

Preparation of Indium Mounts for SIMS Analysis.

Background:

For geological applications epoxy resins have been used routinely for the mounting of specimens and for many applications it's ideal. However, for certain analyses epoxy has major disadvantages such as high H, C, and S contamination as well as degrading the vacuum in the analysis chamber.

Previous studies have used low melting point alloys (e.g. Woods Metal) to mount specimens but they cannot produce the flat surface or the strongly bound specimens required. They also contain numerous elements (Bismuth, Lead, Tin and Cadmium) that may also compromise the analysis.

The Ion Microprobe Facility in Edinburgh (EIMF) has been using Indium mounts for over 20 years. Initially they were used for the mounting of diamonds but now they are frequently used to analyse any material where low backgrounds of H and C are needed or when any additional sample preparation would be detrimental to the work (e.g. surface diffusion studies) or when samples can be mounted unprepared (e.g. micro-fossils).

Why Indium?

Indium is commonly used in liquid crystal displays and because of this wide spread use the material has become readily available (e.g. [Alfa Aesar](#)) in high purity (e.g.99.999%) and at a reasonable price (~£4/g). The physical properties are listed in Table 1. Its hardness (1.2 Mohs Scale) makes it soft enough to enable samples to be pressed into the metal without fracturing or damage. In addition, samples can easily be recovered if another type of analysis (e.g. FTIR) is required. The toxicity is low requiring only basic handling precautions. A MSDS data sheet is linked below.

Table 1
Physical Properties of Indium

Melting Point	156.6 °C
Boiling Point	2072 °C
Hardness (Mohs Scale)	1.2
Density	7.31 g/cm ³

Preparation Procedure:

What is needed:

- Glass slide, Crystalbond or Lakeside, grinding and polishing materials. These are used for the pre-preparation of the samples.
- Ultrasonic bath, beakers, solvents (acetone, ethanol), de-ionised water, oven.
- Hot plate (up to 200°C). Used for the melting of indium, Lakeside and Crystalbond.
- Al-disk with blind hole for the indium metal to sit.
- Hydraulic press (~ 5 ton) for pushing the samples into the indium.
- Melinex sheet (~0.2mm thick). This is used to provide a barrier between the hardened steel plates of the hydraulic press and the indium. It produces a smooth surface to the Indium and prevents the indium 'sticking' to the hardened steel plates of the press.

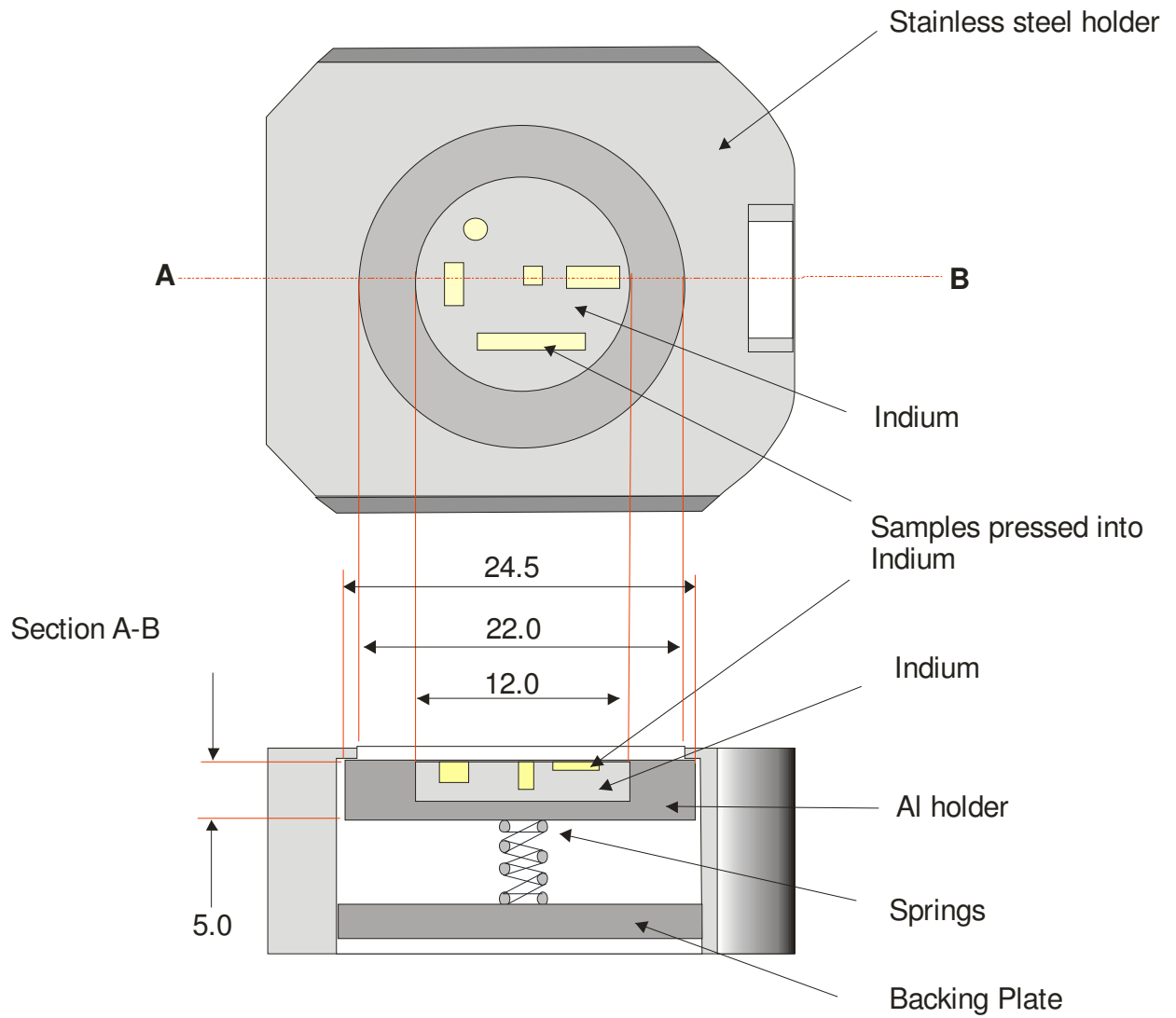
Suppliers of Materials:

1. Indium: Alfa Aesar (<http://www.alfa.com/>)
2. MSDS sheet from Alfa Aesar (<http://www.alfa.com/content/msds/english/00151.pdf>)
3. Melinex sheet: Katco, Performance Materials. (<http://www.katco.uk.com/>)
4. Hydraulic Press: Enerpack RSM50 (<http://uk.rs-online.com/web/>)
5. Lakeside: Lakeside Products (<http://www.lakeside-products.com/>)
6. Crystalbond: SPI supplies (www.2spi.com/) or (<http://www.crystalbond.com/>)

Sample and sample holder dimensions

Dimensions in mm

Polished samples pressed into Indium



Step 1: Prepare the material:

If the samples need to be polished they should be prepared using Crystalbond, Lakeside or an epoxy resin as the embedding medium. Once ground and polished the samples should be removed from the embedding medium, cleaned in acetone, washed with de-ionised water, then dried. The end product should be a polished flat surface with the samples free from their embedding medium.

Step 2: Holder Preparation Steps:



The Al-disk should be cleaned to remove any oils or machining lubricants. The quantity of indium required maybe calculated and then placed in the Al-well. Indium tear-drops can be seen in the bottom right of the picture.

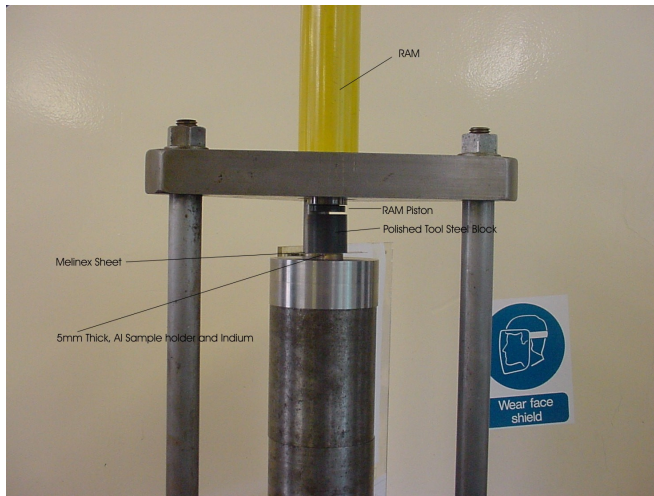


Alternatively, just eye-balling the correct amount of indium works. It maybe advantageous to put less indium in than needed as pressing of the sample can displace enough indium to make the a flat surface across the diameter of the mount.



The holder should be transferred to the hot plate set at $\sim 180^{\circ}\text{C}$ and the indium allowed to melt. Remove and allow to cool.

Step 3: Hydraulic Press:



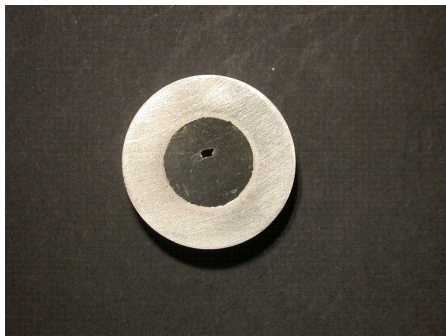
The indium now needs to be pressed into the Al-well. The sample surface is protected from the polished steel block by a sheet of Melinex. This ensures that the steel block does not bond to the surface of the indium and produces a clean scratch free surface. A 5Ton press provides enough pressure to force the indium into the Al-well and remove any trapped pockets of air that may behave as a virtual leaks.

Step 4: Sample insertion:



You should now have an aluminum holder with a central well of indium that has a smooth surface. At this stage small troughs can be gouged into the indium to make room for larger samples, so that on pressing the indium does not flow out of the well.

Ultrasonic clean the block in Acetone.



Gently push the sample into the indium and return the holder to the hydraulic press. Again, using the Melinex sheet compress the samples into the indium. The photograph shows a small fragment of a standard pressed into the indium.

Step 5: Cleaning the sample:

Place the sample in Acetone and allow the solvent to remove any grease. Do not use the ultrasonic bath as this dislodges the samples from the indium.

Remove the holder and wipe the surface with ethanol, de-ionised water and allow to dry. Gold coat the sample ready for analysis.