

Frequently Ask Questions (FAQ) FOR FACTS INSTRUMENTS

General

1. What are materials suitable for instruments in FACTS?

- A. TEM - Metals, Ceramics, Semiconductors, Polymer, Biological Sample*, Organic Sample*
- B. SEM - Metals, Ceramics, Semiconductors, Polymer, Biological Samples*, Organic Samples*
- C. XRD - Metals, Ceramics, Semiconductors, Polymer, Biological Samples*, Organic Samples*
- D. Small Angle X-ray (SAXS) - Metals*, Ceramics*, Semiconductors*, Polymer, Biological Samples*, Organic Samples*
- E. XRF - XRF – Metals, Glass, Ceramics, Building Materials

2. What are the limitations for the materials used in the instruments in FACTS?

A. TEM

- Samples should be preferable non-magnetic
- Polymer, biological samples* organic are easily damaged by beam, preferably used at low kv TEM, e.g 120KV, or cryo-TEM
- Sample preparation must be chosen carefully
- Sample size (to be elaborated)

B. SEM

- Samples should be preferably non-magnetic
- Polymer, biological samples* organic are easily damaged by beam, sample should be coated by conductive materials

C. XRD

- Physical Size on the sample holder

D. SAXS

- SAXS - dispersed in solvent ($\sim 44\mu$ L), powders dispersed on an adhesive tape

E. XRF

- XRF – The pallet size of 40mm

3. What are the physical size of samples suitable to be characterized under the instruments in FACTS?

- TEM - ultra thin $< \sim 100$ nm, thinner the better, 3mm in diameter
- SEM - please check with FACTS Lab in Charge
- XRD - within the sample holder?
- SAXS - dispersed in solvent ($\sim 44\mu$ L), powders dispersed on an adhesive tape
- XRF - The pallet size of 40mm

4. If i want to see the morphology of my samples, which instrument should i use?

SEM, SAXS, AFM. TEM is a transmission technique and produce a 2-D projection traditionally, so it is not the best technique to observe morphology. There is a TEM tomography which probably can achieve this but this technique is not available for our TEM. SAXS is a 2-D information which you need to post process to derive the morphology information. The strength of the SAXS is that it can interpret soft materials morphology and provides a better statistical result

5. The instruments i use cannot give me the data i need. I need a better instrument.

Please contact respective instrument-in-charge to discuss about your data as soon as possible. There are many reasons why you cannot obtain the appropriate the data you are expecting. One of the common reason is due to the sample suitability under the instrument. You need to make sure a suitable sample preparation is used. Another common reason is that your sample synthesis did not successfully synthesize the desired sample you want. The last common reason is that you haven't follow the correct procedure to collect the data. Simply get a known sample to test out and see if you are expecting to

get the right result. Your instrument-in-charge is willing to discuss with you and suggest the appropriate solution for you within their capability.

TEM

1. I want to determine the phase of the my materials, so i would like to use the TEM.

Wherever there is adequate sample, please use the XRD to determine the phase of the materials as it is a simple technique to use. If sample is too little to get any significant signal from XRD, please use the selected area diffraction pattern (SAED) to determine the phase. If you have quantum dots, try to obtain the polycrystalline diffraction data to determine the phase. EDX can only provide elemental information.

2. I have polymer sample, which TEM should i use?

We have a 120 KV TEM, Libra 120 Plus which is suitable to image polymer sample. You do not need a 200 KV TEM to image the polymer sample most of the time because you do not need extremely high resolution. Secondly, carbon is light element and do not scatter electron efficiently, as a result, there is very little contrast when you are using a 200 KV TEM. Thirdly, due to highly energetic electron at 200 KV, your sample will be damaged easily

3. What is the difference between JEOL 2010 UHR and JEOL 2010 HR in FACTS Lab?

The JEOL 2010 UHR have an ultra high resolution pole piece while JEOL 2010 HR have a high resolution pole piece. The point resolution for JEOL 2010 UHR is about 1.9 angstrom while JEOL 2010 HR have a point resolution about 0.22 angstrom. Most user will not be able to differentiate in the performance of the two instruments. However, JEOL 2010 HR have a slightly wider pole piece, therefore in-situ holders which are generally thicker in the sample position are more suitable to be use in this instrument.

4. JEOL 2100F have a Field Emission Gun and therefore the best TEM in FACTS!

User must first evaluate whether their materials is suitable. While the Field Emission Gun give extremely high brightness, due to the high current densities, it can easily damage the sample

One of the advantage of using a Field Emission Gun TEM is that it can form a very fine electron probe. With the scanning coil, it can perform scanning electron microscopy which is useful to combine with EDX elemental mapping and EEL mapping and other techniques.

5. Your peers would say, use FIB, its easy to prepare TEM sample but...

FIB prepared sample does not always guarantee very thin samples. TEM needs very thin sample otherwise, image is very difficult to interpret. In recent years, FIB in combination with other suitable techniques such as argon ion milling is able to thin the sample further, however this will take time to optimize. The hash truth is that there is no easy TEM sample preparation method unless your samples are powders, quantum dots or equivalent.

6. All the papers shows high resolution TEM images (HRTEM). I must have one to be in my publication!

It would be nice to have such image in the publication. However, conventional HRTEM cannot be interpreted directly. Please attend the Advanced Characterization course to understand more.

7. The 120KV TEM have a poorer resolution, I want to be trained in the 200KV TEM.

It really depends on the objective of your research rather than choosing a higher accelerating voltage TEM. The Libra 120 Plus have a 3.4 angstrom resolution. If you do not need high resolution images to interpret lattice images, then perhaps this machine is suitable to your needs. If you have a crystal with a huge lattice, 120KV TEM might also be suitable for you.

SEM/FESEM

1. I would like to use the SEM. Which SEM should i use?

If you are a Final Year Student, you are allowed to use only the JEOL 5310, 5410 and JEOL 6360. Please check the instructions from here

<http://www.mse.ntu.edu.sg/FacilitiesEquipment/Pages/OnlineFacility.aspx>

If you are a post-graduate student, post-doctorate fellows, you are allow to use all the SEM available in FACTS. Again, please follow the instructions from here

<http://www.mse.ntu.edu.sg/FacilitiesEquipment/Pages/OnlineFacility.aspx>

Please determine the smallest sample feature you need to see. Below is the SEM resolution performance

SEM (JEOL 6360, 5410, 5310) : 4 nm

FESEM (JEOL 6340F): 3 nm

FESEM (JEOL 7600F): 1.5 nm

Again, in-house regulation/policy applies, please check from here

<http://facts.ntu.edu.sg/policies.html>

2. I have magnetic samples, can i use the SEM/FESEM?

Please take note that due to the pole pieces and lens system make use of magnetic field to focus the beam, it is highly that magnetic samples have strong interactions with them. All magnetic samples must be highly secure to prevent and magnetic interactions to the lens, e.g magnetic samples is attached to the pole piece, this will not only damage the pole pieces and will cause downtime and repair fee. Your supervisor might have to bear the cost of the repair.

On the other hand, it might be very difficult to focus on magnetic sample, users need to be patient and extra care to take the imaging.

3. I have liquid samples, can i use the SEM/FESEM?

All samples must be dried, oven to ensure minimal outgassing. Failure to do so will damage the vacuum system.

XRD

1. Which XRD machines are suitable for powder and for thin film?

For powder samples the Shimadzu powder, Bruker D8 Advance and Panalytical can be used. For thin film samples the Shimadzu thin film and Bruker D8 Discover can be used.

2. What is the difference between powder XRD analysis and thin film XRD analysis?

The powder XRD analysis is taken in 2theta-theta scan mode with variable incident angle with regard to the sample surface. In this scan mode the X-rays penetrate deeper into the sample to detect all possible crystallites in the sample. As for the Thin Film XRD analysis the incident angle is fixed to avoid convolution of diffracted signals from the thin film and the substrate. To obtain the XRD profile for the thin film sample alone, a glancing angle XRD scan mode has to be used. In this scan mode the X-rays penetrate only the thin film region.

3. What should I do to identify the crystalline phases in my sample?

After obtaining the XRD profile of your sample, you can load it to Match 2 (with the latest database) and perform a search match to identify the phases found in your sample. To make your life easier it is always wise to have additional information about your sample, like its composition and the type of material (alloys, minerals, ceramics, polymer, pharmaceutical, organic/inorganic, semiconductor, etc.).

4. The XRD profile of the sample has very poor quality, like low intensity, broad peaks and few peaks. What could it be improved?

- The low intensity could be improved by increasing the scan time.
- Peak broadening indicates small grain size or low crystallinity, so annealing could be done to increase the grain size and improve the crystallinity. However if the sample undergoes a change in crystal phase, this must be taken into consideration.
- Fewer peaks compared to the database may indicate preferred orientation. For powder samples this could be due to particle morphology and large grain sizes.

Reducing the grain/particle size will improve the randomness of crystal orientation.

5. I want to measure my sample at high-temperature, how should I prepare my sample and which machine should I use?

The Siemens (Bruker) D5005 is equipped with an AntonPaar HTK1000 heating stage, so it can be used for taking XRD profile at non-ambient temperature. During heating the sample chamber is in vacuum to prevent oxidation of the sample and the components found inside. It is also important that the sample must be stable against vacuum and must not evaporate or sublime during heating. Therefore appropriate high temperature limit must be observed and consulted with the instrument manager.

6. Thin films typically exhibit preferred orientation (or texture), what kind of method that is available in FACTS that can be used to characterize the preferred orientation of my sample?

Preferred orientation is characterized by measuring the pole figures (PF) of different (hkl) planes of a crystalline phase. The PF is measured using the Bruker D8 Discover. A subsequent data analysis of the PFs is required in order to identify the degree of crystallographic texture and its components.

SAXS

1. What kind of information can I get from a SAXS experiment?

SAXS measures the scattering of X-rays at low angles ($2\theta = 0-10\text{deg.}$). In this region the scattering of X-rays is due to the particle and thus we are able to evaluate the average size of particles, its shape and morphology. The range of size is between 1 nm and 100 nm.

2. What is the limit in q-range for the SAXS facility in FACTS?

The minimum q-range limit is 0.077nm^{-1} , which corresponds to a lattice distance of about 82 nm. The maximum q-range is 7nm^{-1} .

3. Can we also measure WAXS and what is the q-range limit?

It is possible to measure WAXS and SAXS at the same time. The proper camera attachment and film size must be used. The maximum q-range for WAXS is 27nm^{-1} . The advantage of measuring WAXS and SAXS at the same time is the possibility to evaluate the particle size, shape and the crystalline nature of the sample all at the same time.

4. The SAXS profile of my sample did not show any scattering, what could be the problem?

One reason could be that the particle size is beyond the limit of the SAXS facility. For this issue another method must be used, like microscopy. Another reason could be that not enough particles are dispersed in the solution. Increasing the particle concentration could solve this issue.

5. How should I prepare samples for SAXS?

- For liquid samples (particles suspended in a solution) it is important that it is not alkaline as it will attack the quartz capillary holder. More than enough amount of liquid ($\sim 44\mu\text{L}$) must be provided to fill the quartz capillary.
 - For samples deposited on a substrate, it is important to consider a very thin substrate since the X-ray is transmitted through the sample. If it is too thick the incident X-rays will be absorbed.
 - For powder samples they must be evenly dispersed over an adhesive tape. After which it will be sandwiched between two Cu plates with windows where X-rays can be transmitted.
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