राष्ट्रीय प्रौद्योगिकी संस्थान रायपुर
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Department of Metallurgical Engineering, NIT Raipur

Scanning Electron Microscopy Facility (SEMF, NITRR)

Important notice for SEM End user

All users including institute as well as from outside, are requested to download the latest Requisition Form for SEM/EDX analysis as well as sample preparation work using gold/carbon coater.

- Form for SEM/EDX Analysis (INTERNAL Users ONLY) (doc)
- Form for SEM/EDX Analysis (EXTERNAL Users ONLY) (doc)

All OUTSIDE USERS are required to make a FORMAL WRITTEN REQUEST ON LETTERHEAD through their HEAD OF DEPARTMENT/INSTITUTION to the 'SEM coordinator'. Applicable charges may be paid through a Demand Draft in favour of Director, NIT Raipur, payable at GECT SBI Raipur branch. The charges have to be deposited in ADVANCE only. Outside users may consider doing so on the day of their appointment but they must take into account the following before planning their visit: (a) Time required to deposit the charges (b) Time required to mount samples on the sample holder and (c) Time required to coat the samples with silver if required.

Date and time of allotment will be given only when the samples are absolutely ready for actual analysis.

No Requisition Form will be accepted by SEMF, NITRR if the samples are not properly mounted and coated if required.

SEM facility at NIT Raipur dosen't provide printed micrographs of any kind. So, users are requested to bring FRESH CD for collection of SEM/EDX analysis results.

NO USED CD OR PENDRIVE IS ACCEPTABLE under any circumstances.

Users are requested to report in the SEM facility at NIT Raipur/laboratory at their scheduled appointment.

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Sample Preparation

Guidelines for Preparation of Samples for SEM and EDX analysis

SEM:

1. Keep the size of the samples to about 5-8 mm in size. No dimension of the sample should exceed 10 mm. The ideal shape of a sample is that of a button on your shirt. The thickness should be as small as can be handled easily without damaging the sample or region of interest on the sample. If the sample size is large the region of interest on the sample should be sketched on a paper ahead of SEM/EDEX analysis.
2. Samples have to be mounted on a circular metallic sample holder available with SEMF, NITRR. Users may get their own sample holders fabricated ONLY IF these are acceptable to SEMF, NITRR. You may discuss the design with SEMF, NITRR staff.
3. The samples have to be fixed onto the sample holder rigidly enough so that they do not fall off easily while handling. One may use colloidal silver paste or sticky carbon tape or colloidal graphite all available with SEMF, NITRR. One should never use ordinary gum or any other adhesive without consulting the SEMF, NITRR as it may spoil the coating system and sample both.
4. Samples must be arranged in a circular pattern as displayed in the SEMF, NITRR as it saves a lot of precious time in locating the samples in SEM chamber which is always under vacuum. The fact that the minimum magnification offered by SEM is about 50x only makes identification of samples still harder.
5. Since an electron beam is incident on the samples for SEM analysis it is essential that the samples are electrically conducting. If not, this can be achieved by coating the samples with 20-50 nm thick gold or silver. SEMF, NITRR is equipped with a QUORUM Gold-palladium sputter coater for this purpose.
6. Users are requested to go through published literature so that they already have a reasonably good idea about the magnification or size of features in the samples. Users are also requested to bring along relevant published references if available. Fresh users are also advised to consult other users in their neighborhood to orient themselves for result oriented SEM work.

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EDX:

1. All the requirements mentioned for SEM are also valid for EDX work. However, the samples should be FLAT for accurate QUANTITATIVE analysis (Percentage of elements in the sample). For QUALITATIVE analysis (that is simply finding the elements present in the sample) any shape of the sample is workable.
2. An electrically non-conducting sample needs to be coated with thin layer of carbon. One may still get EDX spectrum showing some peaks of some elements if the samples are coated with gold or silver. BUT, the problem is that gold/silver layer absorbs the x-ray signal from the sample. The key thing to be noted is that this absorption of x-ray signal by gold/silver layer is not (a) uniform for elements across the periodic table, and (b) small peaks in the spectrum are completely suppressed. Users can still use gold silver coating for EDX work if they already have a good idea of percentages of elements present in the sample. The use of any metallic coating other than carbon for EDX work is not advised because the quantitative software in the EDX system assumes that sample is homogenous. The absorption of x-ray signal by carbon coating is negligible on account of its low atomic number and is taken into account while performing quantitative EDX analysis. SEMF, NITRR will soon have the facility of carbon coating for EDX purposes.
3. Some semi-conducting samples can be analyzed through EDX without carbon coating.

   EDX analysis can't detect percentage of carbon, oxygen and hydrogen in a polymer. This is because hydrogen element doe