, Raman spectroscopy and micro X-ray CT, without requiring separate mounts for each technique. Our method uses liquid sulfur as an adhesive, which has been used as an encasing medium for IDPs [1].

Method: We have developed and are testing a novel method of attaching a grain onto the tip of a thin ceramic capillary tube, making use of melted high purity sulfur as the adhesive agent. Sulfur has been used previously to mount samples for TEM analysis [1], but, as far as we can find, has not been used to attach a grain to a multi-functional sample holder. Sulfur, when heated at an appropriate rate, melts at approximately 120 °C to form a low viscosity, pale yellow liquid, it solidifies rapidly if it makes contact with a cold surface.

Our technique involves melting a small quantity of reagent grade powdered sulfur (Sigma-Aldrich; particle size 100 mesh) on a glass slide heated by a small heat pad sitting on a microscope stage. The sulfur is heated until it becomes a clear yellow liquid. The tip of a borosilicate micropipette capillary tube (World Precision Instruments) held by a custom built adapter in a Narishige micromanipulator, is lowered to touch the liquid sulfur. A small quantity of liquid sulfur is drawn into the tube by capillary action (Figure 1). Several tubes can be prepared in one session for later use.

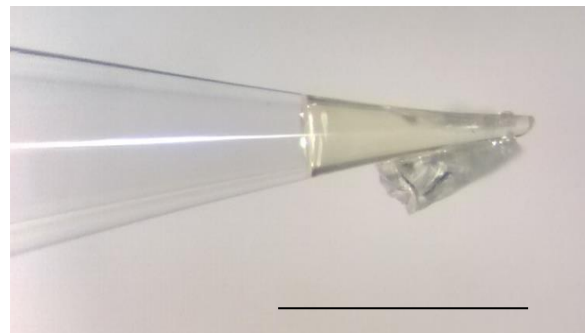


Figure 1. Optical image of capillary with test grain.

The grain to be mounted is placed on a glass slide to the side of the heat pad. The capillary is held by the manipulator just above the heat pad until a droplet of liquid sulfur forms at the capillary tip. The micromanipulator is used to position the tip directly above the grain. The sulphur is allowed to cool (a few seconds) before lifting the capillary away from the slide. We attempted the experiments using capillary tubes with in-

ternal diameters of 5 μm and 30 μm , the best results so far have been achieved with the 5 μm diameter tube. If the grain requires removal and separation from the capillary, gentle heating to melt and vaporize the sulfur bond is readily accomplished using the heat oad-micromanipulator set-up.

Optical and SEM images were taken of the grain mounted on its capillary tube. A specially designed holder was made for the capillary tube (Figure 2), to ensure good contact between holder and capillary as well as ensuring that the tube was stable. The holder acted as a base for storage and transport of the grain.

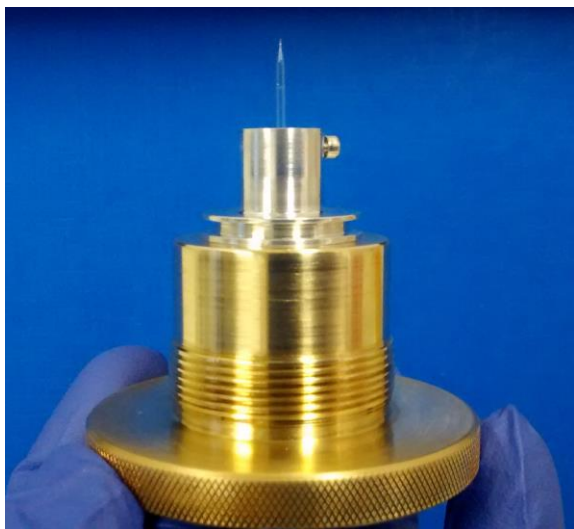


Figure 2: Aluminium capillary holder secured into a brass base holder onto which a plastic cover can be screwed, securing the grain and capillary.

Testing: Security of grain attachment to the capillary tube and the practicality of manipulating the tube was tested by SEM. The grain was not coated, and no further treatment was applied to either the grain or the capillary tube. An SEM image of a test grain on fixed to a capillary tube with sulfur can be seen in Figure 3. Secondary electron imaging proved to be stable at low accelerating voltages in an FEI Quanta 2003D FIBSEM at 2kV and beam current of 0.42nA. In the Zeiss Supra550 FEGSEM, stable images were achieved at 2kV, aperture 30, enabling resolution of features down to a 50 nm scale.

The interface between the grain and sulfur on the external wall of the capillary tube was examined by SEM (Figure 4). No obvious structural faults were identified and we have so far had a grain mounted by this method for approximately 6 weeks with no sign of the sulfur failing. Even when the sample was moved between different buildings, and subsequently between Milton Keynes and London, the grain remained attached to the capillary.

Further testing is required to determine the range of conditions that the sulfur (with its low volatilization temperature) will survive. For example, irradiation by laser for Raman spectroscopy, or a higher beam current in the SEM. It is not certain whether the sulfur would be stable when an SEM is operated in point analysis mode, as the sulfur interface may become if the beam is focused close to the particle-sulfur interface. We are conducting a series of tests to see how close the beam can be focused to the interface.

Conclusion: The resulting grain mounted capillary, while fragile at just 1mm diameter and approximately 3cm length, still requires care when handling but appears to be a viable safe option for small sample grain handling that also allows direct access to the majority of the grain surface for imaging and analysis.

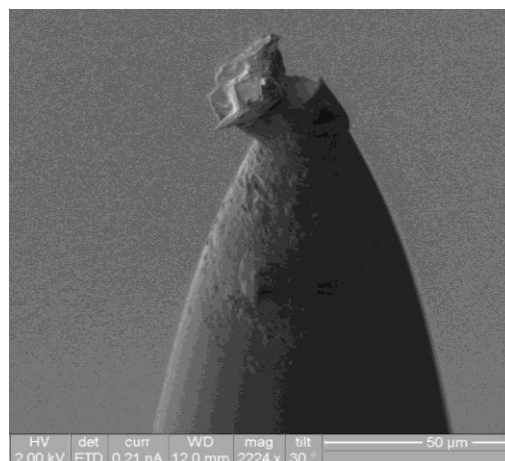


Figure 3: SEM secondary electron image of a test grain on the tip of the capillary tube.

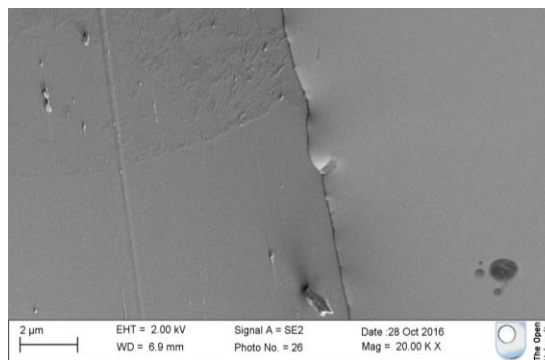


Figure 4: Secondary electron image showing the interface of the capillary (left) with the sulfur (right).

References: [1] Bradley J. P. et al. (1993) *LPSC XXIV*, 173-174.

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