Designation: E 1180 – 03

Standard Practice for Preparing Sulfur Prints for Macrostructural Examination[1]

This standard is issued under the fixed designation E 1180; 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.

1. Scope

1.1 This practice provides information required to prepare sulfur prints (also referred to as Baumann Prints) of most ferrous alloys to reveal the distribution of sulfide inclusions.

1.2 The sulfur print reveals the distribution of sulfides in steels with bulk sulfur contents between about 0.002 and 0.40 weight percent.

1.3 Certain steels contain complex sulfides that do not respond to the test solutions, for example, steels containing titanium sulfides or chromium sulfides.

1.4 The sulfur print test is a qualitative test. The density of the print image should not be used to assess the sulfur content of a steel. Under carefully controlled conditions, it is possible to compare print image intensities if the images are formed only by manganese sulfides.

1.5 The sulfur print image will reveal details of the solidification pattern or metal flow from hot or cold working on appropriately chosen and prepared test specimens.

1.6 This practice does not address acceptance criteria based on the use of the method.

1.7 *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 limitations prior to use.* For specific precautionary statements, see Section 9.

2. Referenced Documents

2.1 *ASTM Standards:*

E 3 Methods of Preparation of Metallographic Specimens[2]

E 7 Terminology Relating to Metallography[2]

E 340 Test Method for Macroetching Metals and Alloys[2]

E 381 Method of Macroetch Testing Steel Bars, Billets, Blooms, and Forgings[2]

E 407 Test Methods for Microetching Metals and Alloys[2]

3. Terminology

3.1 *Definitions*—For definitions of terms used in this practice, see Terminology E 7.

4. Summary of Practice

4.1 The sulfur print provides a means for macroscopic evaluation of the sulfur distribution in steels and cast irons by contact printing using photographic paper soaked in an aqueous acid solution, for example, sulfuric acid, citric acid, or acetic acid.

NOTE 1—No longer are there available emulsion coated half weight fiber based papers suitable for sulfur printing. Emulsion coated single weight fiber base paper is not readily available. Emulsion coated double weight fiber base paper is available, and is preferable to emulsion coated single weight resin coated paper.

4.2 The test specimen is usually a disk or rectangular section, such as used in macroetch evaluations, cut from an as-cast or wrought specimen with either a transverse or longitudinal orientation. The specimen is freshly ground smooth and cleaned to remove cutting oils, scale, abrasives, or other contaminents. The specimen should be at room temperature when sulfur printed.

4.3 A sheet of photographic paper with (usually) a matte surface finish of appropriate size is soaked in the dilute aqueous acid solution, any excess liquid removed, and the emulsion side of the paper is placed on the ground surface of the specimen. After a suitable time, the paper is removed, washed in water, fixed, washed again in water, and dried as flat as possible.

4.4 The distribution of sulfur in the specimen is revealed as a mirror image on the photographic paper as darkly colored areas of silver sulfide embedded in the emulsion.

5. Significance and Use

5.1 The sulfur print reveals the distribution of sulfur as sulfide inclusions in the specimen. The sulfur print complements macroetch methods by providing an additional procedure for evaluating the homogeneity of a steel product.

[1] This practice is under the jurisdiction of ASTM Committee E04 on Metallography and is the direct responsibility of Subcommittee E04.01 on Selection and Preparation of Samples.

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

5.2 Sulfur prints of as-cast specimens generally reveal the solidification pattern and may be used to assess the nature of deoxidation, that is, rimming action versus killed steel sulfur distributions.

5.3 Sulfur prints will reveal segregation patterns, including refilled cracks, and may reveal certain physical irregularities, for example, porosity or cracking.

5.4 The nature of metal flow, such as in various forging operations, can be revealed using sulfur prints of specimens cut parallel to the metal flow direction.

5.5 The sulfur print method is suitable for process control, research and development studies, failure analysis, and for material acceptance purposes.

5.6 The intensity of the sulfur print is influenced by the concentration of sulfur in the steel, the chemical composition of the sulfide inclusions, the aggressiveness of the aqueous acid solution, and the duration of the contact printing between the acid soaked emulsion coated paper and the ground surface of the specimen (this time is the order of seconds rather than minutes). Very low sulfur content steels will produce too faint an image to be useful for macrostructural evaluations. Selection of appropriate printing practices including selection of type of emulsion coated media, acid type and strength, will yield satisfactory prints. Very faint images in the sulfur print can be made more visible by scanning the sulfur print into a PC, and using a photo editor to increase the color saturation. Steels with compositions that produce predominantly titanium or chromium sulfides will not produce useful images.

6. Interferences

6.1 The specimen must be properly cleaned, otherwise dark spots will be produced which may be incorrectly interpreted as a gross sulfide segregate.

6.2 Hydrogen sulfide gas is produced while the paper is in contact with the specimen. The hydrogen sulfide is readily absorbed by the wet emulsion. The hydrogen sulfide reacts with the silver halides in the emulsion to lay down insoluble silver sulfide. If the specimen contains pores or cracks, hydrogen sulfide gas may become entrapped in these openings and may produce a brown color on the paper which may be incorrectly interpreted as a gross sulfide segregate.

6.3 If air is entrapped between the contacting paper and specimen, and is not removed, a white spot may be produced on the print. Air entrapment must be quickly removed by the use of a rubber squeegee or roller to move bubbles to the edge of the specimen.

6.4 Image blurring may result from movement of the paper during contact.

6.5 Specimens with low sulfur contents are often pre-etched before printing to expose more sulfides and enhance the image. If the pre-etchant contains sulfate ions (for example, a stainless steel specimen etched with Marble's reagent), the print will be lightly colored, even if sulfides are not present in the steel. Such etchants should not be used for this purpose.

6.6 If chromium replaces some of the manganese in the sulfide inclusions, the print intensity for a given sulfur level will be reduced. An image will not be obtained, irrespective of the sulfur content, if titanium or chromium sulfides are present.

7. Apparatus

7.1 *Lighting*—If the chosen photographic paper when exposed to the existing room light for 15 min changes from white to light blue and then clears back to white when processed in the sequence of solutions, there is no need to turn of the existing white lighting, and work under amber bulb lighting; never expose the paper to sunlight.

7.2 *Shallow Container*, such as a photographic tray, is required to contain the dilute aqueous acid solution. The container must be large enough to soak the emulsion coated paper without wrinkling.

7.3 *Timing Device*, such as used in a photographic darkroom, is helpful for timing the contact printing time, and the washing and fixing periods.

7.4 *Tank*, of suitable size with cool flowing water, is required for washing the print.

7.5 *Tank, or Covered Tray*, to hold the fixing agent and the print; two can be used sequentially for faster fixing when using emulsion coated double weight fiber based paper.

7.6 *Drying*—Heated drum dryers are no longer made. Heated drying cabinets are available for fiber base prints laid horizontally on a screen. Resin coated papers can be dried with an infra red dryer very quickly. Clothes lines and cork peg boards will also work but the prints do not dry perfectly flat.

8. Reagents and Materials

8.1 Photographic enlarging paper with a matte finish and a fiber base is preferred. Sharper images are produced by the use of glossy photographic printing paper. Due to the smooth surface finish, it is difficult to prevent blurring of the image. Resin-coated paper when used with small area specimens can produce satisfactory results; as the area gets larger it becomes more difficult to have the paper conform to the specimen surface, because the paper base is coated with a thin layer of polyethylene which does not absorb any water and become sufficiently limp, as with fiber base paper. Photographic paper is available in cut sheets and rolls of various widths. Cut sheets are ideal if the specimen size matches the sheet size. Roll dispensed paper can be fed from a "safe" box and cut as needed. The paper sheet should be 12 to 20 mm (½ to ¾ in.) larger than the specimen around the perimeter of the specimen. If the overhang of the paper is too great then the paper will not lie tight to the edge of the specimen.

8.2 Technical or reagent grade acids, sulfuric acid, acetic acid, citric acid, etc., are used to make the solution in which the paper is soaked prior to contact printing; typical concentrations of acid are −2 to 10 % sulfuric acid, 10 to 15 % acetic acid, and 10 to 15 % citric acid.

8.3 A commercial photographic fixing solution (a liquid rapid fixer) is used to fix the sulfur print image after contact printing and washing. The fixer should be tested periodically to ensure that it is still active; set aside a print in the sunlight and if the appearance changes then the fixer is deleted and should be replaced. Used fixer contains silver and should be disposed of in concordance with local regulations. There is not enough silver to justify having the silver recovered from the used fixer.