MILLBROOK
INSTRUMENTS LIMITED

C Samples Suitable for SIMS Analysis

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C.1 Types of Sample

Samples for analysis in the MiniSIMS must be solids (see below for the analysis of liquids in special circumstances).

Samples must be vacuum compatible i.e. they must not contain volatile solvents etc. that will degrade the vacuum in the instrument.

Samples that are electrically conducting (or very weakly insulating) across the surface to be analysed will in general give better results than insulating samples. Insulators cannot be analysed unless the MiniSIMS is fitted with the optional auxiliary electron gun.

For best results, samples should be flat with surface roughness of less than 0.5 mm.

C.2 Sample Handling and Preparation

The critical point to remember is that SIMS is extremely surface sensitive. Whilst this is the reason why the MiniSIMS can provide unique information relating to surface behaviour, the corollary is that surface contamination during handling and preparation will seriously affect, if not render meaningless, the analytical result.

Therefore, all possible care must be taken to ensure that surface contamination does not happen - starting with the education of sample providers. Samples should not be stored or transported in plastic containers, especially plastic bags, because these contain migratory molecules (additives). Even the polyethylene caps of glass bottles should be avoided. Loose wrapping in two layers of non-patterned domestic aluminium foil (dull side inside) is

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recommended. Where appropriate new glass vials with cork stoppers are an alternative. Plain paper envelopes are also usually free of contaminating material. Samples should of course never be directly labeled on the surface of interest.

Taking care with sample handling during loading does result in more consistent instrumental performance over the long-term. In particular:-

Sample stubs should be frequently cleaned using acetone or similar solvents, ideally before each use.

The sample "parking" tray in the sample hatch should similarly be cleaned regularly (e.g. once per week). The tray can be simply lifted out to make this task easier.

Discarded samples should not be allowed to accumulate in the sample parking tray.

Although not essential for a non-UHV instrument, wearing gloves when handling the sample stubs prevents the build up of contamination. Plastic gloves should be both powder and silicone free. If using cotton gloves, take care that no loose fibres are trapped on the sharp edges of the neutralization covers.

When handling the samples themselves, it is essential to avoid contact of the surface with skin. Tweezers should always be used, taking care to avoid making contact with any areas of the surface that will eventually be analyzed.

Thin metal samples can be cut with strong scissors or snips. For material thicker than ~ 0.5 mm a hacksaw will probably be required. Care must be taken to minimize the sticking of particles to the surface, but some is inevitable. This stray material can usually be removed by use of a puff duster (designed to clean camera lenses) or jet of gas from a compressed source. Aerosols are best avoided since many contain halide-based propellants which could leave residues on the surface.

Some semiconductor wafers or devices will fracture easily along the crystal planes, and if the wafer is complete the crystal orientation is often indicated by flats on the outer circumference. Other wafers may be cut to size by scribing straight lines on the sample and pressing gently along the direction of the scribe mark over a slightly raised edge (e.g. a metal rule or glass microscope slide). This operation can be carried out on the reverse side because fine particles are formed during cutting and these can be difficult to remove. However the side of interest will then be in contact with another surface and may become contaminated.

Liquid samples can be attempted by smearing a thin film on to a clean sample stub. The aim is to produce a monolayer thickness, because thicker films will give no greater signal intensity and are likely to be susceptible to charging. The volatile content should be removed (see below) before loading, so only non-volatile components of the original liquid will remain for analysis.

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Powdered samples are best avoided! Loose powder samples will fly about when the sample region is pumped out during sample transfer. Particles could penetrate the vacuum seals of the transfer mechanism or enter the pumps and cause permanent damage. Differential charging may also occur when the charged particle beam strikes the surface, leading to electrostatic repulsion between grains. Internal contamination of the secondary ion optics by powders will lead to a severe decrease in secondary ion collection efficiency. Internal cleaning of the instrument to rectify the problem is not covered under normal warranty and service contracts.

If powders must be analysed, and they cannot be compressed into a pellet, then it is essential to form a firmly compressed, uniform layer of powdered material with as flat a surface as possible. This is best achieved by putting the powder on to double sided conductive tape and pressing using a clean glass coverslide. Alternatively, for conducting materials, the powder can be mounted in indium foil; the "sandwich" method is recommended. Place a piece of foil about 10 mm x 5 mm on a flat surface and cover half the surface with the powder. Fold the foil to form a sandwich and press firmly on the outside. Gently peel open the sandwich and cut to size for mounting.

In either case, any loose powder should be removed by:-

Firmly tapping the edge of the stub on a hard surface to dislodge loose powder
Wiping any excess off the rim of the sample (outside the central analysis area)
Brushing the powder surface lightly with a clean tissue
Blowing with an airjet (compressed gas or lens cleaning bulb)

The above sequence may need to be repeated several times to ensure that all remaining particles of powder are firmly held on the tape or indium foil in a single layer.

C.3 Sample Mounting

Wearing disposable gloves is not essential for this stage but it is recommended to avoid contamination from the skin's natural oils.

For the standard sample stub size, the surface of the sample when mounted on the stub should be 5 mm above the base of the sample platen. For the upgraded sample transfer, these standard stubs are mounted in an adapter of 2 mm thickness. The equivalent height value for stubs designed for use without the adapter is therefore 7 mm.

At lower heights than this, the analysis performance will be progressively degraded. Higher than this, there is a danger of the sample catching and jamming in the instrument (although the arched shape of the sample entry point means that thicker samples of small diameter can be

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loaded with extreme care). For conducting samples, little difference in performance will be seen provided that the sample surface is within 2 mm of the correct height. For insulating samples the height is much more critical.

With conducting samples it is essential to provide good electrical contact between the sample surface and the sample stub. It is also necessary to ensure that the sample cannot be blown off the stub when the air is evacuated during sample transfer.

The sample can be secured in a number of ways, e.g. by the use of adhesive tape, conductive silver paint, molten indium or a metal clip. Prior to mounting a sample, the stub should be cleaned using a lint-free tissue soaked in acetone.

The sample can be fixed to the stub using a brand of double-sided adhesive tape which is low in volatiles and which does not involve a silicone release agent (often used to impregnate the backing paper). The minimum sized piece of tape should be used to avoid contaminating the vacuum unnecessarily. "Conducting" tape should be treated with scepticism unless it has been proved to work! Instead a thin strip of aluminium foil placed under and at right angles to the tape can be folded back over the top of tape before the sample is placed in position; this makes good electrical contact between the stub and the back of the sample.

For a conductive coating on an insulating substrate, the back of the sample is electrically isolated from the surface of interest. Placing a neutralization cover over the sample is one way to make a conductive path from the surface of a sample to the sample stub. Alternatively a very small drop of conductive paint (see below) can be used to make an electrical contact between the edge of the sample surface and the stub.

Conductive silver paint is also a means of bonding the back of a sample to the stub because it remains viscous long enough for the sample to be manoeuvred. Unfortunately, as the paint dries it loses volatile solvents. It cannot, therefore, be introduced into the instrument until this process is complete and often this can be inhibited because the paint is trapped in an irregularity in the underside of the sample. Drying off in a low temperature oven for 5-10 minutes is helpful (a glassware drying cabinet, at about 50 ºC, is ideal). In general the other methods of sample mounting are to be preferred, but if silver paint is used the amount should be kept to the absolute minimum.

Indium metal can also be used as a bonding agent. A small quantity can be melted on the stub, the sample rapidly brought into contact and the metal allowed to re-freeze.

If the sample surface to be analysed is insulating there is no advantage to having a conductive path from the sample surface to the sample stub. In many ways this makes sample mounting less complicated and the sample can simply be stuck down using vacuum compatible double-sided tape.

Samples with a volatile content (and especially those involving organics rather than water)

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should not be loaded directly and allowed to outgas in the sample chamber. This type of sample includes samples that have been freshly mounted using silver conductive paint. Heating and cooling of the sample prior to loading is advisable whenever possible. All such samples should then be allowed to outgas fully in the low vacuum region during loading.

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