Title：**TRANSMISSION ELECTRON MICROSCOPE (TEM)**

報告題名：穿透式電子顯微鏡

Author(s): TSAI-CHEN HSIEH,

Class: 3rd year of Department of Material Science and Engineering

Student ID: D0513682

Course: Surface Analysis

Instructor: Dr. Wei-Chun Lin

Department: Department of Materials Science and Engineering

Academic Year: Semester 1, 2018

Abstract

In this paper, we introduce the transmission electron microscope (TEM). Including the component of TEM and the limitations of TEM. By knowing the principle and the limitation of TEM, we can use it for observed the morphology of our specimen.

Keyword：

Aberration
Aperture
Astigmatism
Application
Characterization
Contrast
Electron Gun
Electromagnetic Lens
Transmission electron microscope

中文摘要

多數材料的改良和研發都藉由微觀結構的改善，因此表面分析技術就更顯得重要。表面分析領域的持續發展為提供科技進步的基礎。其領域中包含的技術有許多種：有進行元素分析的XPS/ESCA(X-射線光電子光譜/化學分析電子光譜)，結構分析的XRD (X-射線繞射分析)和觀察樣品表徵的TEM(穿透式電子顯微鏡)和SEM (掃描式電子顯微鏡)等等。而TEM的分辨率又高於SEM，雖然不如SEM為3D圖示，但也為一非常重要的分析儀器。此篇報告主要著重在穿透式電子顯微鏡(TEM)的介紹，大致分為三部分作介紹，分別為其工作原理、缺點(限制)和應用。在各部分再分割成各自的小部分來講解：工作原理從內部的構造配件開始介紹，缺點以儀器本身所造成的影像失真為主，應用範圍則為現今市場上常介的應用：例如，LED產業、半導體產業等。以淺顯的介紹希望大家可以能大致了解穿透式電子顯微鏡的作用。

關鍵字：

材料分析
穿透式電子顯微鏡
穿透式電子顯微鏡工作原理
穿透式電子顯微鏡構造
穿透式電子顯微鏡影像
結構分析
像差

Table of Content

1. Introduction

1.1History (p.4)
1.2 Component of TEM *(pp.4-6)*
    1.2.1 Electron Gun
    1.2.2 Electromagnetic Lens
    1.2.3 Aperture
    1.2.4 Specimen Holder
    1.2.5 Fluoroscopic Screen
1.3 Sample Preparation *(pp.6-7)*
1.4 Imaging Methods *(p.7)*
1.5Calibrating the TEM *(p.7)*

2. Limitations
2.1 Resolution *(p.8)*
2.2 Aberration *(pp.8-9)*
    2.2.1 Spherical Aberration
    2.2.1 Chromatic Aberration
    2.2.3 Astigmatism
2.3 Contrast *(pp.9-10)*
    2.3.1 Mass Thickness Contrast
    2.3.2 Diffraction Contrast
2.4 Component analysis *(p.10)*

3. Application of TEM *(pp.10-11)*

Reference *(p.12)*

List of Figures

1.1 Component of TEM
1.3 Three common specimen formats
1.4 Spherical aberration
1.5 Spherical aberration vs. No aberration
1.6 Chromatic aberration
1.7 Astigmatism
1.8 Contrast
1.9 TEM image of LED
2.1 TEM image of DRAM
2.2 TEM image of NAND flash memory

List of Tables

1.2 The comparison of electron

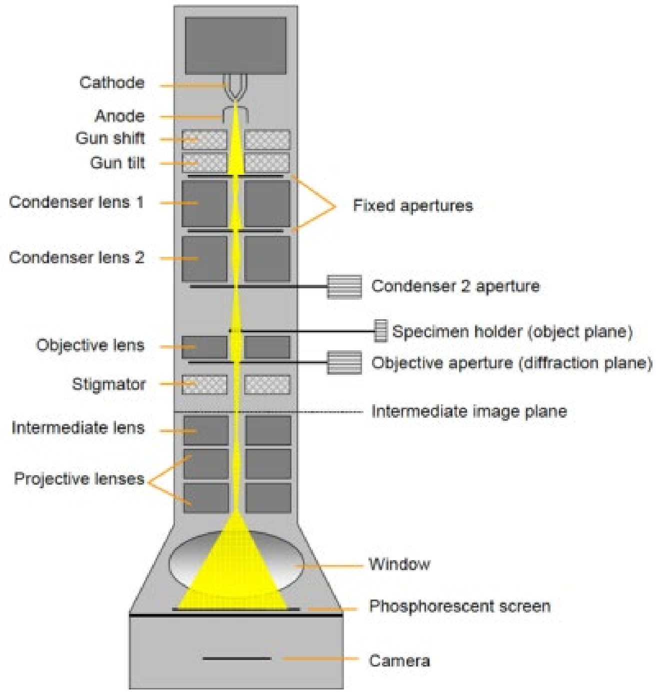
1.1 History

In 1897, J.J. Thomson discovered the electron and it will be deflected by magnetic field.

In 1927, Davisson-Germer discovered electron diffraction.

In 1934, Ernst August Friedrich Ruska designed the first electron microscope.

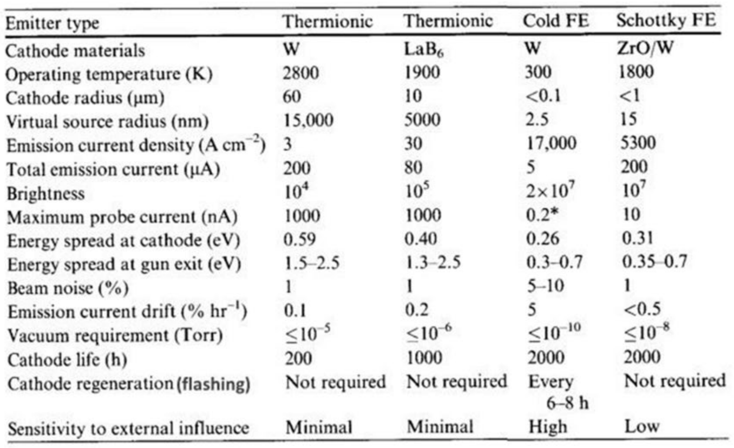
1.2 Component of TEM



1.2.1 Electron Gun

The function of the electron gun is to generate an electron beam, providing a high-energy, sufficiently bright and stable light source. And there are some normal types of electron gun: Thermionic Emission, Field Emission Gun (FEG) and Schottky FEG.

The comparison of electron gun:



Emitter type	Thermionic	Thermionic	Cold FE	Schottky FE
Cathode materials	W	LaB_6	W	ZrO/W
Operating temperature (K)	2800	1900	300	1800
Cathode radius (μm)	60	10	<0.1	<1
Virtual source radius (nm)	15,000	5000	2.5	15
Emission current density (A cm^{-2})	3	30	17,000	5300
Total emission current (μA)	200	80	5	200
Brightness	10^4	10^5	2×10^7	10^7
Maximum probe current (nA)	1000	1000	0.2*	10
Energy spread at cathode (eV)	0.59	0.40	0.26	0.31
Energy spread at gun exit (eV)	1.5–2.5	1.3–2.5	0.3–0.7	0.35–0.7
Beam noise (%)	1	1	5–10	1
Emission current drift (% hr^{-1})	0.1	0.2	5	<0.5
Vacuum requirement (Torr)	$\leq10^{-5}$	$\leq10^{-6}$	$\leq10^{-10}$	$\leq10^{-8}$
Cathode life (h)	200	1000	2000	2000
Cathode regeneration (flashing)	Not required	Not required	Every 6–8 h	Not required
Sensitivity to external influence	Minimal	Minimal	High	Low

1.2.2 Electromagnetic Lens

There are four main form of lenes: Condenser lenses, Objective lenses, Intermediate lenses and Projector lenses.

The condenser lenses is used to adjust the electron beam. The purpose of the condenser lens is to focus the light onto the specimen. This lens is used to form the beam and limit the amount of current in the beam and it is also used to control the diameter of the electron beam.[1]

The objective lenses is the main lenses form of TEM. And it also is the place putting the specimen. The structure of the objective lenses has divided into two parts, the specimen is put in the gap between them.

The "intermediate lens" is placed between the objective lens and the projector lens. The intermediate lens works in the following way. The intermediate lens changes focusing position either on a diffraction pattern or a TEM image produced by the objective lens by adjusting its excitation, and forms the magnified pattern or image on the object plane of the projector lens. Normally, the intermediate lens consists of three parts: The first part mainly selects focusing position, the second part magnifies the focused pattern or image, and the third part mainly achieves rotation-free condition.[2]

Magnification in the electron microscope can be varied from hundreds to several hundred, thousands of times. This is done by varying the strength of the projector and intermediate lenses.

1.2.3 Aperture

The aperture may be fixed in size or variable in size. They can be inserted into the electron beam path or removed, or moved in a plane perpendicular to the electron beam path. Generally TEM has four sets of apertures.

The aperture placed behind the first concentrating lens mainly changes the size of the electron beam and filters out electrons that are too far from the optical axis to increase the quality of the electron beam---enhance coherence and reduce the energy dispersion range.

The aperture (C2 aperture) placed behind the second condenser can change the beam size and the far electrons, and the convergence angle of the specimen.

The aperture placed on the focal plane behind the objective lens, called the objective aperture, is the most important aperture.

The functions of the objective aperture include: (1) filtering electrons that are too far from the optical axis of the objective to improve the focus of the image; (2) improving the image contrast (3) selecting a direct electron beam or a single diffracted electron beam to form a bright field image or a dark field image.

The aperture placed on the first imaging plane is called the selection aperture, and the selection aperture is used to limit the analysis area.

The two apertures closest to the specimen are the most susceptible to contamination and have a life time of one to two years.

1.2.4 Specimen Holder

The function of Specimen Holder is to load the specimen, insert it into the TEM, and provide translation and tilting functions to move the analytically valuable area to the center of the fluorescent screen.

1.2.5 fluoroscopic screen

The fluorescent screen is coated with a fluorescent substance (ZnS). When the electron beam hits the fluorescent substance, the fluorescent substance is excited to emit visible light, so that the electronic image or the diffraction pattern amplified by the projector lens can be converted into a visible light image. In a TEM equipped with a digital camera, the function of the fluorescent screen is to monitor whether or not the maximum brightness is obtained during the adjustment.

1.3 Sample preparation

The TEM specimen is 3 mm. The following figure shows three common three specimen formats:

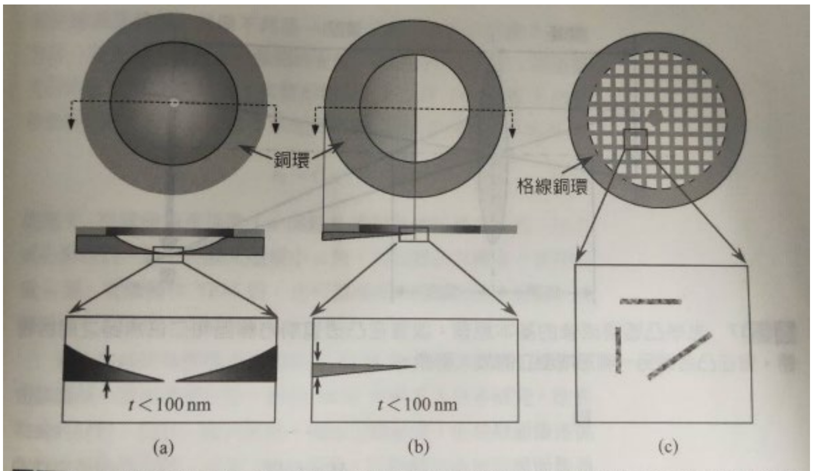


圖1-16 常見 TEM 試片的示意圖：(a) 傳統研磨抛光 + 渦穴研磨型：(b) 改良式研磨抛光型：(c) 碳膜格線銅環支撐微細粉粒型。(a)&(b) 從上而下：俯視圖、剖面側視圖、薄區局部放大圖：(c) 從上而下：俯視圖、局部放大圖。試片的外徑都為 3 厘米。[1]

1.4 Imaging methods

The basic imaging principle of an electromagnetic lens is the same as that of an optical convex lens, and can be expressed by the following formula without considering the rotation of electrons in a magnetic field:

$$+\frac{1}{f_i}=\frac{1}{f}$$

f_0: $object\ \ distance$
f_i: $image\ \ distance$
f: $focal\ \ distance$

Definition of Magnification:

$$M = f_i \,/\, f_0$$

1.5 Calibrating the TEM

An accurate calibration of magnification, diffraction patterns and the relative rotation between the two is critical in quantitative transmission electron microscopy (TEM). Before attempting quantitative TEM analysis, a series of calibrations of the individual TEU should be undertaken. For the magnification calibration of a TEM, the general procedure is to take micrographs of standard samples with known spacings at all magnification values accessible by that standard, Most standards are only suitable for a specific magnification range. New types of calibration samples can now be made with the technology of the semiconductor industry. One major advantage of this type of calibration sample is that the sample can be self-calibrated to accuracies well beyond the resolution of a TEM.[3]

2.1 Resolution

Due to the existence of the diffraction phenomenon, the image formed by the point source passing through the lens is not a point but a disc. The resolution is the smallest distance between the two discs that can be resolved. The smaller the distance, the higher the resolution. The limit that the human eye can distinguish is that the center overlap strength is 81% of the center strength of the disc. In TEM, the resolution is related to the objective lens. The smaller the objective lens, the better the resolution, but too small will make the Airy disc larger due to the diffusion effect.

Most suitable aperture diameter (r):

$$d = 2\alpha f$$

$$\alpha = 0.77\lambda^{\frac{1}{4}}C_s^{\frac{1}{4}}$$

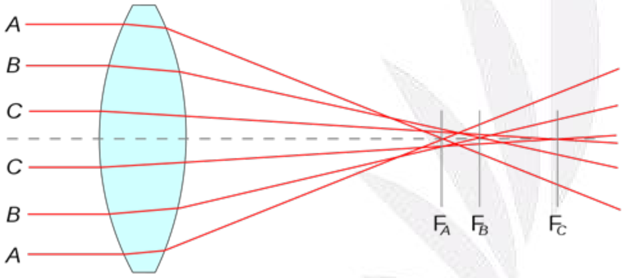
$$r_{m_{i}n} = 0.91\lambda^{\frac{3}{4}}c_s^{\frac{1}{4}}$$

2.2 Aberration

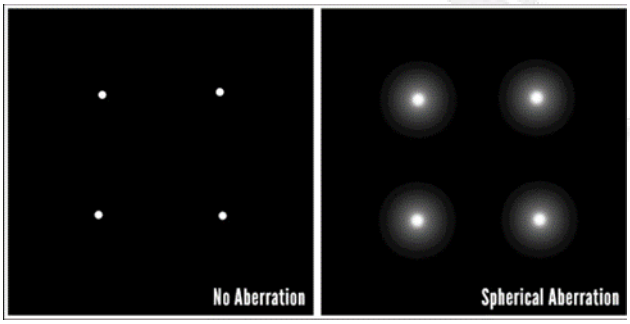
An electron microscope, like an optical microscope, cannot have an ideal lens. There are some main types of lens defects are listed below.

2.2.1 Spherical aberration

The high-energy electrons passing through the edge of the lens are deflected at a greater angle to focus closer to the lens. Therefore, after an ideal point source passes through the lens, its image is no longer an ideal source, but a halo of the disc. In the past, spherical aberration greatly affected the resolution. However, since 1998, TEM has added a spherical aberration corrector. The spherical aberration is significantly improved. So that the resolution of today's TEM can be as low as 1 angstrom.



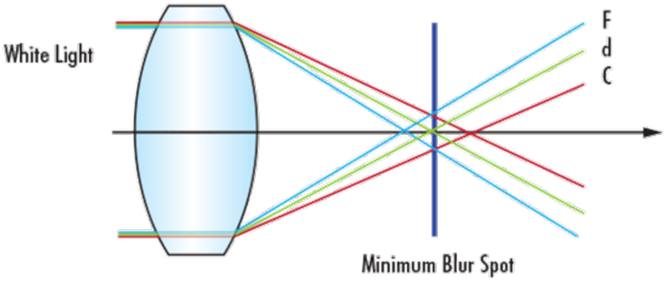
2



2.2.1 Chromatic Aberration

Through high-energy electrons of the same lens radius, the lower energy is deflected at a larger angle, so it is concentrated closer to the lens. This effect is caused by the aperture. The smaller the aperture chromatic aberration, the lower the spherical aberration. A balance needs to be found among the three. The following formula is the minimum radius of an optical disc that is refracted by electrons of different energies:

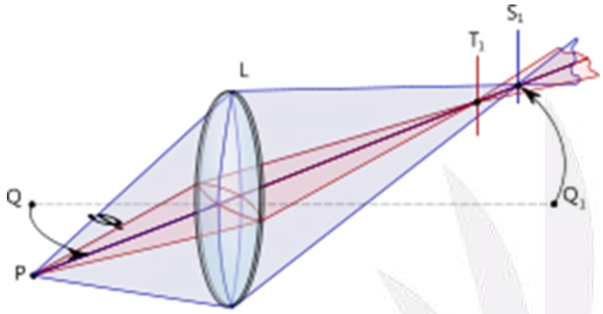
$$r_C = C_C\alpha\left(\frac{\Delta E}{E}\right)$$



3

2.2.3 Astigmatism

Focusing on different distances on different planes through high-energy electrons of the same radius of the lens. This is a defect that must be adjusted to make high-resolution images. It has now been improved with astigmator and computer programs.



2.3 Contrast

Image contrast is an important key to judging information.

2.3.1 Mass thickness Contrast

The contrast of the thickness is formed by the scattering of electrons. The larger the atomic order, the smaller the proportion of direct electrons at a small angle and the darker the image. High angle scattered electrons increase as the thickness of the sheet increases.

2.3.2 Diffraction Contrast

Diffraction contrast: When the lattice plane and the electron beam are at an angle of Bragg will occur a strong diffraction, and the number of electrons that generate strong diffraction is relatively reduced, forming a darker region in the image.

2.4 Component analysis

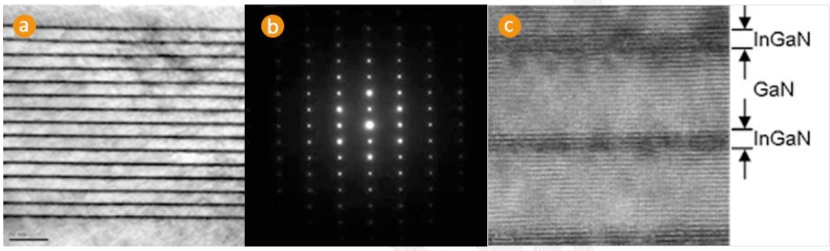
Let’s talk about the three main types of component analysis methods: fixed-point analysis, line profile , and elemental mapping.

Fixed point can be use in both STEM and TEM methods. And the line profile which is used with the STEM method is much better than the TEM does.

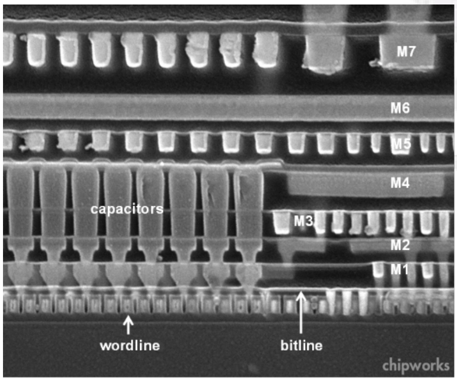
Due to TEM is unable to detect its component signals by image, and must rely on other ancillary equipment such as energy disperser (EDS) and electronic energy loss spectrometer(EELS).

3.Application Of TEM

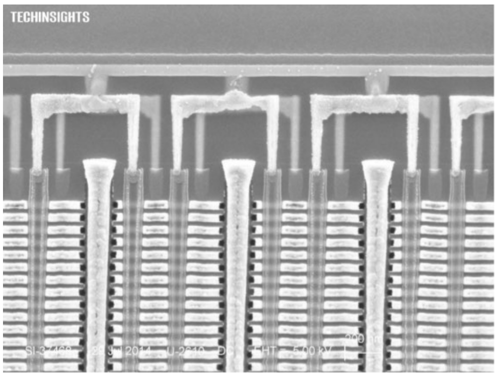
Due to the advancement of TEM technology, TEM has been used in various industries. For example, the LED industry, dynamic random access memory (DRAM) and flash memory (NAND Flash Memory), etc. It can even overcome the unstable state of the liquid under vacuum and detect the liquid sample.[4]



4.TEM image of LED



5.TEM image of DRAM



6.TEM image of NAND flash memory

References

[1] C.Wang, “Lenses and Apertures of A TEM,” *EMA 6518 Course Present.*

[2] “Keywords "intermediate lens" | Keywords | Glossary of TEM Terms | JEOL.” [Online]. Available: https://www.jeol.co.jp/en/words/emterms/search_result.html?keyword=intermediate lens. [Accessed: 23-Dec-2018].

[3] J. P.Mccaffrey, “Calibrating the tem,” 1976.

[4] T.Xu andL.Sun, “Investigation on material behavior in liquid by in situ TEM,” *Superlattices Microstruct.*, vol. 99, pp. 24–34, 2016.

Figure

1.近代穿透式電子顯微鏡實務

2 http://highscope.ch.ntu.edu.tw/wordpress/wp-content/uploads/2009/09/12.jpg

3 https://photographylife.com/what-is-chromatic-aberration

4 TEM image of LED http://www.ma-tek.com/zh-tw/services/index/material/Pro_20

5 TEM image of DRAM https://semimd.com/chipworks/2014/02/07/intels-e-dram-shows-up-in-the-wild/

6 TEM image of NAND flash memory https://www.google.com/search?q=TEM+NAND&rlz=1C1JZAP_zh-TWTW696TW696&source=lnms&tbm=isch&sa=X&ved=0ahUKEwiFvrnijMPfAhWJy7wKHfx7DtcQ_AUIDigB&biw=1366&bih=577#imgrc=ZND46tZNNvB9hM: