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Standard Guide for Reflected–Light Photomicrography[1]

This standard is issued under the fixed designation E883; 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 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 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. Descriptive material is provided where necessary to clarify procedures. References are cited where detailed descriptions may be helpful.

1.3 *This standard does not purport to address all of the safety concerns, 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 requirements 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:*[2]

E3 Guide for Preparation of Metallographic Specimens

E7 Terminology Relating to Metallography

E175 Terminology of Microscopy

E768 Guide for Preparing and Evaluating Specimens for Automatic Inclusion Assessment of Steel

E1951 Guide for Calibrating Reticles and Light Microscope Magnifications

3. Terminology

3.1 *Definitions*—For definitions of terms used in this guide, see Terminologies E7 and E175.

4. Significance and Use

4.1 This guide is useful for the photomicrography and photomacrography of metals and other materials.

4.2 The subsequent processing of the photographic materials is also treated.

5. Magnification

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

5.2 The preferred magnifications for photomicrographs, are: 25×, 50×, 75×, 100×, 200×, 250×, 400×, 500×, 750×, 800×, and 1000×.

5.3 Magnifications are normally calibrated using a stage micrometer. Calibration procedures in Guide E1951 should be followed.

6. Reproduction of Photomicrographs

6.1 Photomicrographs should be at one of the preferred magnifications. A milli- or micrometre marker shall be superimposed on the photomicrograph to indicate magnification, in a contrasting tone. The published magnification, if known, should be stated in the caption.

6.2 Photomicrograph captions should include basic background information (for example, material identification,

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

etchant, mechanical or thermal treatment details) and should briefly describe what is illustrated so that the photomicrograph can stand independent of the text.

6.3 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.

7. Optical Systems

7.1 Microscope objectives are available in increasing order of correction as achromats, semiapochromats (fluorites) and apochromats (see Terminologies E7 and E175). Plan objectives are recommended for photographic purposes because their correction provides a flatter image. The objective lens forms an image of the specimen in a specific plane behind the objective called the back focal plane. (This is one of several possible real image planes, called intermediary planes, where reticles may be inserted as optical overlays on the image.)

7.2 The eyepiece magnifies the back focal plane (or other) 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.

7.2.1 The pupil of the observer's eye must be brought to coincidence with the eyepoint of the visual eyepiece to view the entire microscopical image. High-eyepoint eyepieces are necessary for eyeglass users to see the entire image field.

7.2.2 Most microscopes have built-in photographic capabilities that use an alternate image path through the microscope leading to a camera attachment port or to a viewscreen. A projection eyepiece delivers the image to the camera port or screen.

7.3 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, either fixed or as a zoom system.

7.4 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.

7.5 The resolution of the microscope depends primarily on the numerical aperture of the objective in use (**1**)[3]. The term empty magnification is used to describe high magnifications (above approximately 1100 times the numerical aperture of an objective), which have been shown to offer no increase in image resolution. Nevertheless, some types of information, such as the distance between two constituents, may be more easily obtained from microstructures examined at moderate empty magnifications.

8. Illumination Sources

8.1 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.

8.2 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.

8.3 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. They also contain significant ultraviolet (UV) and infrared (IR) emissions that should be removed for eye safety (and for photographic consistency, with UV); see 8.4, 11.3.1, and 11.5.2.

8.3.1 Xenon arcs produce a spectral quality close to daylight (5600K), with a strong spike at 462 nm. Strong emissions in the IR should be removed. Xenon arcs that do not produce ozone are recommended.

8.3.2 Carbon arcs have a continuous output in the visible portion of the spectrum, with a color temperature near 3800K and a strong emission line at 386 nm.

8.3.3 Mercury arcs have strong UV and near-UV output, and are particularly useful to obtain maximum resolution with a blue filter. The color quality is deficient in red; it cannot be balanced for color photomicrography.

8.3.4 Zirconium arcs have strong spectral output lines in the near IR, requiring filtration. Within the visible region, they are rated at 3200K color temperature.

8.4 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.

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

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

9. Illumination of Specimens

9.1 Photomicrographs are made with a compound microscope comprised at least of an objective lens and an eyepiece with a vertical illuminator between them. Field and aperture diaphragms, with a lamp and lamp condenser lenses, are integral parts of the system. The microscope should allow sufficient adjustment to illuminate the field of view evenly and to completely fill the back aperture of the objective lens with light.

9.2 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 prism systems are used to perform this function.

[3] The boldface numbers in parentheses refer to the list of references at the end of this standard.

9.3 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.

9.4 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. The aperture diaphragm should never be used as a light intensity control.

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

10. Focusing

10.1 Sharp focus is necessary to obtain good photomicrographs.

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

10.2.1 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.

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

10.2.2.1 The simplest case is a transparent spot on a ground-glass containing a fiduciary mark in the film plane. The specimen image is focused 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.

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

10.2.2.3 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.

10.2.2.4 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.

10.3 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.

11. Filters for Photomicrography

11.1 Photomicrographs require filtration of the light source. This section describes filter types and their uses.

11.2 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.

11.2.1 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 8.4 are interference filters.)

11.2.2 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.

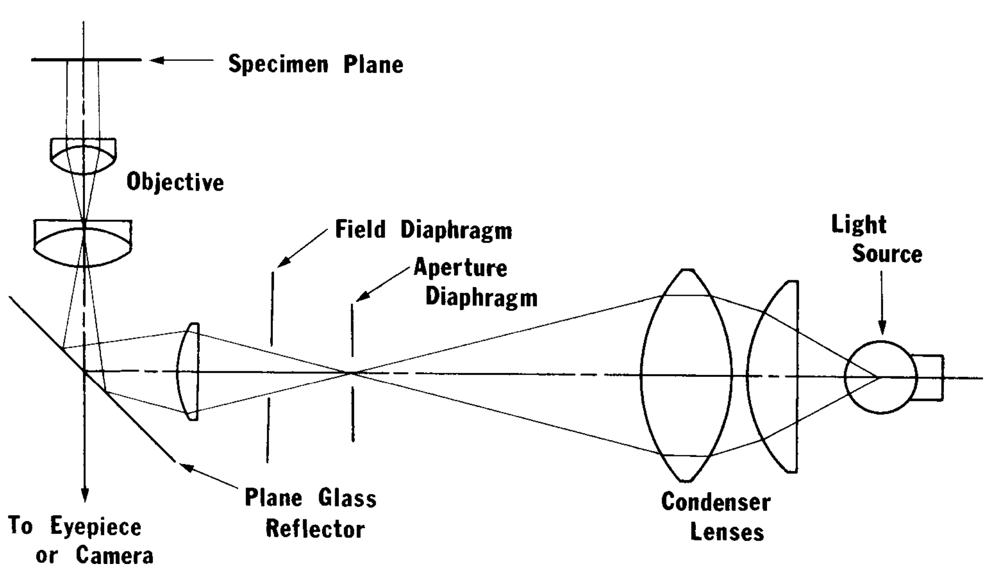

FIG. 1 Vertical Illuminating System for a Metallurgical Microscope

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

11.3.1 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 % $NaNO_2$ is used for a 1-cm path. It should be 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 11.5.2.

11.3.2 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.

11.4 *Filters for Black and White Photomicrography*:

11.4.1 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.

11.4.2 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, a cyan filter for reddish copper plating; a 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.

11.5 *Filters for Color Photomicrography*:

11.5.1 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.

11.5.2 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 11.3.1, and rebalance by adding compensatory blue filters.

11.5.3 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.

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

NOTE 1—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.)

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

12. Illumination Techniques

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

12.2 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 9.2 and Fig. 1). Features such as inclusions and etched grain boundaries have edges that are inclined to the polished surface and reflect light away from the objective lens, making them appear dark.

12.3 *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.)

12.4 *Dark field* illumination is obtained by directing light to the specimen along the outside of the objective, blocking out the illumination passing through the lens. These outside rays are diverted onto the specimen plane obliquely by a conical reflector. No specular reflections enter 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 that would be completely obscured with other kinds of illumination.

12.5 *Polarized light* reveals grain structure and twinning in metals with a hexagonal lattice structure, such as beryllium, tin, titanium and zinc. Polarized illumination is produced by optical components consisting of calcite prisms or Polaroid™ filters. They selectively provide an image consisting of polarized light reflected from a specimen surface and scattered depolarized light from nonplanar surface features. Polarized light reacts differently when reflected from isotropic and anisotropic material lattices. For a cubic material, the microscope field appears dark because most of the light reflected from the specimen is absorbed by the system. 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 system now passes a portion of the reflected light through to the viewing system. Polarized light is also used with optically inactive cubic metals that are treated to produce an anisotropic surface film on the substrate. It is also useful to identify optically active inclusions and phases and in defining domains in ferromagnetic materials.

12.6 *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 system. If a quartz or gypsum sensitive tint filter (also known as a λ compensator, a full-wave plate or a first-order compensator) is used, a magenta color is seen with cubic metals and all birefringent metals appear in vivid color contrasts. Nodular cast iron demonstrates this effect particularly well, if a rotatable stage is used.

12.7 *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 by the specimen, by either path length (feature height) 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 unique advantages: 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 E768; display of surface relief, including changes of a few nanometres at abrupt edges.

13. Instant-Processing Films

13.1 These materials yield photographic images within seconds after exposure (**8**). Both color and black-and-white versions are available. All use variations of the diffusion-transfer process, with each frame developed individually after exposure.

13.1.1 Instant materials should be exposed 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 longer ones will give insufficient separation between the lighter tones.)

13.1.2 The majority of the instant materials 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. 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 with a positive/negative film, the positive will be overexposed, and therefore not considered an acceptable print.)

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

13.2 *Processing Instant Films*—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 goes to completion according to the time-temperature relationship supplied with the film.

13.2.1 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 ¼ to ½ second is suggested for peel-apart materials. Monopack instant materials permit no control over processing time, since the self-developing reaction is controlled by the film holder and proceeds to completion without attention from the user.

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

13.2.3 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.

13.2.4 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. Photographic Materials

14.1 *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 (**4-6**) are recommended reading to learn the complete photographic characteristics of the products, as well as the terminology used to describe them.

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

14.3 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 with similar chemical steps.

14.4 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.

14.5 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.)

14.5.1 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.

14.6 Several properties of negative emulsions that must be considered are: overall light sensitivity (film speed), spectral sensitivity, resolving power, graininess, and contrast potential.

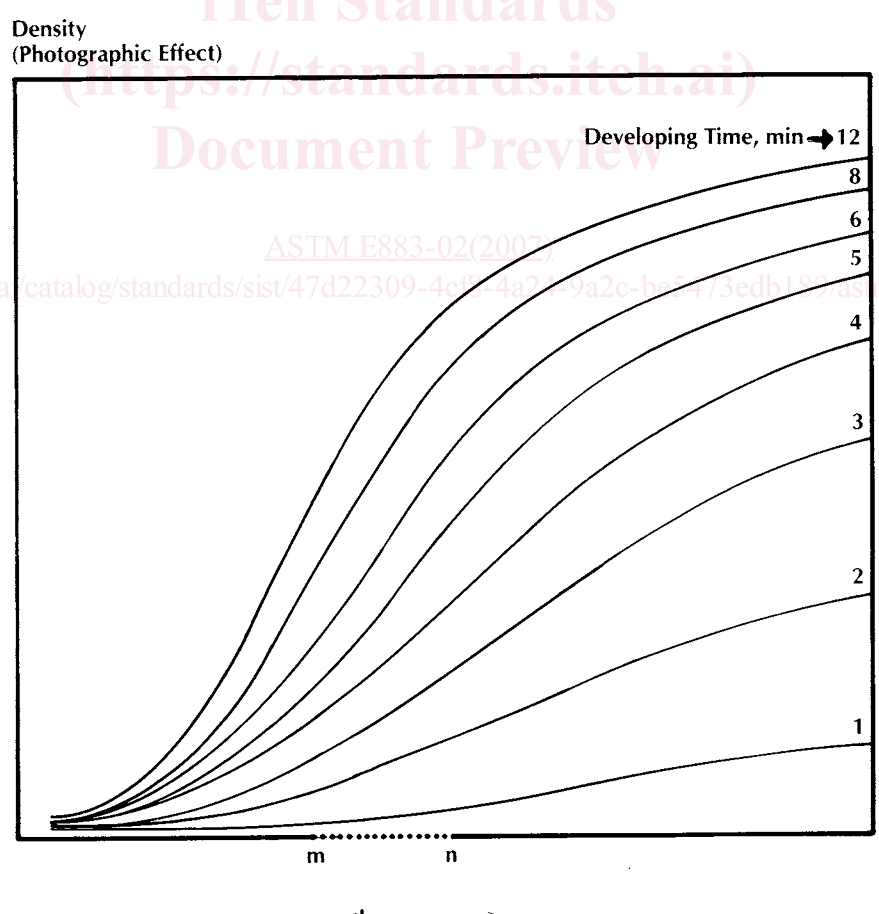

FIG. 2 Optical Density Versus Logarithm of Exposure for Photographic Materials