

MATERIALS CHARACTERIZATION

Introduction to Microscopic and Spectroscopic Methods

Yang Leng

Hong Kong University of Science and Technology



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Preface

This book serves as a textbook for introductory level courses on materials characterization for university students, and also a reference book for scientists and engineers who need basic knowledge and skills in materials characterization. After teaching courses in materials characterization for several years, I strongly feel that a comprehensive single-volume book covering state-of-the-art techniques in materials characterization is clearly needed. The book is based on my teaching notes for an introductory undergraduate course on materials characterization.

This book covers two main aspects of materials characterization: materials structures and chemical analysis. The first five chapters cover commonly used techniques for microstructure analysis including light microscopy, transmission and scanning electron microscopy and scanning probe microscopy. X-ray diffraction is introduced in Chapter 2, even though it is not a microscopic method, because it is the main technique currently used to determine crystal structures. The basic concepts of diffraction introduced in Chapter 2 are also important for understanding analytical methods of transmission electron microscopy. The final five chapters of the book mainly cover techniques for chemical analysis. These chapters introduce the most widely used chemical analysis techniques for solids: fluorescence X-ray spectroscopy, X-ray energy dispersive spectroscopy for microanalysis in electron microscopes, popular surface chemical analysis techniques of X-ray photoelectron spectroscopy (XPS) and secondary ion mass spectroscopy (SIMS), and the molecular vibrational spectroscopy methods of Fourier transform infrared (FTIR) and Raman. Thermal analysis is a rather unique technique and hard to categorize as it is neither a microscopic nor a spectroscopic method. I include thermal analysis in this book because of its increasing applications in materials characterization.

I have tried to write the book in an easy-to-read style by keeping theoretical discussion to a minimal level of mathematics and physics. Only are theories introduced when they are required for readers to understand related working principles of characterization techniques. Technical aspects of preparing specimens and operating instruments are also introduced so as to help readers understand what should be done and what should be avoided in practice. For most engineers and scientists, the interpretation and analysis of characterization outputs are even more important than the technical skills of characterization. Thus, the book provides a number of examples for each characterization technique to help readers interpret and understand analysis outputs. In addition, problems are provided at the end of each chapter for those using the book as a course text.

Note that there are paragraphs printed in italic font in Chapters 2, 3, 4, 5, 6, 7, 9 and 10. These paragraphs provide more extensive knowledge, and are not essential in order for readers to understand the main contents of this book. Readers may skip these paragraphs if they choose.

Finally, I gratefully acknowledge the considerable help I have received from my colleagues as well as my graduate students at the Hong Kong University of Science and Technology during preparation of the manuscript. In particular, the following people helped me to review the manuscript and provided valuable comments: Jiaqi Zheng for Chapter 2, Ning Wang for Chapter 3, Gu Xu for Chapter 4, Jie Xhie for Chapter 5, Lutao Weng for Chapters 7 and 8 and Borong Shi for Chapter 9. Jingshen Wu, who shares teaching duties with me in the materials characterization course, provided valuable suggestions and a number of great micrographs. My graduate students Xiong Lu and Renlong Xin, and especially Xiang Ge, have drawn and reproduced many figures of this book. Also, I am particularly grateful to my friend Martha Dahlen who provided valuable suggestions in English writing and editing.

Yang Leng

The Solutions Manual for this book can be found at <http://www.wiley.com/go/leng>

1

Light Microscopy

Light or optical microscopy is the primary means for scientists and engineers to examine the microstructure of materials. The history of using a light microscope for microstructural examination of materials can be traced back to the 1880s. Since then, light microscopy has been widely used by metallurgists to examine metallic materials. Light microscopy for metallurgists became a special field named *metallography*. The basic techniques developed in metallography are not only used for examining metals, but also are used for examining ceramics and polymers. In this chapter, light microscopy is introduced as a basic tool for microstructural examination of materials including metal, ceramics and polymers.

1.1 Optical Principles

1.1.1 Image Formation

Reviewing the optical principles of microscopes should be the first step to understanding light microscopy. The optical principles of microscopes include image formation, magnification and resolution. Image formation can be illustrated by the behavior of a light path in a compound light microscope as shown in Figure 1.1. A specimen (*object*) is placed at position A where it is between one and two focal lengths from an *objective lens*. Light rays from the object firstly converge at the objective lens and are then focused at position B to form a magnified inverted image. The light rays from the image are further converged by the second lens (*projector lens*) to form a final magnified image of an object at C.

The light path shown in Figure 1.1 generates the real image at C on a screen or camera film, which is not what we see with our eyes. Only a real image can be formed on a screen and photographed. When we examine microstructure with our eyes, the light path in a microscope goes through an *eyepiece* instead of projector lens to form a *virtual image* on the human eye retina, as shown in Figure 1.2. The virtual image is inverted with respect to the object. The virtual image is often adjusted to be located as the minimum distance of eye focus, which is conventionally taken as 250 mm from the eyepiece. A modern microscope is commonly equipped with a device to switch between eyepiece and projector lens for either recording images on photographic film or sending images to a computer screen.

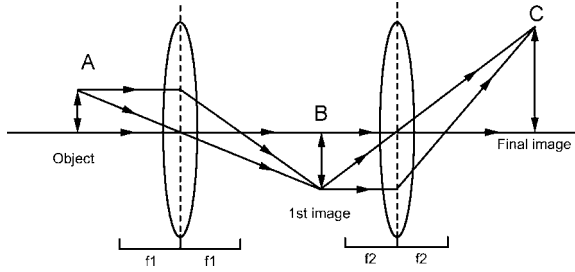


Figure 1.1 Principles of magnification in a microscope.

Advanced microscopes made since 1980 have a more complicated optical arrangement called ‘infinity-corrected’ optics. The objective lens of these microscopes generates parallel beams from a point on the object. A tube lens is added between the objective and eyepiece to focus the parallel beams to form an image on a plane, which is further viewed and enlarged by the eyepiece.

The magnification of a microscope can be calculated by linear optics, which tells us the magnification of a convergent lens M .

$$M = \frac{v - f}{f} \quad (1.1)$$

where f is the focal length of the lens and v is the distance between the image and lens. A higher magnification lens has a shorter focal length as indicated by Equation 1.1. The total

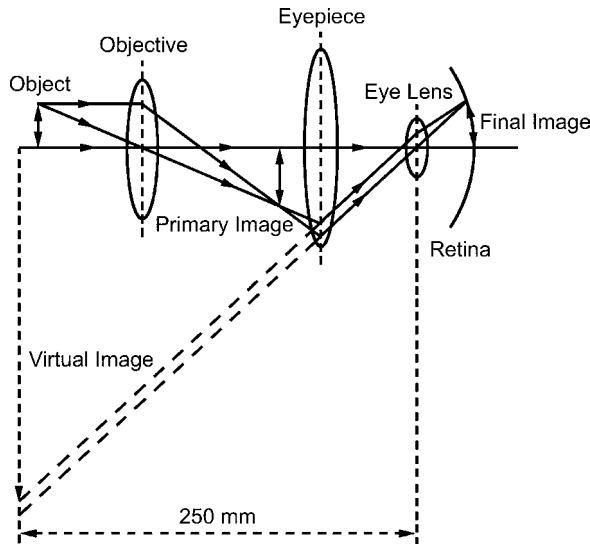


Figure 1.2 Schematic path of light in a microscope with eyepiece. The virtual image is reviewed by a human eye composed of eye lens and retina.

magnification of a compound microscope as shown in Figure 1.1 should be the magnification of the objective lens multiplied by that of the projector lens.

$$M = M_1 M_2 = \frac{(v_1 - f_1)(v_2 - f_2)}{f_1 f_2} \quad (1.2)$$

When an eyepiece is used, the total magnification should be the objective lens magnification multiplied by eyepiece magnification.

1.1.2 Resolution

We naturally ask whether there is any limitation for magnification in light microscopes because Equation 1.2 suggests there is no limitation. However, meaningful magnification of a light microscope is limited by its *resolution*. Resolution refers to the minimum distance between two points at which they can be visibly distinguished as two points. The resolution of a microscope is theoretically controlled by the diffraction of light.

Light diffraction controlling the resolution of microscope can be illustrated with the images of two self-luminous point objects. When the point object is magnified, its image is a central spot (the *Airy disk*) surrounded by a series of diffraction rings (Figure 1.3), not a single spot. To distinguish between two such point objects separated by a short distance, the Airy disks should not severely overlap each other. Thus, controlling the size of the Airy disk is the key to controlling resolution. The size of the Airy disk (d) is related to wavelength of light (λ)

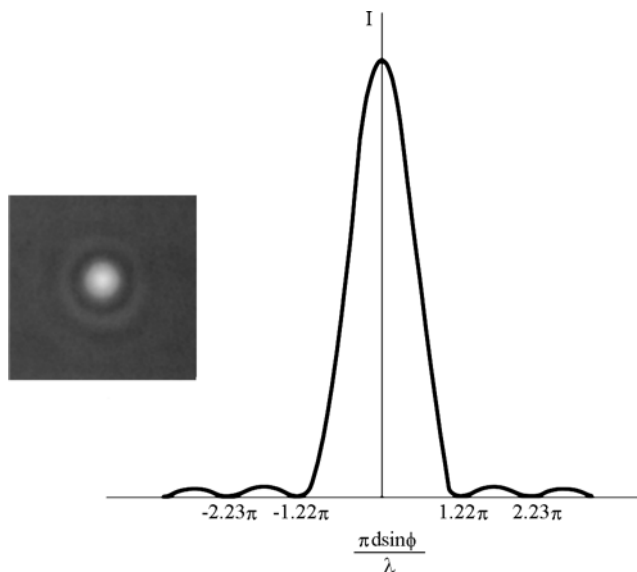


Figure 1.3 A self-luminous point object and the light intensity distribution along a line passing through its center.

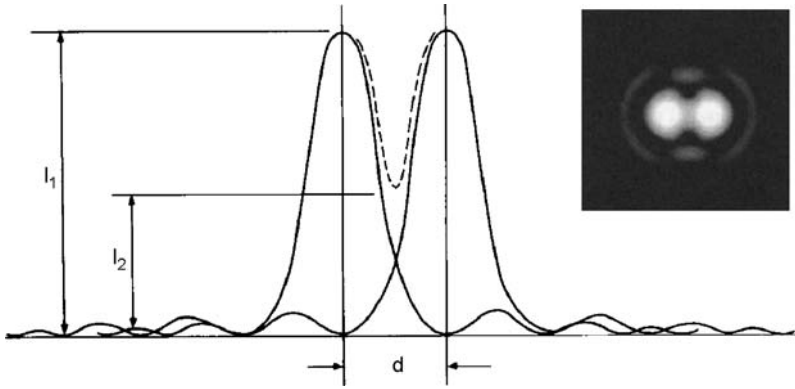


Figure 1.4 Intensity distribution of two Airy disks with a distance $\frac{d}{2}$. I_1 indicates the maximum intensity of each point and I_2 represents overlap intensity.

and angle of light coming into the lens. The resolution of a microscope (R) is defined as the minimum distance between two Airy disks that can be distinguished (Figure 1.4). Resolution is a function of microscope parameters as shown in the following equation.

$$R = \frac{d}{2} = \frac{0.61\lambda}{\mu \sin \alpha} \quad (1.3)$$

where μ is the refractive index of the medium between the object and objective lens and α is the half-angle of the cone of light entering the objective lens (Figure 1.5). The product, $\mu \sin \alpha$, is called the *numerical aperture (NA)*.

According to Equation 1.3, to achieve higher resolution we should use shorter wavelength light and larger NA. The shortest wavelength of visible light is about 400 nm,

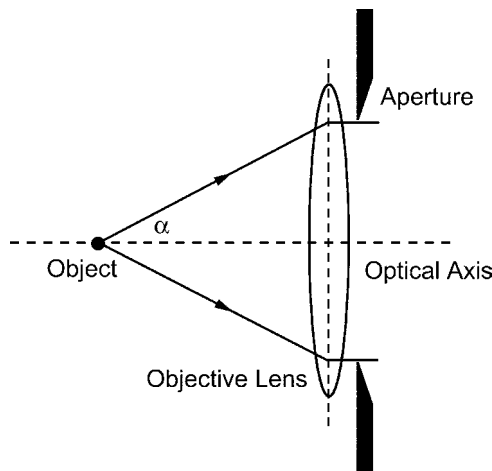


Figure 1.5 The cone of light entering an objective lens showing α is the half angle.

while the NA of the lens depends on α and the medium between the lens and object. Two media between object and objective lens are commonly used: either air for which $\mu = 1$, or oil for which $\mu \approx 1.5$. Thus, the maximum value of NA is about 1.5. We estimate the best resolution of a light microscope from Equation 1.3 as about $0.2 \mu\text{m}$.

Effective Magnification

Magnification is meaningful only in so far as the human eye can see the features resolved by the microscope. Meaningful magnification is the magnification that is sufficient to allow the eyes to see the microscopic features resolved by the microscope. A microscope should enlarge features to about 0.2 mm , the resolution level of the human eye. Thus, the *effective magnification* of light microscope should approximately be $M_{\text{eff}} = 0.2 \div 0.2 \times 10^3 = 1.0 \times 10^3$.

A higher magnification than the effective magnification only makes the image bigger, may make eyes more comfortable during observation, but does not provide more detail in an image.

Brightness and Contrast

To make a microscale object in a material specimen visible, high magnification is not sufficient. A microscope should also generate sufficient *brightness* and *contrast* of light from the object. Brightness refers to the intensity of light. In a transmission light microscope the brightness is related to the numerical aperture (NA) and magnification (M).

$$\text{Brightness} = \frac{(NA)^2}{M^2} \quad (1.4)$$

In a reflected light microscope the brightness is more highly dependent on NA .

$$\text{Brightness} = \frac{(NA)^4}{M^2} \quad (1.5)$$

These relationships indicate that the brightness decreases rapidly with increasing magnification, and controlling NA is not only important for resolution but also for brightness, particularly in a reflected light microscope.

Contrast is defined as the relative change in light intensity (I) between an object and its background.

$$\text{Contrast} = \frac{I_{\text{object}} - I_{\text{background}}}{I_{\text{background}}} \quad (1.6)$$

Visibility requires that the contrast of an object exceeds a critical value called the *contrast threshold*. The contrast threshold of an object is not constant for all images but varies with image brightness. In bright light, the threshold can be as low as about 3% while in dim light the threshold is greater than 200%.

1.1.3 Depth of Field

Depth of field is an important concept when photographing an image. It refers to the range of position for an object in which image sharpness does not change. As illustrated in Figure 1.6, an object image is only accurately in focus when the object lies in a plane within a certain

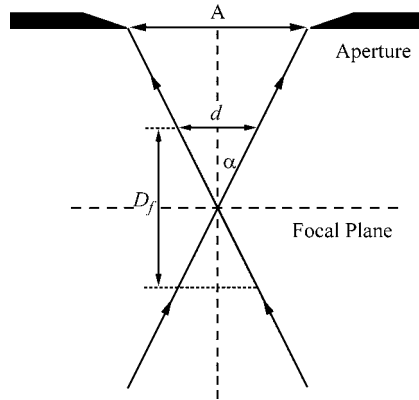


Figure 1.6 Geometric relation among the depth of field (D_f), the half angle entering the objective lens (α) and the size of Airy disk (d).

distance from the objective lens. The image is out of focus when the object lies either closer to or farther from the lens. Since the diffraction effect limits the resolution R , it does not make any difference to the sharpness of the image if the object is within the range of D_f shown in Figure 1.6. Thus, the depth of field can be calculated.

$$D_f = \frac{d}{\tan \alpha} = \frac{2R}{\tan \alpha} = \frac{1.22\lambda}{\mu \sin \alpha \tan \alpha} \quad (1.7)$$

Equation 1.7 indicates that a large depth of field and high resolution cannot be obtained simultaneously; thus, a larger D_f means a larger R and worse resolution. We may reduce angle α to obtain a better depth of field only at the expense of resolution. For a light microscope, α is around 45° and the depth of field is about the same as its resolution.

We should not confuse *depth of field* with *depth of focus*. Depth of focus refers to the range of image plane positions at which the image can be viewed without appearing out of focus for a fixed position of the object. In other words, it is the range of screen positions in which and images can be projected in focus. The depth of focus is M^2 times larger than the depth of field.

1.1.4 Aberrations

The aforementioned calculations of resolution and depth of field are based on the assumptions that all components of the microscope are perfect, and that light rays from any point on an object focus on a correspondingly unique point in the image. Unfortunately, this is almost impossible due to image distortions by the lens called *lens aberrations*. Some aberrations affect the whole field of the image (*chromatic* and *spherical aberrations*), while others affect only off-axis points of the image (*astigmatism* and *curvature of field*). The true resolution and depth of field can be severely diminished by lens aberrations. Thus, it is important for us to have a basic knowledge of aberrations in optical lenses.

Chromatic aberration is caused by the variation in the refractive index of the lens in the range of light wavelengths (*light dispersion*). The refractive index of lens glass is greater for

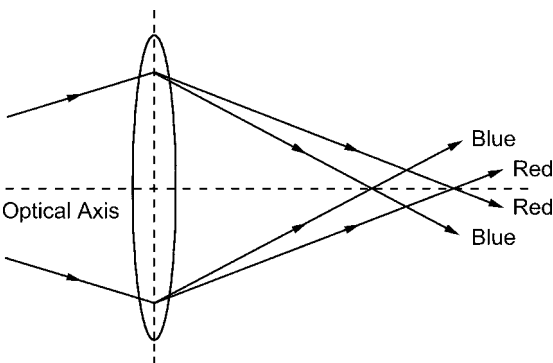


Figure 1.7 Paths of rays in white light illustrating chromatic aberration.

shorter wavelengths (for example, blue) than for longer wavelengths (for example, red). Thus, the degree of light deflection by a lens depends on the wavelength of light. Because a range of wavelengths is present in ordinary light (white light), light cannot be focused at a single point. This phenomenon is illustrated in Figure 1.7.

Spherical aberration is caused by the spherical curvature of a lens. Light rays from a point on the object on the optical axis enter a lens at different angles and cannot be focused at a single point, as shown in Figure 1.8. The portion of the lens farthest from the optical axis brings the rays to a focus nearer the lens than does the central portion of the lens.

Astigmatism results when the rays passing through vertical diameters of the lens are not focused on the same image plane as rays passing through horizontal diameters, as shown in Figure 1.9. In this case, the image of a point becomes an elliptical streak at either side of the best focal plane. Astigmatism can be severe in a lens with asymmetric curvature.

Curvature of field is an off-axis aberration. It occurs because the focal plane of an image is not flat but has a concave spherical surface, as shown in Figure 1.10. This aberration is especially troublesome with a high magnification lens with a short focal length. It may cause unsatisfactory photography.

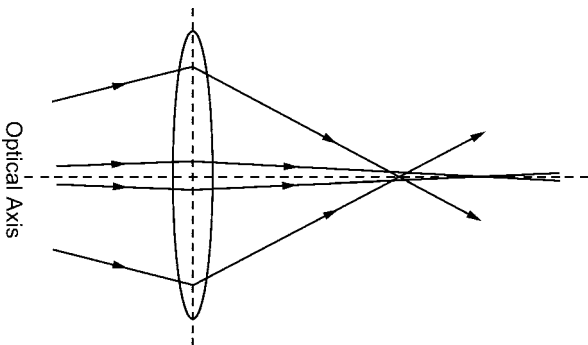


Figure 1.8 Spherical aberration.

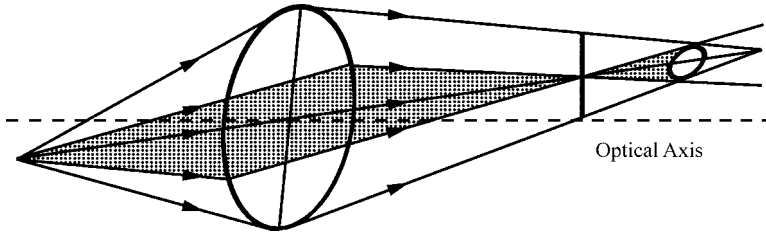


Figure 1.9 Astigmatism is an off-axis aberration.

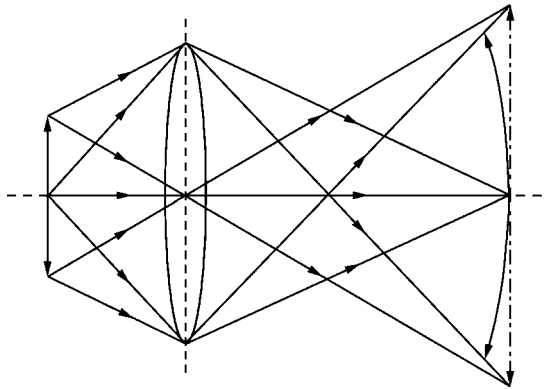


Figure 1.10 Curvature of field is an off-axis aberration.

There are a number of ways to compensate for and/or reduce lens aberrations. For example, combining lenses with different shapes and refractive indices corrects chromatic and spherical aberrations. Selecting single wavelength illumination by the use of filters helps eliminate chromatic aberrations. We expect that the extent to which lens aberrations have been corrected is reflected in the cost of the lens. It is a reason that we see huge price variation in microscopes.

1.2 Instrumentation

A light microscope includes the following main components:

- Illumination system;
- Objective lens;
- Eyepiece;
- Photomicrographic system; and
- Specimen stage.

A light microscope for examining material microstructure can use either transmitted or reflected light for illumination. *Reflected light microscopes* are the most commonly used for metallography, while *transmitted light microscopes* are typically used to examine transparent

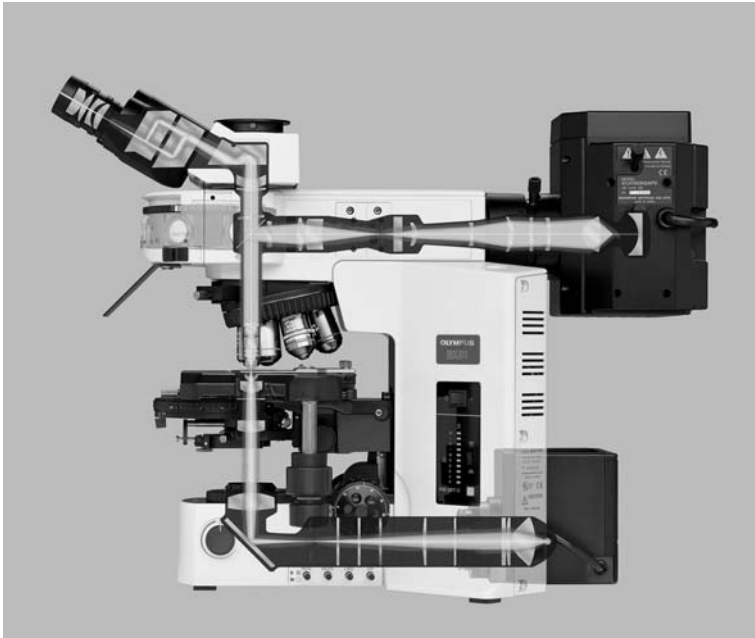


Figure 1.11 An Olympus light microscope used for material examination. The microscope includes transmission and reflection illumination systems. (This image is courtesy of Olympus Corporation.)

or semi-transparent materials, such as certain types of polymers. Figure 1.11 illustrates the structure of a light microscope for materials examination.

1.2.1 Illumination System

The illumination system of a microscope provides visible light by which the specimen is observed. There are three main types of electric lamps used in light microscopes:

1. Low-voltage tungsten filament bulbs;
2. Tungsten–halogen bulbs; and
3. Gas discharge tubes.

Tungsten bulbs provide light of a continuous wavelength spectrum from about 300 to 1500 nm. The *color temperature* of the light, which is important for color photography, is relatively low. Low color temperature implies warmer (more yellow–red) light while high color temperature implies colder (more blue) light. Tungsten–halogen bulbs, like ordinary tungsten bulbs, provide a continuous spectrum. The light is brighter and the color temperature is significantly higher than ordinary tungsten bulbs. The high filament temperature of tungsten–halogen bulbs, however, needs a heat filter in the light path and good ventilation. Gas discharge tubes filled with pressurized mercury or xenon vapor provide extremely high brightness. The more commonly used tubes are filled with mercury, of which the arc has a

discontinuous spectrum. Xenon has a continuous spectrum and very high color temperature. As with tungsten–halogen bulbs, cooling is required for gas discharge tubes.

In a modern microscope, the illumination system is composed of a light lamp (commonly a tungsten–halogen bulb), a *collector lens* and a *condenser lens* to provide integral illumination; such a system is known as the *Köhler system*. The main feature of the Köhler system is that the light from the filament of a lamp is firstly focused at the front focal plane of the condenser lens by a collector lens. Then, the condenser lens collects the light diverging from the source and directs it at a small area of the specimen be examined. The Köhler system provides uniform intensity of illumination on the area of specimen. The system generates two sets of *conjugate focal planes* along the optic axis of a microscope as shown in Figure 1.12. One set of focal planes is for illuminating rays; these are known as the *conjugate aperture planes*. Another set comprises the image-forming rays called the *conjugate field planes*. During normal microscope operation, we see only the image-forming rays through the eyepiece. We can use the illuminating rays to check the alignment of the optical system of the microscope.

There are two important controllable diaphragms in the Köhler system: the *field diaphragm* and the *aperture diaphragm*. The field diaphragm is placed at a focal plane for image formation. Its function is to alter the diameter of the illuminated area of the specimen. When the condenser is focused and centered, we see a sharp image of the field diaphragm with the image of specimen

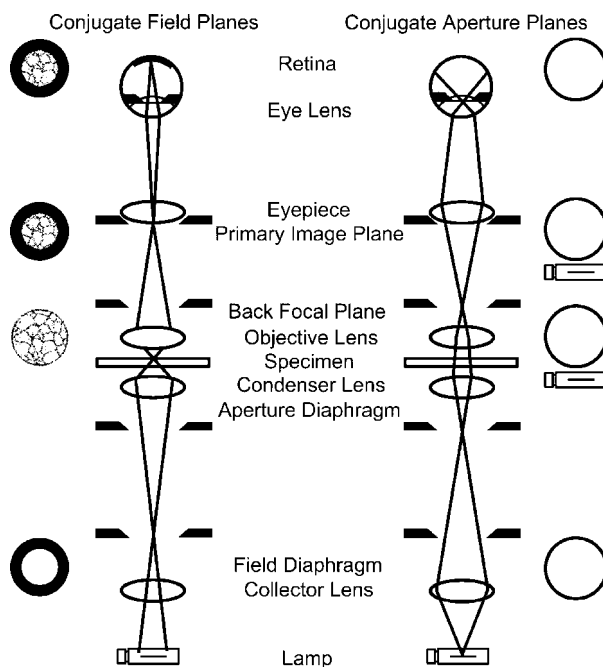


Figure 1.12 Two sets of conjugate focal planes in the Köhler system illustrated in a transmitted light microscope. Image-forming rays focus on the field planes and illuminating rays focus on the aperture planes. The far left-hand and far right-hand parts of the diagram illustrate the images formed by image-forming rays and illuminating rays, respectively. (Reproduced with permission from D.B. Murphy, *Fundamentals of Light Microscopy and Electronic Imaging*, Wiley-Liss. © 2001 John Wiley & Sons Inc.)

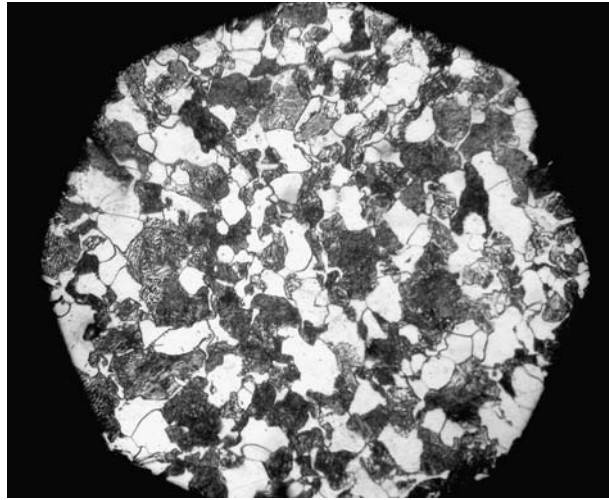


Figure 1.13 Image of the field diaphragm with an image of the specimen. Magnification 100 $\times$.

(Figure 1.13). The field diaphragm restricts the area of view and blocks scattering light that could cause glare and image degradation if they entered the objective lens and eyepiece. The aperture diaphragm is placed at a focus plane of illuminating rays. Its function is to control α , and thus affect the image resolution and depth of field (Section 1.2). We cannot see the aperture diaphragm with the image of specimen. Figure 1.14 illustrates the influence of the aperture diaphragm on the image of a specimen.

The main difference between transmitted light and reflected light microscopes is the illumination system. The Köhler system of reflected light illumination (*epi-illumination*) is illustrated in Figure 1.15 in which a *relay lens* is included. The illuminating rays are reflected by a semi-transparent reflector to illuminate the specimen through an objective lens. There is no difference in how reflected and transmitted light microscopes direct light rays after the rays leave the specimen. There may be a difference in the relative position of the field and aperture diaphragms (Figure 1.12). However, the field diaphragm is always on the focal plane of the image-forming rays while the aperture diaphragm is on a focal plane of the illuminating rays.

Light filters are often included in the light path of illumination systems, even though they are not shown in Figures 1.12 and 1.15. Light filters are used to control wavelengths and intensity of illumination in microscopes in order to achieve optimum visual examination for photomicrography. *Neutral density (ND) filters* can regulate light intensity without changing wavelength. *Colored filters* and *interference filters* are used to isolate specific colors or bands of wavelength. The colored filters are commonly used to produce a broad band of color, while the interference filters offers sharply defined bandwidths. Colored filters are used to match the color temperature of the light to that required by photographic films. Selected filters can also increase contrast between specimen areas with different colors. *Heat filters* absorb much of the infrared radiation which causes heating of specimen when a tungsten-halogen bulb is used as light source.

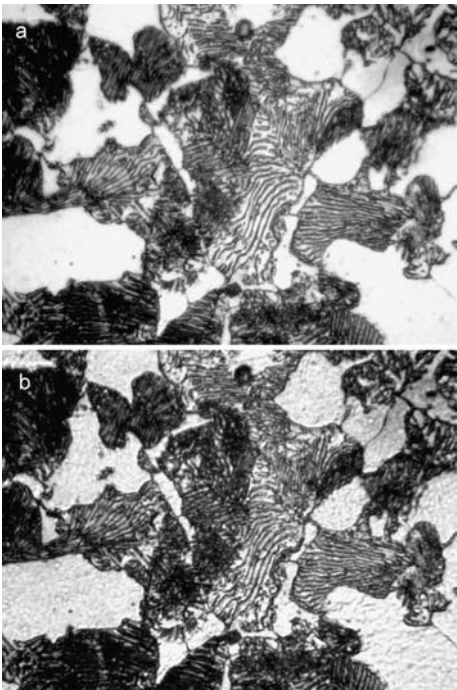


Figure 1.14 Effect of aperture diaphragm on specimen image when: (a) the aperture is large; and (b) the aperture is small. Magnification 500 $\times$.

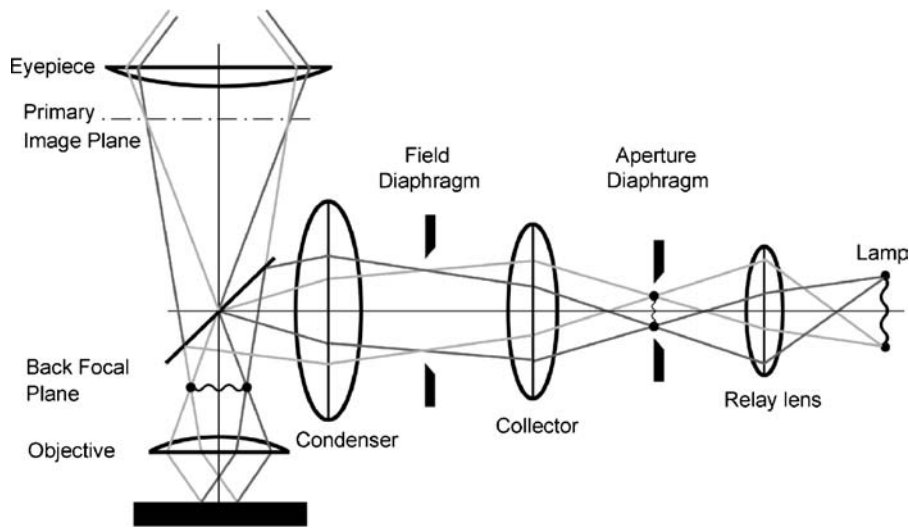


Figure 1.15 Illumination system of a reflected light microscope with illuminating rays.

1.2.2 Objective Lens and Eyepiece

The objective lens is the most important optical component of a light microscope. The magnification of the objective lens determines the total magnification of the microscope because eyepieces commonly have a fixed magnification of $10\times$. The objective lens generates the primary image of the specimen, and its resolution determines the final resolution of the image. The numerical aperture (NA) of the objective lens varies from 0.16 to 1.40, depending on the type of lens. A lens with a high magnification has a higher NA . The highest NA for a dry lens (where the medium between the lens and specimen is air) is about 0.95. Further increase in NA can be achieved by using a lens immersed in an oil medium. The oil immersion lens is often used for examining microstructure greater than $1000\times$ magnification.

Classification of the objective lens is based on its aberration correction capabilities, mainly chromatic aberration. The following lenses are shown from low to high capability.

- Achromat;
- Semi-achromat (also called ‘fluorite’); and
- Apochromat.

The achromatic lens corrects chromatic aberration for two wavelengths (red and blue). It requires green illumination to achieve satisfactory results for visual observation and black and white photography. The semi-achromatic lens improves correction of chromatic aberration. Its NA is larger than that of an achromatic lens with the same magnification and produces a brighter image and higher resolution of detail. The apochromatic lens provides the highest degree of aberration correction. It almost completely eliminates chromatic aberration. It also provides correction of spherical aberration for two colors. Its NA is even larger than that of a semi-achromatic lens. Improvement in quality requires a substantial increase in complexity of the lens structure, and costs. For example, an apochromatic lens may contain 12 or more optical elements.

The characteristics of an objective lens are engraved on the barrel as shown in Figure 1.16. Engraved markings may include the following abbreviations.

- ‘FL’, ‘FLUOR’ or ‘NEOFLUOR’ stands for ‘fluorite’ and indicates the lens is semi-achromatic;
- ‘APO’ indicates that the lens is apochromatic;
- If neither of the above markings appears, then the lens is achromatic;
- ‘PLAN’ or ‘PL’ stands for ‘planar’ and means the lens is corrected for curvature of field, and thus generates a flat field of image;
- ‘DIC’ means the lens includes a Wollaston prism for *differential interference contrast* (Section 1.4.4);
- ‘PH’ or ‘PHACO’ means the lens has a phase ring for phase contrast microscopy (Section 1.1.4.2); and
- ‘number/number’ indicates *magnification/numerical aperture*. Thus, ‘40/0.75’ means the lens has a magnification of $40\times$ and a numerical aperture of 0.75.

The eyepiece is used to view the real primary image formed by the objective lens. In some cases it also completes the correction of aberrations. The eyepiece allows a glass disc with an

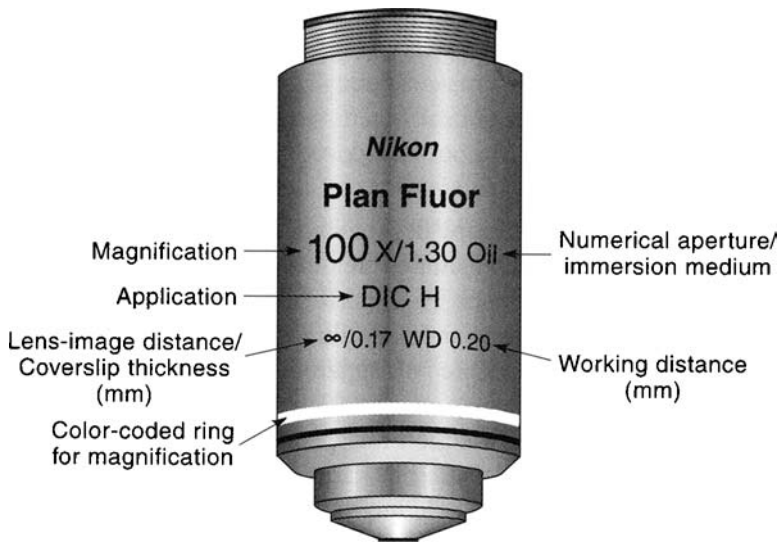


Figure 1.16 Engraved markings on the barrel of an objective lens. (Reproduced with permission from D.B. Murphy, *Fundamentals of Light Microscopy and Electronic Imaging*, Wiley-Liss. © 2001 John Wiley & Sons Inc.)

etched graticule to be inserted into the optical path. The graticule serves as a micrometer for measurement. The eyepiece has either a helical thread or a sliding mount as a focusing mechanism. Importantly, the focusing mechanism of an eyepiece provides a ‘parfocal’ adjustment of the optics so that the same focal plane examined by the eye will be in focus on the film plane of the camera mounted on microscope. Thus, focusing the eyepiece is a necessary step before photographing images in a microscope.

We can summarize the methods for achieving optimum resolution and depth of field in light microscopy. While both resolution and depth of field are crucial for achieving high quality images, one often is achieved at the expense of the other. Thus, compromises must be made while using good judgment.

Steps for Optimum Resolution

- Use an objective lens with the highest NA possible;
- Use high magnification;
- Use an eyepiece compatible with the chosen objective lens;
- Use the shortest possible wavelength light;
- Keep the light system properly centered;
- Use oil immersion lenses if available;
- Adjust the field diaphragm for maximum contrast and the aperture diaphragm for maximum resolution and contrast; and
- Adjust brightness for best resolution.

Steps to Improve Depth of Field

- Reduce NA by closing the aperture diaphragm, or use an objective lens with lower NA;
- Lower the magnification for a given NA;

- Use a high-power eyepiece with a low-power, high-NA objective lens; and
- Use the longest possible wavelength light.

1.3 Specimen Preparation

The microstructure of a material can only be viewed in a light microscope after a specimen has been properly prepared. Metallurgists have developed extensive techniques and accumulated knowledge of metal specimen preparations for over a century. In principle, we can use these techniques to examine not only metallic materials but also ceramics and polymers; in practice, certain modifications are needed and a certain degree of caution must be exercised. The main steps of specimen preparation for light microscopy include the following.

- Sectioning;
- Mounting;
- Grinding;
- Polishing; and
- Etching.

1.3.1 Sectioning

Sectioning serves two purposes: generating a cross-section of the specimen to be examined; and reducing the size of a specimen to be placed on a stage of light microscope, or reducing size of specimen to be embedded in mounting media for further preparation processes. The main methods of sectioning are abrasive cutting, electric discharge machining and microtomy, which is mainly for polymer specimens.

Cutting

Abrasive cutting is the most commonly used method for sectioning materials. Specimens are sectioned by a thin rotating disc in which abrasive particles are supported by suitable media. The abrasive cutoff machine is commonly used for sectioning a large sample. The machine sections the sample with a rapidly rotating wheel made of an abrasive material, such as silicon carbide, and bonding materials such as resin and rubber. The wheels are consumed in the sectioning process. Abrasive cutting requires cooling media in order to reduce friction heat. Friction heat can damage specimens and generate artifacts in the microstructure. Commonly used cooling media consist of water-soluble oil and rust-inhibiting chemicals. The abrasive cutoff machine can section large specimens quickly but with poor precision.

More precise cutting can be achieved by a diamond saw or electric discharge machine (Figure 1.17). The diamond saw is a precision abrasive cutting machine. It sections specimens with a cutting wheel made of tiny diamond particles bonded to a metallic substrate. A cooling medium is also necessary for diamond saw cutting. Electrically conductive materials can be sectioned by an electric discharge machine (EDM). Cutting is accomplished by an electric discharge between an electrode and the specimen submerged in a dielectric fluid. EDM is particularly useful for materials that are difficult to be sectioned by abrasive cutting. EDM may produce significant changes at the machined surface because the electric discharge melts the solid in the cutting path. The solidified material along a machining path must be carefully removed during further preparation processes.

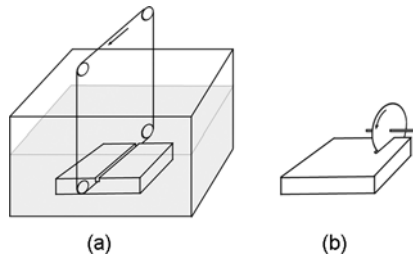


Figure 1.17 Specimen sectioning by: (a) wire cutting with electric discharging; and (b) diamond saw sectioning.

Microtomy

Microtomy refers to sectioning materials with a knife. It is a common technique in biological specimen preparation. It is also used to prepare soft materials such as polymers and soft metals. Tool steel, tungsten carbide, glass and diamond are used as knife materials. A similar technique, *ultramicrotomy*, is widely used for the preparation of biological and polymer specimens in transmission electron microscopy. This topic is discussed in Chapter 3.

1.3.2 Mounting

Mounting refers to embedding specimens in mounting materials (commonly thermosetting polymers) to give them a regular shape for further processing. Mounting is not necessary for bulky specimens, but it is required for specimens that are too small or oddly shaped to be handled or when the edge of a specimen needs to be examined in transverse section. Mounting is popular now because most automatic grinding and polishing machines require specimens to have a cylindrical shape. There are two main types of mounting techniques: *hot mounting* and *cold mounting*.

Hot mounting uses hot-press equipment as shown in Figure 1.18. A specimen is placed in the cylinder of a press and embedded in polymeric powder. The surface to be examined faces the bottom of the cylinder. Then, the specimen and powder are heated at about 150 °C under constant pressure for tens of minutes. Heat and pressure enable the powder to bond with the specimen to form a cylinder. Phenolic (bakelite) is the most widely used polymeric powder for hot mounting. Hot mounting is suitable for most metal specimens. However, if the microstructure of the material changes at the mounting temperature, cold mounting should be used.

In cold mounting, a polymer resin, commonly epoxy, is used to cast a mold with the specimen at ambient temperature. Figure 1.19a shows a typical mold and specimens for cold mounting. Figure 1.19b demonstrates the casting of epoxy resin into the mold in which the specimen surface to be examined is facing the bottom. A cold mounting medium has two constituents: a fluid resin and a powder hardener. The resin and hardener should be carefully mixed in proportion following the instructions provided. Curing times for mounting materials vary from tens of minutes to several hours, depending on the resin type. Figure 1.20 shows the specimens after cold mounted in various resins.