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NENIMF: Specimen Requirements

Introduction

Any vacuum compatible materials including conductors (e.g. metals, alloys, sulfides), non-conductors (e.g. silicate minerals, ceramics, glasses), as well as different biological materials can be analyzed by SIMS. Samples must be flat, polished to within at least 1 um surface relief and be compatible with ultra-high vacuum ($10^{?8}$ to $10^{?9}$ Torr) of the sample chamber.

Sample changes on the Cameca SIMS instruments require a minimum of about 5 minutes, so it is feasible to analyze several sample mounts during a session. However, if concentrations of volatile components represent a main purpose of the analytical work, hydrogen and carbon background signals may take hours to decay to baseline conditions after sample change. Thus, it is beneficial to mount as many unknown samples as possible in each mount.

We emphasize that improper sample preparation has resulted in many wasted analytical hours. We strongly recommend that users discuss sample preparation with the facility management so that the NENIMF staff can help in preparing the mounts. In such a case, we recommend that specimens and relevant standards be sent to NENIMF at least two weeks in advance of the users' scheduled visit. The user fee will include time spent for the preparation and documentation of sample mounts if this is left solely to NENIMF staff. The user fee will not be charged if user comes to NENIMF in advance to prepare the samples.

Sample dimensions

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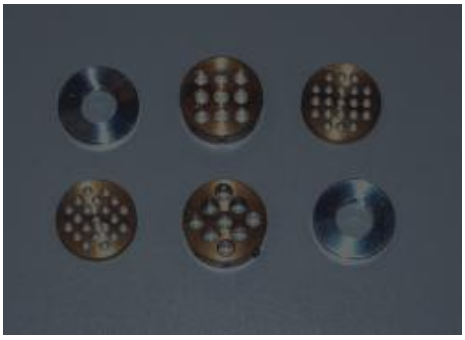
Cameca sample holders



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Sample mounts available in NENIMF

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1" diameter sample rings for grain mounts

The Cameca IMS 3f and 1270 accept the same specimen holder having 24.5-25.0 mm (~1") in diameter and less than 12 mm (~0.5") thick. Care must always be taken to place the specimens in the center of the disc, as secondary ion extraction is affected by the edge of the sample holder. Samples should be positioned within 8mm of the center to ensure access to the location within the machine and best possible transmission/consistency for all measurements.

Several adapters have been constructed so that a wider variety of samples can be accommodated into the Cameca holders:

- 24-25 mm (~1") diameter thin section, epoxy block or polished samples pressed into Indium;
- 24-25 mm (~1") outer diameter with 12.5 mm (~0.5") of exposed (usable) area;
- 5 mm (0.197") diameter tube mounts;
- 6mm Ta or stainless steel mask mount.

Our sample holders use samples mounted in 1" diameter rings and have 10mm, 14mm, 19mm and 24mm apertures.

Sample Preparation

Multiple samples can be mounted in a prepared disk of epoxy or onto a 1" diameter round glass slide (with minimal epoxy). Thin wafers (suitable for infrared spectroscopic analysis) can also be glued to a glass slide with a drop of "superglue" or pressed into double-sided (vacuum compatible) sticky tape.

All epoxy resins degas and will contribute to the H and C background in the instrument. They should therefore be used sparingly and solid grain mounts should be as thin as possible. Acrylic resins should not be used. Based on our experience, we recommend using Buehler Epo-Thin low viscosity epoxy resin and hardener or Struers EPOES Resin and EPOAR Hardener for sample preparation (for more detail see also: http://www.geos.ed.ac.uk/facilities/ionprobe/EpoxyResins/Results.html). For the lowest levels of detection, polished samples can be pressed into indium metal because the indium metal does not contribute to hydrogen and carbon background signal.

Note that residual polishing media, the rests of conducting coat deposited on the sample, inadvertent contamination left occasionally on the sample surface may negatively contribute to the quality of analytical data.

Insulating samples need to be coated with a thin layer of Au (10-30nm); they can also be easily carbon coated with a carbon evaporation accessory and carbon yarn or rod. However, we recommend that visitors consider the elements they want to study before final sample preparation. For example, applying a carbon coat would be clearly inappropriate for analysis of carbon in silicate glass.

Furthermore, polishing with Al2O3 suspension may cause problems in analysis for trace Al in silicates and oxides. One of the most serious contamination problems is observed for boron, as B contents of diamond polishing compounds are high. We would thus recommend the technique of sample preparation for analyses of B concentrations and isotopic composition given by Chaussidon et al. (1997, Geostand. Newsletter. 21: 7-17).

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