



## Standard Guide for Reflected-Light Photomicrography<sup>1</sup>

This standard is issued under the fixed designation E 883; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

*This standard has been approved for use by agencies of the Department of Defense.*

### 1. Scope

1.1 This guide outlines various suggested methods which may be followed in the photography of metals and materials with the reflected-light microscope. Methods are included for preparation of prints and transparencies in black-and-white and in color, using both direct rapid and wet processes.

1.2 Descriptive material is provided where necessary to clarify procedures. References are cited where detailed descriptions may be helpful. Guidelines are suggested to yield photomicrographs of typical subjects and, to the extent possible, of atypical subjects as well. Information is included concerning techniques for the enhanced display of specific material features.

1.3 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific precautionary statements are given in X1.7.

1.4 The sections appear in the following order:

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### 2. Referenced Documents

#### 2.1 ASTM Standards:

- E 3 Methods of Preparation of Metallographic Specimens<sup>2</sup>
- E 7 Terminology Relating to Metallography<sup>2</sup>

<sup>1</sup> This guide is under the jurisdiction of ASTM Committee E-4 on Metallography and is the direct responsibility of Subcommittee E04.01 on Sampling, Specimen Preparation, and Photography.

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 03.01.

E 175 Terminology of Microscopy<sup>3</sup>

E 768 Practice for Preparing and Evaluating Specimens for Automatic Inclusion Assessment of Steel<sup>2</sup>

### 3. Significance and Use

3.1 This guide is useful for determining appropriate conditions for photomicrography of metals (see Methods E 3) and materials with the reflected-light microscope and the subsequent processing of the photographic materials. It is limited to these applications.

### 4. Magnification

4.1 Photomicrographs shall be made at preferred magnifications, except in those special cases where details of the microstructure are best revealed by unique magnifications.

4.2 The preferred magnifications for general use in making photomicrographs, expressed in linear units, are: 25 $\times$ , 50 $\times$ , 75 $\times$ , 100 $\times$ , 200 $\times$ , 250 $\times$ , 400 $\times$ , 500 $\times$ , 750 $\times$ , 800 $\times$ , and 1000 $\times$ .

4.3 Magnifications are normally calibrated using a stage micrometer. When precision calibration is required, a certified stage micrometer shall be used.

### 5. Reproduction of Photomicrographs

5.1 Photomicrographs submitted for publication shall be enlarged or reduced to the nearest standard magnification, if necessary. A milli- or micrometre marker shall be superimposed on the photomicrograph to indicate magnification, in a contrasting tone. The actual linear magnification of the print shall be stated in the caption.

5.2 Photomicrograph captions should include basic background information (for example, material identification, etchant, mechanical or thermal treatment details) and should briefly describe what is illustrated so that the photomicrograph can stand independent of the text.

5.3 Arrows or other markings, in a contrasting tone, shall be used to designate specific features in a photomicrograph. Any marking used shall be referenced in the caption.

### 6. Optical Systems

6.1 The microscope objective forms an image of the specimens in a specific plane within the microscope called the

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 14.02.

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intermediary plane. Objectives are available in increasing order of correction as achromats, semiapochromats (fluorite), and apochromats (see Terminology E 7 and E 175). Plan objectives are recommended for photographic purposes due to their correction to provide flatness of field.

6.2 The eyepiece magnifies the intermediary image for observation or photomicrography. Eyepieces are sometimes also used to accomplish the full correction of the objective's spherical aberration and to improve the flatness of field. The pupil of the observer's eye must be brought to coincidence with the eyepoint of the eyepiece while viewing the microscopical image.

6.3 Intermediate lenses (relay or tube lenses) are often required to transfer the specimen image from the intermediary plane of the objective to that of the eyepiece. They may also add their own magnification factor, as in the case of zoom systems.

6.4 The objective, the eyepiece, and the compound microscope (including any intermediate lenses) are designed as a single optical unit. It is recommended to use only objectives and eyepieces which are intended for the microscope in use.

6.5 The resolution of the microscope depends primarily on the numerical aperture of the objective in use (1)<sup>4</sup>. High degrees of print or visual magnification (above approximately 1100 times the numerical aperture) do not add information content to the image and are called empty magnification. Magnification above this limit may be useful in certain cases, for example, as in measuring the distance between two points.

## 7. Illumination Sources

7.1 Metallographic photomicrography typically uses Köhler illumination. To obtain Köhler illumination, an image of the field diaphragm is focused in the specimen plane, and an image of the lamp filament or arc is focused in the plane of the aperture diaphragm. Specific steps to obtain Köhler illumination vary with the microscope used. The manufacturer's instructions should be followed closely.

7.2 For incandescent lamps, the applied voltage determines the unit brightness and the color temperature of the source. Evaporated tungsten blackens the envelope, resulting in diminished brightness and color temperature as the lamp ages. Tungsten-halogen lamps minimize envelope blackening, maintaining constant brightness and color temperature for most of their life. The high brightness and 3200 K color temperature of these lamps makes them especially suitable for color photomicrography.

7.3 With arc sources, brightness per unit area is substantially higher than that from any incandescent source. Their spectral output contains high energy spikes superimposed on a white-light continuum. Xenon arcs produce a spectral quality close to daylight (5600 K) with a strong spike at 462 nm. There are also strong emissions in the infrared, which should be removed (see 7.4). Carbon arcs have a continuous output in the visible portion of the spectrum, with a color temperature near 3800 K and a strong emission line at 400 nm. Mercury arcs,

with their strong UV and near-UV output, are particularly useful to obtain maximum resolution. Their color quality is deficient in red and cannot be balanced for color photomicrography. Zirconium arcs have strong spectral output lines in the near infrared, requiring filtration. Within the visible region, they are rated at a 3200 K color temperature.

7.4 Arc lamps require heat protection for filters and other optical components, and certainly for eye safety. Infrared removal may be obtained by: "hot" mirrors in the illumination beam to reflect IR while transmitting visible light; heat-absorbing filters to transmit visible light while absorbing IR, for example, solid glass filters or liquid-filled cells. Xenon arc lamps that do not produce ozone should be used.

7.5 A detailed discussion of illumination sources and the quality of illuminants is given by Loveland (2).

7.6 Some advice on using metallographic microscopes for visual observation has been compiled in Appendix X1.

## 8. Illumination of Specimens

8.1 The goal of an illumination system is to establish an optical train from light source to specimen plane which illuminates the field of view evenly and completely fills the aperture of the objective.

8.2 Photomicrographs are made with a compound microscope comprising at least an objective and an eyepiece with a vertical illuminator between them. Field and aperture diaphragms, with associated lamp condensing optics, are integral with the system.

8.2.1 The vertical illuminator is a thin-film-coated plane glass reflector set at 45° to the optical axis behind the objective. It reflects the illumination beam into the objective and transmits the image beam from the objective to the eyepiece. In some microscopes a prism is used to perform this function.

8.2.2 The field diaphragm is an adjustable aperture which restricts the illuminated area of the specimen to that which is to be photographed. It eliminates contrast-reducing stray light. The field diaphragm is also a useful target when focusing a low-contrast specimen.

8.2.3 The aperture diaphragm establishes the optimum balance between contrast, resolution, and depth of field. It should be set to illuminate about 70 % of the objective's aperture diameter. This can be observed by removing the eyepiece and inspecting the back of the objective, either directly or with a pinhole eyepiece. Some instruments have "Bertrand" lenses for this purpose. The aperture diaphragm should never be used as a light intensity control.

8.2.4 See Fig. 1 for an illustration of a typical vertical illumination system.

## 9. Focusing

9.1 Sharp focus is necessary to obtain good photomicrographs.

9.2 There are two systems for obtaining sharp focus: ground-glass focusing and aerial image focusing.

9.2.1 For ground-glass focusing, relatively glare-free surroundings and a magnifier up to about 3× are required. To focus, the focusing knob is oscillated between underfocus and overfocus in succeeding smaller increments until the image is sharp.

<sup>4</sup> The boldface numbers in parentheses refer to the list of references appended to this guide.

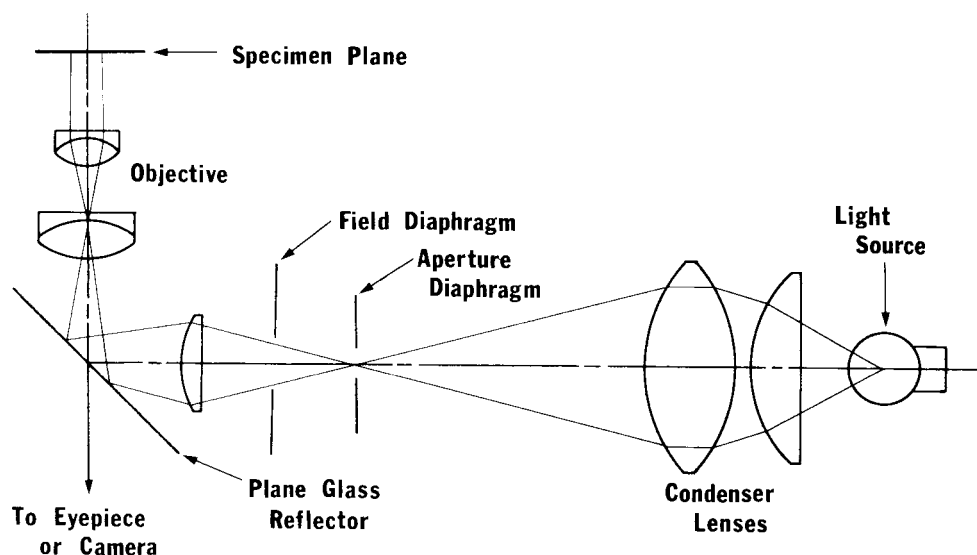


FIG. 1 Vertical Illuminating System for a Metallurgical Microscope

9.2.2 There are four possible variations for focusing an aerial image.

9.2.2.1 The simplest case is a transparent spot on a ground-glass containing a fiduciary mark in the film plane. To focus, the specimen image is made to coincide with the fiduciary mark, using a magnifying loupe of about  $3\times$  to  $5\times$ . When the focus is correct, the specimen image and the fiduciary mark will not move with respect to each other when the operator's head is moved.

9.2.2.2 A second case uses a reticle fixed within the optical system. Focusing is a two-step process: focus the eyepiece on the reticle; bring the image into focus against the reticle figure.

9.2.2.3 In the third case, a reticle is inserted into a focusing eyepiece. Depending on equipment used, this can be either a two or three-step process: focus the reticle within the eyepiece; next, set the proper interpupillary distance, if required (some equipment requires a specific interpupillary distance for eyepiece focus to coincide with camera focus); then focus the image coincident with the reticle.

9.2.2.4 The fourth case uses a single-lens reflex camera body, where the camera focusing screen is the plane of reference. An eyepiece magnifier for the camera is an important accessory for this case. An aerial image focusing screen is preferred.

9.3 The critical focus point is affected by both the principal illumination wavelength in use and the size of the aperture diaphragm. Final focusing should be checked with all filters, apertures, and other components set for the photomicrograph.

## 10. Filters for Photomicrography

10.1 The production of high-quality photomicrographs requires filtration of the light emitted from light sources. This section describes filter types and their uses.

10.2 Each filter selectively removes some wavelengths from the transmitted beam of light. Two types of filters, interference and absorption, can be used for this purpose.

10.2.1 Interference filters act as selective mirrors. By means of coatings on a glass substrate, they selectively transmit certain wavelengths while reflecting all others. These filters

may be used in high-energy light beams. The mirrored side of the filter should face the light source. (The hot mirrors in 7.4 are interference filters.)

10.2.2 Absorption filters are dyed substrates of glass, plastic, or gelatine. They absorb some wavelengths of light and transmit the balance. Through their absorption, they can become overheated and damaged if placed in high-energy light beams without protection. The usual protection is either an interference filter or a liquid-filled cell placed in the beam before the absorption filter. Wratten gelatine filters are used below as examples (3). Many similar glass and plastic filters are also available.

10.3 Certain general purpose filters have application in both color and black-and-white photomicrography.

10.3.1 Ultraviolet light can be removed with an interference filter, a glass or gel filter from the Wratten #2 series, or a liquid cell filled with a sodium nitrite solution (2%  $\text{NaNO}_2$  for a 1-cm path, proportionately stronger or weaker for other cell path lengths). Ultraviolet light must be removed from arc lamps for eye safety, and should be removed for color photomicrography, as explained in 10.5.

10.3.2 Gray neutral density filters reduce the intensity of a light beam equally across the visible spectrum. They are made in interference and absorption types in many different densities, for example, the Wratten #96 series. They are useful for eyepiece work with an arc source, and to modify the brightness of any tungsten source without changing its color temperature.

### 10.4 Filters for Black and White Photomicrography:

10.4.1 Generally, a monochromatic filter is used to optimize the resolution of the objective. With achromats, a green centered around 550 nm is used; for apochromats and semi-apochromats, a blue centered around 486 nm provides slightly better resolution, but with a penalty of more difficult visual focusing.

10.4.2 Cases arise where the visual contrast can be improved to emphasize a colored feature in the microstructure. The color will reproduce darker in the photomicrograph if a filter is used with a color complementary to that of the feature

(for example, cyan filter for reddish copper platings; blue for yellow carbonitride particles). When maximum detail in a colored phase must be shown, choose a filter with the same color as the phase.

10.5 *Filters for Color Photomicrography:*

10.5.1 Color photomicrography generally requires filtration to balance the light at the image plane to the color temperature specified by the film's manufacturer. Most transparency and negative color films are balanced for use with daylight at 5600 K. Some films are balanced for tungsten source lighting at either 3200 K or 3400 K.

10.5.2 Color films record ultraviolet light as blue. Since different metals reflect varying amounts of ultraviolet light, the simplest solution is to remove all ultraviolet light, as in 10.3.1, and rebalance by adding compensatory blue filters.

10.5.3 Table 1 lists filter recommendations appropriate for color photomicrography. These include strong conversion filters (the blue 80 series and the orange-yellow 85 series) and weaker light balancing filters (the yellow 81 series and the blue 82 series). Because of individual variations in equipment and other filtration (for example, IR and UV removal), some fine tuning is usually required with color correction filters. These filters are commonly used in color printing, and are available in sets containing various strengths of red, yellow, green, cyan, blue, and magenta.

10.5.4 Proper color balance for any color film can be determined using a first-surface mirror as the specimen (see 13.11). After the recommended filtration in Table 1 has been inserted, a series of test exposures is made with several color correction filters, until a neutral gray result is obtained. (Due to differences in manufacture, films with the same color temperature ratings may require slightly different groups of filters to achieve the correct color balance.)

**11. Illumination Techniques**

11.1 Metallographic specimens should be illuminated to reveal significant structural details with optimum contrast and resolution, and with sufficient brightness for accurate photographic recording.

11.2 With bright field illumination, polished areas of the specimen that are perpendicular to the light path reflect incident vertical illumination back into the objective lens and appear bright (see 8.2.1 and Fig. 1). Features such as inclusions and etched grain boundaries have edges which are inclined to the polished surface and reflect light away from the objective lens, making them appear dark.

11.3 Oblique illumination is similar to bright field, but is nonspecular, with the light impinging on the specimen at an oblique angle to the optical axis. It is obtained by decentering

the aperture diaphragm, or by tilting the specimen slightly (4). The technique is useful to enhance specimen surface relief and to determine if specific features are pits or projections, since shadows are cast by nonplanar features. Resolution decreases as the illumination is made more oblique. (It is important that the decentered diaphragm be completely imaged in the rear focal plane of the objective to keep the illumination reasonably uniform across the field.)

11.4 Dark field illumination is obtained by directing light to the specimen along the outside of the objective, blocking out the center. These rays are diverted onto the specimen plane obliquely by a reflector. No specular reflections enter the front lens of the objective. Only features that are tilted with respect to the surface (for example, grain boundaries, pits, and inclusions) will reflect light into the objective. These features will appear bright against a dark background. Image contrast is higher in dark field illumination than in other modes and will frequently reveal specimen detail which would be completely obscured with other kinds of illumination.

11.5 In polarized light illumination, light passes through a plane polarizing device, called the polarizer, located in the illumination system prior to the vertical illuminator; it is thus incident on the specimen as plane polarized light. After the polarized beam is reflected from the surface of a specimen, most or all of the light is absorbed by a second plane polarizing device located after the vertical illuminator, called an analyzer. The axes of the polarizer and the analyzer are oriented at 90° to each other. Plane-polarized light reacts differently when reflected from isotropic and anisotropic material lattices. For a cubic metal, the microscopic field appears dark because all of the light reflected from the specimen is absorbed by the analyzer. With an anisotropic material, the plane polarized beam reflected from the specimen surface either becomes elliptically polarized or the polarization plane is rotated. In both cases, the analyzer system now passes a portion of the reflected light through to the viewing system. Polarized light, with appropriate specimen preparation, reveals grain structure and twinning in metals with a hexagonal lattice structure, such as beryllium, tin, titanium, and zinc. Polarized light is also used with optically inactive cubic metals that are treated to produce an anisotropic surface film directly oriented with the substrate. Contrast in anodic films on aluminum or other metals can be improved with polarized light. It is also useful to identify optically active inclusions and phases, and in defining domains in ferromagnetic materials.

11.6 *Sensitive Tint*—Many metals and nonmetallic crystals are birefringent; plane polarized light is reflected from them as elliptically polarized light, which has a component not extinguished by the analyzer. If a sensitive tint filter is used, a magenta color is seen with cubic metals and all birefringent metals appear in vivid color contrasts. Aluminum or nodular cast iron demonstrate this effect particularly well, if a rotatable stage is used.

11.7 *Phase Contrast*—Phase contrast is an effective method in displaying the difference in level among grains, crystal edges, and other diffractive detail. This illumination technique produces enhanced contrast in the microscopical image by separating the undeviated image rays (for example, from a

**TABLE 1 Suggested Filtration for Color Photomicrography**

Film Color Balance Light Source	Daylight	3200 K	3400 K
	Wratten Filter Number		
Tungsten	80A + 82A	82A	82C
Tungsten-halogen	80A	None	82A
Zirconium arc	80A	None	82A
Carbon arc, 4.5 amp	80C	81C	81A
Carbon arc, 10 amp	82C + 82C	81EF	81C
Xenon arc	None	85B	85

planar bright field area) from those deviated by reflection or diffraction. There may be an edge effect (a light or dark line) that is not present with differential interference contrast illumination. However, with gross structure this can be an advantage. A raised structure results in a bright phase contrast, while a trough results in a dark phase contrast. To form the image, a circular slit at the rear condenser aperture is imaged in the rear focal plane of the microcroscope (the eyepoint) by Köhler illumination. A phase plate is placed in one of these planes, usually at the eyepoint with metallographic microscopes. An annular ring in the plate, of different thickness, covers the image of the light source whose rays constitute undeviated light. These rays then spread over the entire image area. Very slight deviations in their angle will cause the rays to fall outside the annulus. If the optical path (thickness  $\times$  refractive index) of the light through the annulus is one-quarter wavelength more or less than that through the rest of the area of the phase plate, the two segregated beams will meet and interfere or reinforce in the image plane. This is because diffraction itself causes an advancement or retardation of one-quarter wavelength and this becomes one-half wavelength at the image plane. There is normally a neutral density coating over the annulus to prevent the brightness of the direct beam from overwhelming the interference effect.

11.8 *Differential Interference Contrast*—(DIC or Nomarski illumination) This illumination technique shows edges of discontinuities on specimens as variations in brightness. Color contrast can be added as an additional indication of level variation. The method is termed differential because very minor discontinuities are emphasized, whereas slightly angled slopes are displayed almost as if they were perfectly normal to the optical axis; for example, a cylindrical phase looks flat with fairly sharp edges. A modified Wollaston prism located at the rear focal plane of the objective splits the illumination beam into two parallel beams, separated in phase by one-quarter wavelength. Any alteration of the optical path of the specimen, by either path length or refractive index, produces an interference pattern in the image beams. As the beams return through the DIC prism, they are reunited and the interference effect appears as a variation in brightness and color. Most microscopes allow translation of the DIC prism to produce different color displays as well. DIC has several advantages over phase contrast: since the full back aperture is illuminated, the full resolution of the objective is utilized; the interference plane is very shallow, keeping out-of-plane detail from interfering; there is an oblique appearance as an additional clue to level differences. Useful applications of DIC are: judging adequacy of specimen preparation for automated microscopy, as in Practice E 768; display of surface relief, including changes of a few nanometres at abrupt edges.

## 12. Photographic Materials

12.1 *Instant-Processing Films*—This class of materials yields photographic images within seconds after exposure. Both color and black-and-white varieties are available. All use variations of the diffusion-transfer process, with each frame developed individually after exposure.

12.2 The majority of the instant materials, including all black-and-white versions, are of peel-apart construction, where

the positive print is detached from the processing packet after development and the rest of the unit is discarded. A useful variation of this provides a transparent negative as well, for multiple print production by wet-process darkroom methods.

12.3 Black-and-white metallography using the high-speed versions is convenient for noncritical work, if only a single print is required and very fine structures or very long tonal ranges are not present (see 12.10 and 12.11.3). The slower, medium-speed emulsions reproduce longer tonal ranges and are satisfactory for all single-print metallographic use. Excellent photographic prints can be made from the negative instant films with great degrees of enlargement possible. (In order to optimize the exposure of the negative in a positive/negative film, the positive will be overexposed, and therefore not considered an acceptable print.)

12.4 Peel-apart color materials provide satisfactory prints, providing that care is taken in filtration and exposure. They are available in daylight balance only.

12.5 The pack materials, both black-and-white and color, require adaptors that are unlike those normally fitted to metallographic equipment. No processing control is possible (see 14.4). With some exceptions, pack prints should not be cut. The use of the more adaptable peel-apart materials may be the better choice for metallography.

12.6 *Wet-Process Materials: General and Black-and-White*—Conventional photographic materials provide an almost unlimited choice of conditions for recording an image. Many of the readily available products can be usefully employed in metallography. References 4, 5, and 6 are recommended reading to learn the complete photographic characteristics of the products, as well as the terminology used to describe them.

12.7 The essential construction of photographic materials consists of a carrier base with a light-sensitive layer of silver halides in gelatine, commonly called the emulsion. Intermediate negative and projectable emulsions are on transparent glass or flexible acetate or polyester film bases, while reflection print materials have white paper or paper/plastic composite bases.

12.8 The most common materials are negative-acting, that is, exposure to light and subsequent chemical processing displays an image on a film wherein the tonal values of the original scene (microscopical field) are reversed. This is subsequently printed by light exposure through the negative onto photographic paper, where a positive image (the negative of the negative film image) is reproduced again with similar chemical steps.

12.9 Some materials, either by controlled pre-exposure during manufacture or by specialized processing, yield a positive image directly and are called positive-acting. The principal uses are for projectables (slides) and negative duplication.

12.10 Negative film materials are of the most concern for metallography. The film chosen to record a microscopical image must be able to reproduce the tonal values in the image in their correct relationship to produce satisfactory prints. The film choice is in part dictated by the subject matter to be recorded—a simple steel image in bright-field may have only a brightness ratio of 1:3, dark field and polarized light images

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can exceed a 1:100 ratio. Reflection prints can at best reproduce a 1:30 brightness range. A film chosen for the first example should be capable of expanding the brightness range (contrast) by exposure and development control. A film for the latter example should compress the contrast of the original image. Typically, a film classified as high-contrast would be used in the first case, while a medium-to-low contrast material would be chosen for the other. (The extremely high-contrast lithographic films used for graphic arts purposes are excluded here. Their useful range of tonal reproduction is too restricted.)

12.10.1 The contrast potential of any film material is most easily expressed graphically as the film's characteristic curve published for all films in the manufacturers' literature. As an example of a film's potential, such a curve is schematically represented in Fig. 2. As the exposure increases on the horizontal axis, the corresponding photographic effect (blackening of the film) increases on the vertical axis. This effect becomes more prominent with increasing time of development, as indicated by the individual numbers on the curves. The useful part of a film's sensitivity range is the mid-portion, where the slope is relatively constant, indicating a proportional change in density with a proportional change in exposure (shown on Fig. 2 by range m-n). The slope of the curve rises more steeply as development proceeds and thus the contrast of the film image increases with increasing development.

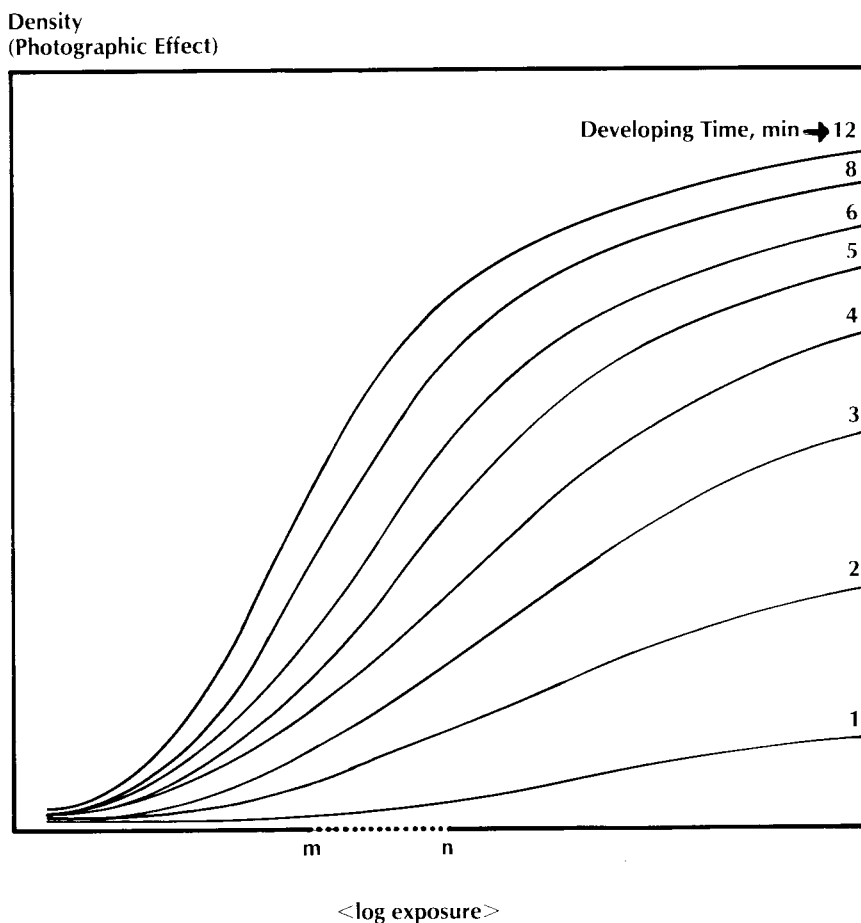
12.11 Several properties of negative emulsions that must be considered are: overall light sensitivity (film speed), spectral

sensitivity, resolving power, graininess, and contrast potential. All of these qualities cannot be optimized at once in any film, hence choices must be made to suit the needs of the photomicrograph.

12.11.1 Films are rated for general pictorial purposes by film speed numbers, for example, ISO speeds (formerly ASA) or DIN indices, with the higher rankings having increased light sensitivity. These rankings are not usually significant in metallography, since it is seldom important to make a rapid exposure. A faster film will probably be more convenient with a dim image, but if exposures over several seconds are required, the degree of departure from reciprocity will usually be the controlling consideration in film choice (see 13.7).

12.11.2 Some films record in the green and blue wavelengths much more efficiently than their overall film speeds would indicate and are thus good choices for black-and-white photomicrography. Orthochromatic films are especially useful; their red-blindness is inconsequential with green or blue filtration while permitting use of a red safelight in the dark-room.

12.11.3 The resolving power of an emulsion defines the closest spacing of points in an image that can be reproduced by the film as individual points. In general, any film which can resolve 20 or more lines per mm (10 line pairs per mm) with a low contrast image will be adequate for making same size (contact) prints. Films with higher resolutions are required for enlarged prints, with the enlarging factor controlling the film



**FIG. 2 Optical Density Versus Logarithm of Exposure for Photographic Materials**

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resolution needed (for example, a 4× enlargement would require an emulsion capable of resolving 4 × 20, or 80 lines/mm).

12.11.4 Graininess of a photographic emulsion is usually a function of the emulsion speed (ISO index), with faster films tending to be grainier. The presence of grain is more an annoyance factor than a technical defect. However, when enlargements are required, the choice of finer-grained emulsions will produce superior prints.

12.11.5 A film with a suitable contrast index should be chosen to balance the contrast class of the specimen photographed. Therefore, flat, low-contrast images require a high contrast index material, in the order of 1.4 to 1.6, to produce a satisfactory print. Most metallographic images have more inherent contrast and can be adequately recorded with a contrast index of 1.0. Extremely contrasty images (some dark field or polarized light images) are best recorded with materials of contrast index near 0.7 (which is in the useful range for general photography).

12.12 Negative materials must be printed to provide a usable image. Any reflection print using a glossy stock is restricted to reproducing a brightness range to a maximum of 1:30. Smooth nonglossy, matte, and textured paper stocks will produce decreasing brightness ranges, in the order listed. For a maximum definition of metallographic images, glossy stock is always suggested.

12.13 Like films, printing papers have different speed emulsions available. The only important distinction is between those intended for contact printing (relatively low sensitivity) and those for projection printing (higher sensitivity). A variety of contrast grades are available in both types, to balance the image contrast on the film (7). Some enlarging papers are offered in multiple-contrast versions, where the paper contrast can be changed with appropriate magenta and yellow filtration.

12.14 Printing materials have emulsions coated on either paper or plastic-coated (commonly called RC) bases. While the traditional fiber paper base is likely to have the longest useful life and reproduce a slightly higher brightness range, the plastic-coated types are much more convenient to handle, especially in a nonautomated darkroom.

12.15 *Wet-Process Materials: Color Considerations*—All of the considerations discussed in 12.6 through 12.11 also apply to color materials, but there are others as well. All color films are really tri-pack films, each with one emulsion layer responding to ultraviolet/blue, green, and red radiation, respectively. Because of this complexity, little variation in exposure or development is possible and the manufacturer's instructions must be followed closely. Especially important is the color temperature of the light (see 10.5.1) and the exposure time required.

12.16 Color positive (slide) films are convenient to use. The emulsion exposed in the microscope is reversal processed to yield a transparency directly. Emulsions are available which are balanced for daylight quality, 3400 K light and 3200 K light. All brands except Kodachrome<sup>™</sup> can be user-processed. Direct-reversal print-making processes are available, or an internegative film can be prepared from the slide for subsequent color printing.

12.17 Color negative film must be printed (to either a reflection print or a transparency) for viewing. Except for a few sheet and large roll films balanced for 3200 K, all are designed for daylight-quality illumination and all may be user-processed. Since printing is required, a gray scale (see 13.12) should be exposed for each different microscope set-up as a guide for the printer. Alternatively, an instant color print can be used for a sample.

### 13. Photographic Exposure

13.1 *Exposing Instant Materials*—Very little exposure manipulation is possible with instant-access photographic materials. Those furnishing prints alone should be exposed according to the maximum brightness in the image (see 13.3). The varieties that provide a permanent negative are best optimized for the negative by using the minimum (shadow) brightness in the image (see 13.4) and by usually overexposing (print is lighter) the positive by about a factor of two.

13.2 *Exposing Wet-Process Materials: General and Black-and-White*—After a film is chosen according to the criteria of 12.11.5, a suitable exposure must be made to record all of the tonal values present in the image on the straight-line portion of the characteristic curve, as shown by m-n in Fig. 2. The immediate problem is to determine the proper exposure, which is approached differently for negative and reversal (positive) materials.

13.3 The simplest case in photomicrography involves reversal materials. The exposure must be sufficiently long so that the lightest (white) tone in the image is reproduced as the brightest tone in the picture. (Shorter exposures will produce muddy whites, while much longer ones will crowd all of the light tones at the high end of the characteristic curve and give insufficient separation between them.) The correct exposure time, which is a function of the brightness at the film plane, must be experimentally determined to calibrate the system, as explained below. Factors influencing film-plane brightness are: type of lamp and voltage or power setting, filtration, aperture diaphragm opening, magnification at the film plane, and the numerical aperture of the objective. Once calibrated, the same conditions will reproduce the same film exposure without further testing.

13.3.1 A calibration exposure series holds all of the film-plane brightness factors from 13.3 constant, while varying the exposure time around an estimated time. (The estimated time can be quickly approximated from trial exposure with an instant-print material of approximately the same white-light film speed.) A suggested sequence of exposures is 1/8, 1/4, 1/2, 1, 2, 4, and 8 times the estimated exposure. With roll film, one frame per exposure level is exposed. With sheet film the entire series can be put on one sheet, using a premarked dark slide to mask successive portions of the frame. (In this case, because the exposures will be additive, the series should be 1/8 + 1/8 + 1/4 + 1/2 + 1 + 2 + 4 times the estimated exposure, with the dark slide advanced into the image area after each exposure, according to the markings.) The dried film is directly compared to the microscopical image to judge the most successful exposure. This time value is used in one of the following ways, depending on the sort of light-measuring equipment available at the microscope.

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13.3.1.1 With no exposure measuring device, the calibration becomes strictly empirical. The microscope, filtration, lamp (including voltage for filament lamp or power setting for arc lamp), aperture diaphragm setting, film plane magnification and numerical aperture of the objective, as well as the exposure time, must all be recorded as the calibration conditions with that film processed in that way. Obviously, changes in any of the conditions will require another calibration. This can often be avoided by making an instant-film test with the new conditions, and using the relationship:

$$\text{new time} = \text{old (calibrated) time} \\ \times (\text{new instant print time})/(\text{old instant print time}) \quad (1)$$

13.3.1.2 A simple brightness meter, either built into the microscope or used as an external accessory, makes exposure determination simpler. External probes are used either in the eyepiece or at the ground-glass screen of a sheet film camera back. Built-in meters sample the image beam light in a fixed location within the microscope. With the film type and film processing necessarily held constant, the only factors that need to be known are the correct exposure time for a certain brightness value with a specified filtration. (Photocells often have color sensitivities that differ across the spectrum from film color sensitivities. Thus, any filtration change will require a new calibration.) As the brightness value changes, a proportional change in the exposure time will be required. A simple two-point calibration graph will usually suffice for external probes, while microscope manufacturers provide calculation guides for built-in brightness devices. In cases where the microscope eyepiece magnification need not be the same as the film plane magnification (usually applies to microscopes with a bellows camera station), the indicated exposure time must be modified if the film plane magnification is changed, unless a film-plane brightness probe is used. The calculation is:

$$\text{new time} = \text{old (calibrated) time} \times \left( \frac{\text{new magnification}}{\text{old magnification}} \right)^2 \quad (2)$$

13.3.1.3 The next step in sophistication is a photometer with built-in provision for entering film speeds, typically marked as ASA or ISO indices. In this case, the meter calculating dial is adjusted after calibration to show the effective exposure index for the film that gives the experimentally determined exposure time at the brightness value used in the calibration. Thereafter, with the same film, processing and filtration, the effective exposure index is simply dialed in, the reading made, and the calculator dial shows the correct exposure time directly. (In the case of a modified conventional exposure meter at the eyepiece with a bellows-type camera back, the extra calculation in 13.3.1.2 is still necessary for film magnifications other than the calculated one.)

13.3.1.4 Some microscopes will automatically make the correct exposure directly upon releasing the exposure button, based upon an automatic integration of brightness and programmed film exposure index. A one-point calibration is all that is required to find the proper exposure index for each film/processing/filtration condition.

13.4 Negative materials must have sufficient exposure to record the darkest (but nonblack) part of the image on the film, so that it can be printed in its proper relationship to all of the

other tones in the image. All of the other (brighter) tones in the image will therefore record as darker images on the negative. Calibration for this follows the scheme of 13.3.1, but photographic printing of the test negatives is required to judge the optimum exposure to use for the film calibration.

13.5 All of Section 13 has thus far applied to relatively bright microscopical images—bright field, differential interference contrast, sensitive tint or phase contrast. With polarized light and dark field, the images are typically very dark overall, with important brighter spots. New calibrations are required for these, and they will usually remain empirical, since very few exposure-measuring devices have sufficient sensitivity to respond to the dim images produced. Furthermore, the correct exposure is that which properly records the bright areas (which may be only points) without regard to the relative brightness of the darker background.

13.6 Almost all exposure-measuring devices respond to substantially all of the microscopical image field, integrating the brightness from all points sampled. If a photomicrograph is required where the brightness varies substantially across the image field (for example, showing the metal-mount interface), the integrated brightness will be less than that from a uniformly bright field, and the exposure instrument will call for an overexposure to satisfy its calibration criteria. In this case, the exposure measurement should be taken from an adjacent field of uniform brightness, then the specimen is returned to the chosen field for the exposure.

13.7 All film emulsions will only obey the exposure/brightness relationship that was calibrated in 13.3 over a restricted range of exposure times. Generally, exposure times greater than two seconds will produce underexposure when the calibration conditions are followed. When the indicated exposure time exceeds about ten seconds, the extent of underexposure becomes quite serious. This departure from reciprocity (the reciprocal time/brightness relationship for correct exposure) must be included in calibrations when low-brightness images are used (8). In like manner, exceedingly brief exposure times will also result in a degree of underexposure, with departure from reciprocity starting at about 1/1000 of 1 s. (These very short exposure times are possible only with electronic flash illumination, a rare circumstance in metallography.)

13.8 Printing papers can be empirically exposed by either contact or projection, with stepped exposure testing similar to the concept developed in 13.3.1, but with smaller exposure intervals. A commercially available exposure guide, consisting of different gray densities in pie-shaped segments of a circle on a transparent base, is a more useful means to this end. A brightness meter probe, either on a contact printer stage or on an enlarging easel, is easily calibrated to yield consistent exposures (for example, negatives made at any time can be printed to a common matrix brightness by using the meter probe on the negative image of the matrix). Because of the variety of such instruments available, no directions are given here. The specific manufacturer's instructions should be followed.

13.9 Different contrast grades will usually require varying exposure times, but these will be in a consistent ratio within



any one brand of paper, thus negating the need for individual calibration by contrast grade.

13.10 *Exposing Wet-Process Materials: Color Considerations*—As mentioned in 10.5.1, color emulsions are balanced in manufacture to properly respond to light of a specified color quality, either daylight (at 5600 K) or tungsten source (3200 or 3400 K). Inevitably, filtration is required to produce the correct color temperature in metallography. Other components in a microscope system (such as heat absorbers, lenses, prisms, and mirrors) will alter the illumination quality of the beam, even if the lamp has the same color balance as the film emulsion.

13.10.1 The blue-sensing layer of a color emulsion depends on a consistent amount of ultraviolet to respond correctly. Metals vary widely in their UV reflectance, making this a constant source of variation. The most efficient solution is to filter out all of the ultraviolet and then experimentally add blue to the light to rebalance.

13.11 It is important to have a standard to balance the effective illumination of the system to photographic neutrality. Aluminum is photographically neutral throughout the visible and UV wavelengths. A first-surface aluminum mirror can be used as a repeatable standard. (A protective chromium overcoating destroys the neutrality, but a thin silicon monoxide protective layer is acceptable.) A seven-level step wedge (see 13.3.1) is made with the aluminum standard as target, using the predicted filtration. The predicted correct exposure should be the fourth of the seven steps to provide a series of gray levels. Any deviation from neutral gray observed can be corrected with color correction filters (see 10.5.3).

13.12 When using color negative film, a special problem is encountered. No clue exists in the negative to assist the technician in establishing color filtration and exposure for printing. An internal calibration can be made, whereby one frame of each color negative series is exposed to the neutral aluminum reflector to provide a repeatable test target that receives the same processing as the rest of the exposures. With trial-and-error printing, test prints can be made from the test negative with trial filter packs to find neutrality. This same pack should be used for the entire batch of negatives. For those laboratories with a color printing analyzer, the test frame can be used to set filtration directly. The exposure for the gray frame should yield a light-to-medium gray print when properly printed, which can be achieved with a microscope exposure of one fourth the exposure normally used for a bright-field exposure. (Some microscopes will have different color balance requirements for each objective lens used. This can only be determined by trial. If this is the case, a gray frame for each objective used in the photographic series is required.)

## 14. Photographic Processing

14.1 *Processing Instant Materials*—All instant materials, both peel-apart and monopack, are processed by pulling the picture unit through an accurately-spaced roller pair. A chemical pod at the leading edge is ruptured and the contents spread throughout the unit by the rolls, starting the development action. The reaction effectively goes to completion according to the time-temperature relationship supplied with the film.

14.1.1 The picture unit must be pulled in a straight line

through the rollers, at a constant speed, to secure uniform processing. A pull-time of  $\frac{1}{4}$  to  $\frac{1}{2}$  second is suggested.

14.1.2 The roller pair should be kept clean, since even fine debris on the rolls will cause uneven reagent spreading and picture nonuniformity.

14.2 Black-and-white peel-apart materials are best processed for at least the recommended time for the type involved. Extended processing up to three minutes is permissible. Beyond this, problems may be encountered as the units are peeled.

14.3 Improved contrast and color saturation can be achieved with color peel-apart materials by processing to a two-minute standard, rather than the recommended one minute. The color balance will shift toward cyan, which can be corrected by adding some red to the filter pack.

14.4 Monopack instant materials permit no control over processing time, since the self-developing reaction proceeds to completion without attention from the user.

14.5 *Processing Wet-Process Materials: General and Black-and-White*—Roll films are processed on small reels within light-tight developing tanks, where chemicals are sequentially poured in and out. Adapters permit sheet materials to use similar tanks, but usually sheet film is loaded into flat hanger frames and processed by immersion in a series of tanks, each containing one of the chemicals required. (A few sheets can also be processed in trays, like prints.) Whatever the method, consistency in processing (on which the exposure calibrations rely) can only be achieved by using repeatable techniques with respect to chemical step timing, constant temperature for the whole processing sequence, regular efficient agitation, and known chemical activity of the processing solutions.

14.5.1 The time for each chemical step is determined by the film and the chemicals used. Photographic manufacturers publish explicit instructions (8, 9) for their use, and these should be followed.

14.5.2 The recommended temperature for processing is 20°C (68°F). If the darkroom ambient temperature is normally at another temperature, this can also be used. The time for the developing step will require modification with change in temperature, but this time is readily determined from published time-temperature compensation data that are plotted on semi-logarithmic paper similar to Fig. 3 (8, 9).

14.5.3 Agitation during processing is required to remove depleted chemical layers from the film surface and replace with fresh solution. Agitation must be uniform to ensure good mixing in all parts of the tank, yet sufficiently random to prevent streaking. The resulting contrast is also influenced in part by degree of agitation. Reference 8 lists satisfactory agitation procedures for all of the methods of development mentioned in 14.5.

14.5.4 The working strength of processing solutions can be maintained by replenishment, with total chemical replacement well before exhaustion of activity. Users of roll-film tanks may find it more convenient to use total loss developers, whereby fresh chemicals are used for each batch.

14.6 For reasons of water economy and time, the use of a fixer clearing agent is recommended after film processing.

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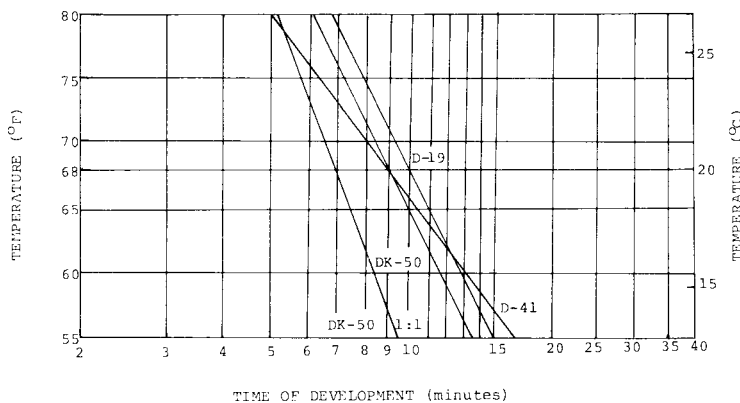


FIG. 3 Time Corrections for Variations in Development Temperature

These baths reduce the amount of washing required by at least a factor of five.

14.7 High-contrast process (not lithographic) films are very useful for low-contrast metallographic specimens, but their published developers apply to graphic arts uses. An appropriate developer for contrast index ranges useful in metallography is Kodak's D-76™ formulation, modified by addition of 0.2 % benzotriazole in the proportion of 5 mL per litre.

14.8 Many automatic film processors are available. Their use is a great help in maintaining uniform processing for laboratories with sufficient work volume to justify them.

14.9 Safelights should be used carefully when processing film. The best usage is to direct the beam against a wall or ceiling, rather than directly at the film. The bulb wattage specified for the filter and film in use should not be exceeded. Orthochromatic films can tolerate constant use of a dark red light, while panchromatic materials are best processed in total darkness. A very dark green safelight can be used for a few seconds only with panchromatic film, as when changing from tank to tank. The minimum safelight-to-film spacing in any case should be 1.2 m (4 ft).

14.10 Reflection prints are processed through the same steps as films: developer, stop bath, fixer, and wash. Consistency in time, temperature, and agitation is important to repeatable results, as is the use of proper-strength chemical solutions. The usual manual procedure involves processing one or a few prints at one time through shallow trays of solutions, using a moderately-bright orangish safelight. While packaged chemical solutions recommend 20–21°C (68–70°F) for all processing steps, including wash, any consistent room temperature between 18–24°C (64–75°F) can be used with little change in procedure or results. The important factor is temperature consistency within a predictable narrow range, rather than absolute temperature accuracy. The steps discussed below are confined to tray use. The use of efficient, comparatively inexpensive automatic processors for prints has become commonplace in laboratories, making printing a much faster operation. Important considerations about chemical mixing, replenishment, and machine maintenance, as described in the operating manuals, must be followed to achieve consistency. If plastic/paper composite materials are used, their wet-time must be restricted, according to the instructions packaged with the stock. Conventional fiber-based printing papers have much greater tolerance to prolonged immersion.

14.10.1 Any standard cold-tone (nonportrait-type) developer may be used. Dependent on the paper used, development should be for either 60 or 90 s, with intermittent agitation. An incorrect exposure can be compensated by variation of the development time, but this should be kept to within 15 s of the aim time. Multiple prints can be processed concurrently, with constant interleaving of the prints in the tray to expose the emulsion to fresh developer at frequent intervals. Care should be taken, especially with the stiffer plastic/paper composites, to avoid scratching the emulsion of one print with the corner of another. Most developers have a capacity of about 120 sheets per litre, 10 × 12.7 cm (4 × 5 in.), but only if used within the same day in the tray. Aerial oxidation degrades the developer activity. Fresh solution should be used every day.

14.10.2 Immediately after development, the print should be drained and put into an acetic acid stop bath for about 30 s, with agitation, to stop development action. The alkalinity of the developer is neutralized by the stop bath, thus protecting the mildly acidic pH of the fixing bath, which is next. A suitable stop bath can be made by adding 48 mL of 28 % acetic acid to one litre of water. (Dilute 3 parts of concentrated acetic acid into 8 parts of water to make the 28 % stock solution.) A safe practice is to change the stop bath along with the developer to prevent inadvertent exhaustion of activity. Commercial stop bath concentrates are available with a dye indicator that changes color when the bath is exhausted.

14.10.3 A fixing bath removes unused silver from the emulsion as silver thiosulfate. Either conventional hypo baths of sodium thiosulfate or quick-acting ammonium thiosulfate can be used with fiber-based papers, while the composites require the ammonium version. Fixing baths used for film should not be used for paper, since they contain iodides which may stain the paper. Prolonged immersion in fixers should be avoided because of both a bleaching effect and the tendency to deposit compounds difficult to wash out later. As in other solutions, frequent agitation is necessary. Fixers mixed for prints generally have a capacity of 100 prints per litre, 10 × 12.7 cm (4 × 5 in.). More efficient fixing can be achieved with two sequential fixing baths, with the print immersed for one-half the total time in each. After 100 prints are processed, discard the first bath, move the second bath into its place, and make a new second bath. The maximum useful life of fixer in a tray is 1 week.

14.10.4 Washing is essential to remove residual chemicals

for a print that is stain-free and durable. The wash temperature should be close to the processing temperature.

14.10.4.1 Conventional paper prints need about a 1 h wash, with a water flow sufficient to change the entire bath every 5 min. Frequent agitation is required to prevent prints from sticking to each other and washing unevenly. Wash time can be reduced to 10 min if a fixer clearing agent is used. Typically, the print is rinsed in water for 1 min, bathed in the clearing agent with agitation for 2 to 3 min, then transferred to the wash for 10 min. Capacities of these solutions vary somewhat with the brand used.

14.10.4.2 Composite plastic/paper prints are washed in running water for only 3 to 5 min. No advantage is gained with these materials by using a fixer clearing bath.

14.10.5 Conventional paper prints should be squeegeed after washing, then dried in heated dryers, or in blotter arrangements, to prevent curl. Glossy stock can be rolled onto ferrotype tins or dried on a heated ferrotyping drum to produce the highest gloss and detail retention. Plastic/paper prints are squeegeed, then dried without touching other materials, such as by hanging from one corner. Plastic/paper prints may alternatively be immersed in a wetting agent solution and immediately hung by one corner over a sink. This allows the water to rapidly flow off of the print and prevents spotting. Heated dryers especially made for these materials can be used. Unlike fiber-based prints, glossy surfaces of plastic/paper prints should never be ferrotyped. This would spoil both the ferrotype medium and the print.

14.11 Reasonable care is necessary when storing, mixing, and using photographic chemicals. Contact dermatitis can develop from skin exposure to many of the solutions, as well as the more obvious dangers of eye contact, breathing, and ingestion. Photographic emulsions are adversely affected by contamination with chemical vapors, solutions, or powdery residues in the air.

14.11.1 All photographic chemicals are packaged with warnings about their proper handling, mixing, and possible health hazards, usually including steps to take if accidents happen. These instructions should be read frequently and carefully followed. They should be posted along with a Poison Control Center telephone number, wherever chemicals are mixed or used. Safety equipment, such as eyewash provisions, should be nearby. Protective gear for mixing should include gloves, aprons, eye protection, and, if powders are involved in quantity, chemical dust masks. Processing steps should minimize contact with the solutions. Rubber gloves are suggested for processing in tubes or tanks, while tray processing should be done with tongs rather than bare hands.

14.11.2 Chemical mixing, especially if it involves solids, should be done in a location different than where photographic materials are exposed or processed.

14.11.3 The darkroom should be damp-cleaned frequently and chemicals should be stored away from photosensitive materials.

14.12 *Processing Wet-Process Materials: Color Considerations*—Because of the complex nature of all color film and print emulsions, even more consistency is required during processing than is true with black-and-white. Instruc-

tions with the chemical processing kit to be used should be thoroughly understood and the procedural steps should be planned in advance. Especially important is the temperature. While some processing kits allow use over a short range of temperatures, once the temperature for use is selected, it must be firmly controlled. Most development steps, for example, require  $\pm 1/4^{\circ}\text{C}$  ( $\pm 1/2^{\circ}\text{F}$ ) stability for development steps and little more range for the other solutions and washes. Step timing (including drain times) and agitation techniques must be accurate and repeatable.

14.12.1 Commercial laboratories can usually process color materials more inexpensively than an in-house laboratory. This is especially true with films, where a standardized processing without operator judgment is all that is required. Prints from slides are no more difficult, except that the magnifications offered by commercial houses may not match those recommended (see 4.2). Prints from color negatives, in addition to the magnification consideration, are seldom successfully printed unless a close relationship is established with the printing source. The use of standardizing gray targets (see 13.12) is especially valuable here.

14.12.2 Temperature stability for the processing solutions and the processing tank(s) is most easily maintained with a water bath. For water economy, a thermostated water bath can be used rather than a constant stream of tempering water. Sufficient fresh water must be available for adequate washing, however, this must be at or near the temperature of the processing solutions.

14.12.3 Most roll films (and small batches of sheet film) are processed in a small tank, which is inverted for agitation and where solutions are sequentially poured in and out (see 14.5). This arrangement usually utilizes chemical kits with the capacity for a specific quantity of film, and usually with extended processing times after the initial use. Control of chemical strength is automatic in this case.

14.12.4 Larger film volumes use individual tanks for each solution (and another for washing), with maintenance of chemical activity through replenishment. Calculation and record-keeping is required here. Almost daily usage is required to use up the chemicals' capacity before aerial oxidation depletes their strength. Agitation in a tank line is especially critical and is usually achieved with gaseous-burst arrangements (nitrogen in the oxygen sensitive chemical tanks, air in the others).

14.12.5 Large batches of color prints can be simultaneously processed in tank lines similar to 14.12.4, with the prints contained within plastic-mesh baskets. The problems with maintaining chemical activity and with agitation are the same as those for film.

14.12.6 Smaller print volumes are processed individually or a few at one time in various types of drum and tube processors. Small volumes of single-use chemicals are required for each run, with no replenishment considerations. While the rotating drum devices have their own built-in agitation, the addition of a mechanical rotator for a processing tube will provide more even agitation over that achieved by manually rolling the tube.

14.12.7 Sheet films meant for printing without enlargement are best placed emulsion-to-emulsion with the printing paper in



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a printing frame, and then illuminated by an enlarger, since all color emulsions are projection-speed materials.

14.12.8 All positive and negative color print emulsions are coated on plastic or plastic/paper composite bases. As with the similar black-and-white materials, air drying or drying in specialized print dryers is required.

## **15. Keywords**

15.1 metallography; microscopy; photography; photomicrography; photomicroscopy

## **APPENDIXES**

### **(Nonmandatory Information)**

#### **X1. SUGGESTIONS FOR VISUAL USE OF METALLOGRAPHIC MICROSCOPES**

X1.1 All optical microscopes are so designed and optically corrected that the user should have no discomfort if proper illumination levels are used and the eyes can be relaxed for infinity viewing (with eyeglasses, when necessary). The manufacturer's instructions should be consulted for each microscope to ensure proper use of that particular instrument.

X1.2 Illumination levels should be reduced to the lowest intensity that permits comfortable observation. Small detail is better resolved when glare is absent and only 3 to 5 footcandles are required at the eyepoint. It is convenient to have a low-voltage incandescent source for sustained visual work and an arc source for photomicrography.

X1.3 While arc lamps are valuable for many applications because of their high brightness, their illumination must be reduced at the visual eyepiece to prevent eye damage. Filtration is the preferred method of reducing brightness. Colored or neutral density absorbance filters, or both, are usually adequate for incandescent sources, while the high-heat, high-brightness arc sources will require interference-type filters. (Absorption filters can be used with arc sources, if protected by a water cell on the lamp side.) Several filter density strengths are suggested to accommodate the ranges of specimen brightness and observer comfort level.

X1.4 Illumination levels should not be reduced by closing the aperture diaphragm beyond its optimum point, for example, where about 70 % of the back lens of the objective is filled with light. Smaller openings coarsen detail and degrade the resolving power of the system by introducing diffraction effects from the diaphragm blade edges.

X1.5 Eye fatigue can develop rapidly on the microscope, if one eye is kept closed or sees an unsharp image. The criterion to satisfy is that both eyes should be totally relaxed (focused at infinity) and open.

X1.5.1 With monocular microscopes, an opaque black eye-shield large enough to completely block the unused eye's field of vision should be used, to permit comfortable "two-eyed viewing."

X1.5.2 With binocular microscopes, two types of construction exist, both requiring initial standardization of the user's eyes: (a) one fixed and one variable-length eyetube; and (b) both eyetubes of variable length. In either case, the use of a fixed reticle within the microscope system (see 9.2.2.2) is the preferred set-up target, with the specimen deliberately out of focus. Alternatively, a specimen with fine high-contrast detail should be used. In case (a), the target is brought into critical focus using the eye at the fixed eyetube and the microscope's focusing control, then the variable-length eyetube is adjusted to give the same focus for the other eye. With case (b), the target is individually made sharp for each eye by changing the length of the respective eyetube.

X1.6 Eyeglasses can be removed when using the microscope, if the eye defect is limited to near- or far-sightedness, where the microscope focusing will accommodate the deficiency. If cylindrical correction (astigmatism) is involved, however, the eyeglass corrections are necessary and high-eyepoint eyepieces are recommended. (Eyeglass users are reminded that the distance vision part of bifocal or trifocal lenses is the correct choice for microscopy.)

X1.6.1 An alternative to using eyeglasses at the microscope is a lens cap containing a lens with the user's optical prescription. The caps are available from most microscope manufacturers, and local optical shops can supply the lenses. (This can become awkward when several instruments must be used, each with a different outer diameter for its eyepieces.)

X1.7 Summarizing, three cautions for eye comfort and safety should be observed:

X1.7.1 Do not use illumination brighter than necessary.

X1.7.2 Make adjustments to keep both eyes at infinity with equally sharp images (or mask off the unused eye with monocular microscopes).

X1.7.3 Do not remove eyeglasses unless other means are provided for correcting the eye's vision.

X1.7.4 When using arc illumination sources, filters to remove ultraviolet and infrared radiation should be used.

## **X2. GUIDE FOR METALLOGRAPHIC PHOTOMACROGRAPHY**

### **X2.1 Introduction and General Principles**

X2.1.1 The photomacrographic magnification range is  $0.1\times$  to  $50\times$ . (Photomicrographs are generally made at  $25\times$  and higher.)

X2.1.2 Photomacrographs are made with only a conventional camera body and lenses, usually with supplementary lenses or means to extend the lens-to-film spacing beyond that provided by the normal focusing mechanism.

X2.1.3 A magnification verifier (ruler or other size gauge) should be included in the photographic field whenever possible.

X2.1.4 Photomacrographs may require lengthy set-up times and long exposures. Movement-free camera and subject supports are important.

X2.1.5 General photographic principles regarding film and print materials and their processing have been treated in the main E 883 text. These considerations are equally valid for photomacrographic use.

X2.1.6 There are no preferred print magnifications for publication of photomacrographs. For reader convenience, however, magnifications should be adjusted to even-numbered tenths in the final print (for example,  $1.2\times$  and  $3.0\times$ , rather than  $1.1\times$  and  $2.95\times$ ).

X2.1.7 As much of the required final print magnification as possible should be obtained on the negative.

X2.1.8 For three-dimensional subjects, camera viewpoints should be selected which require the least depth of field.

X2.1.9 Use as wide an aperture as possible, consistent with required field depth.

### **X2.2 Magnification Techniques**

X2.2.1 Nonremovable lens cameras can achieve close focusing and larger film-plane images with supplementary lenses, available in strengths from  $+1$  to  $+10$  diopters. Approximate image magnification can be calculated through the relation  $M = FD/1000$ , where  $M$  is the magnification,  $F$  is the prime lens focal length in millimetres, and  $D$  is the diopter number. Image size is also dependent upon lens-to-film distance with focusing cameras. Careful measurement of lens-to-subject distance is important, requiring use of tables furnished with supplementary lens sets. Frequently used magnifications may justify construction of focal frames.<sup>5</sup> Supplementary lens arrangements are easy to use, require no exposure compensations, and can be hand-held if there is sufficient light and focal frames are used. Disadvantages are film-plane magnifications under  $1\times$  and the necessity of using small apertures (and slower shutter speeds) to compensate for image degradation from the action of the supplementary lens.

X2.2.2 Cameras with removable lenses are preferred for photomacrography. Increasing the lens-to-film distance will increase the magnification on the film. For most roll-film cameras, rigid extension tube sets are available, permitting a

convenient series of fixed magnifications. Most cameras with removable optics can use bellows connections to the camera body, yielding a continuous range of magnifications.

X2.2.3 The choice of lenses in photomacrography is manifold, dependent on the magnification required. The shorter the focal length, the higher the magnification achieved on the film with the same bellows extension. Conversely, longer focal lengths generally allow more working distance between the lens and the subject. They also provide more natural perspective relationships, which may be important for three-dimensional subjects. A practical rule is that the lens focal length should be at least 1.5 times the length of the specimen at magnifications above  $1\times$ .

X2.2.4 Conventional lenses can be mounted in a reversed position on extension tubes or bellows for increased sharpness at low to intermediate magnifications. Enlarger lenses can also be used for this purpose when reverse mounted. For intermediate and high magnifications, reversed 16 mm cine lenses and conventional microscope objectives can be used. Special-purpose macro lenses are available in focal lengths from 15 mm to 150 mm which can be used alone or with extension tubes or bellows to cover the entire photomacrographic range.

X2.2.5 Increasing the lens-to-film distance beyond that provided by the normal focusing mechanism requires an increase in exposure. Single-lens reflex cameras with exposure meters in the camera automatically provide the necessary correction. Fig. X2.1 illustrates the extent of correction required for other cameras.

X2.2.6 Camera-equipped stereomicroscopes are ideal for photomacrography over their working ranges (typically  $5\times$  and higher). Those with vertical illuminator attachments greatly simplify the set up of axial illumination (see X2.3.5).

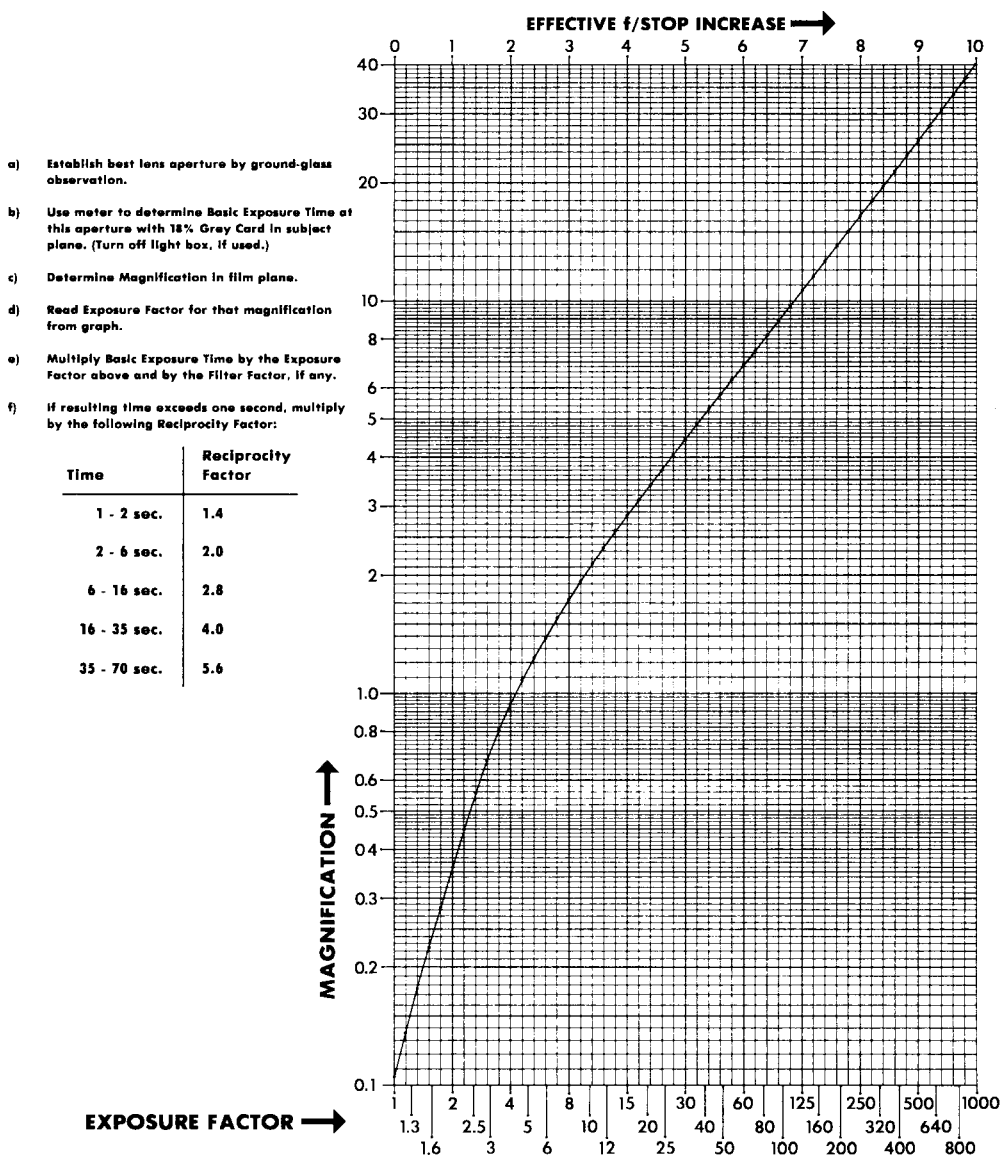
### **X2.3 Illumination Techniques**

X2.3.1 Technical documentation requires the description of a problem or condition in a clear, concise, and unambiguous manner. In photomacrography, these criteria are largely filled through the choice and arrangement of appropriate lighting. All details of a specimen should be shown with good definition and contrast. One main light source should be used, falling from the top half as the photo will be viewed. Supplementary illumination to fill shadows, define textures, and accent local areas should all be subordinate and should cause no competing shadows. An important exception is a flat, nontextured subject, such as an etched slice, where even, nondirectional lighting is often required.

X2.3.2 The size of the light source is dictated by the smallest detail to be resolved in the specimen. The use of an overlarge source causes light to wrap around small features, effectively obscuring them. Point-lights from specular or other highly reflective details will merge with each other if a large source is used, resulting in glare spots where detail should be resolved.

X2.3.3 Metallographic photography often involves documentation at magnifications of  $3\times$  and less. Conventional studio flood and spot luminaires are usually sufficient. Fill-in

<sup>5</sup> Close-up Photography and Photomacrography, Kodak Publication N-12, Rochester, NY, 1977.



**FIG. X2.1 Photomacrography Exposure Computer**

illumination is often simplified by using white cards with paper or matte foil surfaces or mirrors. A focusing fresnel spotlight is useful for providing skimming illumination to show surface textures. Many specimens can be back-lighted (for example, with an illuminator for X rays) to define their edges or to drop out competing shadows. Black nonreflecting fabrics are useful as shadow-free backgrounds. Three-dimensional specular or machined specimens are often best shown with tent lighting (a translucent paper or fabric enclosure, externally lighted to cast nondirectional transmitted light on all parts of the subject). An alternative to tent lighting is white cards reflecting light to the subject. Flat nonspecular specimens are easily lit by a light source surrounding the lens, either a ring light or a reflector lighted by floodlamps. Coarse-grained etch slices and flat artwork are best photographed with floodlights at 45° to the surface. (All of the above sources are also available in electronic flash versions, but continuous light sources are necessary for set-up prior to taking the photographs.)

**X2.3.4 Lighting of small specimen areas for photomacro-**

graphs from about 3× and higher is complicated by the small lens-to-subject working distances. Specimens that can use nondirectional illumination are best lighted with ring lights or small tents. Directional illumination is conveniently supplied with fiber optic lights, small focusing spotlights, or microscope illuminators. Samples with fine textural details or small pointlights require as small a light source as possible. Bare microscope illuminator lamps with the smallest dimension of the filament facing the specimen are often ideal, as are bare-bulb electronic flash lamps and AG flashbulbs (pointed end toward the subject).

**X2.3.5 Axial (bright field) illumination** is necessary when photographing flat, specular specimens. The set-up is critical: the specimen must be normal to the lens axis; a thin glass plate must be located on the optic axis between the specimen and the lens, inclined at 45° to the specimen surface; a directional light (microscope illuminator or small spotlight) must be arranged exactly parallel to the specimen surface, illuminating the side of the glass plate facing the specimen; the specimen must be



shaded from direct light from the lamp. In use, light is reflected from the lamp to the specimen by the glass, then returns through the glass to the lens. Defects in a polished specimen

surface, too large for conventional photomicrography, can be illustrated, as well as fine detail in all similar subjects.

### X3. VIDEO IMAGING PRINTING

X3.1 Recently, reproduction of microscopical and macroscopical images via video technology, both black-and-white and color, has become a practical and cost effective method of photodocumentation. While photographic techniques are capable of producing images with greater resolution, digital video techniques can provide images with acceptable resolution for noncritical work.

X3.2 A system for producing high quality video prints requires both a high resolution video camera and a high resolution video printer. The coupling of a high resolution component with a lower resolution component will produce prints with the lower resolution. The range of the camera output signal should coincide with the print range of the printer in order not to truncate the whitest or blackest portion of the signal. Interposing equipment (monitors, image analysis/processing systems) between the camera and printer can increase noise in the digital signal resulting in decreased resolution and/or loss of detail in the print.

X3.3 Video printers have controls for adjusting the contrast, brightness, and color tint of prints. Images should be optimized for these parameters by adjusting the illumination before printing, as manipulation of the image by use of the printer controls can reduce the range of grays (or colors) available to the printer.

X3.4 Magnifications of video prints should be calibrated by

use of a print of two measuring devices, one placed on each axis of the print. This calibration print should be produced at the same magnification as the prints of interest. Care must be taken to ensure that the aspect ratio of the object is accurately reproduced in the print, as the x and y dimensions of the final print can be adjusted independently through controls provided on some printers. When printing systems allow, it is recommended that magnification markers be placed directly on video prints. Magnifications used should follow the guidelines provided in 4.2 and X2.1.6 of this standard.

X3.5 Most high quality video printers will allow some adjustment of the final print dimensions. Major adjustments to magnification should be made by use of camera lenses or microscope objectives. Increasing of magnification by use of video printer controls is not recommended due to degradation of resolution.

X3.6 Significant manipulation of final prints by use of image processing systems, video camera settings, or video printer controls is not recommended, but in some cases may be acceptable. However, under no circumstances shall visual images be misrepresented by the use of "enhanced" video prints. For video prints that have undergone meaningful alteration by the use of video techniques, the type and extent of the manipulation is to be reported.

### REFERENCES

- (1) Vander Voort, G. F., *Metallography: Principles and Practice*, McGraw-Hill Book Co., New York, NY, 1984.
- (2) Loveland, R. P., *Photomicrography*, John Wiley and Sons, Inc., New York, NY, 1970, (Reprinted by Robert E. Krieger Publ., Malabar FL, 1981), Vol 1, Chapters 7, 8, 9.
- (3) *Kodak Filters for Scientific and Technical Use*, Kodak Publication B-3, Rochester, NY, 1981.
- (4) Loveland, R. P., *Photomicrography*, John Wiley and Sons, Inc., New York, NY, 1970, (Reprinted by Robert E. Krieger Publ., Malabar, FL, 1981), Vol 1, Chapter 4, p. 179.
- (5) Todd, H. N. and Zakia, R. D., *Photographic Sensitometry*, 2nd Ed., Morgan and Morgan, Dobbs Ferry, NY, 1974.
- (6) Loveland, R. P., "Characteristics and Choice of Photographic Materials for Photomicrography," *The Microscope*, Vol 19, 1971, pp. 177-204.
- (7) *Quality Enlarging with Black-and-White Papers*, Kodak Publication G-1, Rochester, NY, 1982.
- (8) *Kodak Plates and Films for Scientific Photography*, Kodak Publication P-315, Rochester, NY, 1981.
- (9) *Polaroid Black and White Films*, Polaroid Corp., Cambridge, MA, 1980.

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