Booking Guidelines

A. IIT KGP Users

IIT Kharagpur faculty members and students can book SATHI facilities using the ERP. The booking has to be initiated by a Faculty Member only.

Detail guidelines have already been circulated to all the Faculty Members.

B. External (Non-IIT KGP) Users

1. User writes to Chief Operating Officer (COO) SATHI Foundation IIT Kharagpur (coosathi@iitkgp.ac.in) regarding the analysis of the sample
2. If the user wants the NDA to be signed, COO send the template/format and both the parties execute the NDA.
3. COO acknowledges and sends the relevant sample details form to the user
   a. HRTEM
   b. ToF-SIMS
   c. Surface Profilometer
4. User fills the forms and send it to the COO.
5. COO checks the details and requests the missing details, if any. COO then forwards it to the Facility In-charge for the approval:
   a. To check whether the sample is acceptable for the analysis
   b. To clarify any technical doubts, if the user has any
6. Facility In-charge approves / rejects or seeks the clarification.
7. Once the Facility In-charge approves the sample, COO prepares an estimate and sends it to the user
8. User conveys his / her willingness to go ahead and request the next steps
9. COO sends the bank details for the payment
10. User makes the payment using NEFT / RTGS and shares the UTR number with the COO
11. COO confirms the receipt of the payment to the user
12. COO shares the softcopy of the invoice with the user and mails the hardcopy (optional).
13. COO shares the sample preparation and submission guidelines with the user (through an email or a portal link), if necessary
14. COO allocates the machine time slot to the user for analysis (date, time, location, operator etc.) and conveys it to the user.
15. User submits the sample physically or through courier
16. COO shares the results of the sample analysis with the user through mutually agreed media

Forms Download:

1. HRTEM Sample and Payment Details Form
2. ToF-SIMS Sample and Payment Details Form
3. Profilometer Sample and Payment Details Form

JEOL JEM ARM 300 F2 HRTEM

1. **Type of Analysis:** The analysis modes are TEM / HRTEM / SAED / EDS / STEM EDS / EDS Mapping / ADF / EELS

2. **Sample Types**

   (i) Non-magnetic inorganic samples are allowed. For powdered samples that do not degas under vacuum are allowed. However, sample preparation is a must for them.

   (ii) Magnetic samples in powder form and organic samples are strictly not allowed

   (iii) Sample types allowed are Powder (prepared on grid), self-standing film and disc.

   (iv) For compounds and alloys containing magnetic elements such as iron, cobalt and nickel, the user should provide couple of FESEM images of the powder sample (along with EDS-preferred) prepared on grid.

3. **Sample size**

   For the HRTEM (High-Resolution Transmission Electron Microscope), the acceptable sample size typically depends on several factors related to the specimen holder and the specific capabilities of the instrument. Here are some general considerations:

   Grid Size: HRTEM instruments like the JEM ARM 300 F2 often use standard TEM grids for sample mounting. The most common grid sizes are:

   - **Standard Size**: 3.05 mm in diameter with a mesh size of 100 to 400 mesh.

   Sample Thickness: The sample thickness should be suitable for transmission electron microscopy (TEM) analysis. Typically, samples should be thin enough to allow electrons to pass through for imaging and analysis. For thin films, thicknesses are often in the range of tens to hundreds of nanometers.

   Compatibility: Ensure the sample grid and holder are compatible with the JEOL JEM ARM 300 F2 HRTEM stage and holders.

4. **Sample Preparation Guidelines**

   Proper sample preparation is crucial to obtain accurate and meaningful results. Here are the sample preparation guidelines for various types of samples:

   (i) General Sample Preparation Considerations:

   - **Cleanliness:** Ensure that your sample is free from dust, contaminants, and residues.
   - **Thin Sections:** For TEM analysis, samples should be thin enough (typically <100 nm) to allow electrons to pass through.
   - **Stable Mounting:** Mount the sample securely on a TEM grid or holder.

   (ii) Material Science Samples:

- **Polishing:** Polish bulk materials to achieve a smooth surface.
- **Ion Milling:** For cross-sectional analysis, use ion milling to create thin lamellae.
- **Replica Technique**: Create a replica of the sample surface using a carbon film for shadowing.

(iii) Nanoparticles and Nanomaterials:

- **Dispersion:** Disperse nanoparticles in a suitable solvent.
- **Deposition:** Deposit a drop of the dispersion onto a TEM grid.
- **Drying:** Allow the solvent to evaporate, leaving the nanoparticles on the grid.

(iv) Inorganic Crystals and Thin Films:

- **Sectioning:** Cut thin sections using a diamond knife or focused ion beam (FIB).
- **Ion Milling (Optional):** Thin the sample further using ion milling.

Kindly note that specific sample preparation steps may vary based on the material type, research goals, and the specific analysis you intend to perform.

Drop casting for powder samples (nanoparticles and nanomaterials)

(i) For the drop casting of the sample, the sample should be dispersed in isopropyl alcohol
    a. In case of any challenges with isopropyl alcohol, then Acetone or distilled water can be used
(ii) Sample (Metal disc / after drop casting on the grid) must be oven dried for 1 day.
    a. Temperature depends on the type of sample (User need to take care of the morphology of the sample)
(iii) Put the sample overnight in the desiccator before the day or two of the analysis
(iv) Bring the sample along with the desiccator for analysis
(v) Users requiring sample preparation services from the SATHI Foundation IIT Kharagpur need to indicate or mention the same in the communication
(vi) A demo video is available on YouTube https://www.youtube.com/watch?v=rVT98ywELvE

5. Sample Submission Guidelines

(i) Users can send the sample through courier. It takes about a week. Care must be taken to ensure that samples remain in the upright position and in dry condition.
(ii) Numbering should be properly with waterproof ink in and preferably in the grid box for easy identification
(iii) SATHI Foundation IIT Kharagpur is not responsible for any damage that may happen during the transit. If due to some reason, the sample is found to be damaged, it will not be taken for analysis and the same will be reported to the user
(iv) Users requiring Grid for the sample analysis, need to specify the type of grid required. SATHI Foundation shall provide the grid (subject to availability)

6. Support

A. For any Technical query

- Prof. Debabrata Pradhan, Professor-In Charge – deb@matsc.iitkgp.ac.in
  Office 03222- 281798, Mob: 974994697
- Prof. Ahin Roy, Professor-In Charge, ahinroy@matsc.iitkgp.ac.in
  Office 03222-283988, Mob: 7348903906

B. For operational issues

- Dr. Avinash Joshi – COO, SATHI Foundation IIT Kharagpur - sathiops@iitkgp.ac.in ,
  (Direct Dial): 03222-214792, Mob: +91 9850753614,

PHI nanoToF 3, ToF-SIMS

Type of Analysis:

Following three (3) type of analyses can be carried out using SIMS-ToF:

(i) Spectrum
(ii) Image
(iii) Depth Profile (up to 100 Nano meters only)

Sample Types:

The acceptable sample types typically include:

(i) Solid Surfaces: This includes a wide range of solid materials such as metals, semiconductors, ceramics, polymers, and thin films.

(ii) Coatings and Thin Films: Samples with coatings or thin films are suitable, as ToF-SIMS is particularly useful for depth profiling and characterization of thin layers.

(iii) Organic Materials: Organic samples can also be analyzed, including biological samples (though preparation methods might vary).

(iv) Composite Materials: Samples consisting of multiple layers or materials can be analyzed to characterize interfaces and layer compositions.

(v) Semiconductors and Electronic Materials: ToF-SIMS is often used in the semiconductor industry for process control and failure analysis.

(vi) Polymer Films and Coatings: Polymers are commonly analyzed due to their diverse chemical compositions and surface properties.

Sample size

Thin, flat squares of dimension. 8mm x 8 mm x 4 mm (maximum) are permitted

Sample Preparation Guidelines

Before analysis, it's crucial to ensure that the sample preparation is appropriate for ToF-SIMS analysis, which typically involves cleaning the surface to remove contaminants and optimizing the surface condition for accurate analysis

The sample preparation steps for submitting a sample for ToF-SIMS analysis can vary depending on the type of sample being analyzed. Here's a breakdown of sample preparation steps tailored to different types of samples commonly analyzed using ToF-SIMS:

(i) Solid Surfaces (Metals, Semiconductors, Ceramics)

- **Cleaning**: Clean the surface thoroughly to remove contaminants using solvent cleaning (e.g., acetone, isopropanol) or plasma cleaning.
- **Drying**: Ensure the surface is completely dry to prevent water or solvent residues.
- **Mounting**: Secure the sample on a suitable holder ensuring stability during analysis.
- **Surface Smoothing**: Polish or flatten the surface if necessary for improved analysis resolution.
- **Masking**: Use masking techniques if specific areas need to be analyzed separately.
- **Storage**: Store in a clean environment to prevent contamination before analysis.

(ii) Polymer Films and Coatings

- **Cleaning:** Remove any surface contaminants using mild solvent cleaning or plasma cleaning.
- **Drying:** Ensure thorough drying to avoid solvent residues.
- **Mounting:** Secure the film or coating on a suitable substrate or holder.
- **Surface Smoothing:** Flatten or ensure uniformity if necessary for accurate analysis.
- **Masking:** Use masking if analyzing specific regions of the film or coating.
- **Storage:** Store in a clean, controlled environment to prevent contamination.
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(iii) Organic Materials (Biological Samples, Polymers)

- **Cleaning:** Clean gently to avoid damage using mild solvents or detergents suitable for organic materials.
- **Drying:** Ensure gentle drying to avoid structural damage.
- **Mounting:** Handle delicately and mount on a suitable substrate or holder.
- **Surface Smoothing:** Ensure the surface is flat and uniform if necessary.
- **Masking:** Use masking to isolate specific areas of interest.
- **Storage:** Store in appropriate conditions to maintain sample integrity.
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(iv) Semiconductor Devices and Thin Films

- **Cleaning**: Clean with solvents suitable for semiconductor materials or use plasma cleaning.
- **Drying**: Ensure thorough drying to remove any residues.
- **Mounting**: Secure on a suitable holder for stability during analysis.
- **Surface Smoothing**: Polish if necessary for improved surface analysis.
- **Masking**: Use masking for specific areas or features.
- **Storage**: Store in a controlled environment to avoid contamination.

Additional Considerations:

- **Sample Conditioning**: Some samples may require specific conditioning (e.g., annealing) to stabilize or enhance surface features.
- **Documentation**: Label samples clearly with relevant information (e.g., sample type, preparation steps) for traceability.

- **Consult Manufacturer Guidelines**: Always refer to manufacturer's ToF-SIMS instrument manual or guidelines for specific recommendations tailored to your sample type and analysis goals.

By following these tailored sample preparation steps, you can ensure that your samples are optimized for accurate and reliable analysis using ToF-SIMS instrument, regardless of the material type being analyzed.

Sample Submission Guidelines:

(i) Sample must be properly secured in a sponge like cover to ensure that the surface to be tested is protected.
(ii) Proper care must be taken to ensure the sample is dry and dust free
(iii) The sample surface to be analyzed should be kept in upright position and clearly identifiable
(iv) Keep a small piece of paper with following details along with the sample for proper identification.
   a. Numbering should be done with waterproof ink if the number of samples are more than one
   b. Contact details (Name, phone number, email address)
   c. Type of test(s) to be carried out
(v) Sample submission in-person time at the SATHI COO's office is between 9 AM to 11 AM on working days

Support

C. For any Technical query
   - Prof. Rahul Mitra: Professor-In Charge – rahul@metal.iitkgp.ac.in
     Office 03222-283292, Mob: +91 9434034270

D. For operational issues
   - Dr. Avinash Joshi – COO, SATHI Foundation IIT Kharagpur - sathiops@iitkgp.ac.in ,
     (Direct Dial): 03222-214792, Mob: +91 9850753614,

KLA Alpha-Step D-600 Surface Profilometer

Type of Analysis:

The Alpha-Step D-600 stylus profiler by KLA is capable of measuring 2D and 3D step heights ranging from a **few nanometers to 1200 µm**. Here are the sample types and applications supported by the Alpha-Step D-600:

(i) Step Height Measurement:
- The instrument accurately measures step heights, allowing you to quantify material added or removed during processes such as etching, sputtering, SIMS, deposition, spin coatings, CMP, and more.
- It covers a wide range of step heights, making it suitable for various applications.

(ii) Texture Measurement:
- The Alpha-Step D-600 assesses 2D and 3D texture, providing information about sample roughness and waviness.
- This feature helps characterize surface quality and topography.

(iii) Form Measurement:
- It quantifies wafer bow and determines the height and radius of curvature of structures (e.g., lenses).
- Useful for understanding the shape and form of samples.

(iv) Stress Measurement:
- The instrument accurately measures sample bow using a stress chuck to support the sample in a neutral position.
- Stress analysis is essential for various applications, including semiconductor manufacturing and materials research.

Sample Types

The Alpha-Step D-600's direct stylus measurement is independent of material properties, making it suitable for a wide variety of structures and materials

However, there are certain sample types that may not be suitable for measurement using this profiler:

(i) **Soft or Delicate Samples**:
- The D-600 is designed to measure soft or delicate samples without causing damage.
- However, extremely fragile materials may still be challenging to measure accurately.

(ii) Materials with Curvature Smaller Than Stylus Width:
- If a sample has features with a radius of curvature smaller than the width of the stylus tip, distortion in measured feature width may occur.
- While the height measurement remains accurate, the lateral dimensions may be affected.

(iii) Highly Transparent Materials:

- The D-600's stylus measurement technique is independent of material properties, but highly transparent materials (such as certain polymers or glass) may pose challenges due to light scattering or refraction.

(iv) Samples with Excessive Roughness or Surface Irregularities:

- While it can measure roughness, excessively rough surfaces may lead to stylus tip wear or inaccurate results.
- Extremely irregular surfaces may also affect the stylus movement.

(v) Samples with Extreme Chemical Reactivity:

- Although the instrument is versatile, samples with extreme chemical reactivity (e.g., highly corrosive materials) should be handled with caution.

Sample size

The instrument allows for a sample size up to 200mm in diameter and a maximum height of 30 mm.

Support

A. For any Technical query

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