Designation: E 883 – 99

Standard Guide for Reflected–Light Photomicrography[1]

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 (ϵ) 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:

2. Referenced Documents

2.1 *ASTM Standards:*

E 3 Methods of Preparation of Metallographic Specimens[2]
E 7 Terminology Relating to Metallography[2]
E 175 Terminology of Microscopy[3]
E 768 Practice for Preparing and Evaluating Specimens for Automatic Inclusion Assessment of Steel[2]

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×, 50×, 75×, 100×, 200×, 250×, 400×, 500×, 750×, 800×, and 1000×.

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

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[1] This guide is under the jurisdiction of ASTM Committee E-4 on Metalography and is the direct responsibility of Subcommittee E04.01on Sampling, Specimen Preparation, and Photography.

Current edition approved April 10, 1999. Published July 1999. Originally published as E 883 – 82. Last previous edition E 883 – 94.

[2] *Annual Book of ASTM Standards*, Vol 03.01.

[3] *Annual Book of ASTM Standards*, Vol 14.02.

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)**[4]. 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 filtation. 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 succeedingly smaller increments until the image is sharp.

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[4] The boldface numbers in parentheses refer to the list of references appended to this guide.

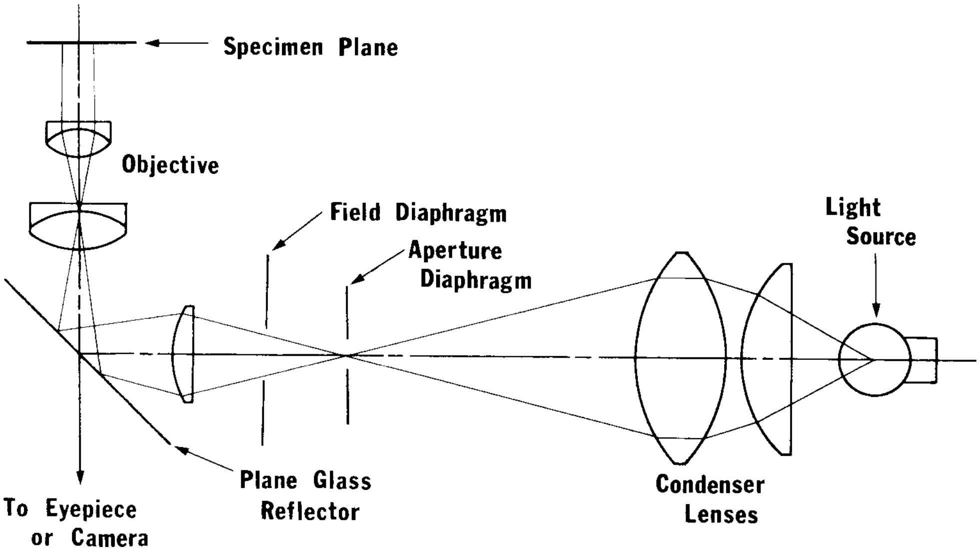

FIG. 1 Vertical Illuminating System for a Metallurgical Microscope

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 groundglass 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× to 5×. 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 interpupiliary distance, if required (some equipment requires a specific interpupiliary 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 % $NaNO_2$ 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 semiapochromats, 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.)

TABLE 1 Suggested Filtration for Color Photomicrography

Film Color Balance	Daylight	3200 K	3400 K
Light Source	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

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 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 microcrope (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 × 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-andWhite*—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 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 darkroom.

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

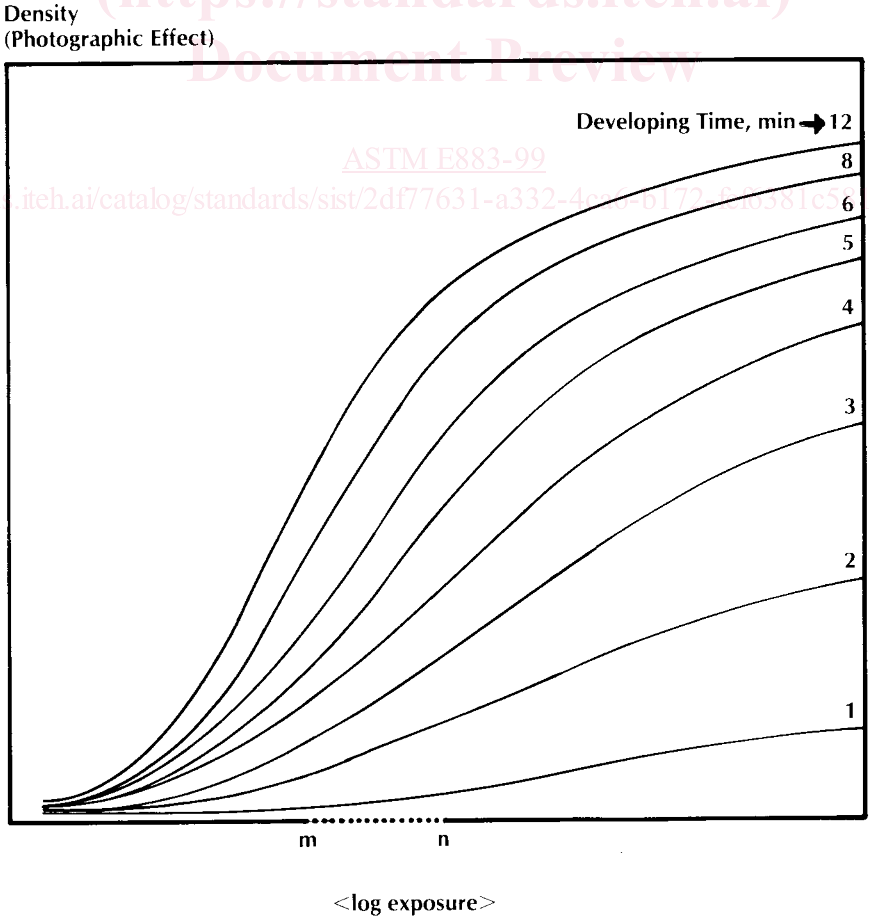

FIG. 2 Optical Density Versus Logarithm of Exposure for Photographic Materials