THIN FILM RESEARCH LABORATORY

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NEW SERVICES IN THIN FILM DEPOSITION AND MICROFABRICATION

The GCM extends its range of services by making available to the industry a whole series of instruments for thin film deposition and microfabrication.

For details, please consult: **www.gcmlab.ca/pdf/Thin_Film_Deposition.pdf**

SAMPLE PREPARATION : THE KEY FOR SUCCESSFUL ANALYSIS

In collaboration with Suzie Poulin, research associate, GCM

When planning an analysis, people often devote a lot of time to pick the right analytical technique, while neglecting sample preparation. Yet, for many analyses, good sample preparation is critical to get reliable and repeatable results. We will give in this newsletter several tips for careful sample preparation for various types of analysis.

Sample representativeness

Most analyses of offered by the GCM require small amounts of matter, in the range of a few grams or a few millilitres. Accordingly, samples sent for analysis often represent a small portion of the product or the batch under study. For a homogeneous product, this does not present any particular challenge. However, for an inhomogeneous product, one can either shake it to improve its uniformity in the case of liquids or powders, as long as it does not alter its properties, or run the analysis on several samples taken from the product or on several areas of the product in the case of solids.

Physical limitations

Before any analysis, it is a good practise to check the maximum sample dimensions acceptable by the instrument. Some apparatus, like Atomic Force Microscopes (AFM) or Fourier Transform Infrared Spectrometers (FTIR), can accommodate relatively large samples of a few tens of centimetres, while surface analysis instruments are limited to dimensions of 1 cm x 1 cm x 6 mm thick in the case of GCM'S XPS, or 8 cm x 6 cm x 2 cm for GCM's TOF-SIMS.

If the sample size must be reduced, we recommend keeping an area completely virgin, that is an area that has not been in contact with any other surface whatsoever. For instance, for an AFM measurement, it is possible to decrease the sample size to a few cm's while taking care not to affect the central area of 100 µm x 100 µm that will be used for the analysis. Each technique analyzes a different area, so it is recommended to discuss sample preparation beforehand.

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Sample preparation for mechanical measurements

For surface mechanical analysis like those offered by the GCM (microhardness, micro scratch, wear tests, etc), it generally suffices to avoid any contact with chemical reactants that might cause a surface modification, like an acid. An accidental contact with a low reactivity chemical or liquid (like water) will not normally influence the measurement quality.

Sample preparation for chemical analysis

Chemical measurements almost always call for rigorous sample preparation. The lower the concentration or the depth for the analysis, the more meticulous the handling has to be to minimize contamination risks. We recommend avoiding hand contact because finger grease increases the concentration of carbon, oxygen, sodium and calcium on the sample surface. This precaution is even more critical when the depth of the analysis is low such as is the case for surface analysis techniques (XPS and TOF-SIMS). It therefore matters to handle the sample with gloves without powder and with clean pliers to avoid any contact with the surface to analyze. Some techniques require that the samples be put in high vacuum; it is then necessary to check the vacuum compatibility of the sample.

Sample preparation for Scanning Electron Microscope (SEM) or Atomic Force Microscopy (AFM)

In general, recommendations for chemical analysis also apply to microscopy. For SEM, one must also check for vacuum compatibility of the sample, since many organic samples tend to degas in the SEM chamber. To avoid contamination, one can use a SEM with an environmental chamber.

Packaging

The ideal package is a container that does not affect the composition of the sample and that is insensitive to heat. For solid samples, we suggest aluminum paper (like the one used for cooking) because it rarely reacts with solids and it is remarkably stable in temperature. In general, we prohibit paper bags and plastic bags ("Ziploc" type) because this type of package, when exposed to heat, tends to stick to the sample and can then modify its chemical properties. Liquid samples and powders can be put in a closed plastic or glass container, keeping in mind that fluoroware leaves a fluor contamination detectable by XPS and TOF-SIMS for instance.

CHECKLIST FOR SAMPLE PREPARATION

- Is the sample size suitable for the analysis?
- What sample quantity is needed?
- Is the sample representative of my product?
- Is the analytical technique a vacuum technique? If yes, is my sample vacuum compatible?
- Did I handle the sample with gloves and pliers to avoid contamination (in the case of chemical analysis and microscopy)?
- Did I put my sample in a suitable package like aluminum paper for solids or a glass or plastic container for powders or liquids?

Suzie Poulin received her B.Sc. degree in chemistry from the Université de Montréal in 1976 and then pursued a master's degree in polymer crystallography at UQAM. After two years as research associate in Université de Montréal's crystallography research group, she joined the physics department of École Polytechnique, in 1980, to work in electron paramagnetic resonance (EPR). A year later, she was in charge of the ERD and RBS techniques of the GCM. In 1985, she took charge of the Surface Analysis Laboratory that only had an XPS at the time. New equipments were later added to the laboratory like AFM microscope, FTIR systems and a TOF-SIMS on which she develops her expertise on various solid materials.