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StandardPractice for Preparing Sulfur Prints for Macrostructural Evaluation[1]

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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 0.010 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 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.8 *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:*[2]

E3 Guide for Preparation of Metallographic Specimens
E7 Terminology Relating to Metallography
E340 Test Method for Macroetching Metals and Alloys
E381 Method of Macroetch Testing Steel Bars, Billets, Blooms, and Forgings
E407 Practice for Microetching Metals and Alloys

3. Terminology

3.1 *Definitions*—For definitions of terms used in this practice, see Terminology E7.

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.

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 contaminants. 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.

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.

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

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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.

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 off 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 paper is a multilayer paper coated with a gelatin emulsion containing about 80 mg per square meter of silver as a halide (Cl and/or Br) supported by a paper base that is nominally single or double weight (110 or 235 g/sq. m). The speed and contrast characteristics are of no importance when sulfur printing. The paper base may be fibre base or resin coated. A thin layer of baryta may separate the emulsion and the base in order to provide a more visible image. A glossy emulsion is preferred to a matte emulsion if image sharpness is important; the problem is that a glossy emulsion may slide on the steel surface and cause blurring. A fibre base is preferred to a resin coated base because the fibre base tends to better conform to the steel surface; in addition it has less tendency to slip when smoothing the paper over the steel surface. Note that photo paper for digital photo printing contains no silver halide emulsion and is not suitable for sulfur printing. The advantage of resin coated photographic paper, over fibre base paper, is that the paper base is sealed from contact with the dilute acid, the rapid fixer, and the water during washing; hence the processing time, including drying time, is much less, especially if double weight paper is used. 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 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