Lab Sessions Guide Advanced Methods for Materials Characterization

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Advanced Methods for Materials Characterization

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本书详细介绍了广泛应用于材料科学与工程、物理、化学和其他相关领域的实验方法，并介绍了这些实验的目标、原理、实验设备及操作流程，通过一步一步的操作来指导读者熟悉实验流程，发现实验之美。

本书适合作为以上专业学生及研究人员的实验指导书。

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Preface

This brochure is prepared as a "Lab Sessions Guide" for the course of "Advanced Methods for Materials Characterization", which is one of the core courses for undergraduate students in the School of Materials Science and Engineering, University of Shanghai for Science and Technology (USST). In this course, students are expected to explore the beauty and scientific significance of a number of experimental approaches frequently used in the fields of materials science and engineering as well as physics, chemistry, biology and related disciplines. The principal goal of this guide is to offer the students step-by-step instruments during the lab sessions; nonetheless, it may also be helpful to our colleagues and graduate students for their research purpose as an abridged manual of the apparati included.

We are greatly indebted to our colleagues, Drs. Xianying Wang, Dunliang Jian, Shengjuan Li and Feng Tian, for their wonderful help during the preparation of this guide. We also wish to thank Profs. Xia Wang, Fengcang Ma, Huijuan Zhang and Xiaohong Chen for their encouragement and constructive suggestions to make this course possible. Thanks are also due to the authors of the original materials we referenced herein.

Xuyan Liu Qiang Li Deng Pan
Dengguang Yu Yuedong Sun Fang Liu

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Lab Session 1:

Scanning Electron Microscopy (SEM)

1. Objective

The objective of this experiment is 1) to enhance the understanding of structure and working principle of SEM; 2) to observe the morphology and Z contrast of specimen; and 3) to explore the spatial resolution of an SEM.

2. Working Principle

In some ways, SEMs work in the same way as key copying machines. When you get a key copied at your local hardware store, a machine traces over the indentations of the original key while cutting an exact replica into a blank key. The copy isn't made all at once, but rather traced out from one end to the other. You might think of the specimen under examination as the original key. The SEM's job is to use an electron beam to trace over the object, creating an exact replica of the original object on a monitor. So rather than just tracing out a flat one-dimensional outline of the key, the SEM gives the viewer more of a living, breathing 3-D image, complete with grooves and engraving.

As the electron beam traces over the object, it interacts with the surface of the object, dislodging secondary electrons from the surface of the specimen in unique patterns. A secondary electron

detector attracts those scattered electrons and, depending on the number of electrons that reach the detector, registers different levels of brightness on a monitor. Additional sensors detect backscattered electrons (electrons that reflect off the specimen's surface) and X-rays (emitted from beneath the specimen's surface). Dot by dot, row by row, an image of the original object is scanned onto a monitor for viewing (hence the "scanning" part of the machine's name).

Of course, this entire process wouldn't be possible if the microscope couldn't control the movement of an electron beam. SEMs use scanning coils, which create a magnetic field using fluctuating voltage, to manipulate the electron beam. The scanning coils are able to move the beam precisely back and forth over a defined section of an object. If a researcher wants to increase the magnification of an image, he or she simply sets the electron beam to scan a smaller area of the sample.

While it's nice to know how an SEM works in theory, operating one is even better.

Key components of an SEM are shown in Fig. 1-1

1) Electron gun: Electron guns aren't some futuristic weapon used in the newest Vin Diesel movie. Instead, they produce the steady stream of electrons necessary for SEMs to operate. Electron guns are typically one of two types. Thermionic guns, which are the most common type, apply thermal energy to a filament (usually made of tungsten, which has a high melting point) to coax electrons away from the gun and toward the specimen under examina-

tion. Field emission guns, on the other hand, create a strong electrical field to pull electrons away from the atoms they're associated with. Electron guns are located either at the very top or at the very bottom of an SEM and fire a beam of electrons at the object under examination. These electrons don't naturally go where they need to, however, which gets us to the next component of SEMs.

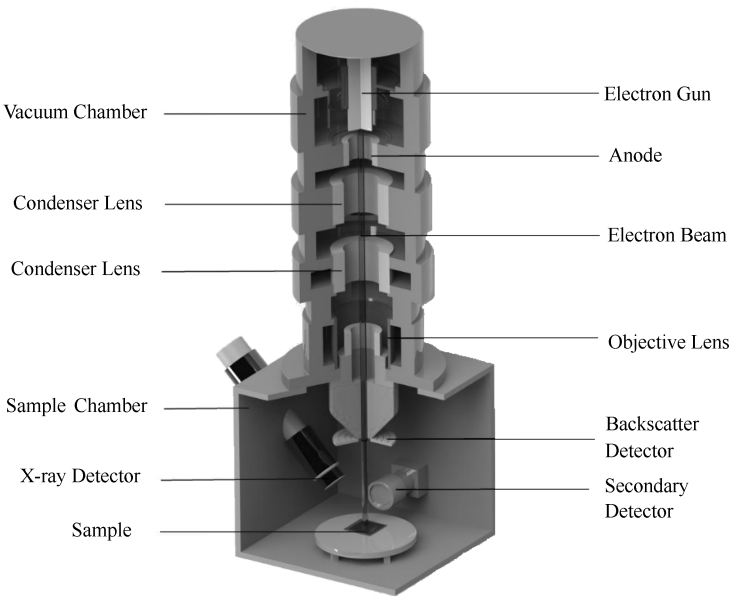

Fig. 1-1 Schematic of SEM key components

2) Lenses: Just like optical microscopes, SEMs use lenses to produce clear and detailed images. The lenses in these devices, however, work differently. For one thing, they aren't made of glass. Instead, the lenses are made of magnets capable of bending the path of electrons. By doing so, the lenses focus and control the

electron beam, ensuring that the electrons end up precisely where they need to go.

3) Sample chamber: The sample chamber of an SEM is where researchers place the specimen that they are examining. Because the specimen must be kept extremely still for the microscope to produce clear images, the sample chamber must be very sturdy and insulated from vibration. In fact, SEMs are so sensitive to vibrations that they're often installed on the ground floor of a building. The sample chambers of an SEM do more than keep a specimen still. They also manipulate the specimen, placing it at different angles and moving it so that researchers don't have to constantly remount the object to take different images.

4) Detectors: You might think of an SEM's various types of detectors as the eyes of the microscope. These devices detect the various ways that the electron beam interacts with the sample object. For instance, Everhart-Thornley detectors register secondary electrons, which are electrons dislodged from the outer surface of a specimen. These detectors are capable of producing the most detailed images of an object's surface. Other detectors, such as backscattered electron detectors and X-ray detectors, can tell researchers about the composition of a substance.

5) Vacuum chamber: SEMs require a vacuum to operate. Without a vacuum, the electron beam generated by the electron gun would encounter constant interference from air particles in the atmosphere. Not only would these particles block the path of the electron beam, they would also be knocked out of the air and onto the specimen, which would distort the surface of the specimen.

(Source:http://www. howstuffworks. com)

3. Instrumentation(Fig. 1-2)

Fig. 1-2 QUANTA FEG450 SEM equipped with a field emission gun

Secondary electron resolution:

- High vacuum mode:1. 2nm (30kV)
- Low vacuum mode:1. 4nm (30kV)
- Back scattering electron resolution:2. 5nm (30kV)
- Accelerating voltage:0. 2～30kV
- Magnification range:12×～ 1,000,000×
- Current:200nA max.

Sample chamber:

- Sample stage:5-axis servo auto-centering stage
  $X \geqslant 100$mm

$Y \geqslant 100$mm

$Z \geqslant 100$mm

$T = -5° \sim +70°$

$R = 360°$continuous rotation

■ Sample size: horizontal length > 280mm

4. Experimental Procedure

1) Check the circulating water system first to make sure the pressure index is about 4.5 and the temperature is about 18 to 20℃.

2) Check 'LINE' and 'INV' on the power display panel to make sure they are lit, and one of the six indicators above is on.

3) Turn on the controlling computer, and start the user interface.

4) Before loading the sample, check the beam on/accelerating voltage and make sure it is turned off, then press the 'VENT' button. Load the sample with gloves on, and double check the sample height below the limit before securing the sample stage.

5) Apply a force to the sample chamber door with one hand, then press the 'PUMP' button.

6) Pump the vacuum to below 5×10^{-3} Pa, then apply high voltage. Make sure 'SE' or 'TLD' in the 'Detector' menu is selected, press 'beam on' button, then a sound of V6 valve opening shall be heard, and wait for the button color turning yellow.

7) When the image shows up, adjust the image in focus, and press 'OK'.

8) Hold left 'Shift' key on the keyboard and right-click the mouse to remove the astigmatism.

9) Press 'F6' to scan the image frame, then save the image by selecting 'Save As...' in the 'File' pulldown menu.

10) After the session is done, press the 'beam on' button then wait a few seconds for the V6 valve sound, button color turns to gray from yellow, then pump the air into the sample chamber, remove the sample, then restore the chamber vacuum.

5. Other Requirements

Other requirements for this lab sessionand lab report will be delivered by the instructor at the session.

Lab Session 2:

Transmission Electron Microscopy (TEM)

1. Objective

The objective of this experiment is 1) to enhance the understanding of the structure and working principle of TEM; and 2) to explore the spatial resolution of a TEM.

2. Working Principle

The transmission electron microscope uses a high energy electron beam transmitted through a very thin sample to image and analyze the microstructure of materials with atomic scale resolution. The electrons are focused with electromagnetic lenses and the image is observed on a fluorescent screen, or recorded on a film or digital camera. The electrons are accelerated at several hundred kV, giving wavelengths much smaller than that of light: 200kV electrons have a wavelength of 0.025Å. However, whereas the resolution of the optical microscope is limited by the wavelength of light, that of the electron microscope is limited by aberrations inherent in electromagnetic lenses, to about 1～2Å.

Because even for very thin samples one is looking through many atoms, one does not usually see individual atoms. Rather the high

resolution imaging mode of the microscope images the crystal lattice of a material as an interference pattern between the transmitted and diffracted beams. This allows one to observe planar and line defects, grain boundaries, interfaces, etc. with atomic scale resolution. The brightfield/darkfield imaging modes of the microscope, which operate at intermediate magnification, combined with electron diffraction, are also invaluable for giving information about the morphology, crystal phases, and defects in a material. Finally the microscope is equipped with a special imaging lens allowing for the observation of micromagnetic domain structures in a field-free environment.

The TEM is also capable of forming a focused electron probe, as small as 20Å, which can be positioned on very fine features in the sample formicrodiffraction information or analysis of X-rays for compositional information. The latter is the same signal as that used for EMPA and SEM composition analysis (see EMPA facility), where the resolution is on the order of one micron due to beam spreading in the bulk sample. The spatial resolution for this compositional analysis in TEM is much higher, on the order of the probe size, because the sample is so thin. Conversely the signal is much smaller and therefore less quantitative. The high brightness field-emission gun improves the sensitivity and resolution of X-ray compositional analysis over that available with more traditional thermionic sources.

Restrictions on Samples:

Sample preparation for TEM generally requires more time and experience than for most other characterization techniques. A TEM specimen must be approximately 1,000Å or less in thickness in the area of interest. The entire specimen must fit into a 3 mm diameter cup and be less than about 100 microns in thickness. A thin, disc shaped sample with a hole in the middle, the edges of the hole being thin enough for TEM viewing, is typical. The initial disk is usually formed by cutting and grinding from bulk or thin film/substrate material, and the final thinning done by ion milling. Other specimen preparation possibilities include direct deposition onto a TEM-thin substrate (Si_3N_4, carbon); direct dispersion of powders on such a substrate; grinding and polishing using special devices (t-tool, tripod); chemical etching and electropolishing; lithographic patterning of walls and pillars for cross-section viewing; and focused ion beam (FIB) sectioning for site specific samples.

Artifacts are common in TEM samples, due both to the thinning process and to changing the form of the original material. For example surface oxide films may be introduced during ion milling and the strain state of a thin film may change if the substrate is removed. Most artifacts can either be minimized by appropriate preparation techniques or be systematically identified and separated from real information.

(Source: http://www.stanford.edu/group/snl/tem.htm)

3. Instrumentation(Fig. 2-1)

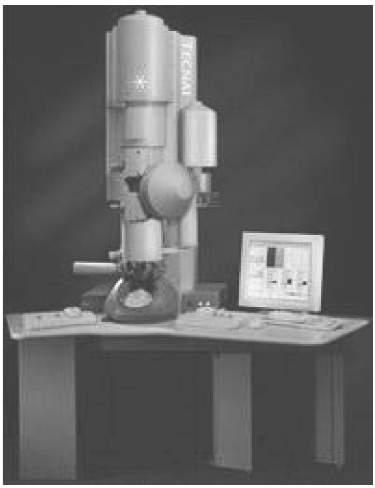

Fig. 2-1 TF30 TEM equipped with a field emission gun

Specifications:

- Schottky field emission filament
- Accelerating voltage:50～300kV
- TEM magnifications:60×～1,000,000×
- Spot resolution:0.20nm
- Line resolution:0.10nm
- Information resolution:0.14nm
- Minimum beam spot:0.3nm
- Maximum angle of convergence:±12°
- STEM magnifications:50×～3,000,000×(+8×zoom)
- STEM image resolution:0.17nm
- Max. tilting angle of sample stage:±40°