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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Practice for Preparing Sulfur Prints for Macrostructural Examination¹

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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 0.005 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 Specimens²

E 7 Terminology Relating to Metallography²

E 340 Test Method for Macroetching Metals and Alloys²

E 381 Method of Macroetch Testing Steel Bars, Billets, Blooms, and Forgings²

E 407 Test Methods for Microetching Metals and Alloys²

3. Terminology

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

¹ This practice is under the jurisdiction of ASTM Committee E-4 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.

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, or an equivalent, soaked in an aqueous acid solution, for example, sulfuric acid, citric acid, or acetic acid.

NOTE 1—There are light weight emulsion coated papers suitable for sulfur printing currently on the market.

4.2 The test specimen is usually a disk, such as used in macroetch evaluations, cut from an as-cast or wrought specimen with either a transverse or longitudinal orientation. The disk is ground smooth and cleaned.

4.3 A sheet of photographic paper (or equivalent) with a (usually) matte surface finish of appropriate size is soaked in the dilute aqueous acid solution and the emulsion side of the paper is placed on the ground surface of the disk. After a suitable time, the paper is removed, washed in water, fixed, washed and dried.

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

5. Significance and Use

5.1 The sulfur print reveals the distribution of sulfur as sulfide inclusions in the test sample. The sulfur print complements macroetch methods by providing an alternative 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.

5.3 Sulfur prints will reveal segregation patterns 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 materials 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. 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 species, and aqueous concentration will yield satisfactory prints. Steels with compositions that produce predominantly titanium sulfides or chromium sulfides will not produce useful images.

6. Interferences

6.1 The test sample 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 disk. If the disk 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 disk and is not removed, a white spot may be produced on the print.

6.4 Image blurring may result from movement of the paper during contact which will make interpretation difficult.

6.5 Disks with low sulfur contents are often pre-etched before printing to expose more sulfides and enhance the image. If the etchant contains sulfate ions (for example, a stainless steel disk 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 the sulfides are present as titanium sulfides or as chromium sulfides.

7. Apparatus

7.1 *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 media (for example, photographic paper) without excessive folding of the paper.

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

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

7.4 *Tank*, to hold the fixing agent and the print, is required.

7.5 *Print Drier* (optional), useful for aid for drying the sulfur print after processing.

8. Reagents and Materials

8.1 A matte-finish type, photographic emulsion coated printing media, of normal or thin thickness is used for the contact print. Sharper images are produced by use of glossy type photographic printing paper. However, due to their smoother surface finish, it is more difficult to keep the paper motionless during printing to prevent blurring of the image. Bromide-type papers used for enlarging are preferred to chloride-type papers. Resin-coated papers produce satisfactory results.

8.2 Reagent grade acids, sulfuric acid, acetic acid, citric

acid, etc., are used to make the sensitizing solution in which the paper is soaked prior to contact printing.

8.3 A commercial photographic fixing solution is used to fix the sulfur print image after contact printing and washing.

9. Hazards

9.1 Sulfuric acid, H_2SO_4 , is a highly corrosive, dangerously reactive, strong oxidizing agent. It reacts with water releasing substantial heat. Add sulfuric acid very slowly to the water with constant stirring. Contact with concentrated sulfuric acid must be avoided. The dilute solution used to soak the prints is not particularly dangerous but exposure to it should be minimized and hands should be washed after any contact. The use of rubber gloves should be considered. Use tongs to handle the paper in the soaking solution. The other acids recommended for sensitizing the paper are less aggressive than sulfuric acid, however, appropriate care should be taken in mixing and handling.

10. Sampling and Test Specimens

10.1 Samples are generally selected in the same manner and extent as for macroetching, as described in Methods E 3, E 340, and E 381. Samples are normally prepared to sample the entire cross section, and are oriented either transversely or longitudinally, depending on the purpose of the examination.

10.2 The number, orientation, and location of test pieces may be subject to producer-purchaser agreement.

10.3 Disks should be cut in a region away from any effects from hot shearing or burning.

10.4 Samples should be thin enough for ease of handling, generally 12 to 25 mm (0.5 to 1 in.) thick. The surface to be contact printed should be smooth ground and carefully cleaned. Edges should be free of flash or burrs.

10.5 Excessively smooth surfaces, such as produced by polishing, will promote slippage between the paper and disk resulting in blurred images.

10.6 Surface preparation (see Methods E 3, E 340 and E 381) should not produce excessive cold work at the test surface.

11. Procedure

11.1 Soak the photographic paper (or equivalent) in the selected aqueous acid solution. The strength of the solution will depend upon the acid selected, the sulfur content of the steel and the desired printing time. SAE 10XX steels with sulfur contents between 0.015 wt % and 0.035 wt % are typically printed using a 2% sulfuric acid solution. SAE 11XX series steels are more successfully printed with a 15 % citric acid solution. Steels with low sulfur contents (under 0.010 wt %) may be printed using 5 to 10 % sulfuric acid solutions. When large size prints are desired, a long printing time may be required, necessitating a weaker acid solution.

11.2 Soak the paper in the solution for 1 to 5 min. A 3-min soak time is most commonly used. Periods in excess of 5 min may cause swelling of the emulsion.

11.3 After soaking the required time, remove the paper from the solution and allow excess solution to drip off the paper into the bath. To minimize paper movement during printing, it may be advisable to place the paper on a glass plate and squeegee out excess moisture.