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JY/T 011-1996

General rules for transmission electron microscopy

现代分析仪器分析方法通则

透射电子显微镜方法通则

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Table of Contents

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Foreword

The drafting format and method of this Standard complies with the requirements of GB/T 1.1-1993 "Directives for the work of standardization - Unit 1: Drafting and presentation of standards - Part 1: General rules for drafting standards" and GB/T 1.4-88 "Directives for the work of standardization - rules for drafting chemical analysis standards".

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General rules for transmission electron microscopy

1 Scope

This General Rules specify the conventional analytical method of transmission electron microscopy; it is are applicable to high-voltage transmission electron microscopy, high-resolution transmission electron microscopy, ordinary transmission electron microscopy AND simple transmission electron microscopy (hereinafter referred to as TEM).

2 Definitions

This Standard adopts the following definitions:

2.1 Resolution

TEM's capability that can clearly distinguish the minimum distance between two object points and crystal faces.

2.2 Astigmatism

An image blurring that is caused by non-rotational symmetry of electromagnetic lenses' magnetic field, in which the lenses have different focusing ability in mutually perpendicular directions.

2.3 Electron diffraction

The effect whereby electron beams are scattered in crystals; only in the direction of satisfying the Bragg's Law, are there emitting of diffracted beams that are mutually reinforced.

3 Principle

The determinant of TEM imaging is the specimen scattering to incident electron, including elastic scattering and inelastic scattering. When thin-specimen is imaging, the un-scattered electrons shall constitute the background. However, the image contrast depends on different scattering characteristics of each part of the specimen to the electrons. Different experimental conditions shall produce different image contrasts.

TEM can not only show the microstructure morphology of the specimen, but

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3.2 Diffraction modality

If the specimen is crystal, its electron diffraction pattern shall be presented on the focal plane of the objective lens. Change the current of intermediate lens to make the image formed on the focal plane of the objective lens. The electron diffraction pattern on the plane is enlarged by the intermediate lens and the projector lens. Then the enlarged image of electron diffraction pattern shall be obtained on the phosphor screen.

4 Instruments

4.1 Instrument structure

4.1.1 Lighting system

It is composed of electron gun and condenser. Electron gun provides stable electron source of small size and emits high-brightness electron beams. Condenser converges electron beams and irradiates the specimen.

4.1.2 Specimen room and imaging system

The specimen room is under the lighting system. Placed on the specimen holder, the specimen can move or tilt within the specified range. Composed of objective lens, intermediate lens and projector lens, the imaging system enlarges the image and presents it on the phosphor screen.

4.1.3 Observing and recording system

The observing room is under the projector lens. The image on the phosphor screen can be observed through windows. The photographic plate is placed under the phosphor screen. When phosphor screen is erected, it shall expose and record the image.

4.1.4 Other systems and accessories

Other systems and accessories include vacuum system, power system, security system and cooling system. Some are equipped with scanning accessories, micro diffraction apparatus, electron energy loss spectrometer (EELS) and X-ray energy dispersive spectrometer (EDS), etc.

4.2 Technical indicators

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6.2 Test preparation

6.2.1 Centering adjustment

Increase high voltage and filament current. After light spot appears on the phosphor screen, center the lighting system and imaging system. Adjustment of “current center” or “voltage center” shall be strictly in accordance with the instructions for use.

6.2.2 Astigmatism calibration

Use the well-known specimens, for example, micro sieve. Observe the hole at high magnification. Alternately adjust the focus of the objective lens and the anastigmator. When the objective lens is under-focused or over-focused, the uniform and clear Fresnel Fringes image shall be obtained at the edge of the hole.

6.2.3 Magnification calibration

Because of hysteresis effect of electromagnetic lens, there are 5%~10% error between the actual magnification and the reading value. If the magnification is less than 50,000 times, use grating replica (2000 pcs/mm) with 50 nm fringe space to calibrate. For greater magnification, use thin crystal with well-known grating space to calibrate. Shoot photos of different magnifications and obtain the calibration curve by calculation.

6.2.4 Camera constant calibration

Camera constant is the product of diffraction camera length L and wavelength of the incident electron beams k. Because of uncertainty of the length of TEM camera, it needs to use a gold polycrystalline thin film to calibrate.

6.2.5 Magnetic rotation calibration

In the selective-area, when diffraction modality is switched to imaging modality, the change on excitation conditions of intermediate lens shall make the image and the diffraction pattern rotate at different angles, comparing to the actual orientation of specimen crystal. If the magnetic rotation of the image is Φ_i and the magnetic rotation of the pattern is Φ_d. then the magnetic rotation of the image to the pattern shall be $\Phi=\Phi_i-\Phi_d$. Usually, it utilizes external features to directly reflect the calibrated magnetic rotation of MoO_3 thin crystal. Therefore, it shall only need the calibration value of crystal rotation represented by the diffraction pattern to represent the actual rotation of the crystal.

6.2.6 Specimen height calibration

During magnification calibration and selective-area diffraction, in order to

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them coincide. Switch to diffraction modality. Pull out objective lens aperture. The electron diffraction pattern, which reflects the crystallographic characteristics of specimen in micro-area, can be obtained on phosphor screen.

For electron diffraction in selective-area, the size of selective-area is limited by the effect of spherical aberration of objective lens and image focus error. For high-resolution TEM, the diameter of minimum selective-area is about 1 μ. In order to obtain smaller selective-area, it uses micro-diffraction techniques (μ diffraction). In experiment, use scanning transmission operation mode, the beam-spot diameter is reduced to less than 100 nm. The scope of micro-area of specimen that participates in diffraction is limited by the beam-spot diameter.

6.4.3 Bright-field image and dark-field image of diffraction contrast

Usually, the analysis on bright-field imaging and dark-field imaging is always combined with electron diffraction of selective-area, so as to determine the phase's microscopic morphology, lattice type and parameters. If objective lens aperture is used to block diffraction beams, and only transmission beams are allowed to go through the aperture hole to imaging, then, the image obtained shall be called bright-field image of diffraction contrast. If objective lens aperture is used to block transmission beams and most of diffraction beams, and only some diffraction beams are allowed to go through the aperture hole to imaging, obviously, the area contributed to the diffraction beams of specimen or the bright contrast represented by phase in dark field shall be called the dark-field image of diffraction contrast.

Generally speaking, central dark field imaging produces small aberration. Make selective-area diffraction of specimen first. Tilt the specimen to make some diffraction spot to be the brightest (dual-beam conditions) except the transmission spot. Adjust the lighting electron beam to tilt. Move the transmission beam to the original bright diffraction spot. Then the weak spot which is symmetrical to the original bright spot is moved to optical axis and shall get brighter. Allow only this diffraction beam to get through the aperture hole of objective lens to form the central dark-field image. Use specific diffraction beams to form the central dark-field image of diffraction contrast is one of the effective methods to analyze complicating diffraction figure and display each single-phase crystallographic characteristics.

6.4.4 Dark-field image of weak-beam

It is widely used in studies on crystal defects, for example, the bright-field image and central dark-field image have higher resolution. In experiment, make specimen relatively far away from Bragg reflection position, comparing to electron beam. Use the weak first-order diffraction beam to make central dark-field image. Near the center of the crystal dislocation, there are always some

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For example, use accessories of convergent diffraction, add the third condenser above the objective lens OR adopt scanning transmission mode to reduce the specimen height.

6.4.7 Lattice type of crystal and determination of lattice parameters

For the well-known crystal, it can use the standardization of one or several electron diffraction patterns to determine the phase. However, for the unknown phase with complicated structure, it must determine its three-dimensional structure. The spatial periodicity of crystal structure reflected on electron diffraction pattern is the reciprocal space corresponding to its periodicity. Therefore, if a set of electron diffraction patterns, that are of different crystal belts of same-crystal and that has the correlation, can be obtained, then, according to its correlation, its crystal lattice can be rebuilt by calculation, the lattice parameters can be determined, and the phase can be identified.

For this purpose, it shall need dual-tilting or tilting-rotating specimen-holder to make the analyzed crystal to rotate around some low-index crystal orientation, so as to adjust & find the electron diffraction pattern of relevant crystal belt.

6.4.8 Test of small size and shape

When testing the size and shape of micro-particle specimen, in order to eliminate systematic errors of TEM and make the tested specimen to be measured precisely, the most effective method shall be the Internal Standard Method. That is, put a standard specimen of well-known size and the tested specimen on a same copper grid carrier. Put them into TEM to observe and shoot photos. It shall not cause magnification error because the standard specimen and the tested specimen are on a same photographic plate under same shooting conditions. The magnification calculated by standard specimen shall be the magnification of tested specimen. The common use specimen for internal standard method at high magnification is polystyrene sphere or crystal of well-known grating space, e.g., beef liver enzyme hydroperoxide (8.6± 0.2nm).

When magnification is low, it usually uses grating replica as external standard specimen. In order to eliminate the magnification error, the selected magnification shall not be adjusted during specimen and standard specimen observation AND shooting.

6.4.9 Electron energy loss spectroscopy analysis

It is to study the primary process after high-energy electrons are incident to the specimen, motivating electrons of atomic inner shell to ionize. It shall be realized by electron energy loss spectrometer (EELS). EELS is composed of electron energy analyzer, spectrum display, recording system and data

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processing system. After the specimen get through the spectrometer, it shall scatter according to amount of electron energy loss, record the appropriate strength and display the spectrum of electron energy loss. Analyze different spectral region, then information about specimen thickness, chemical composition, electronic density and electronic structure can be obtained.

6.4.10 X-ray energy spectrum analysis

After high-energy electrons are incident to the specimen, electrons of atomic inner shell are motivated to ionize. Then, X-ray is generated when atoms are reverting to basic state. The energy spectrum analysis is to study the motivation process of this characteristic and provide information about chemical composition in micro-area of specimen. X-ray energy dispersive spectrometer (EDS) collects the characteristic X-ray from specimen, enlarge it, and sent it to multichannel analyzer. Scatter according to its different characteristic energy, record the corresponding strength and display X-ray spectrum. Identify the elements contained in the specimen according to qualitative characteristic energy of spectrum peak. Use data processing system to run thin film quantitative analysis program. Calculate the percentage content of each element according to intensity value.

6.5 Instrument check and power off after testing

Take out the specimen. Exit from aperture of objective lens and aperture of selective-area. Adjust to low magnification. Defocus the condenser. Successively cut off filament current, high voltage, main power supply and power supply of other accessories. Cover the back plate of observation room. After 15 mins, turn off cooling water system and regulated power supply.

7 Expression of analysis results

7.1 TEM image is usually provided in photo and interpreted according to mass-thickness contrast, diffraction contrast and phase contrast imaging theory.

7.2 Photographic plate of electron diffraction is standardized and analyzed according to basic theory of electron diffraction to determine crystallographic orientation relationship or crystal structure.

7.3 Provide results of qualitative or quantitative analysis on chemical composition in micro-area of specimen.

8 Safety precautions

8.1 Operating rules of TEM must be strictly obeyed.

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8.2 When power or water is suddenly cut off, it shall sequentially turn off and leave the specimen in original place. After power or water supply is restored, reboot the system to continue the work.

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