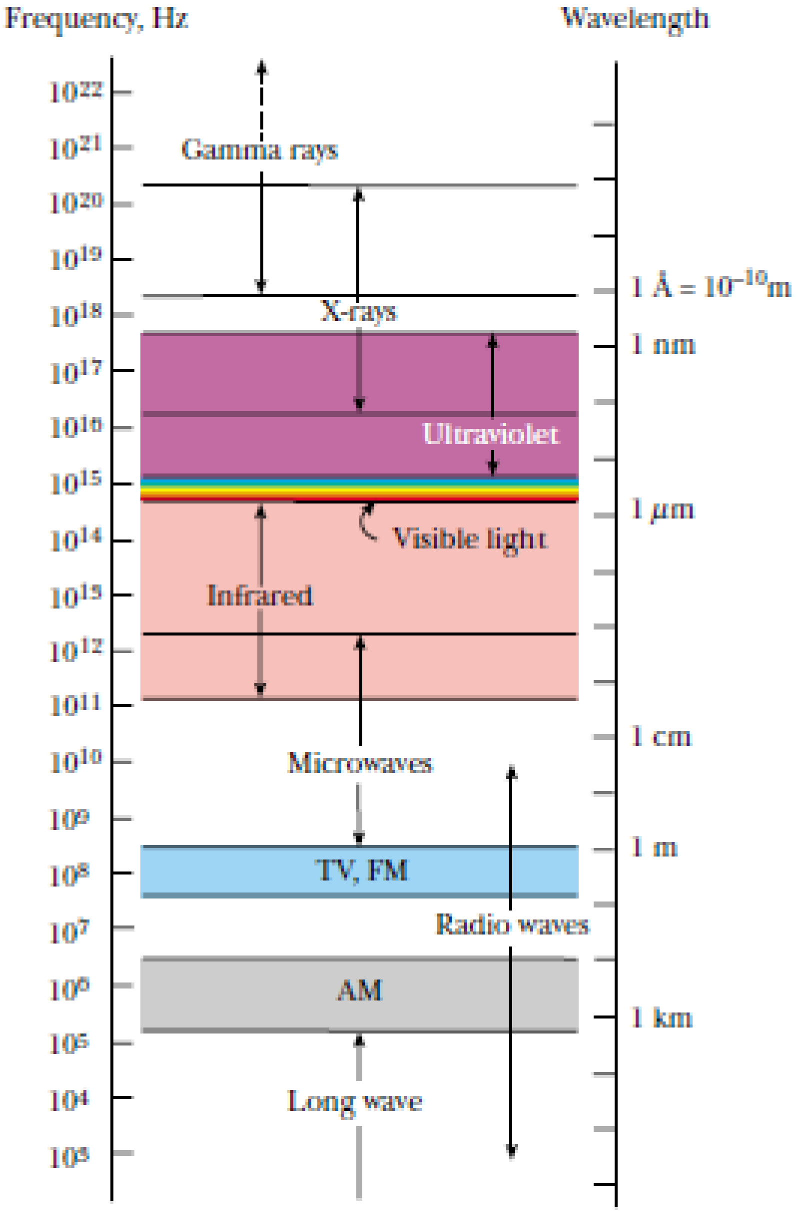
Microscopes

1. Optic Microscopes
2. Electron Microscopes

We use mostly optic (metal) microscopes in metallogaphy.



The electromagnetic spectrum. Note the overlap between adjacent wave types.

Optic Microscopes for Metallogaphy Basic pieces

1. Light source
2. Objective lens
3. Half-silvered mirror
4. Eyepiece (or ocular)

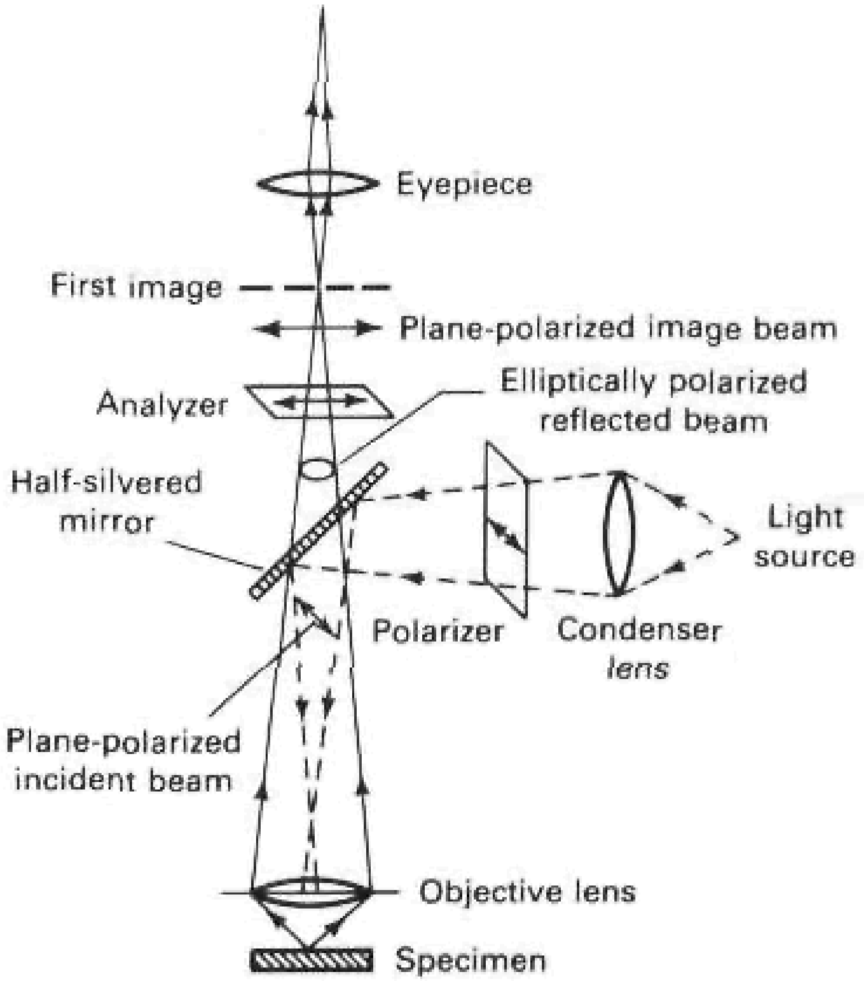


Fig. 3 Principles of polarized light microscopy

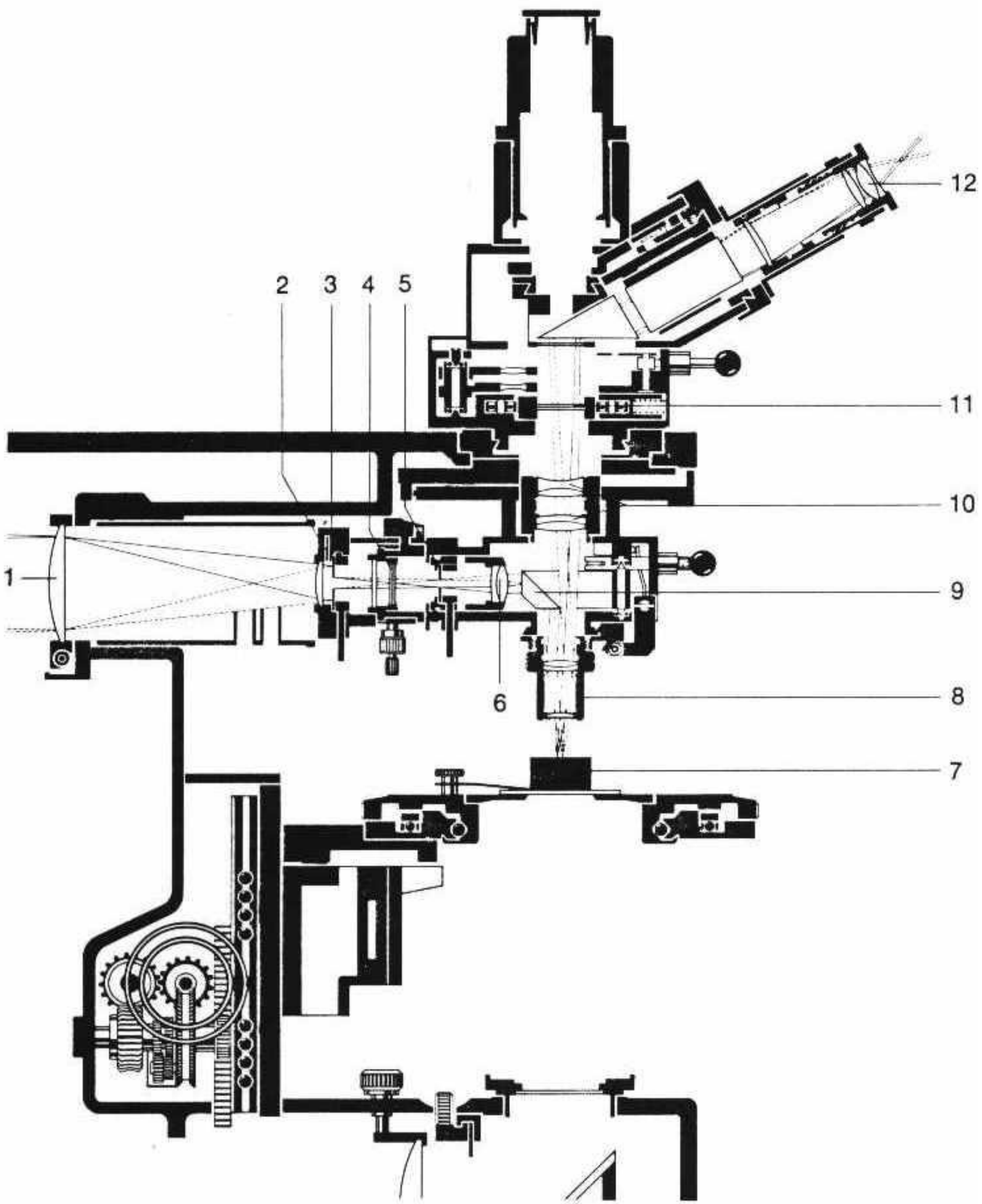


Fig. 26 Light path in an incident-light polarizing microscope. 1, Hinged lens; 2, half stop; 3, aperture diaphragm; 4, filter or prism polarizer; 5, field diaphragm; 6, centrable lens, used to center the field diaphragm; 7, polished section; 8, objective; 9, compensating prism, with switchover against optical-flat reflector; 10, tube lens (intermediate optical system); 11, rotating analyzer; 12, eyepiece with focusing eyelens. (E. Leitz, Inc.)

Classification of microscopes

1. Upright incident-light microscope
2. Inverted incident-light microscope

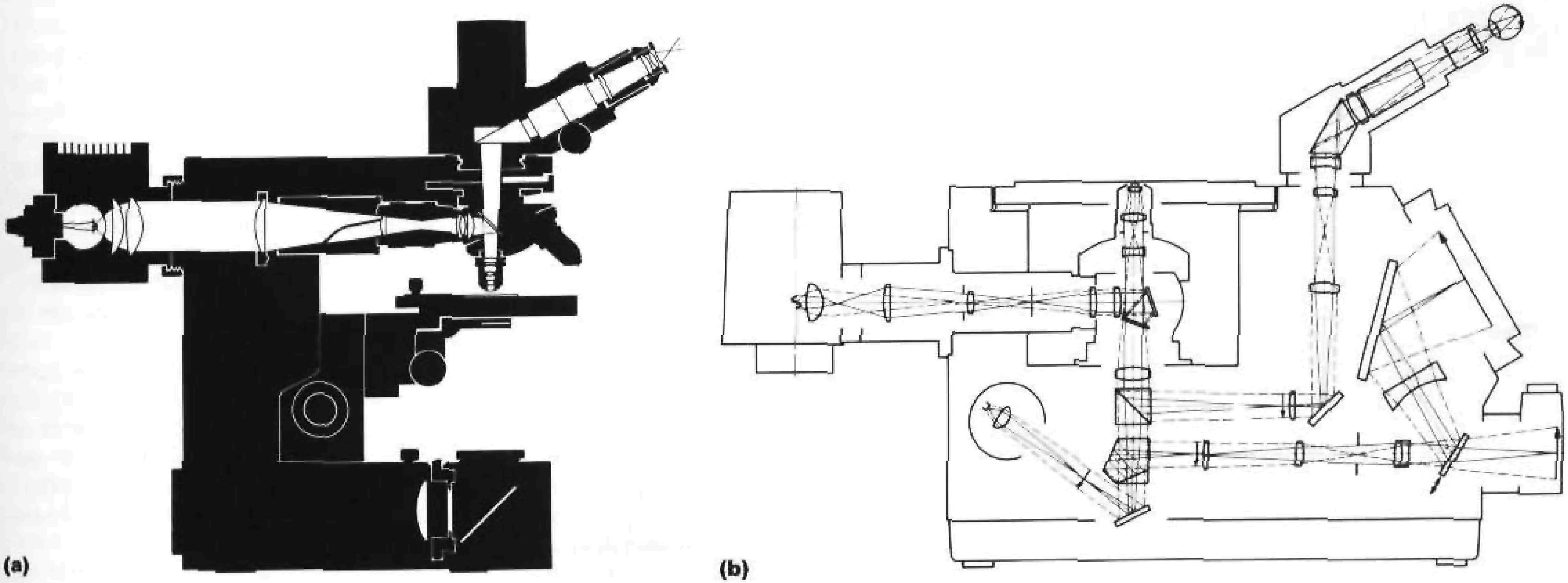
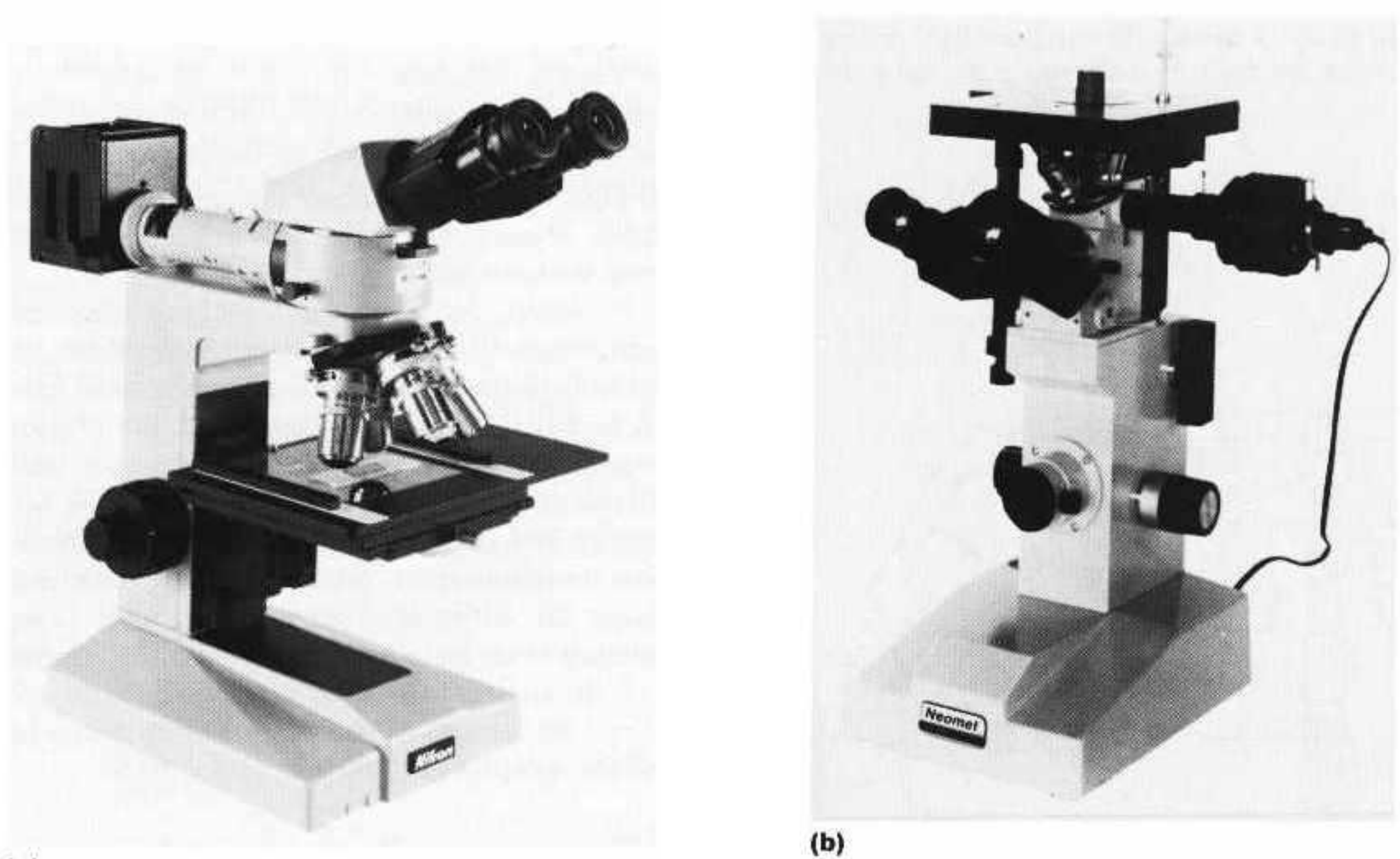


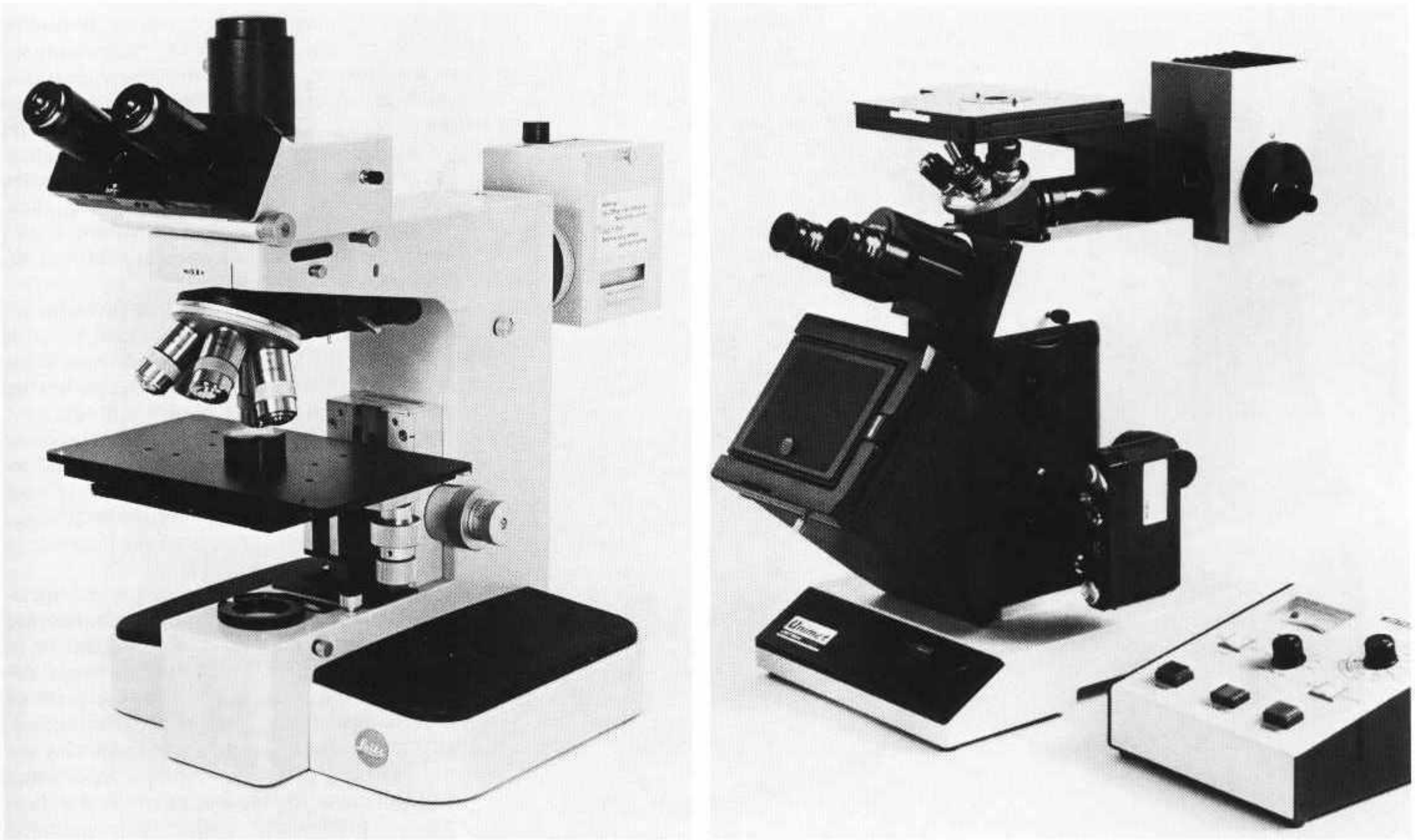
Fig. 1 Light paths in (a) an upright incident-light microscope and (b) an inverted incident-light microscope. (E. Leitz, Inc.; C. Zeiss, Inc.)



(a)

(b)

Fig. 2 (a) Upright bench microscope. (b) Inverted bench microscope (Nikon, Inc.; Unitron Instruments, Inc.)



(a)

(b)

Fig. 3 Research-quality optical microscopes. (a) Upright. (b) Inverted. (E. Leitz, Inc.; Unitron Instruments, Inc.)

Fig. 4 Moderately priced inverted metallograph. The small box to the right is an automatic exposure control. (Nikon, Inc.)

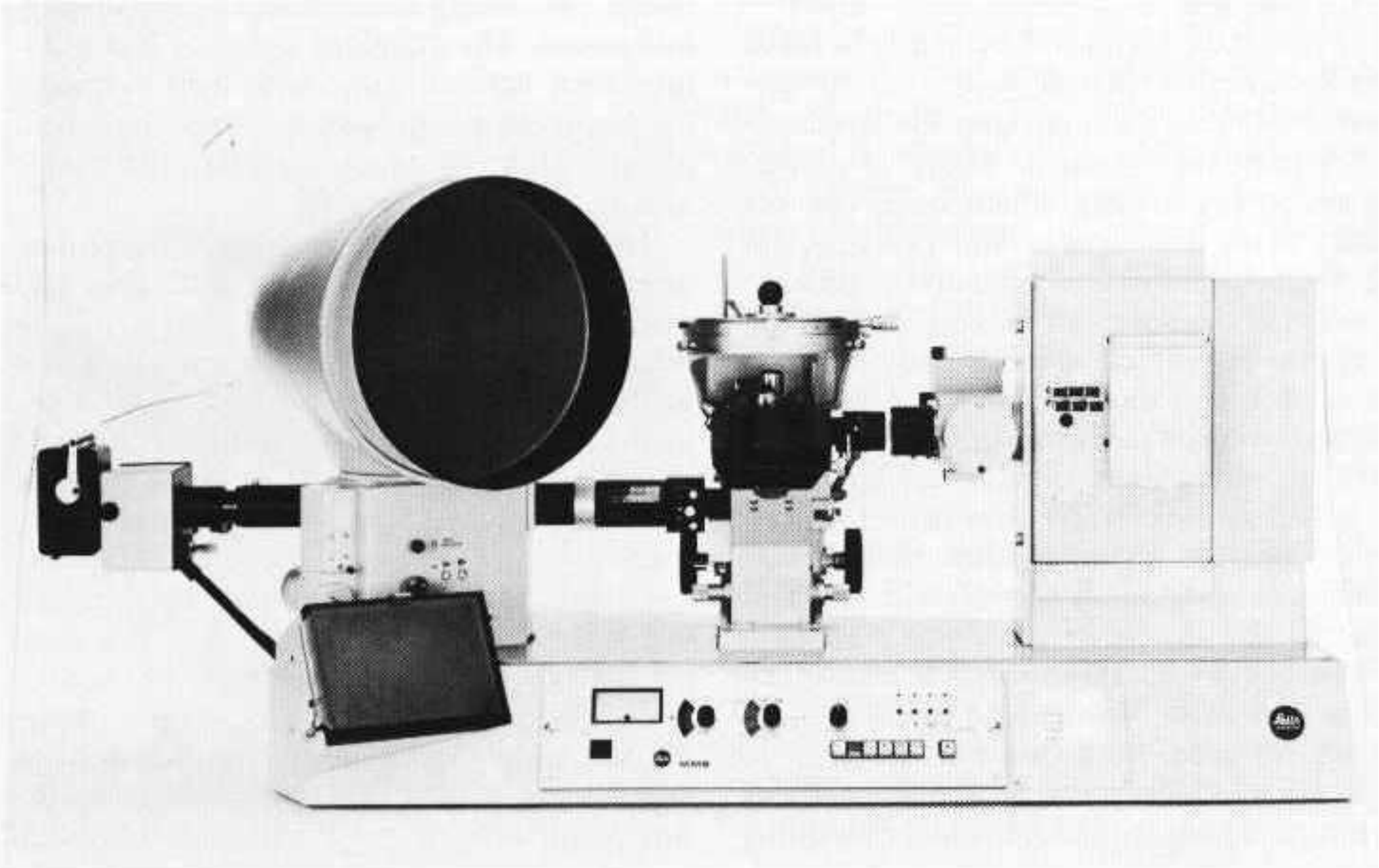


Fig. 5 Research-quality metallograph with a projection screen for group viewing. (E. Leitz, Inc.)

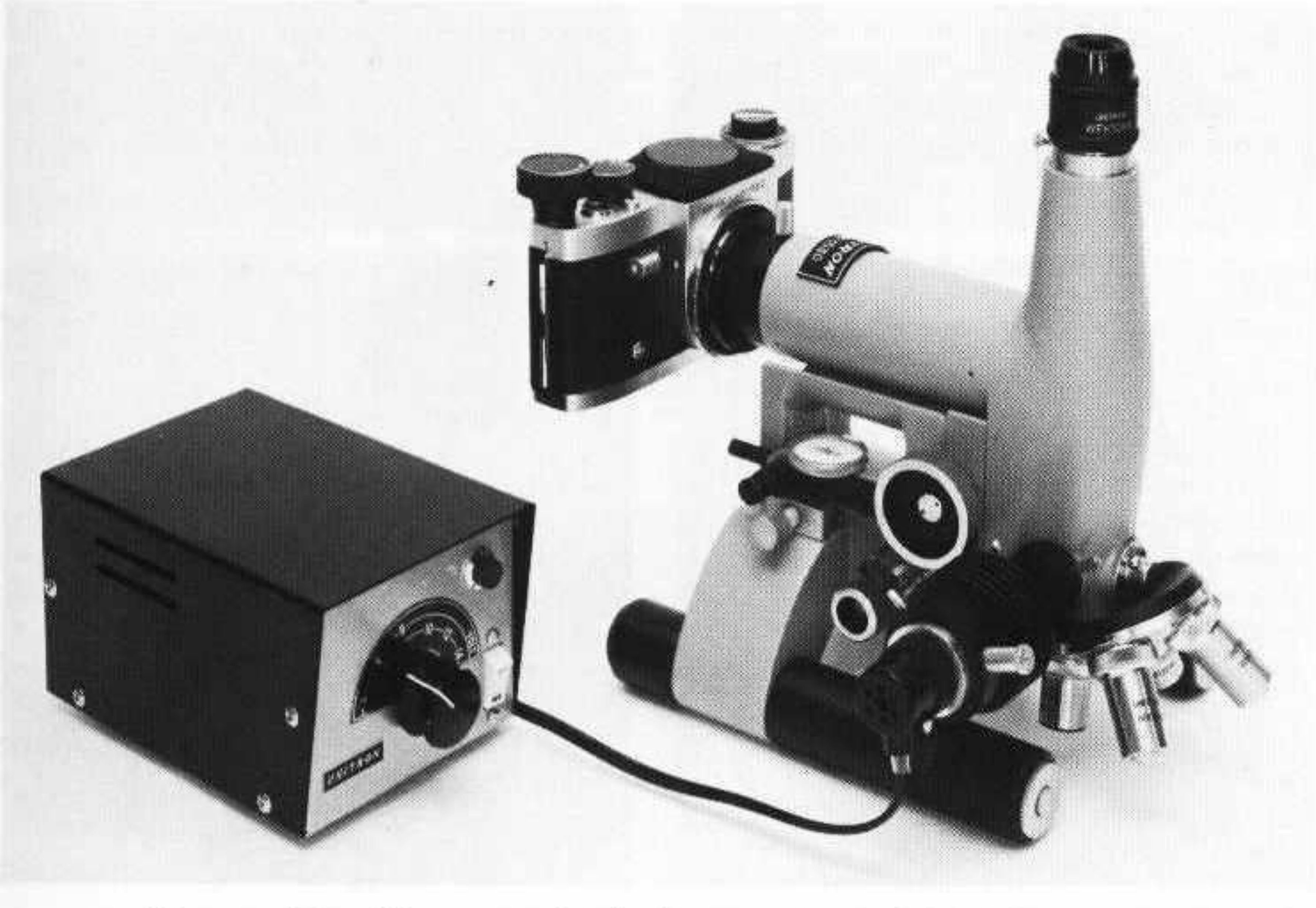


Fig. 45 Microscope designed for use away from the laboratory. Batteries for the light source are contained in the cylindrical stand of the instrument. (Unitron Instruments, Inc.)

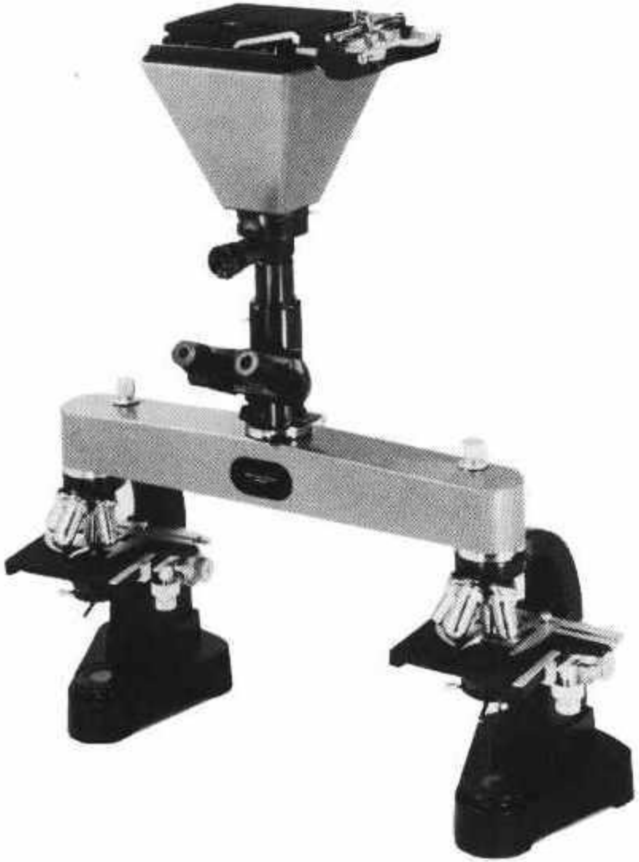


Fig. 46 Comparison microscope, which allows simultaneous viewing of two specimens. (E. Leitz, Inc.)

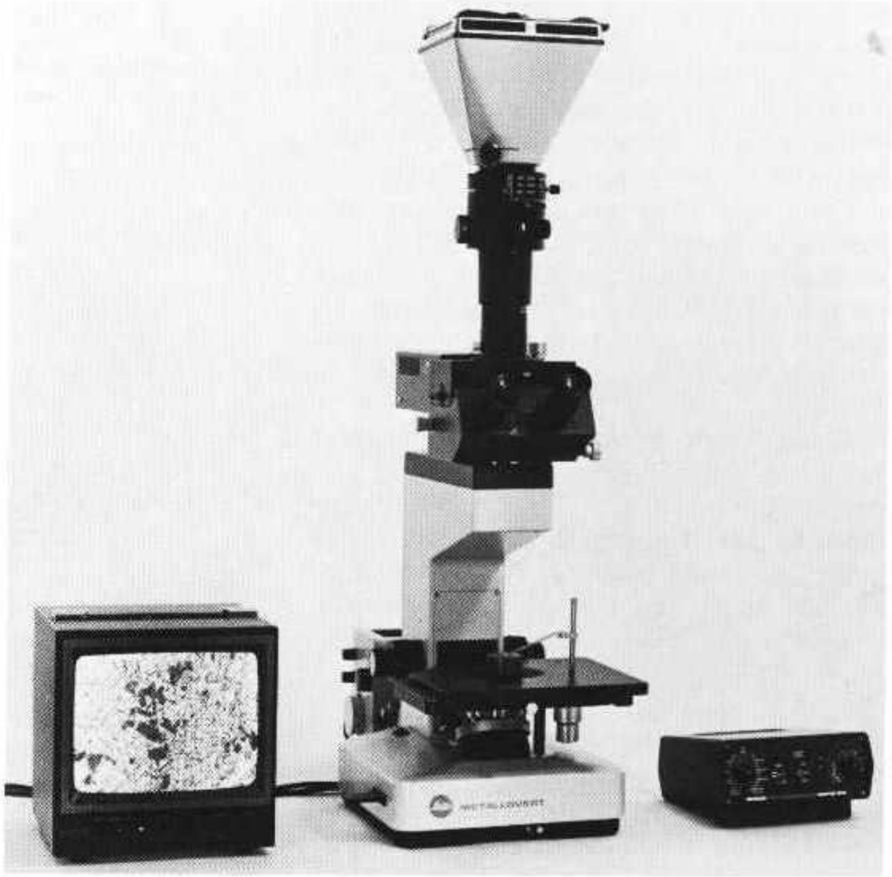


Fig. 47 Television monitor for group viewing attached to an inverted-type microscope. (E. Leitz, Inc.)

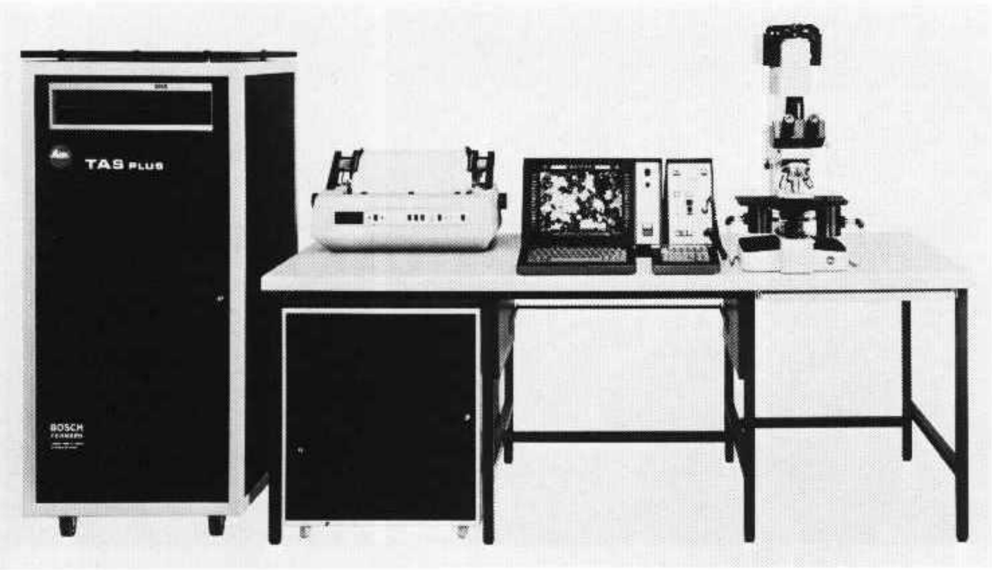


Fig. 48 Fully automatic image analyzer. Although these devices can be quite expensive, they have stimulated interest in stereology and its application to structure-property correlations. (E. Leitz, Inc.)

Fig. 49 Semiautomatic image analyzer. With this system, the operator controls detection of features by tracing with a light pen. (C. Zeiss, Inc.)

Properties of Objective Lens

- Magnification (on lens)
- Numerical aperture (N.A)
- N.A.=n SIN(alpha)
- Where n= is the minimum reflection index of the material (air or oil) between the specimen and the lens (n almost 1 for air)
- alpha = is the half-angle of the most oblique light rays that enter the front lens of the objective. Light-collecting ability increases with alpha
- Resolution (d)
- d= k*lamda/(N.A.)
- where k is 0.5 or 0.61
- lamda is wavelength
- Depth of field (Tf)
- Tf= lamda*(n^2-NA^2)^0.5/NA^2

$$NA = n \sin \alpha$$

$$d = \frac{k\lambda}{NA}$$

$$T_{\mathrm{f}} = \frac{\lambda \sqrt{n^2 - NA^2}}{NA^2}$$

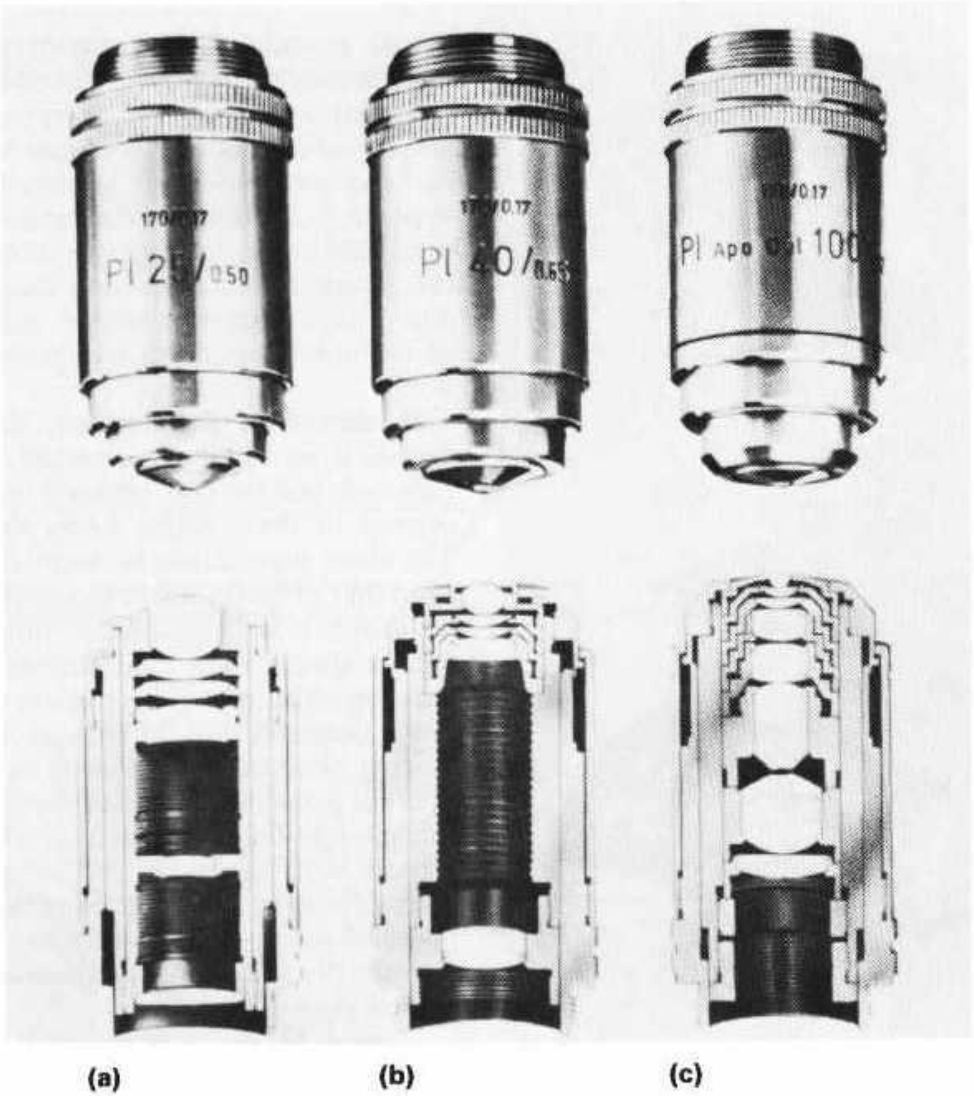
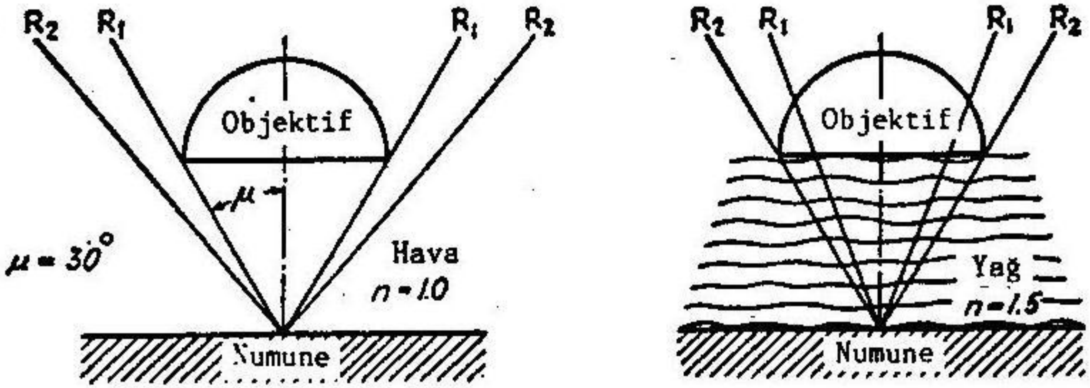


Fig. 6 Plano-type objective lenses and cross sections through each. The lens shown in (c) is a 14-element oil-immersion objective, with a numerical aperture (*NA*) of 1.32. Because the lens and specimen must be cleaned between each use, oil immersion is rarely used; it does provide higher resolution and a crisper image, which is valuable for examining low-reflectivity specimens. (E. Leitz, Inc.)

$$\text{N.A.} = \eta \cdot \sin \mu$$



(a) Kuru objektif (b) Yağlı objektif

Şekil 3-2 — Kuru ve yağlı objektiflerde nümerik açıklığın şematik gösterilişi

dır. Havanın kırılma indisi $\eta = 1$ olduğundan, kuru objektifte $\text{N.A} = \sin \mu$, objektif yağının kırılma indisi $\eta = 1{,}5$ olduğundan, yağlı objektifte $\text{N.A} = 1{,}5 \sin \mu$'dır.

c) *Ayırt Etme Gücü :* Bir objektifin ayırt etme gücü, numunedeki birbirine yakın detayları farkettirebilme yeteneğinin ölçüsüdür. Bu güç, objektifin nümerik açıklığına ve numuneyi aydınlatan ışığın dalga boyuna bağlıdır. Eğer, objektifin açıklığı tamamen ışıklandırılmış ise, maksimum ayırt etme gücü elde edilmiş olur ki, bu da;

$$\text{Ayırt etme gücü} = \frac{2(\text{N.A})}{\lambda} \tag{3-1}$$

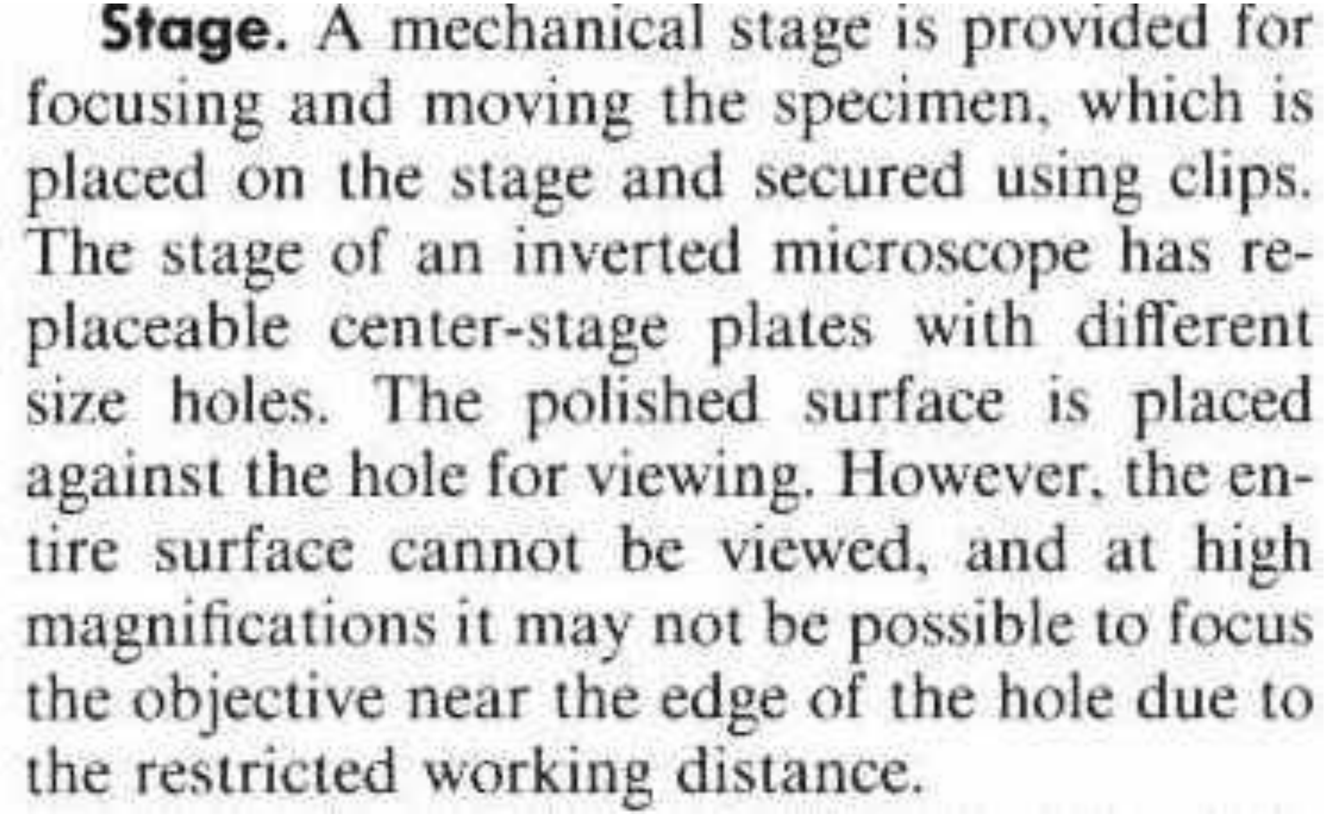
Properties of Ocular Lens

- Magnification (on lens)

Total Mag = Mag of Ob. X Mag. of Oc. X k

Light source

- Low-voltage tungsten-filament lamp
- Carbon-arc light source
- Xenon-arc light source
- Zircon-arc light source
- Sodium-arc light source
- Quartz-iodine light source
- Mercury-vapor lamps



Stage. A mechanical stage is provided for focusing and moving the specimen, which is placed on the stage and secured using clips. The stage of an inverted microscope has replaceable center-stage plates with different size holes. The polished surface is placed against the hole for viewing. However, the entire surface cannot be viewed, and at high magnifications it may not be possible to focus the objective near the edge of the hole due to the restricted working distance.

Using the upright microscope, the specimen is placed on a slide on the stage. Because the polished surface must be perpendicular to the light beam, clay is placed between the specimen bottom and the slide. A piece of lens tissue is placed over the polished surface, and the specimen is pressed into the clay using a leveling press. However, pieces of tissue may adhere to the specimen surface. An alternative, particularly useful with mounted specimens, is to use a ring instead of tissue to flatten the specimen. Aluminum or stainless steel ring forms of the same size as the mounts (flattened slightly in a vise) will seat on the mount rather than the specimen.

Stand. Bench microscopes require a rigid stand, particularly if photomicroscopy is performed on the unit. The various pieces of the microscope are attached to the stand when assembled. In some cases, the bench microscope is placed on a separate stand that also holds the photographic system

Lens Defects

Many lens defects result from the laws of reflection and refraction. The refractive index of a lens varies with the wavelength of light, and the focal length of the lens varies with the refractive index. Therefore, focal length will change for different colors of light. A separate image for each wavelength present is focused at different distances from the lens (Fig. 10). This is longitudinal chromatic aberration. Moreover, magnification varies with focal length, altering the size of the image. This is lateral chromatic aberration (Fig. 11). These differences must be eliminated to produce color photographs. Because achromats have limited corrections for these problems, they must be used with yellow-green light filtering to obtain sharp images.

Spherical aberration (Fig. 12) occurs when light from a point object on the optical axis is more strongly refracted at the center or at the periphery of the lens, producing a series of focal positions in which the point image appears as a circle of finite area. This can be minimized by using an aperture that restricts use of the objective to the central portion. Lens design also can correct part of this problem.

Because the image surface of optimum focus is curved, compensating eyepieces with equal but opposite curvature are used to produce a flat image (Fig. 13). Other problems, such as coma and astigmatism, can impair image quality unless corrected.

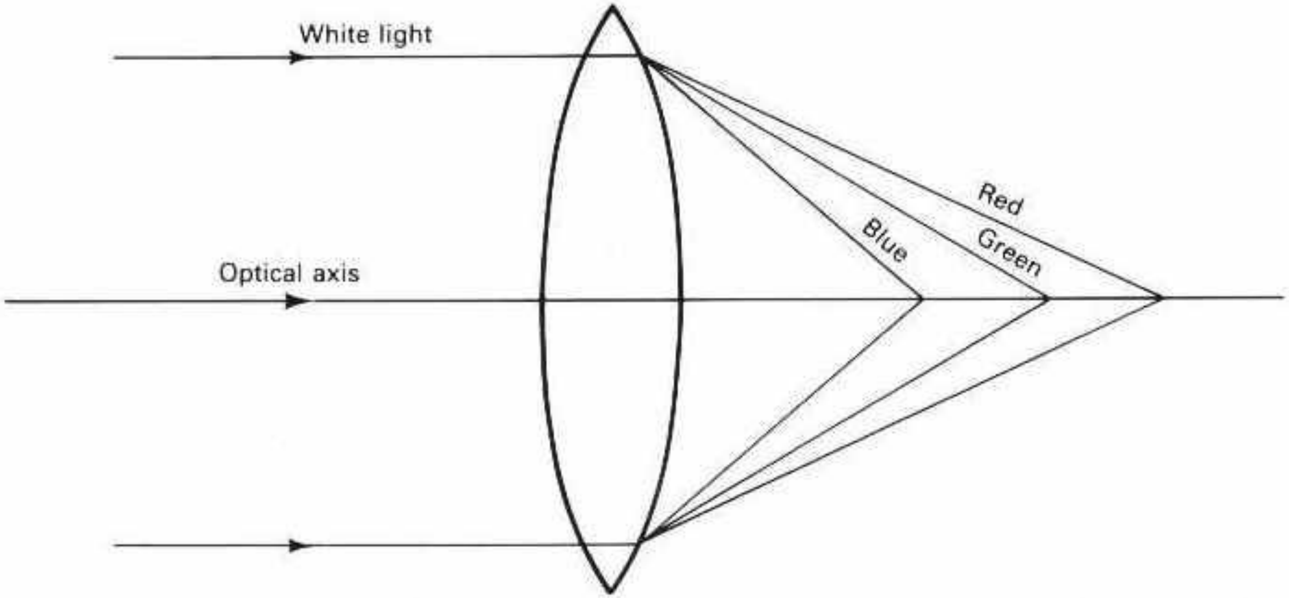


Fig. 10 Longitudinal chromatic aberration in an uncorrected lens. Different wavelengths cause each of the three primary colors to be focused at a different point along the optical axis.

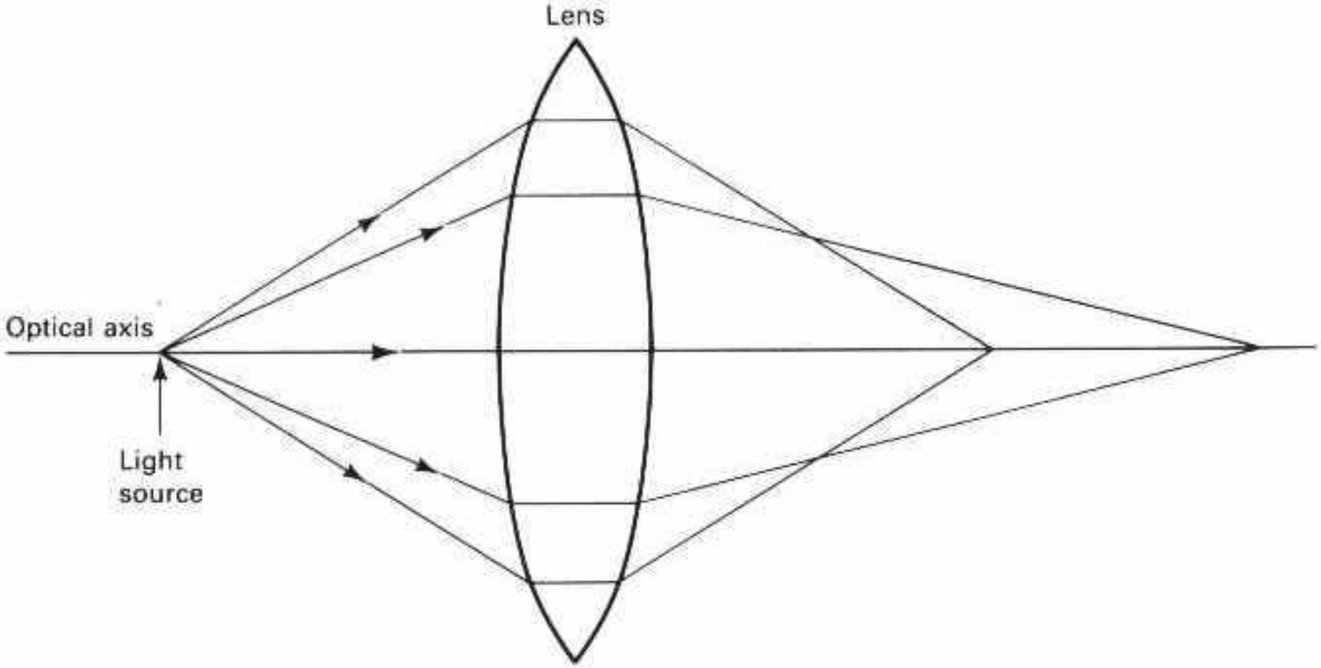


Fig. 12 Spherical aberration. Light rays passing through the outer portion of the lens are more strongly refracted than those passing through the central portion and are focused at a different point along the optical axis. This problem can be minimized by using an aperture to restrict the light path to the central part of the objective.

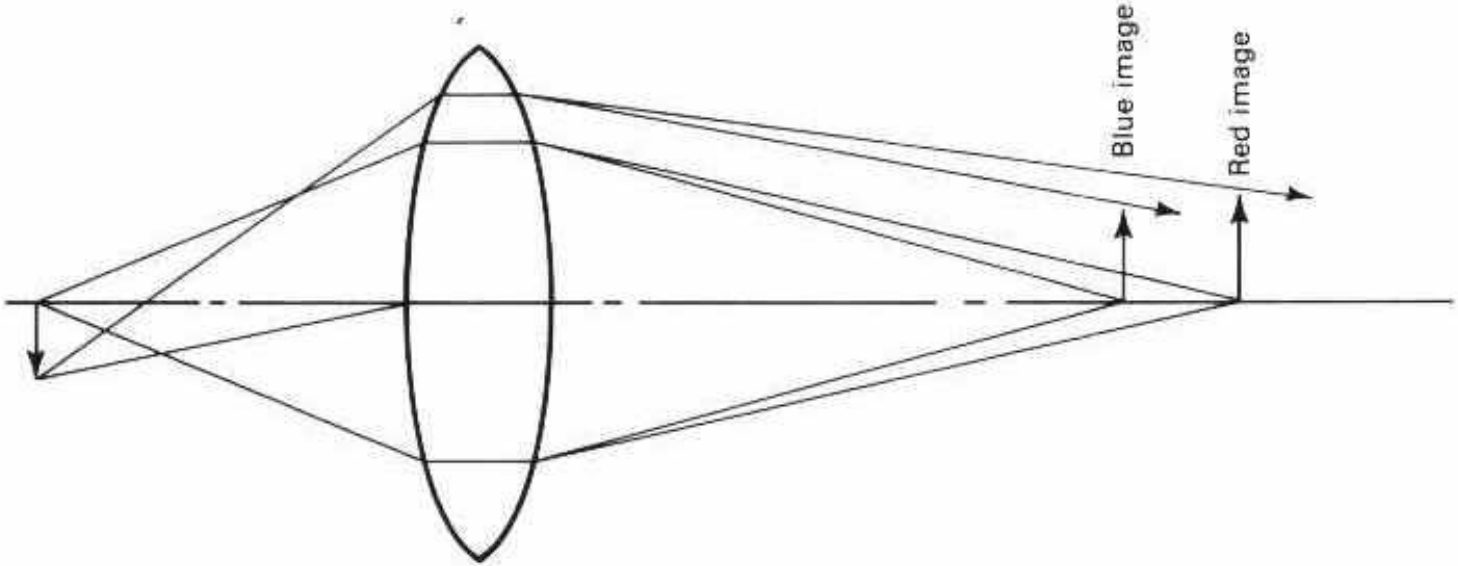
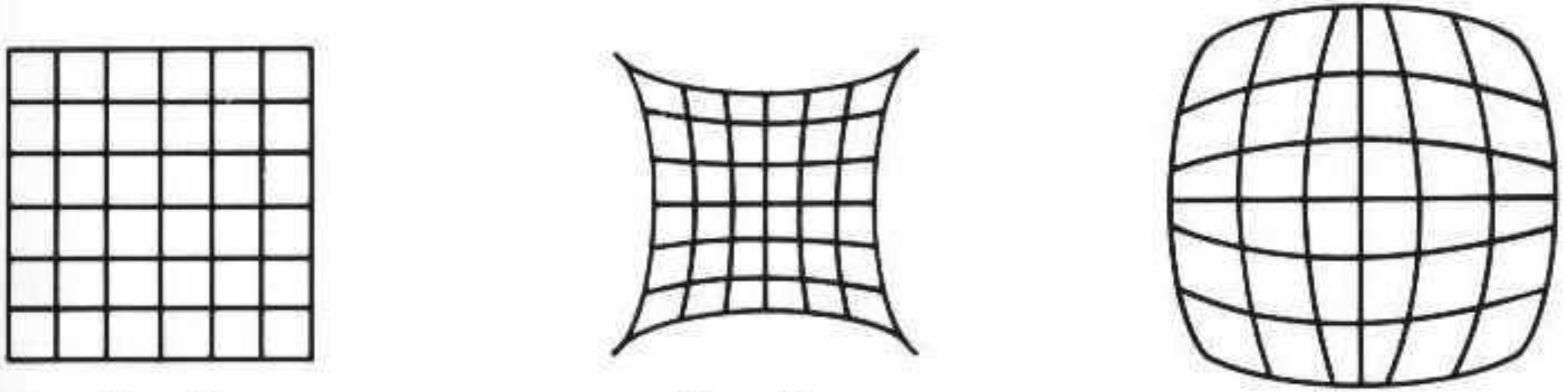


Fig. 11 Lateral chromatic aberration. As focal length is varied, magnification changes, altering image size.



Normal image

Pincushion image

Barrel image

Fig. 13 Image distortions caused by curvature in the image surface of best focus. A compensating eyepiece, with a curvature equal to but opposite of that of the image surface, must be used to produce a normal image.

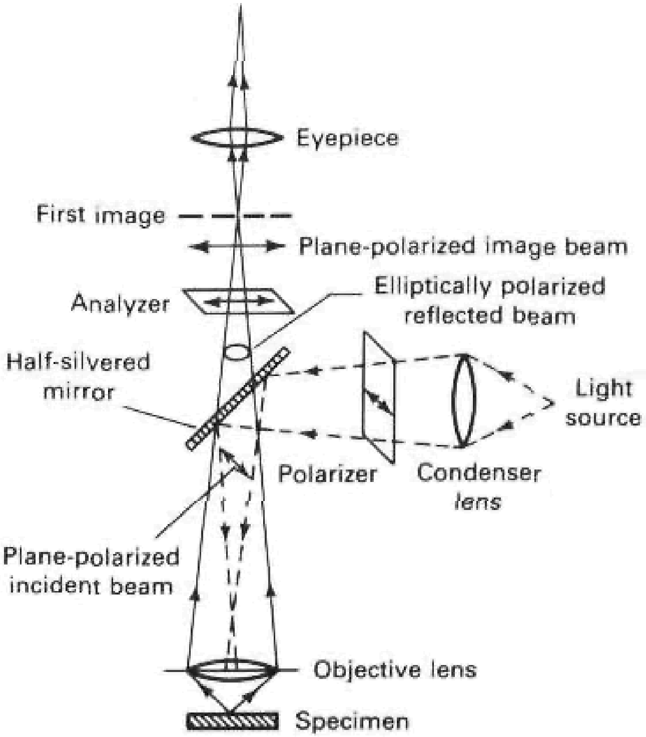
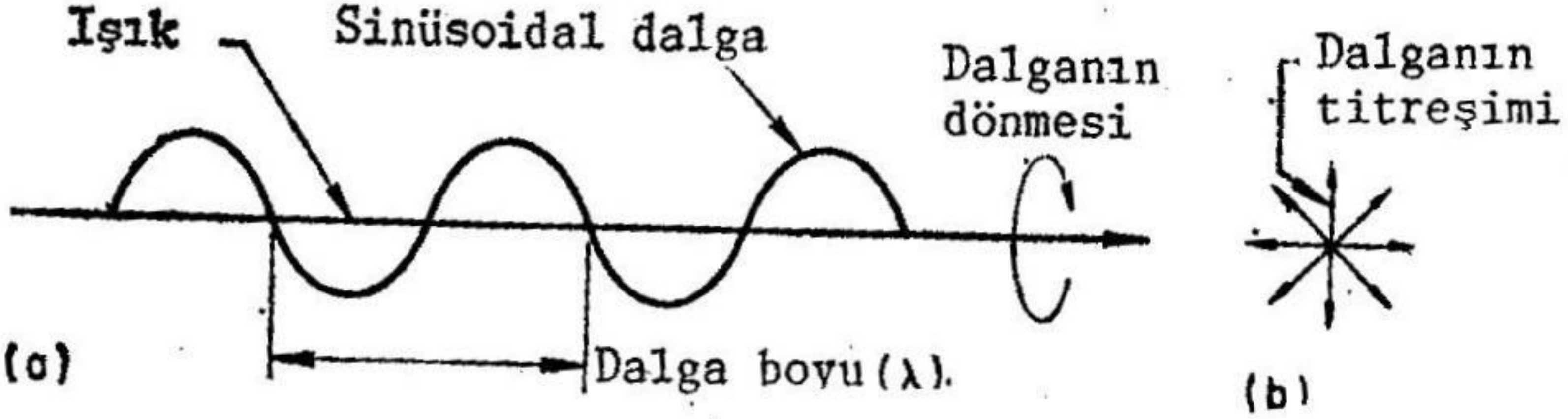
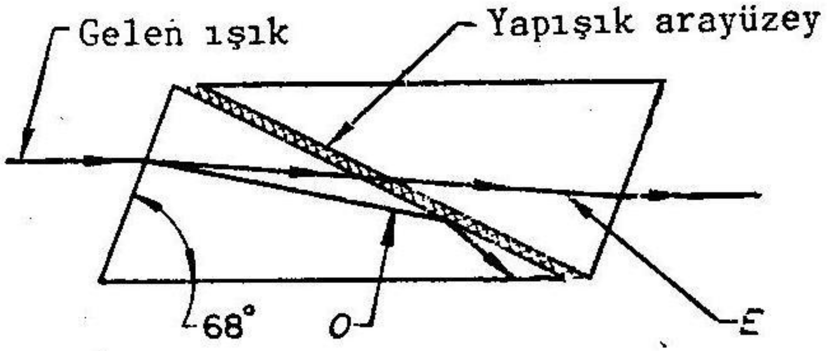


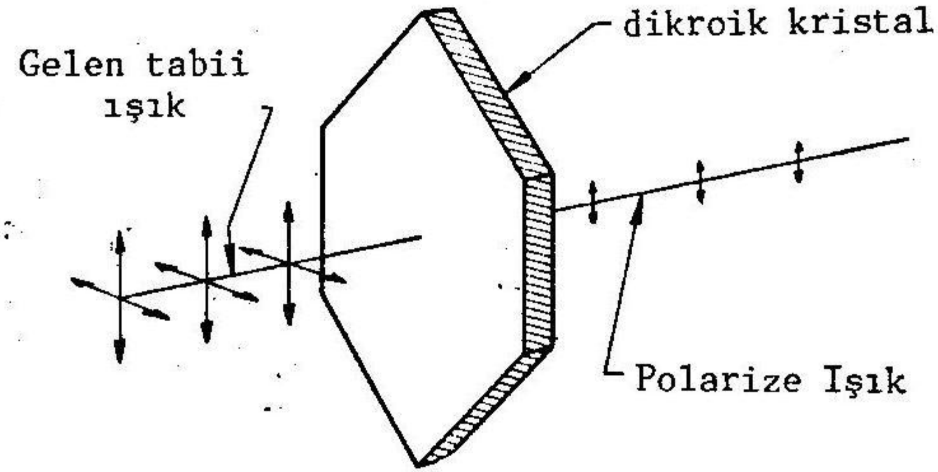
Fig. 3 Principles of polarized light microscopy



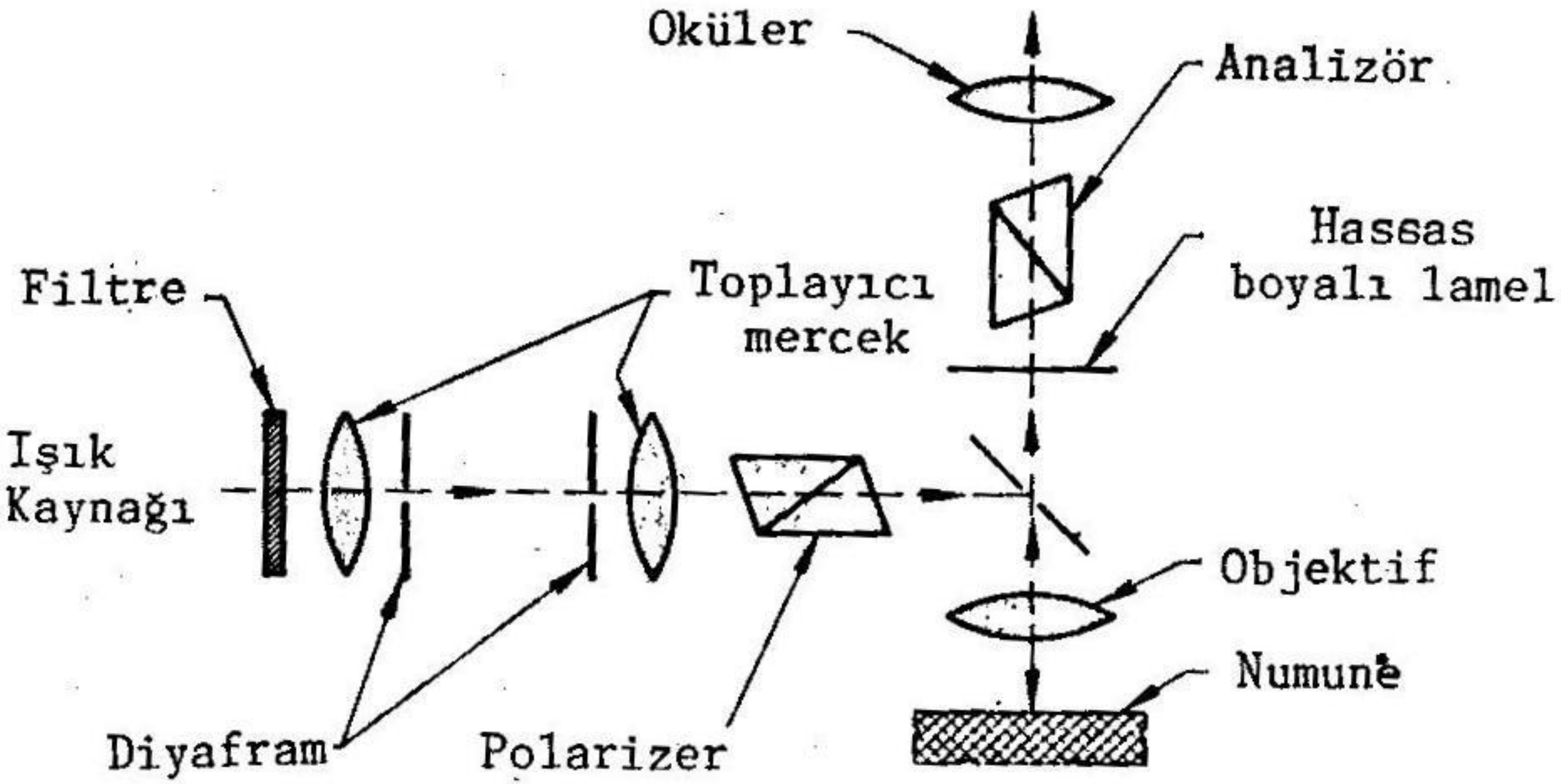
Şekil 3-7 — Işığın sinosoidal dalga şeklinde gösterilmesi. a) Düşey düzlemde, b) Enine kesit



Şekil 3-8 — Nikol Prizmasının prensibi. Gelen ışık «ordinary» ve «Ekstra ordinary» olarak ikiye ayrılmaktadır



Şekil 3-9 — Dikroik kristalden geçen tabii ışıktan polarize ışık elde edilmesi. Kristal yatay titreşimleri tamamen absorbe etmekte düşey titreşimleri ise kısmen absorbe etmektedir



Şekil 3-10 — Optik mikroskopta, polarize ışık düzeninin yeri

Polarize Işığın Metalografide Kullanılması

Pratikte, mikroskoba yerleştirilen polarize ışık düzeni Şekil 3-10'da görüldüğü gibidir. Polarizörden çıkan polarize ışık numuneye gider ve numuneden yansıyan ışık Analizör'den geçer. Analizör, 0-90° arasında dönebilir. Polarizör ile Analizör birbirine dik olunca ışık tamamen yok olur. Eğer ışık izotropik yüzeyden yansıyorsa, analizörün döndürülmesi ile tamamen yok olur. Buna karşılık eğer ışık anizotropik yüzeyden yansırsa, analizör döndürüldüğünde görüntü her 90°de bir alternatif olarak karanlıktan aydınlığa doğru değişir. Bunun sebebi, anizotropik malzemelerde optik özelliklerin kristalografik yön ile değişmesidir.

Anizotropik metaller kübik olmayan metallerdir. Örneğin Sb, Be, Cd, Mg, Sn, Ti, U, Zn, Zr gibi. Bu metallerde özellikle tane yapısı polarize ışık altında gayet iyi bir şekilde ayırt edilir. Polarize ışık aynı zamanda martensitik çeliklerde primer ostenit tane boyutunu saptamada da kullanılmaktadır. Anisotropik davranış, dağlanmış martensit dilimlerinin oyuklardaki oblik yansımadan kaynaklanmaktadır.

Isotropik metaller kübik yapıdadır. Örneğin Al, Cu, Cr, Fe, Mn, Mo, Ni, W, V gibi. Kübik yapıdaki metaller parlatıldıktan sonra polarize ışık altında karanlık görülürler. Fakat dağlamadan sonra yüzey topografyası değişerek veya dağlanan yüzeyde anizotropik film oluşturarak polarize ışığa karşı duyarlık kazanır.

Polarize ışık çok fazlı alaşımların etüdünde de başarı ile kullanılmaktadır. Çok fazlı alaşımlar başlıca üç grupta toplandığında;

a) *İki izotropik faz içeren alaşımlar:* Bu durumda polarize ışıkta her iki faz ayırt edilemez. Ancak uygun bir dağlama reaktifi kullanılarak fazlardan birinin optik aktivitesi diğerine kıyasla arttırılır. Örneğin, Cu-Ni-Sn üçlü sisteminde teta fazı, sigma fazından (her ikisi de izotropik) özel dağlama uygulanarak polarize ışık altında birbirinden ayırt edilebilir.

b) *İzotropik ve anizotropik faz içeren alaşımlar :* Bu alaşımlarda anizotropik faz polarize ışığa duyarlı olduğundan fazlar kolayca birbirinden ayırt edilebilir.

c) *İki anizotropik faz içeren alaşımlar:* Bu alaşımlarda fazlar polarize ışık altında farklı duyarlık gösterdiklerinden birbirinden ayırt edilebilirler.

Examination Modes

- Bright-Field illumination
- Oblique illumination
- Dark-Field illumination
- Polarized light
- Color etching

To achieve the resolution capability of the selected objective, image contrast must be adequate. Image contrast depends on specimen preparation and optics. Differences in light reflectivity from the specimen surface produce amplitude features visible to the eye after magnification. Phase differences created by light reflection must be rendered visible by the use of phase-contrast or interference-contrast attachments to the microscope.

Bright-Field Illumination. Bright-field vertical illumination, the most widely used method of observation, accounts for the vast majority of micrographs taken. In operation, light passes through the objective and strikes the specimen surface perpendicularly. Surface features normal to the incident light reflect light back through the objective to the eyepieces, where the surface features appear bright. Surfaces oblique to the light beam reflect less light to the objective and appear darker, depending on their angle.

Oblique Illumination. With some microscopes, it is possible to decenter the condenser assembly or the mirror so that the light passing through the objective strikes the specimen surface at a nonperpendicular angle. Roughness on the specimen surface will cast shadows, producing a three-dimensional appearance. This allows determination of features that are in relief or are recessed. However, very little obliqueness can be introduced, because this technique causes lighting to become nonuniform and reduces resolution.

In dark-field illumination, the light reflected from obliquely oriented features is collected, and the rays reflected from features normal to the incident beam are blocked. Therefore, the contrast is essentially reversed from that of bright-field illumination; that is, features that are bright in bright-field illumination appear dark, and features normally dark appear bright. This produces very strong image contrast, with the oblique features appearing luminous. Under such conditions, it is often possible to see features not visible using bright-field illumination. This method is particularly useful for studying grain structures. However, the low light intensity makes photomicroscopy more difficult, a problem lessened by the use of automatic exposure-control devices.

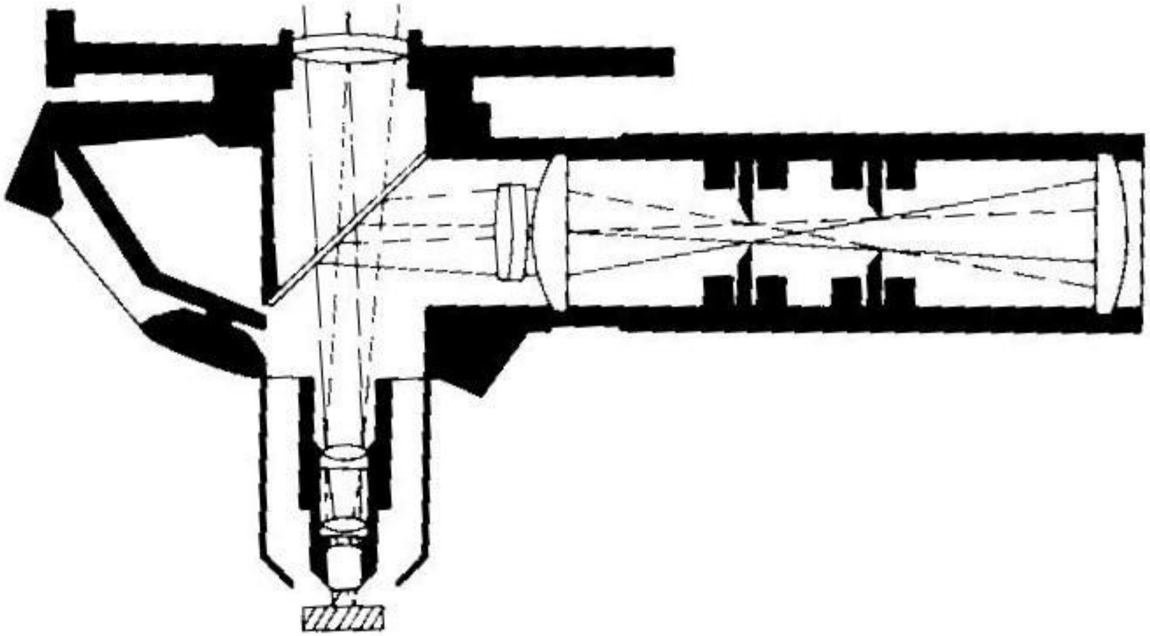


Figure 4-13 Optical path in the vertical illuminator of the metallurgical microscope in the bright-field illumination mode. *(Courtesy of E. Leitz, Inc.)*

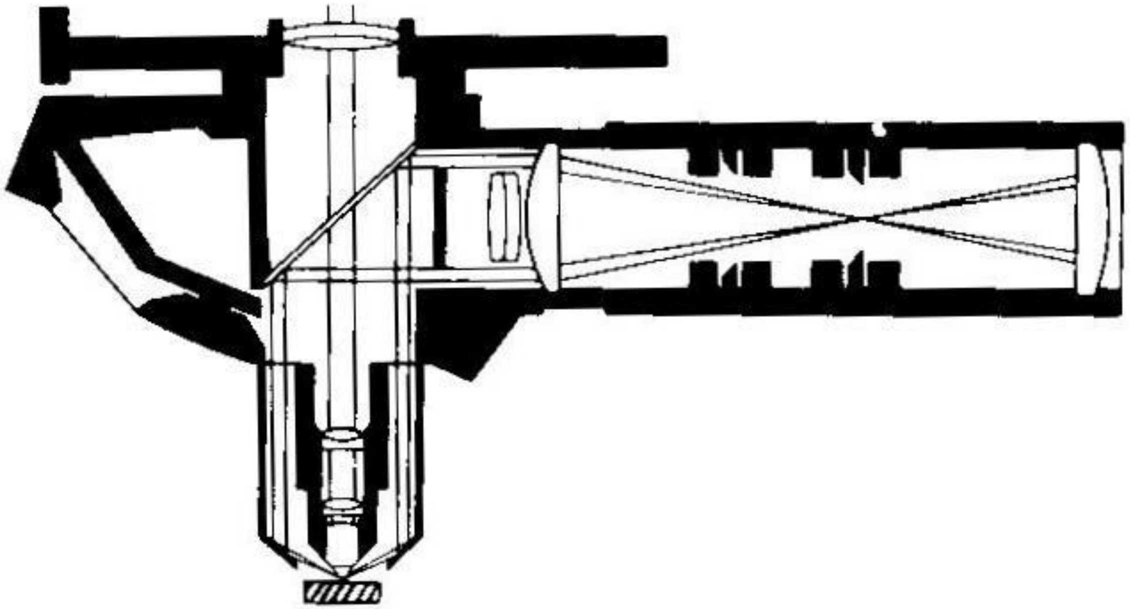


Figure 4-14 Optical path in the vertical illuminator of the metallurgical microscope in the dark-field illumination mode. *(Courtesy of E. Leitz, Inc.)*

Figures 16 to 18 illustrate the value of dark-field illumination for examining grain structure. Figures 19 to 21 show the eutectic in the copper-phosphorus system in bright-field, dark-field, and interference-contrast illumination. Note the strong contrast at the lamellae in dark-field. Figures 22 to 25 show martensite formed in a copper-aluminum alloy using bright-field, dark-field, polarized light, and interference-contrast illumination. Note how the latter three illumination modes produce greater detail of the structure than bright-field illumination (even if the specimen is etched).

Polarized light (Ref 12-14), as used in metallography, has generally been limited to observation of certain optically anisotropic metals, such as beryllium, α-titanium, zirconium, and uranium, that are difficult to etch but respond well to polarized light when properly polished. Before development of the electron microprobe analyzer (EMPA) and energy-dispersive spectroscopy (EDS), polarized light examination was an integral part of the procedure for identifying inclusions. Since the development of these instruments, polarized light has been used less frequently for this purpose, because identification with the EMPA or EDS techniques is more definitive.

Most metallurgical microscopes now use synthetic Polaroid filters. The "polarizer" is placed in the light path before the objective, and the "analyzer" is placed in the light path after the objective, generally just below the eyepiece. Figure 26 shows the light path in the incident-light polarizing microscope.

Light consists of transverse waves vibrating in all directions at right angles to the direction of propagation. These vibrations occur symmetrically around the direction of propagation and are unpolarized. When light passes through a polarizing filter, the vibrations occur in only one plane in the direction of propagation, and the light is termed plane-polarized. This plane will change as the filter is rotated. When the analyzer filter is placed in the light path, plane-polarized light will pass through it if the plane of vibration of the light is parallel to the plane of vibration of the analyzer. If the plane of vibration of the analyzer is perpendicular to that of the light, the light will not pass through, and extinction results.

When plane-polarized light is reflected from the surface of an isotropic metal (any metal with a cubic crystallographic structure, such as iron), then passes through the analyzer in the crossed position (plane of vibration perpendicular to that of the plane-polarized light), the image is extinguished, or dark. However, in practice, because the metallurgical microscope will not produce perfectly plane-polarized light, complete extinction will not occur. This is not a serious problem, because polarized light is used only in a qualitative manner in metallography. Strain-free objectives, usually achromats, must be used. Fluorite or apochromatic objectives are unsuitable. A strong white-light source is required to produce accurate color effects.

If an optically anisotropic, polished metal is placed under the light beam with the polarizer and analyzer crossed, the microstructure will be revealed (Fig. 27 and 28). The quality of specimen preparation is very important, and the surface must be perpendicular to the light path. Rotation of the specimen under the beam changes light intensity and color. Because it may be difficult to set the polarizer and analyzer in the crossed position accurately when an anisotropic specimen is in place unless the crossed positions are marked on the polarizer and the analyzer, it is best to find this position first using an isotropic specimen.

When plane-polarized light strikes an anisotropic metal surface, reflection occurs as two plane-polarized components at right angles to each other. The directions vary with crystal structure. The strength of these two perpendicular reflections can change, and a phase difference exists between them. These differences vary with each metal and depend on the crystal orientation. No reflection is obtained when the basal plane of hexagonal or tetragonal crystals is perpendicular to the light beam. Maximum reflectance occurs when the principal symmetry axis of the crystal is perpendicular to the light beam. The resultant image is predominantly influenced by these orientation effects; phase differences are of little significance.

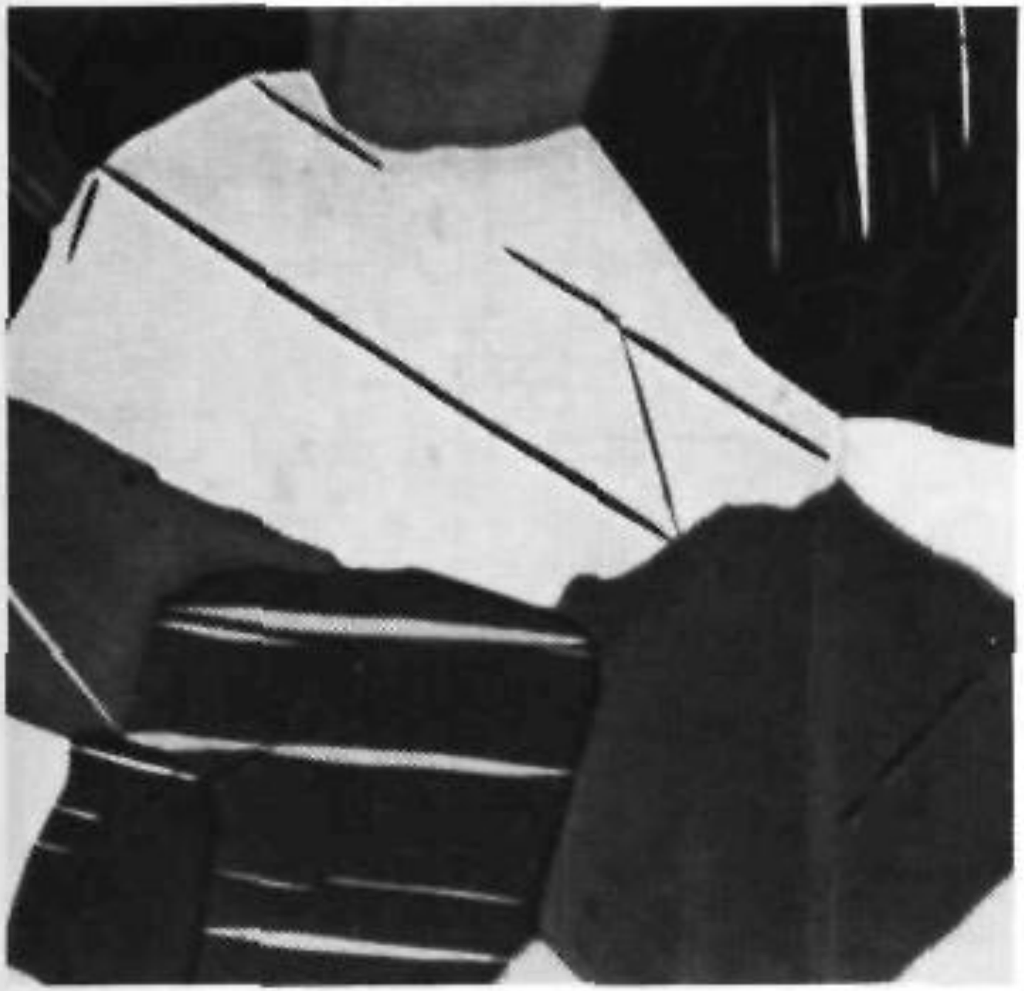


Fig. 4 Grains and deformation twins revealed by polarized light on an as-polished section of cast bismuth. 50×

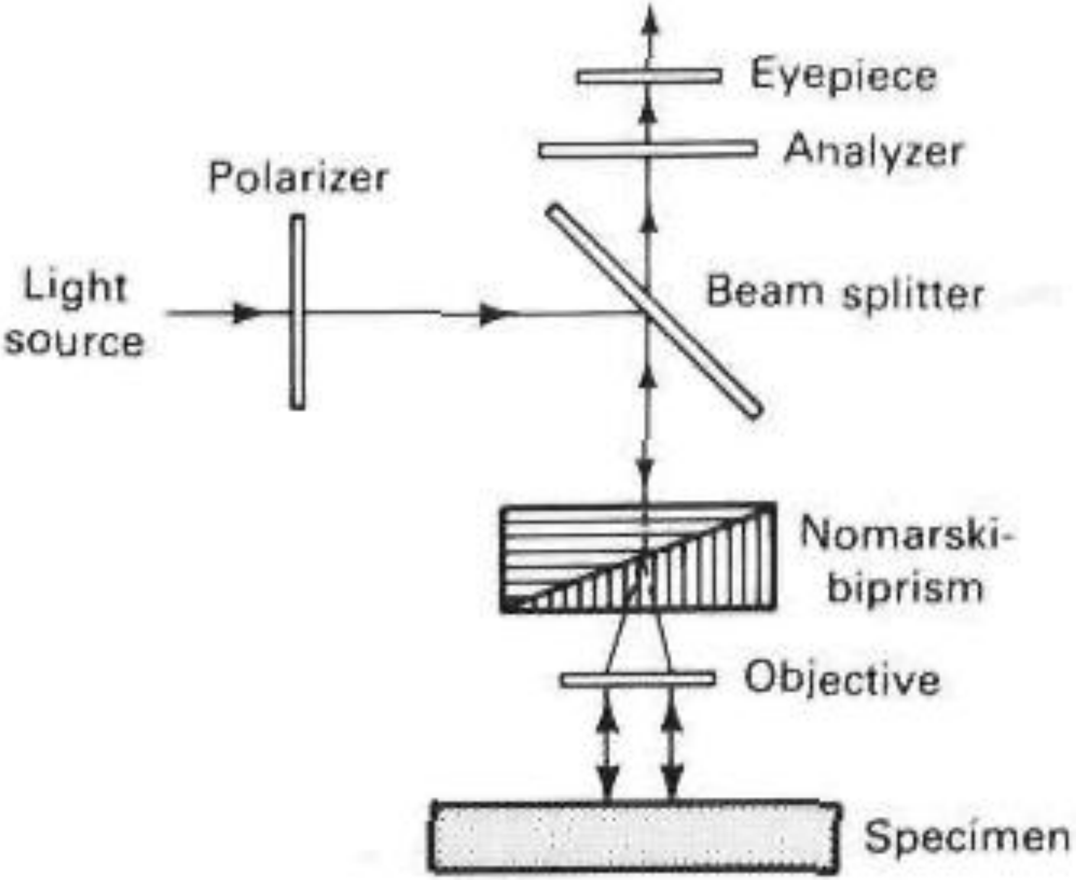


Fig. 6 Principles of differential interference contrast after Nomarski

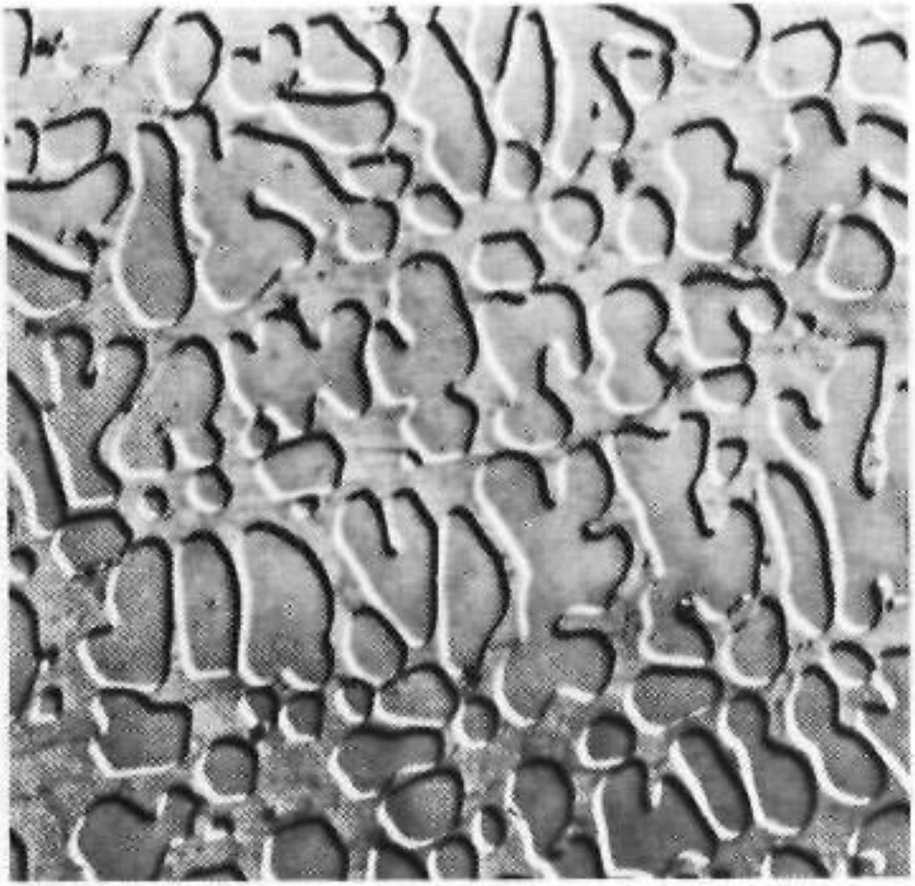


Fig. 7 Differential interference contrast after Nomarski showing the two-phase structure of a U-33Al-25Co (at.%) alloy. Electrolytically etched. 250×

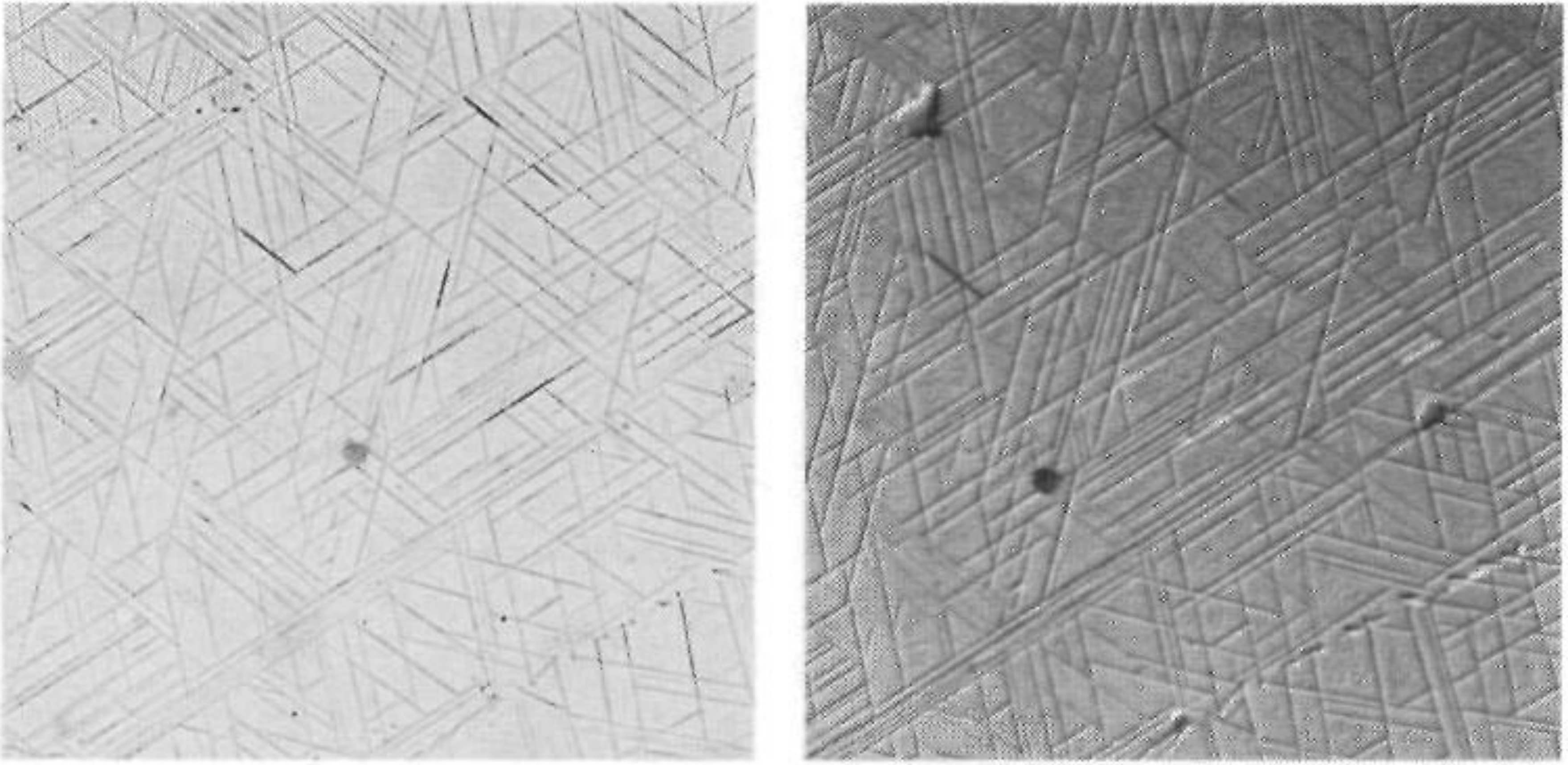


Fig. 38, 39 Inconel 718 heat treated 100 h at 870 °C (1600 °F) to produce needlelike orthorhombic Ni_3Nb. Fig. 38: bright-field illumination. Fig. 39: differential interference-contrast illumination. Particles in relief in Fig. 39 are niobium carbides; particles flush with the surface are niobium nitride. As-polished. 400×

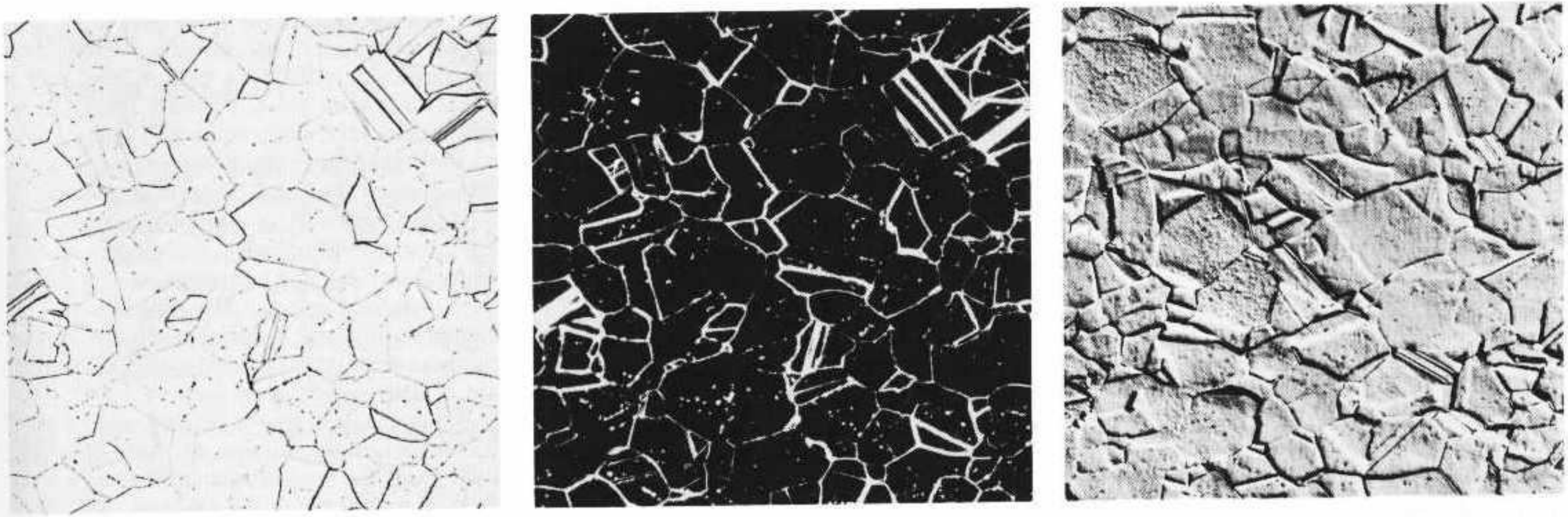


Fig. 16, 17, 18 Austenitic stainless steel (Fe-20Cr-33Ni-2.5Mo-3.5Cu and Nb + Ta), solution annealed. Fig. 16: bright-field illumination. Fig. 17: dark-field illumination. Fig. 18: differential interference-contrast illumination. 15 mL HCl, 10 mL acetic acid, 10 mL HNO_3, and 2 drops glycerol. 400×

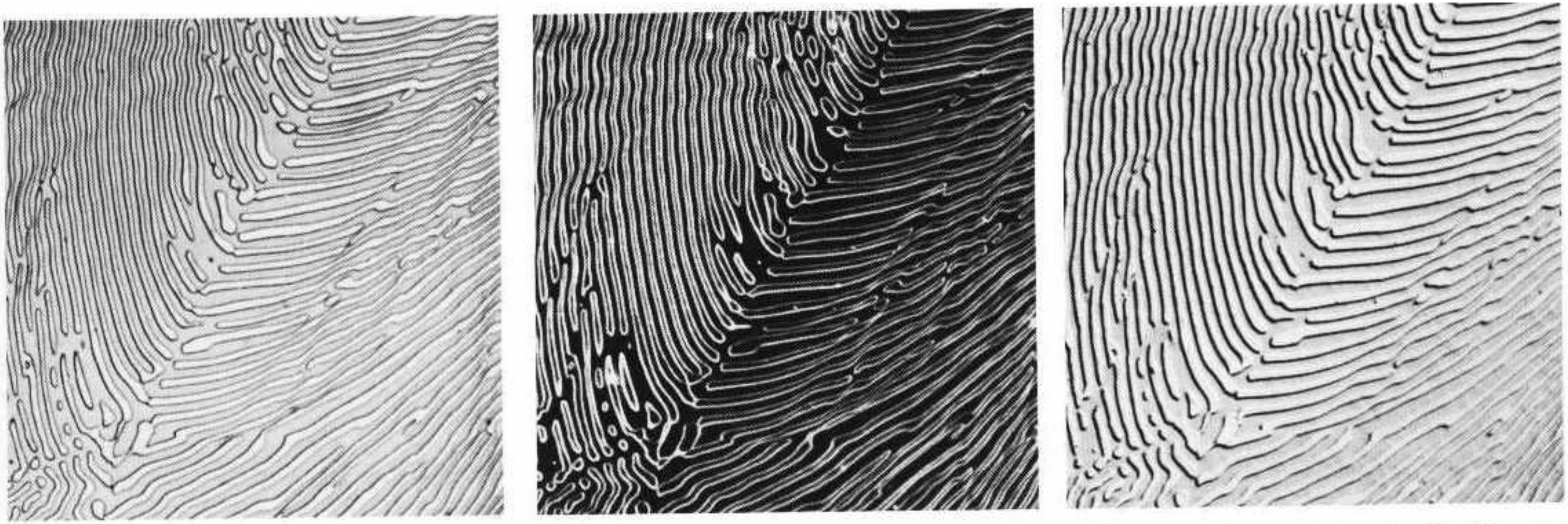


Fig. 19, 20, 21 Cu-8.9P sand cast alloy showing the α + Cu_3P eutectic. Fig. 19: bright-field illumination. Fig. 20: dark-field illumination. Fig. 21: differential interference-contrast illumination. Swab etched using an aqueous solution of 3% $(NH_4)_2S_2O_8$ and 1% NH_4OH. 1000×

Fig. 22, 23, 24, 25 Cu-11.8Al (aluminum bronze), heat treated, with martensite in the microstructure. Fig. 22: bright-field illumination. Fig. 23: dark-field illumination. Fig. 24: differential interference-contrast illumination. Fig. 25: crossed polarized light illumination. As-polished. 200×

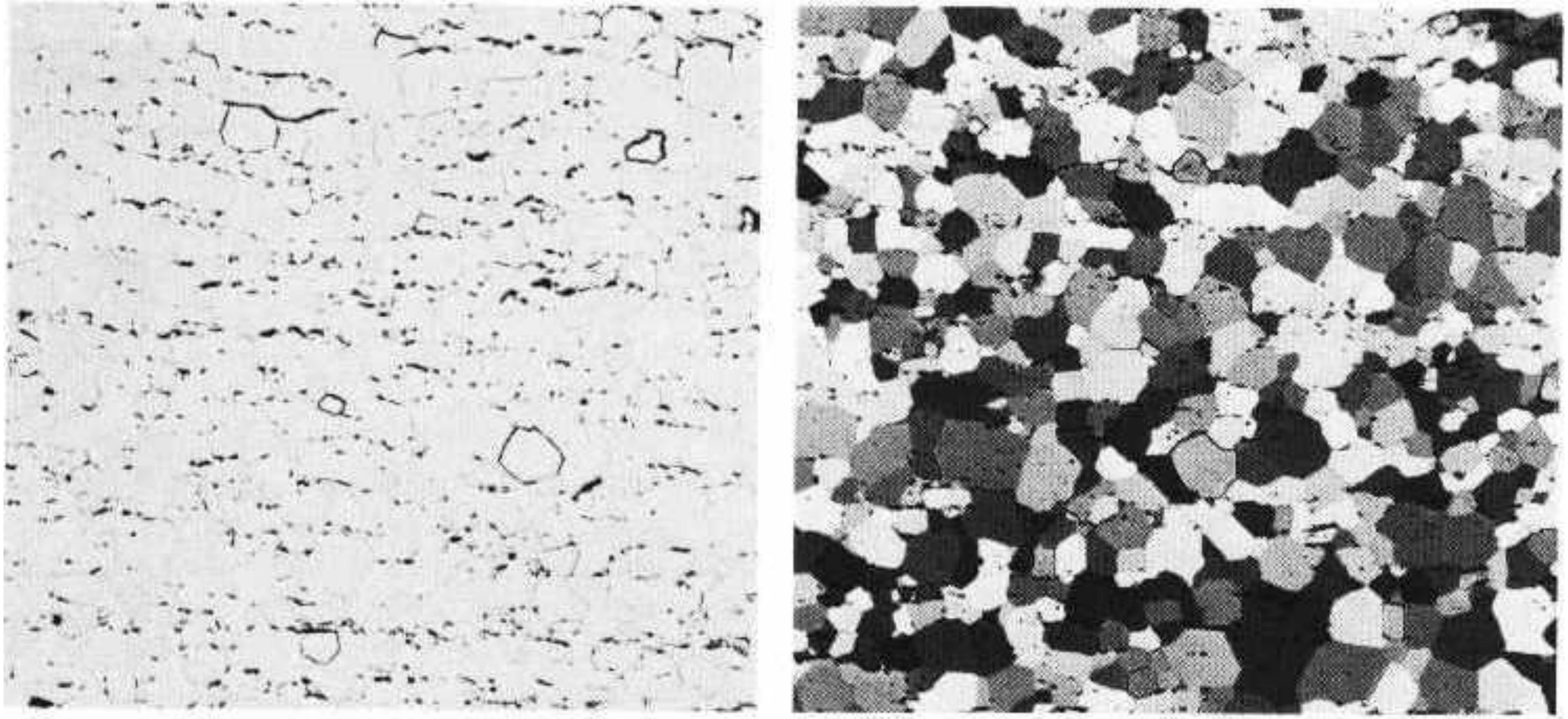


Fig. 27, 28 Polycrystalline zirconium. Fig. 27: bright-field illumination. Fig. 28: crossed polarized light illumination. Chemically polished in 45 mL HNO_3, 45 mL H_2O_2, and 10 mL HF. 100×

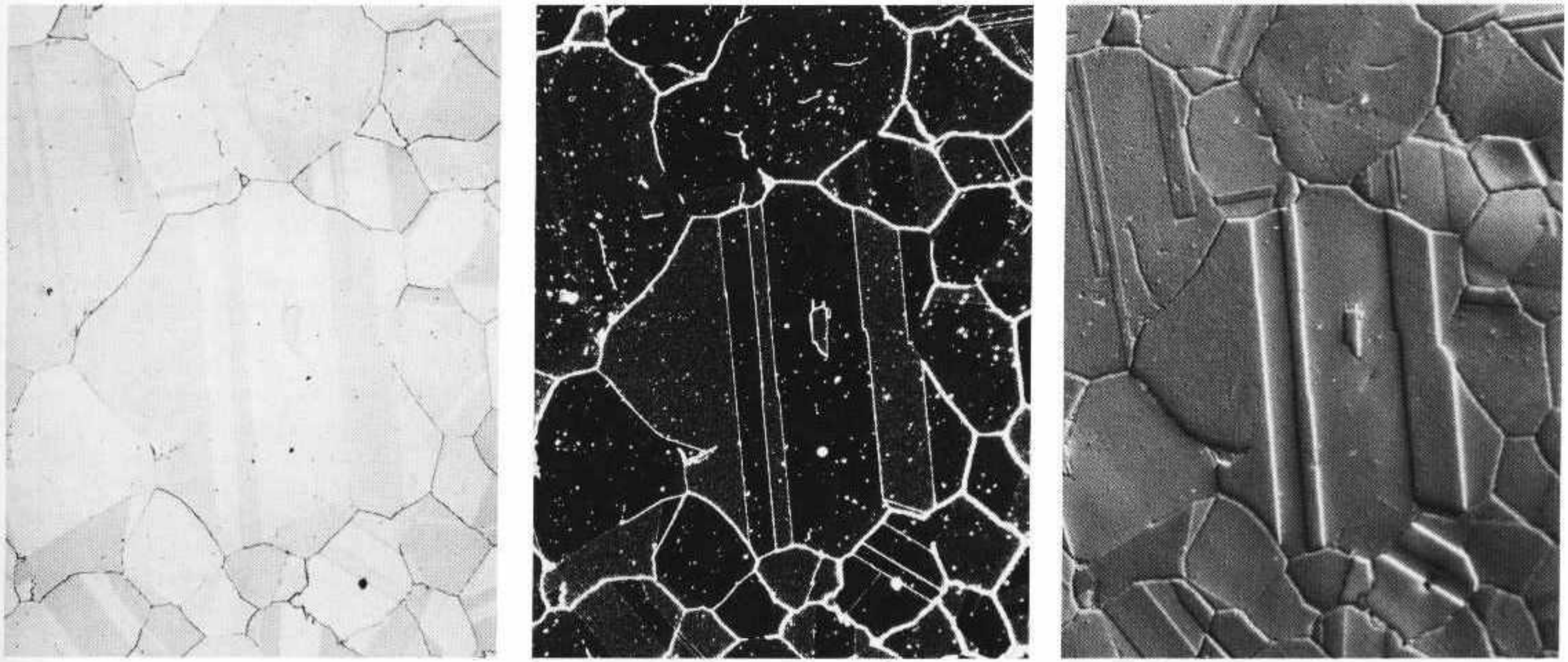


Fig. 35, 36, 37 Solution-annealed and aged Waspaloy. Fig. 35: bright-field illumination. Fig. 36: dark-field illumination. Fig. 37: differential interference-contrast illumination. Glyceregia. 200×

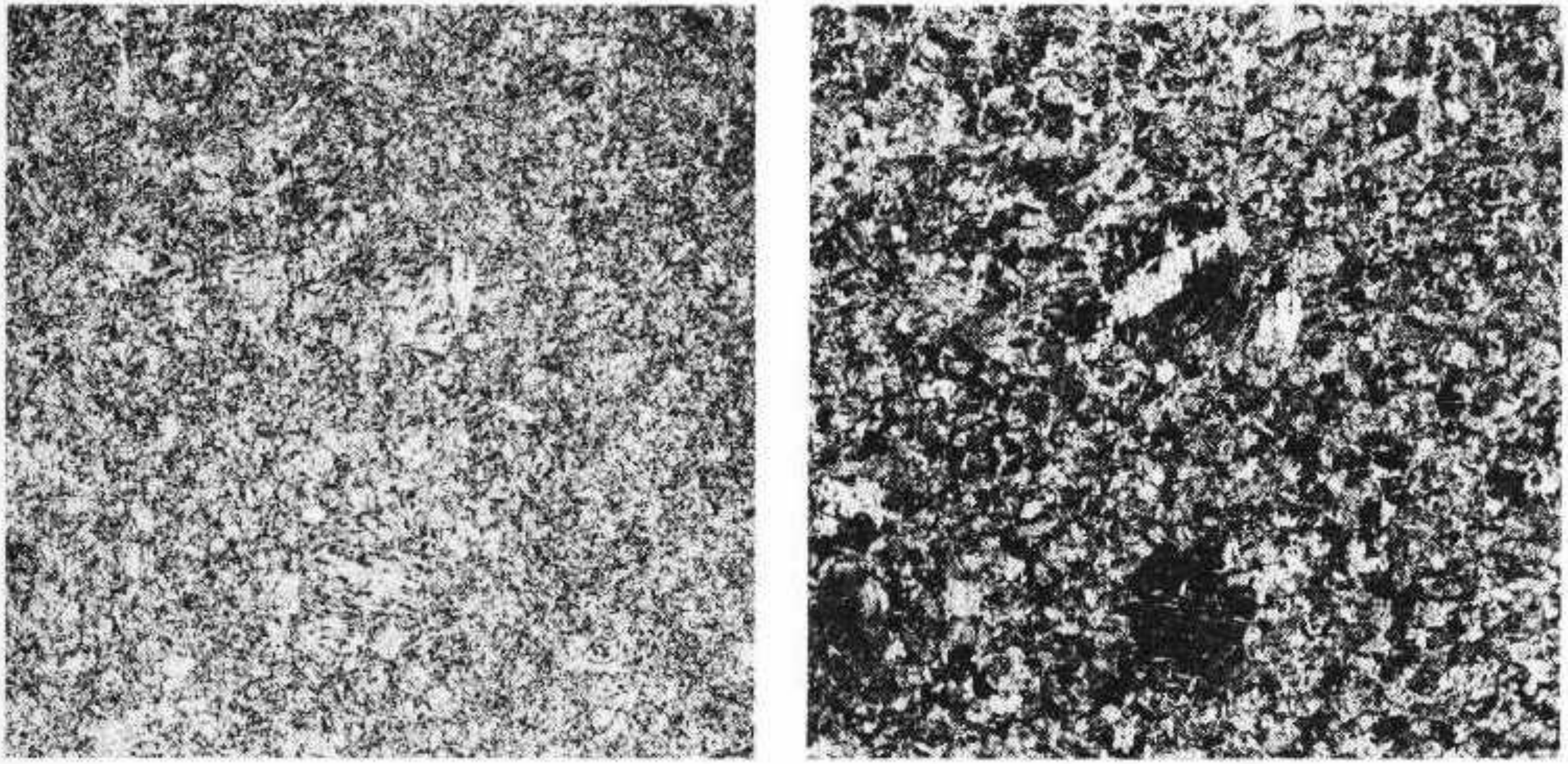


Fig. 32, 33 AF 1410 alloy steel. Fig. 32: Highly tempered lath martensite is difficult to study under bright-field illumination. Fig. 33: Crossed polarized light reveals the packet size by contrast differences. Tint etched in 10% $Na_2S_2O_5$. 100×

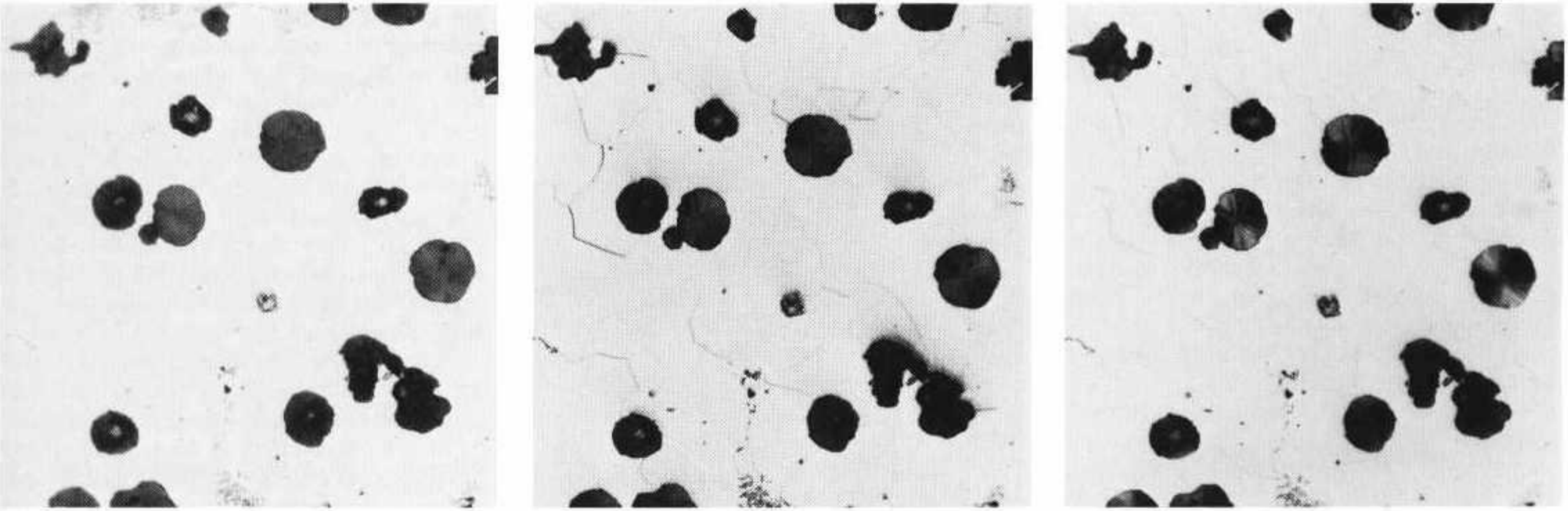


Fig. 29, 30, 31 Graphite nodules in cast iron. Fig. 29: bright-field illumination. Fig. 30: differential interference-contrast illumination. Fig. 31: crossed polarized light illumination. 2% nital. 400×

Stereomicroscope

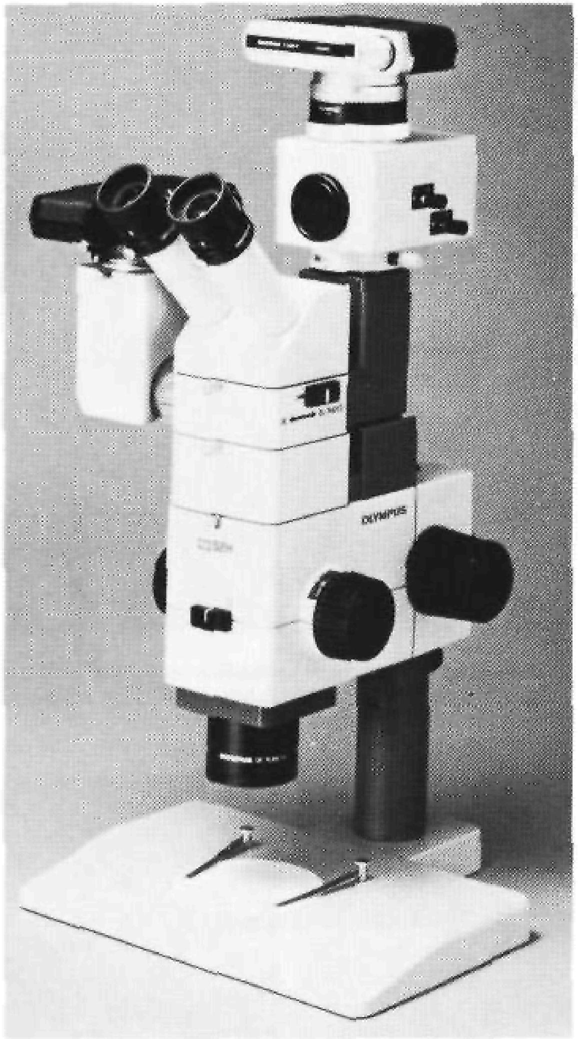
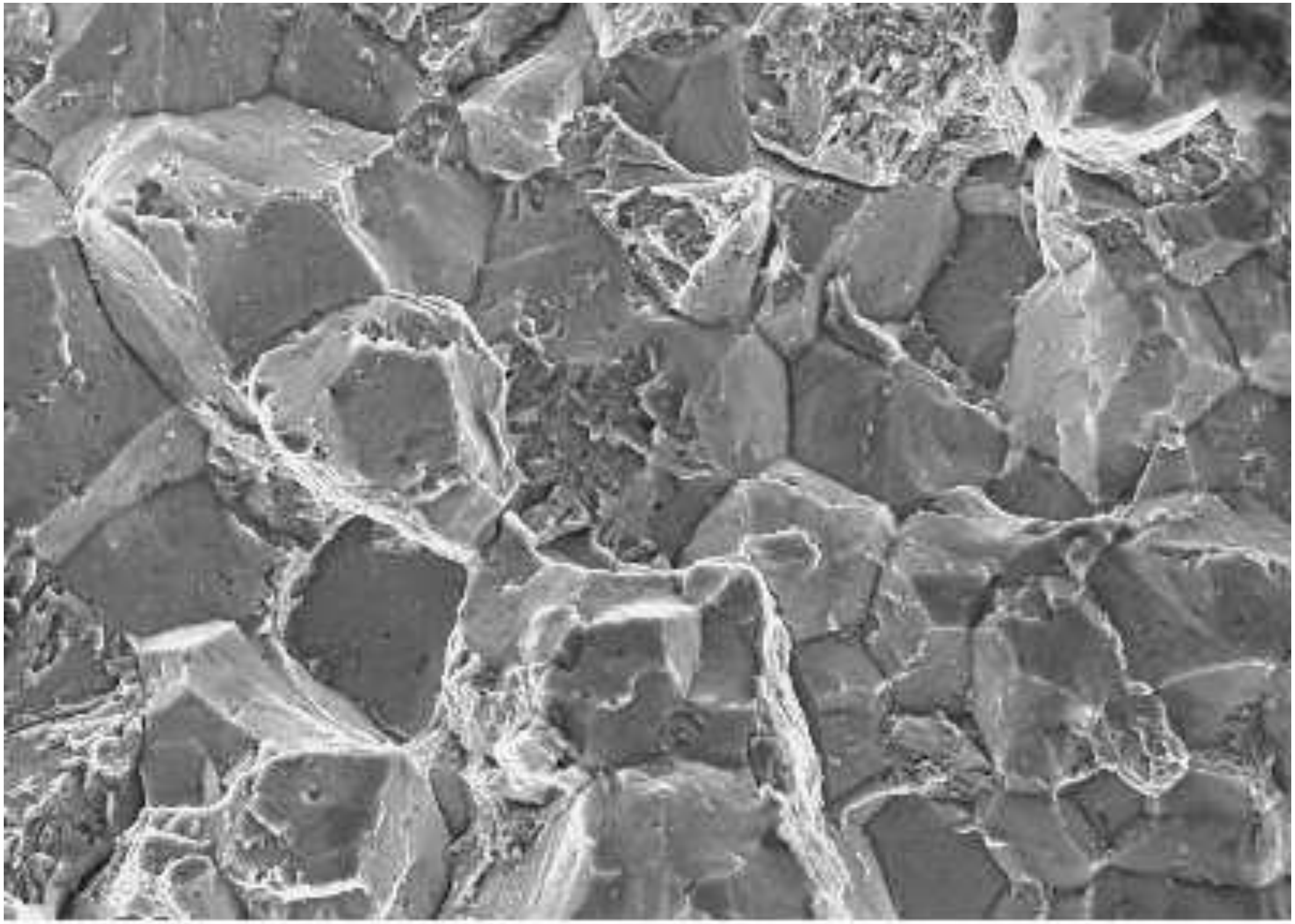
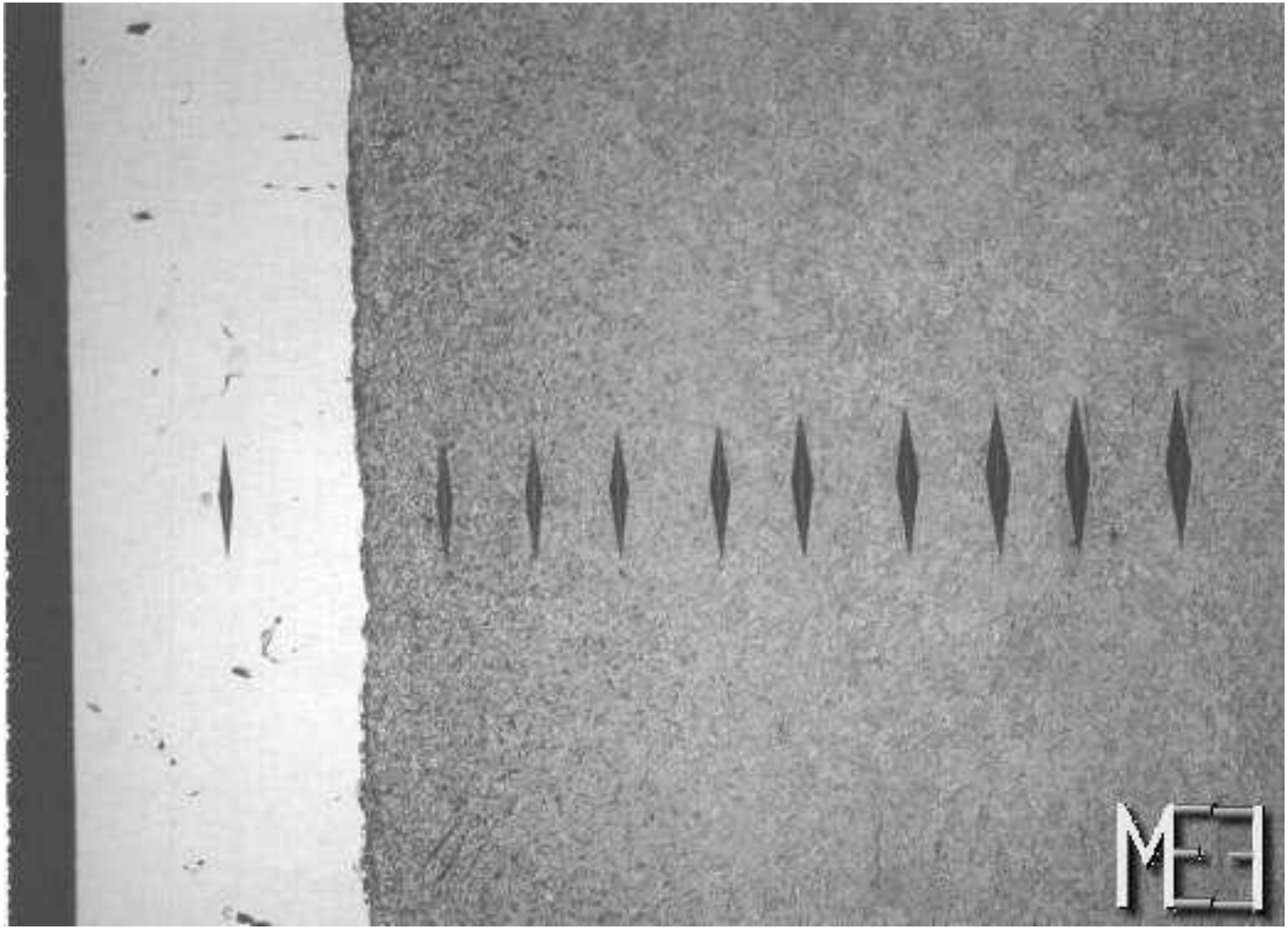


Fig. 66 Typical stereomicroscope. This type of instrument is useful for macroexamination and can be used in preliminary examinations to point out specific features for more detailed study. (Olympus Corporation of America)



Intergranular Fracture



Hot-Stage-Microscope

- It is used for the observation of phase transformation at high temperatures

- For example in Cu- 40 % Zn alloy,
- formation of Widmanstatten-alfa rods

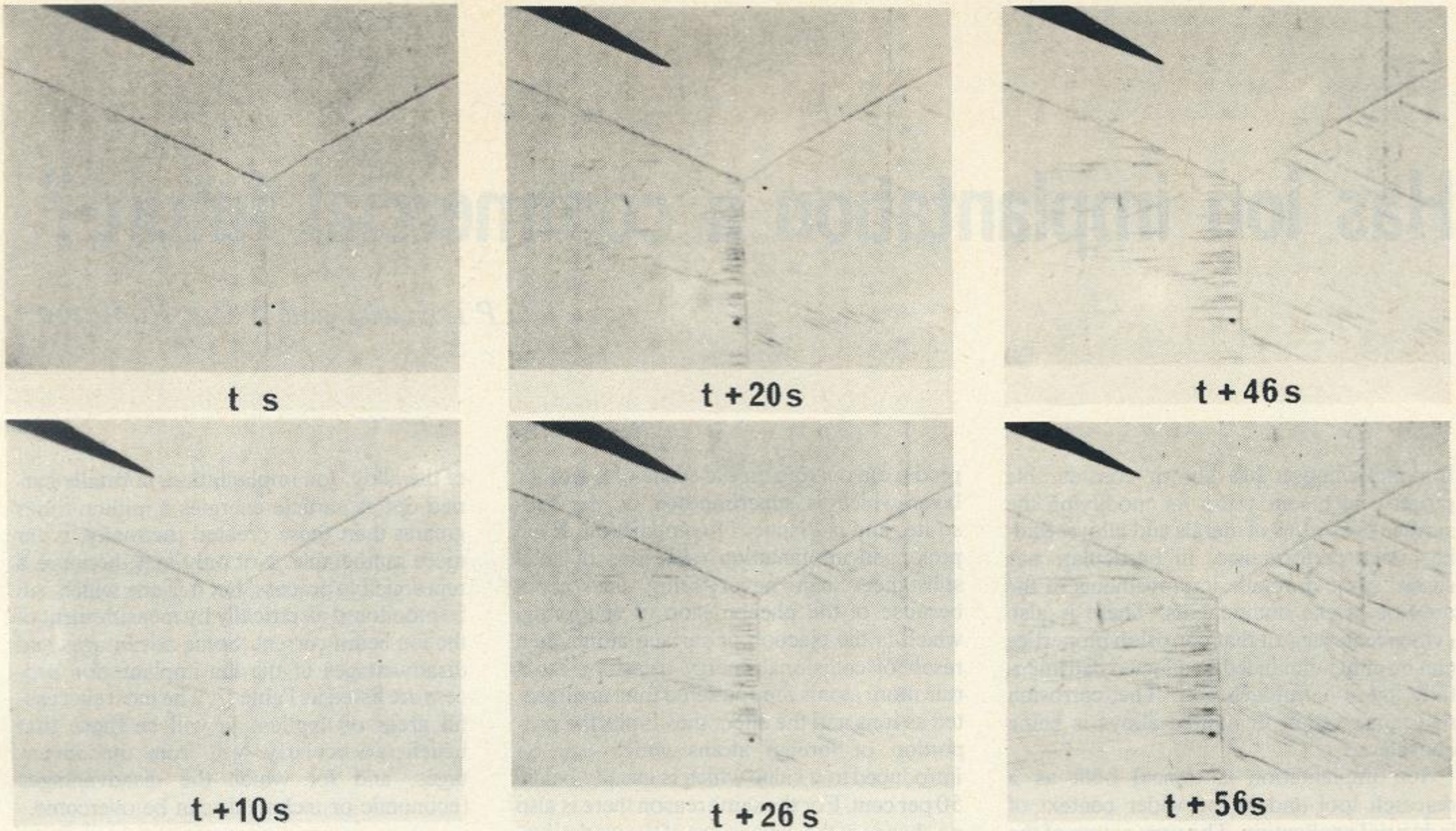


Fig 9. Hot stage microscopy: Growth of Widmanstätten α rods during isothermal ageing the metastable β' phase in a Cu-40% Zn alloy using a hot stage microscope (Ciné film sequence).

Scanning Electron Microscope

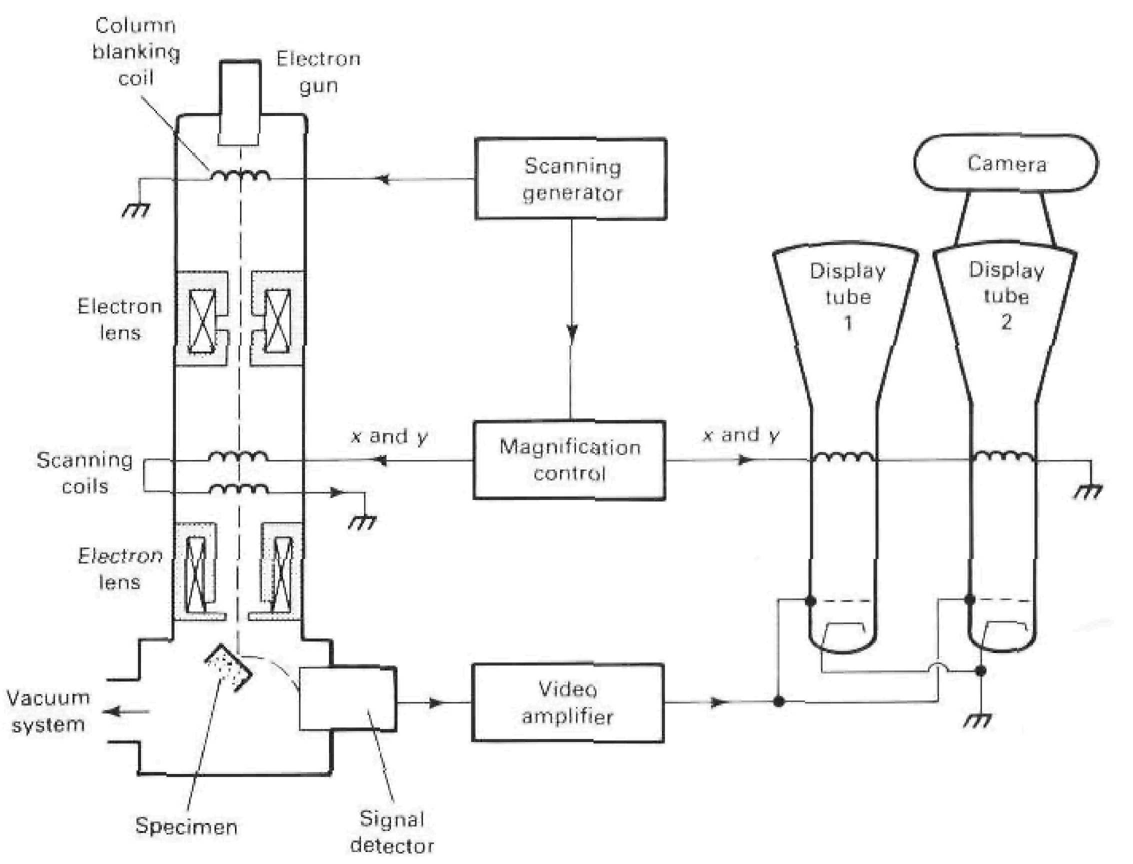


Fig. 1 Typical design (schematic) of the scanning electron microscope for secondary electron imaging. (Ref 2)