MATERIALS SCIENCE *and* ENGINEERING *Lab Manual*

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PART I

Metallographic Laboratory Practice

1–1 Safety Rules for the Materials Science Laboratory

There are some procedures and precautions that should be faithfully observed to ensure the safety and security of students in the materials science laboratory. These include what a student must do in case of an emergency situation such as a fire or an explosion, as well as preventive measures that, if taken, can minimize or eliminate the possibility of accidents. Students should follow these safety rules:

1. You should be aware of the locations of the emergency switch buttons, which can be used to cut off all electric circuits in the laboratory rooms, except the lights.
2. You should be aware of the locations of fire extinguishing equipment and fire alarms in the laboratory.
3. You should write the university emergency telephone number on a piece of paper that you can keep handy at all times.
4. Any malfunctioning of laboratory equipment should be promptly reported to your instructor, as should plugged drains or other safety hazards.
5. You should be aware of the location of the first aid kit and know how to use the emergency eye wash (and shower) in the materials science lab.
6. You should not work in the materials science laboratory unless an instructor or qualified technician is present.
7. Minor cuts or burns should not be neglected. You should report them immediately to the instructor, who will decide whether to carry out first aid procedures or to send you to the university medical center for treatment.
8. You must wear safety glasses for eye protection when you are operating a grinding wheel, a power saw, or a cut-off wheel. Some precaution must also be taken when using metal cutting tools such as chisels and hammers, and when handling acids or caustic solutions. Should a droplet of any of these liquids get into your eye, promptly and continuously flood the eye with water and seek assistance from the instructor.
9. Because a materials science laboratory contains many hazardous corrosive and/or poisonous liquids, you should never drink from the laboratory glassware.
10. The alcohol that is available in the laboratory for preparing specimens for metallographic examination is denatured methyl alcohol, a highly poisonous liquid that should never be drunk or swallowed.
11. Take care to avoid spilling any liquids on the floor or other flat surfaces in the laboratory.

1–2 Laboratory Report Preparation

The ability to communicate clearly both orally and in writing is of great importance to professional engineers. After graduation, you will spend a good part of your time explaining your ideas and points of view both to your superiors and to the technicians under your supervision. You are, therefore, advised to work on improving your written communication skills, and the preparation of laboratory reports provides you with an excellent opportunity to do so. Unlike a newspaper article, a technical report has a certain standard format that must always be adhered to. In addition, the style of technical and scientific writing is different from that used in, say, books of literature. Colloquial language must never be used; instead, explain your ideas in simple and grammatically correct English using clear and short sentences. Try to get to the point directly and avoid unnecessary elaborations and wordy text. Also, it is a well-established tradition in scientific writing to report in the third person (i.e., avoid using "I" and "we") and in the past tense. Let us now discuss in detail the standard technical report format.

In most cases, a technical report or laboratory report should consist of the following components:

1. *Title page* The title page should indicate the university, the college, and the department in which the materials science laboratory course is offered. It should also show the title and the serial number of the experiment performed, your name, the name of the laboratory instructor to whom the report is submitted, and the date on which the report is submitted.

2. *Objective of the experiment* Briefly state the purpose or the goals of the experiment. This part of the report should not exceed two fairly short paragraphs.

3. *Equipment* List the equipment used in the experiment and briefly mention the specific characteristics of each (e.g., capacity, accuracy of reading).

4. *Experimental Procedure* Write in your own words the actual experimental procedure followed. The procedure should reflect the facilities available at the laboratory where the experiment was conducted and may, therefore, differ slightly from the standard procedure mentioned later in this manual.

5. *Experimental results* In this part of your report, raw data taken during the course of the experiment should be presented in an appropriate form such as tables, graphs, figures, or photographs. These must be numbered and supplemented by captions that briefly describe what the figures or photographs are all about. Whenever graphics are used, special attention should be given to the drawing scale in order to yield meaningful curves that clearly indicate the significance of the results. Miniature and over-enlarged graphs should be avoided. Finally, remember to plot the independent variables (such as time or the temperature at which a sample is soaked) along the abscissa, and the dependent variable (such as the hardness of a sample after thermal treatment) along the ordinate.

6. *Discussion of results* This part of the report should constitute a discussion of the experimental data obtained as well as the possible sources of experimental error and in what way these may have affected the results. You can also include a correlation

and/or a comparison of your experimental results with those that can be predicted from theories or the use of theoretical analysis, and try to explain any discrepancies.

7. *Conclusions* In this section, you present what you can conclude from the results of the experiment and the analysis applied in the previous discussion. It may also be appropriate to add your personal opinion whether the experiment served its goal of reinforcing what was covered in the materials science lectures.

8. *References* Here you provide a numbered list of scientific books or articles to which you refer in the text. Make sure that each reference was actually cited and numbered in the text, that each number corresponds to the correct reference, and that the numbers are in sequential order. You can also provide a bibliography, which is a general list of supporting materials that were not specifically referred to in the text. There is a standard format for writing a reference; use the bibliography at the end of this manual as a guideline.

1–3 The Use of Standards

During an experiment, or a test that is carried out to determine the physical, chemical, or mechanical properties of a material, there are always variables whose magnitudes affect the readings taken and, consequently, the results obtained. Experimental results would not, therefore, be meaningful unless the variables affecting the test are fully specified. As a consequence, it is obvious that the results obtained from two tests can be compared only when the corresponding variables in both tests are set to be identical. This leads to the need to standardize the specifications and the methods of testing, so that the results of identical tests can be compared.

The process of specifying the procedure and the parameters affecting an experimental test is not an easy one. It should be, and actually is, based on striking a balance between accuracy and practicality. Needless to say, the targeted or desired accuracy and the availability of facilities differ from one country to another, and so do the standards for the methods of testing. It is for this reason that engineering societies, governmental institutes, and manufacturers' associations in each country work together by establishing committees of specialists to compile standards for each commonly used test that should be adhered to whenever that test is carried out.

In the area of material testing and metallurgy, we in the United States should always follow the standards developed by the American Society for Metals (ASM) and the American Society for Testing and Materials (ASTM). For each test, there is a publication in which the standard conditions for performing that test are precisely specified. In the publication detailing the standards for a tension test, for example, the shape, dimensions, and surface finish of the test specimen are accurately covered. If you use a specimen having a shape or dimensions different from the standard ones, the results you obtain would not truly indicate the properties of the material tested, and could not be compared with the data given in handbooks. As you may have expected, each of these publications has an alphanumeric code or designation to which you should refer when ordering the publication or when writing a test report. A brief summary of the standards for commonly used tests is given in Part 3 of this manual.

1–4 Hardness Testing Methods

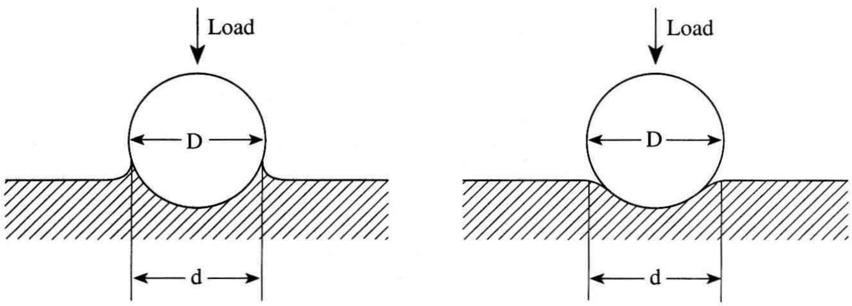
Hardness can be defined as the resistance of a material to scratching, abrasion, or penetration. Obviously, any hardness index is a manifestation of the combined effect of several related properties, which may include the yield point, ultimate tensile strength, malleability, work hardening characteristics, wear-resistance properties, etc. Therefore, hardness measurements must be interpreted with caution and full consideration of their attendant limitations. In fact, past methods for determining hardness, like the file hardness test, were not fully reliable because they were dependent on the skill of the technician performing the test. It was not until 1900 that Dr. Brinell of Sweden proposed a new, reliable method whereby the hardness of a metal could be indicated by its resistance to indentation. Nowadays, hardness testing has found extensive industrial applications and is an essential tool in the quality control of metals, alloys, and metal products. The most commonly used hardness testing methods are the Brinell and the Rockwell hardness tests, which we will discuss in detail. Other, specialized hardness tests are, we believe, beyond the scope of this book.

1–4–1 Brinell Hardness Test This test involves forcing a hardened steel ball into the metal specimen under a definite static load, then measuring the size (diameter) of the impression produced by that ball penetrator. In this case, the hardness index, which is called the Brinell Hardness Number, is the static load acting on the penetrator divided by the spherical area of the impression, the unit being kilogram force per square millimeter. According to ASTM specifications (Designation: E 10), the penetrator used in a standard Brinell hardness test is a spherical ball having a diameter of 10 mm. These standards also specify the load as well as the duration of its application, namely 3000 kg and at least 10 seconds for ferrous metals and 500 kg and at least 30 seconds for nonferrous metals. The diameter of the impression is usually obtained by optical magnification projected on a screen, where it can be measured accurately using a vernier calliper. Alternatively, a special measuring-type microscope is sometimes used. In either case, a number of measurements must be made across different diameters. These measurements are then averaged in order to obtain a value that truly represents the diameter of the impression. The Brinell Hardness Number corresponding to this value can be obtained from tables, thus eliminating the need for calculations.

It must always be borne in mind that the ball penetrator will inevitably undergo elastic deformation when forced into the metal specimen during the test. For any specific applied load, say 3000 kg force, the magnitude of that deformation will depend on the resistance of the metal specimen to penetration (i.e., its hardness) and, obviously, on the hardness of the ball penetrator as well. The higher the hardness of the metal specimen, the more sensible the amount of deformation in the ball would be. Ordinary high carbon steel ball penetrators can only be used when the hardness of the metal specimen does not exceed a Brinell Hardness Number of about 500. For higher values of hardness (up to Brinell Hardness Number 700), the use of a tungsten carbide ball penetrator is recommended.

In medium-hard ferrous metals, there is usually a raised ridge of metal around the impression caused by the penetrator, while in brass and bronze the impression is surrounded by a depressed surface. Consequently, the diameter of the impression appears slightly larger in brass or bronze than what it really should be, as indicated in Figure 1–1. Because of that undesirable secondary plastic deformation (and work hardening), hardness measurements should not be taken close to the edges of the specimen or close to each other. The minimum distance between the center of the impression and the edge of the test specimen should be 2.5 times the diameter of the impression. Also, as a precaution in this test, the thickness of the test specimen must not be less than 6 mm (0.25 in.), so that the anvil's support will not have any influence on the penetration, a problem that results in erratic hardness values. For thinner test specimens, lighter loads and smaller penetrators are used. Finally, the condition of the test surface specified in the standards must be adhered to. The surface of the specimen must be flat, reasonably smooth, and free from defects in order to obtain meaningful results.

As we will see later, the obvious advantages of the Brinell hardness testing method are that only one theoretical linear scale of hardness is used, regardless of the metal being tested, and that the hardness of the metal on that scale and its ultimate tensile strength are indeed correlated.



(a) Medium-hard ferrous metal (b) Brass/Bronze

FIGURE 1–1 Schematic illustration of the Brinell hardness test.

1–4–2 Rockwell Hardness Test Similar to the Brinell hardness test, the Rockwell hardness test involves forcing an indentor into the test specimen under static load. However, in the Rockwell testing method, the hardness index is determined by measuring the increment of depth (of the impression) as a result of applying a primary and a secondary load, instead of measuring the diameter. Consequently, there is no need for optical measurements or calculations and the Rockwell hardness number is readily shown on a dial indicator. The Rockwell hardness test is, therefore, commonly used in industry because of its simplicity and the ease with which it can be performed. Figure 1–2 illustrates a Rockwell hardness testing apparatus.

There are basically two standard indentors that are used with two hardness scales to determine the Rockwell hardness numbers for nearly all the common metals and alloys (ASTM Designation: E 18). These indentors (penetrators) are a hardened steel ball hav-

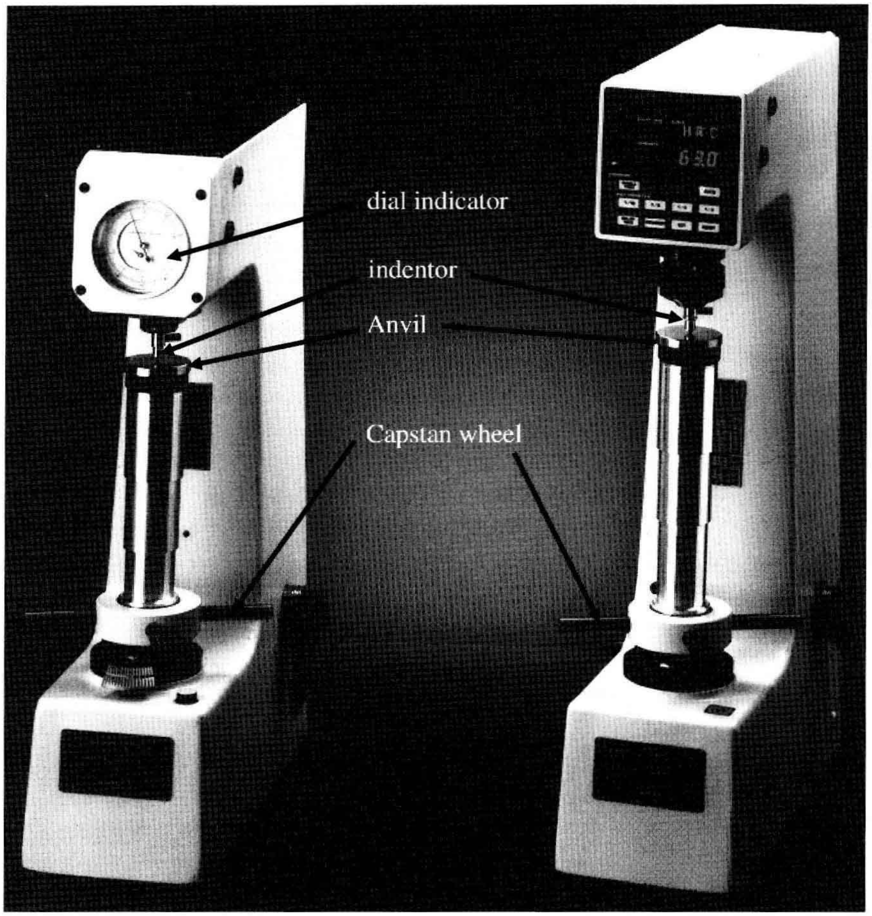


FIGURE 1–2 A photograph of a Rockwell hardness testing apparatus. *(Courtesy of Buehler Ltd.)*

ing a diameter of 1.59 mm (1⁄16 in.) and a diamond cone having an apex angle of 120° and a rounded tip 0.2 mm in radius (called the Brale), and are used with scales designated as B and C, respectively. The working range of scale B, which is used for nonferrous metals and annealed low carbon steels, is from Rb 0 to Rb 100. For the sake of measurement accuracy, when the hardness of the material being tested exceeds Rb 100, you must switch to scale C; if the hardness is less than Rb 0, another appropriate Rockwell hardness scale should be used. The useful range of the C scale, which is used for hardened and tempered steels, is from Rc 20 (equivalent to Rb 97) to slightly above Rc 70. Owing to inherent inaccuracies associated with shaping the Brale, the C scale should not be used for measuring hardness below Rc 20; instead, the hardened steel ball and scale B are usually employed.

Figure 1–3 shows the procedure for performing a Rockwell hardness test on the C scale. First the test specimen is placed on the anvil at the upper end of the elevating screw. The capstan wheel is then rotated so as to bring the surface of the test specimen

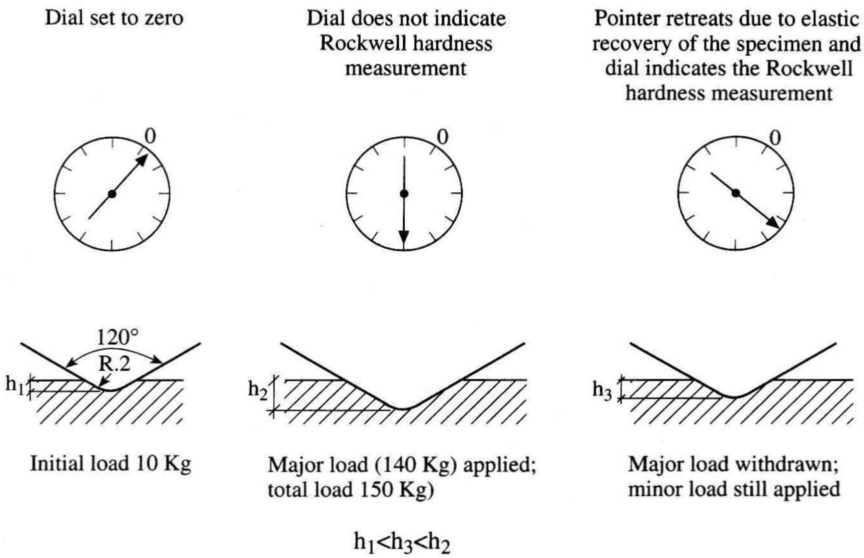


FIGURE 1–3 The procedure for performing a Rockwell hardness test (on the C scale).

in contact with the penetrator. By further rotation of the wheel, the test specimen is forced against the indentor and a minor load of 10 kg is slowly applied in order to seat the specimen firmly. At this moment, the dial indicator of the apparatus (whether mechanical or optical) is set to zero. Next, an additional load of 140 kg (90 kg in a test on the B scale) is applied by means of a release handle mounted on the side of the apparatus. The total major load will now be 150 kg and the duration of its application should be at least 10 seconds. Obviously, the application of that load would force the penetrator into the specimen to an additional depth. Still, that depth must not be considered as an indication of hardness because it includes an elastic as well as a plastic deformation. Therefore, the additional load is released without removing the minor load, and the hardness index is then shown on the dial indicator. That reading reflects the permanent or plastic increment of penetration depth resulting from the increment of load between the minor and major loads. It does not indicate the total depth of penetration of the indentor. Again, as in the case of the Brinell hardness test, care must be taken to ensure that the surface conditions of the test specimen—its flatness and its thickness—are within the limits specified by the standards.

Review Questions on Hardness Testing

1. How do you define hardness?
2. What is the theory on which Brinell hardness testing is based?
3. What are the advantages of the Brinell hardness testing method?

4. What is the theory on which Rockwell hardness testing is based?

5. What is the purpose of the minor load in Rockwell hardness testing?

6. What are the advantages of the Rockwell hardness testing method?

7. What are the disadvantages of the Rockwell method?

8. How thick must the specimen be in order to get an accurate Rockwell hardness reading?

9. How close can a Rockwell hardness indentation be to another without getting an error in the reading?

10. What is the main difference between a regular hardness tester and a microhardness tester?

1–5 The Metallurgical Microscope

The optical microscope is the tool that is commonly used for examining and photographically recording the microstructures of metals and alloys. Since metals and alloys are always opaque and do not allow light to pass through, no matter how thin they are, the metallurgical microscope differs from the biological type in the manner by which the specimen under investigation is illuminated. As can be seen in Figure 1–4, which indicates a schematic illustration of the metallurgical microscope, a special external source of light (called the illuminator) is employed. It is also evident that the metallurgical microscope is composed of two distinct and separate optical lenses, namely the objective and the eyepiece. In fact, we will see later that each one of them can, and usually is, replaced by an optical system of lenses in order to improve the clarity with which the microscopic image is observed.

Let us now discuss the principles of operation of the metallurgical microscope. A horizontal bundle of rays of light is emitted from the illuminator and diverted downward, by means of a plane glass reflector to pass through the objective and fall onto the specially prepared surface of the metal specimen. The light that is reflected from the surface of the specimen will pass again through the objective to form an enlarged primary image of the illuminated area. The eyepiece is then used to further magnify the

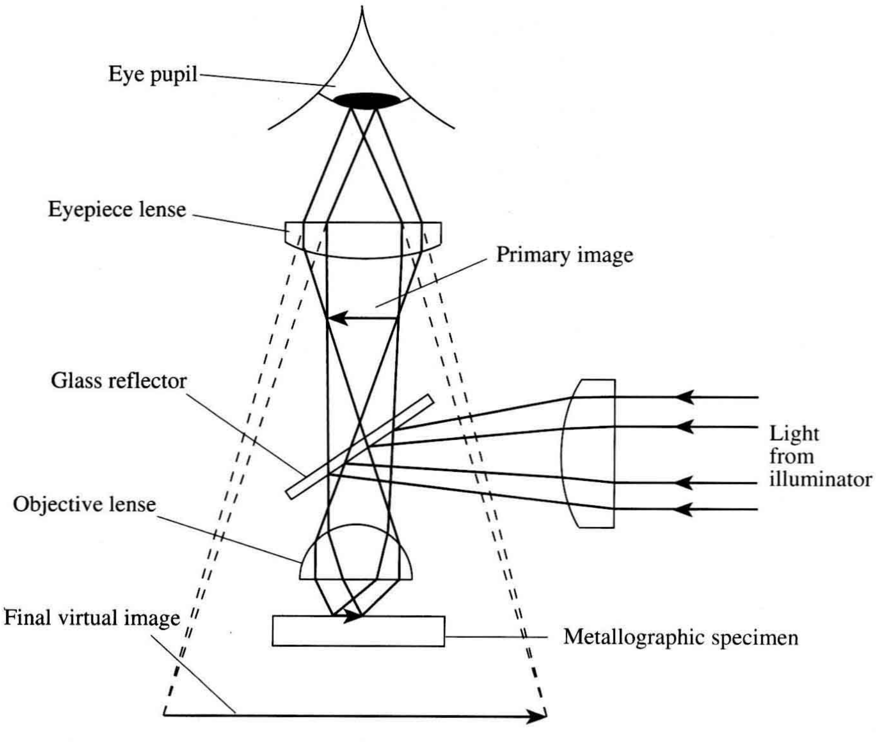


FIGURE 1–4 Schematic illustration of the metallurgical microscope.

primary image for visual examination or to project it onto a photographic film so that it can be permanently recorded. The power of magnification of the microscope is the product of the powers of magnification of the objective and the eyepiece. In a modern metallurgical microscope, like those shown in Figure 1–5, the objective can be changed easily in order to obtain a higher or lower magnifying power. Magnifications of up to 1000× can be obtained and are quite common, but the clarity of the obtained image decreases at higher magnifications.

In order to be corrected for two defects occurring in simple lenses, the objective must be composed of an optical system of lenses. These two defects are the chromatic and the spherical aberrations and are responsible for destroying sharp definition in the image. Chromatic aberration occurs because white light is actually a mixture of color lights that have different wave lengths and are, therefore, refracted by the optical glass of the lens to different degrees. As can be seen in Figure 1–6, the final outcome will be three images of different colors, the violet or blue image being closer to the lens and the red image farthest. The remedy for this problem involves the use of either an achromatic

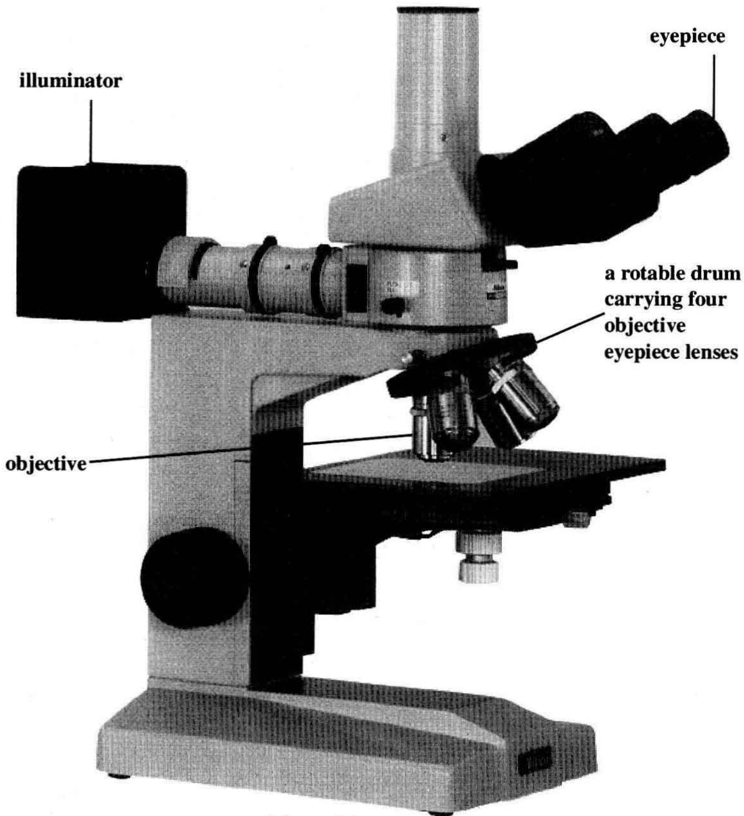
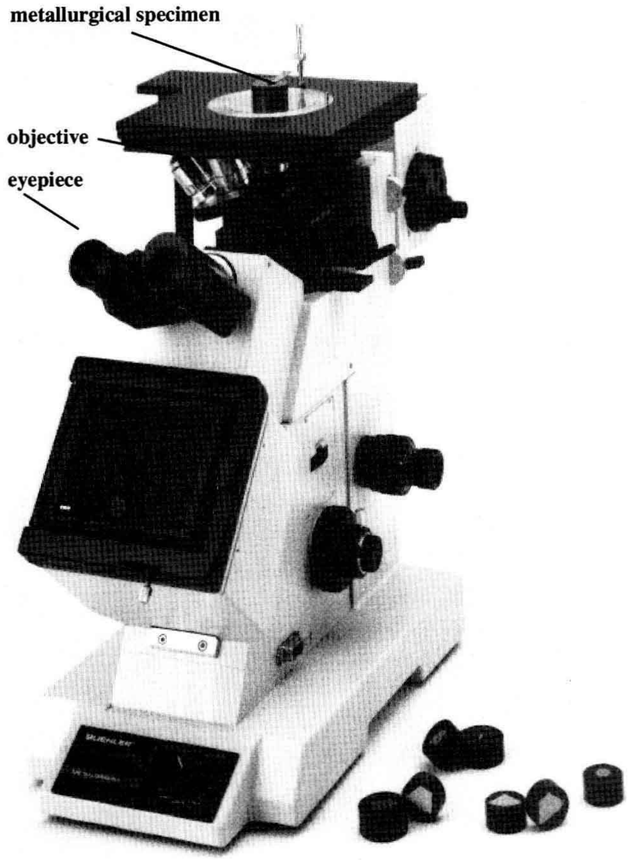


FIGURE 1–5 Photographs of modern metallurgical microscopes. ((a) *Courtesy of Nikon;* (b) *Courtesy of Buehler Ltd.*)



(b) inverted-stage type

FIGURE 1–5 (continued)

or apochromatic objective. As shown in Figure 1–7, the achromatic objective is corrected to bring green and red images to a focus in the same plane, whereas the apochromatic objective is highly corrected and brings the violet, green, and red images to a single sharp focus. The spherical aberration is caused by the fact that the light beams passing through the outermost margins of a lens are refracted to a greater degree than are

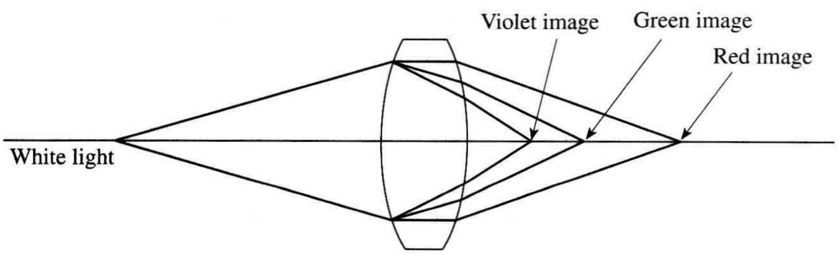


FIGURE 1–6 Schematic illustration of chromatic aberration.