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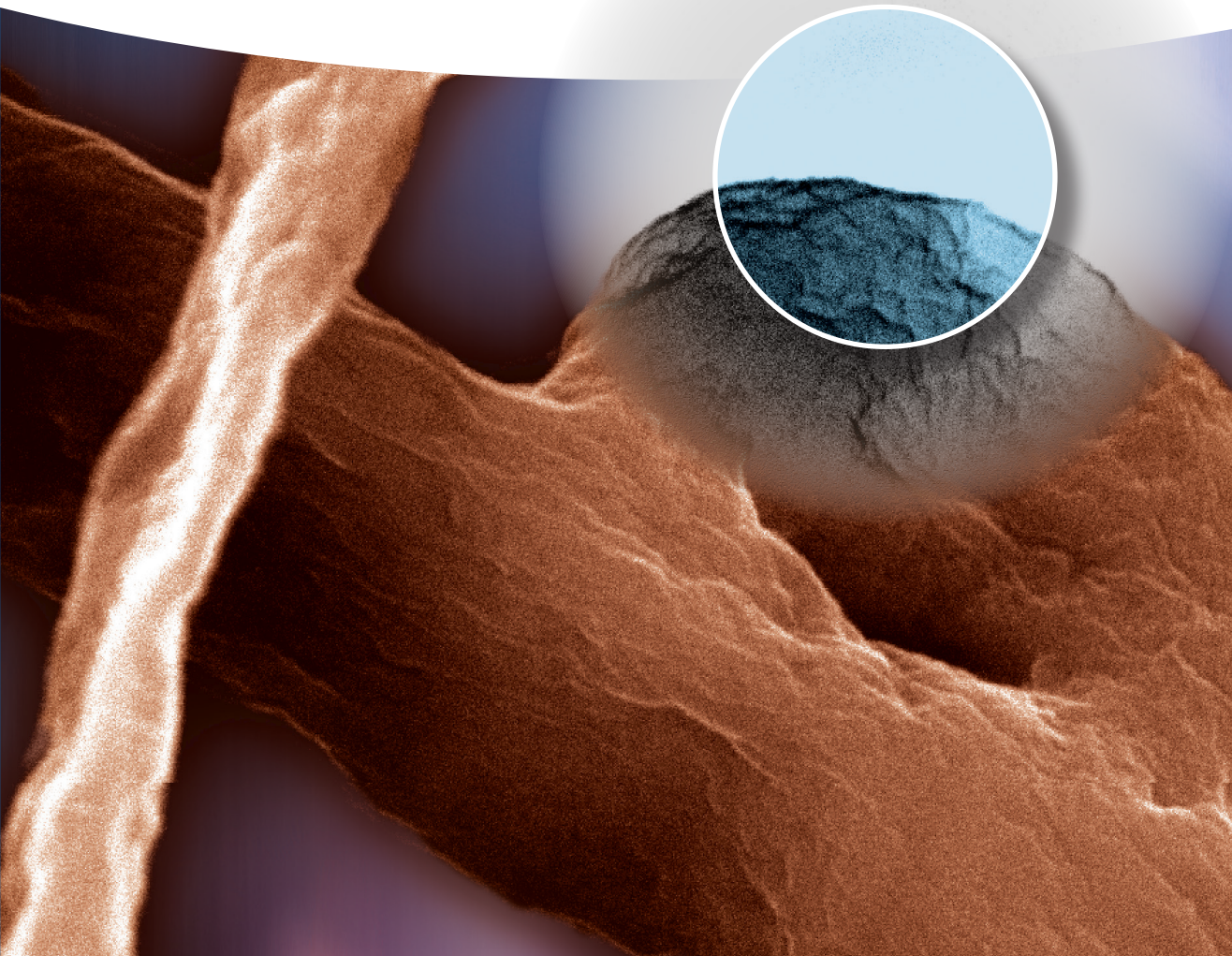
Materials Characterization

Introduction to Microscopic and Spectroscopic Methods

Second Edition



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Contents

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 metals, 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 first 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

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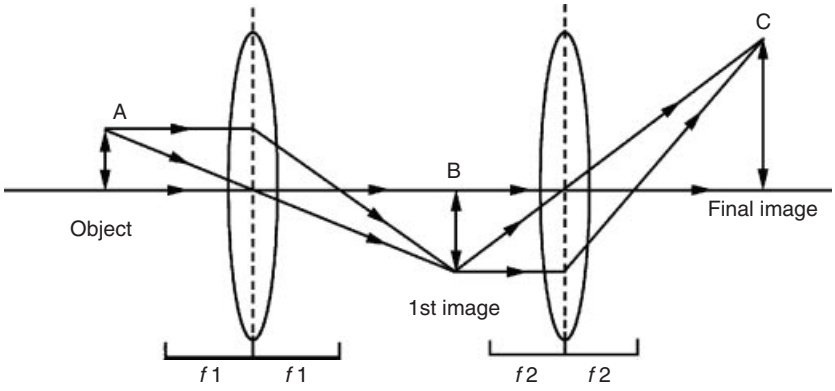

Figure 1.1 Principles of magnification in a microscope.

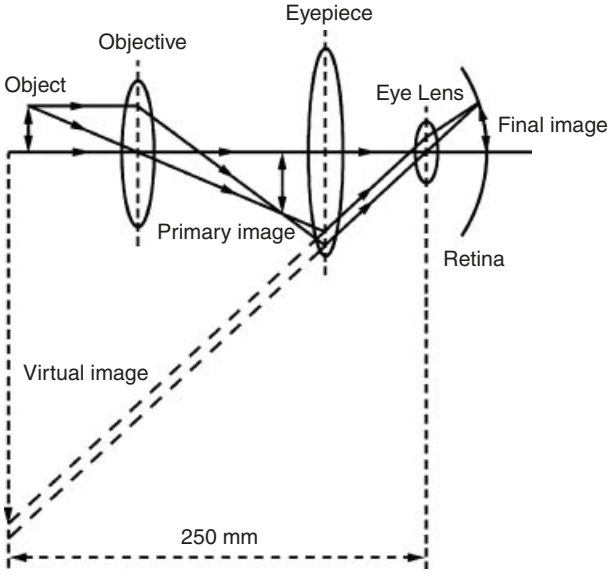

Figure 1.2 Schematic path of light in a microscope with an eyepiece. The virtual image is reviewed by a human eye composed of the eye lens and retina.

as 250 mm from the eyepiece. A modern microscope is commonly equipped with a device to switch from eyepiece to projector lens for either recording images on photographic film or sending images to a computer screen.

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:

$$M = \frac{v - f}{f} \tag{1.1}$$

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 Eq. (1.1). The total 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.

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

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

1.1.2 Resolution

We naturally ask whether there is any limitation for magnification in light microscopes because Eq. (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 the wavelength of light (λ) and the 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:

$$R = \frac{d}{2} = \frac{0.61\lambda}{\mu \sin\alpha} \tag{1.3}$$

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 Eq. (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, while the NA of the lens depends on α and the medium between the

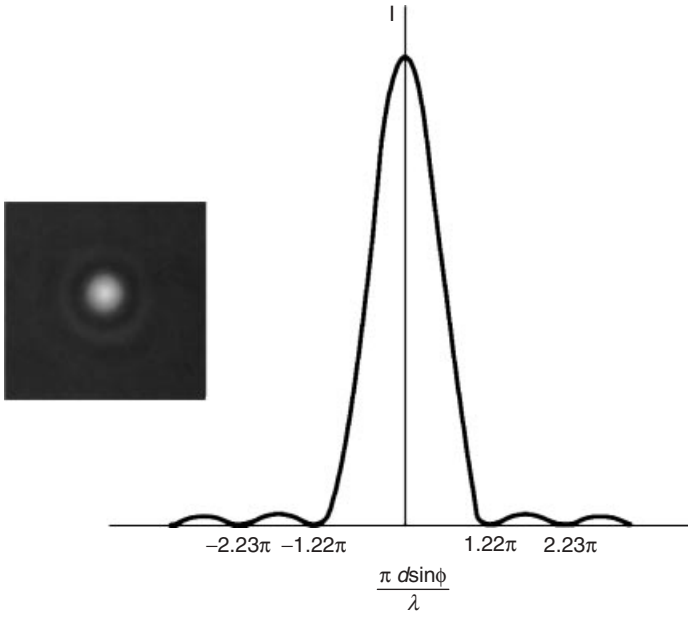

Figure 1.3 A self-luminous point object and the light-intensity distribution along a line passing through its center.

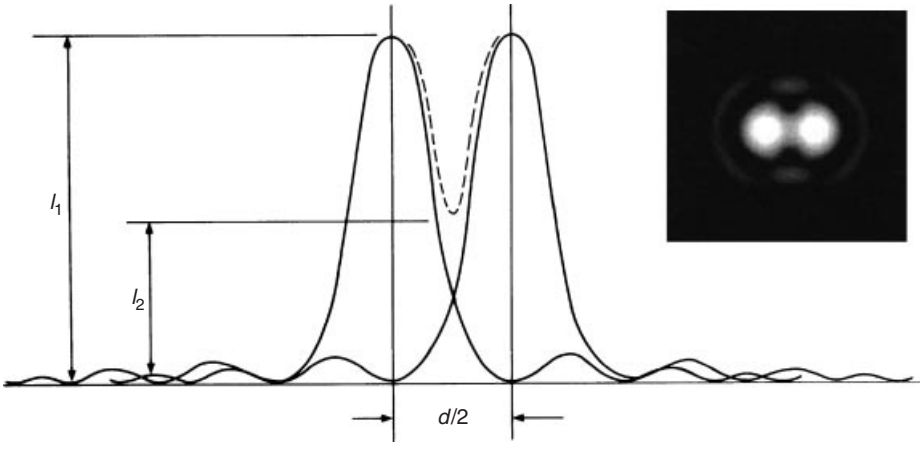

Figure 1.4 Intensity distribution of two airy disks with a distance $d/2$. I_1 indicates the maximum intensity of each point and I_2 represents the overlap intensity.

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 Eq. (1.3) as about 0.2 μm.

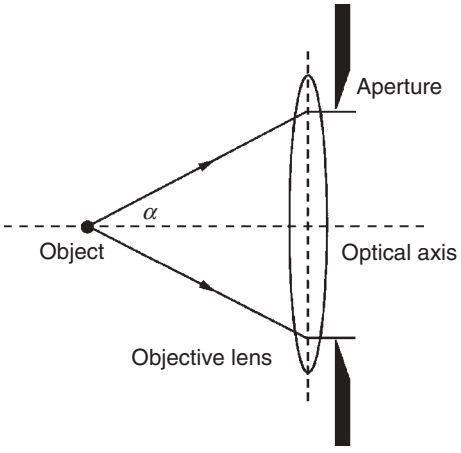

Figure 1.5 The cone of light entering an objective lens showing α is the half-angle.

1.1.2.1 **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. This means that the microscope resolution multiplying the effective magnification should be equal to the eye resolution. Thus, the *effective magnification* of a 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.

1.1.2.2 **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).

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

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

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

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.

$$\text{Contrast} = \frac{I_{\text{object}} - I_{\text{background}}}{I_{\text{background}}} \tag{1.6}$$

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 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.

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

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.

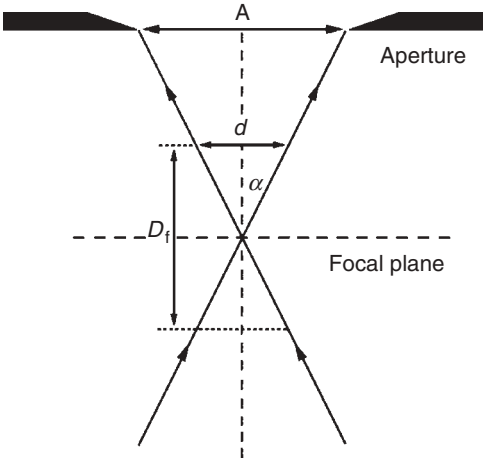

Figure 1.6 Geometric relation among the depth of field (D_f), the half-angle entering the objective lens (α), and the size of the Airy disk (d).

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 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

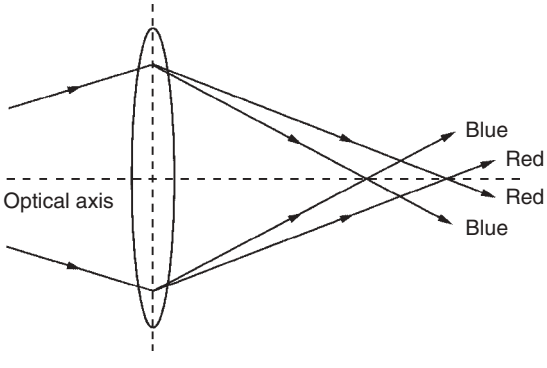

Figure 1.7 Paths of rays in white light illustrating chromatic aberration.

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.

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.

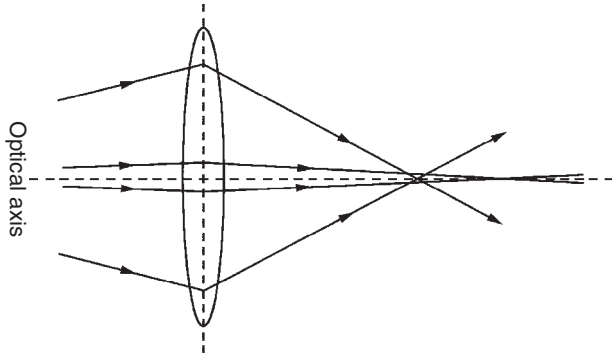

Figure 1.8 Spherical aberration.

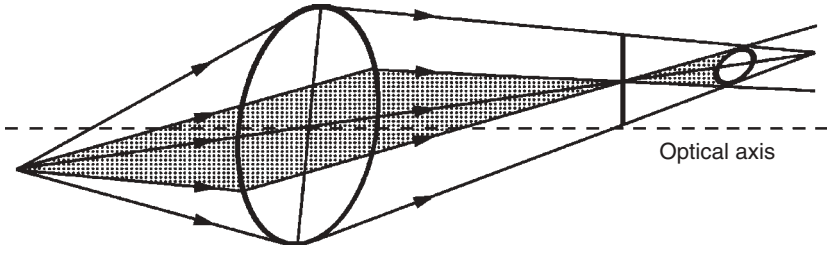

Figure 1.9 Astigmatism is an off-axis aberration.

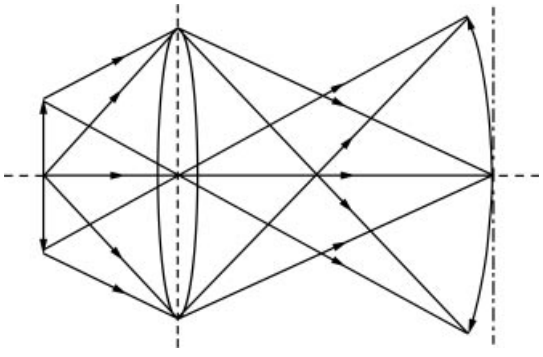

Figure 1.10 Curvature of field is an off-axis aberration.

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 or semitransparent 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 a 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. Their *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

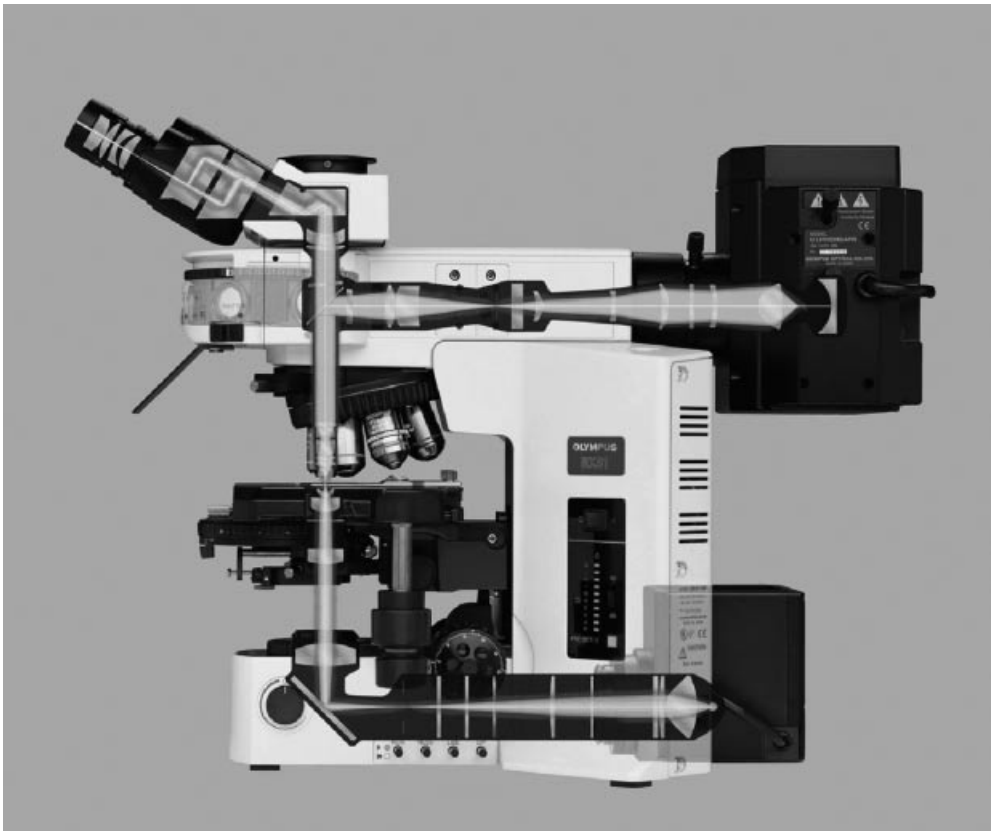

Figure 1.11 An Olympus light microscope used for material examination. The microscope includes transmission- and reflection-illumination systems. (This image is courtesy of Olympus Corporation.)

spectrum. Their 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 first 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

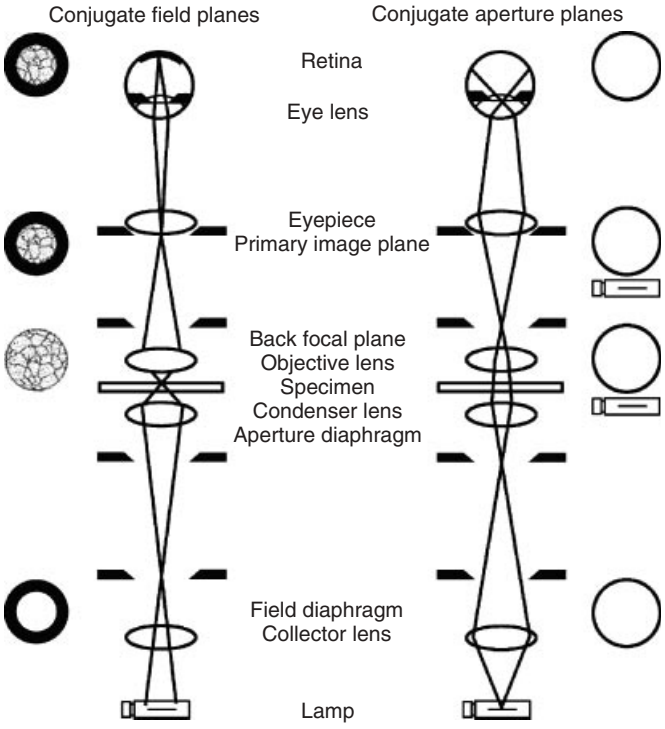

Figure 1.12 Two sets of conjugate focal planes in the Köhler system illustrated in a transmitted-light microscope. Image-forming rays focus on the field planes and illuminating rays focus on the aperture planes. The far left-hand and far right-hand parts of the diagram illustrate the images formed by image-forming rays and illuminating rays, respectively. (Reproduced with permission from Ref. [1]. © 2001 John Wiley & Sons Inc.)

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 the image-formation rays. 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 1.13). 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 the illuminating rays. Its function is to control α, and thus affect the image resolution and depth of field

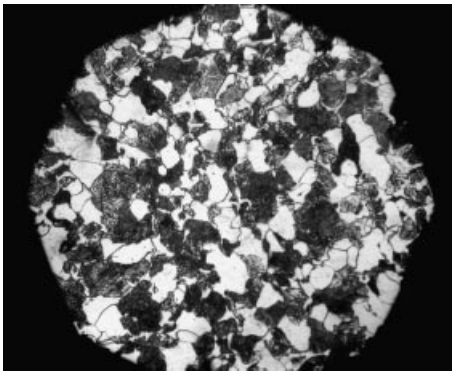

Figure 1.13 Image of the field diaphragm with an image of the specimen. Magnification 100×.

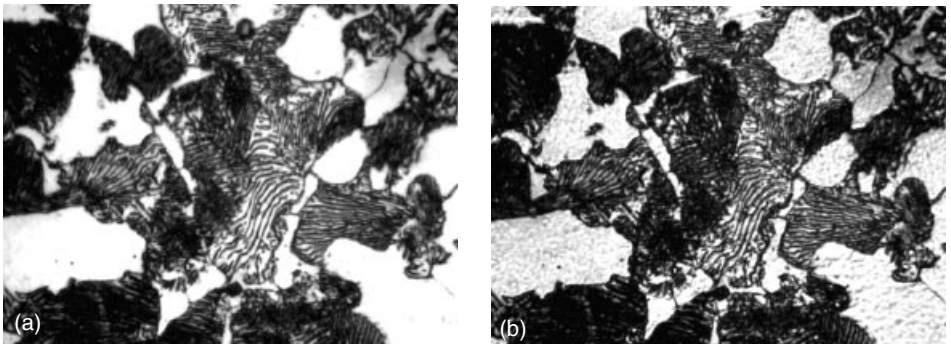

Figure 1.14 Effect of aperture diaphragm on specimen image when: (a) the aperture is large and (b) the aperture is small. Magnification 500×.

(Sections 1.1.2 and 1.1.3). 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 semitransparent 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 the wavelengths and intensity of illumination in microscopes in order to

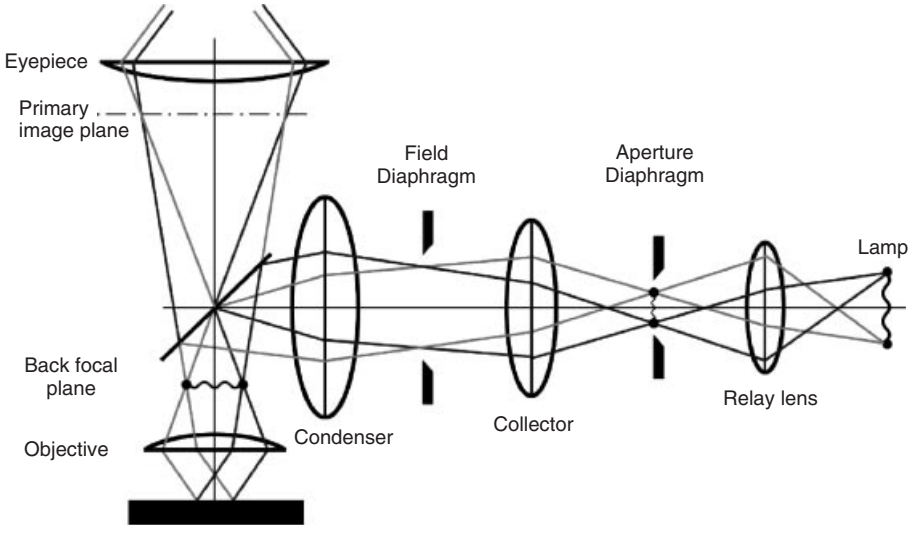

Figure 1.15 Illumination system of a reflected-light microscope with illuminating rays.

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 offer 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 that causes heating of specimen when a tungsten–halogen bulb is used as light source.

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×. 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× 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;
- semiachromat (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 of black and white photography. The semiachromatic 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 semiachromatic lens. Improvement in quality requires a substantial increase in the 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 semiachromatic;
- ''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;

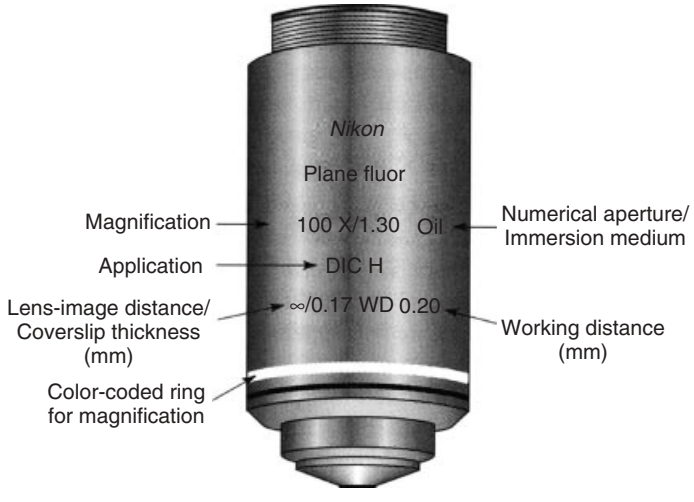

Figure 1.16 Engraved markings on the barrel of an objective lens. (Reproduced with permission from Ref. [1]. © 2001 John Wiley & Sons Inc.)