Designation: E 1351 – 01

Standard Practice for Production and Evaluation of Field Metallographic Replicas[1]

This standard is issued under the fixed designation E 1351; 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.

INTRODUCTION

Replication is a nondestructive sampling procedure which records and preserves the topography of a metallographic specimen as a negative relief on a plastic film. The microstructural replica can be examined using a light microscope (LM) or scanning electron microscope (SEM) for subsequent analysis. Specimens examined in the SEM are vacuum coated with vaporized carbon or a suitable metal to provide contrast and conductivity. The convenience of the replication process makes it suitable for obtaining microstructures from field locations for subsequent examination and analysis in a laboratory. The proper preparation of the test surface and of the replica itself is of paramount importance and must receive careful attention. Because of the diversity of metallographic equipment available and the wide range of environments in which replication is conducted, the preparation of replicas of high quality should be viewed as a skilled process for which there exists a variety of techniques that achieve satisfactory results.

This practice presents some guidelines on the preparation of metallic surfaces and production of replicas and guidelines on evaluation of replica quality. It does not attempt to limit the variations in technique developed by skilled metallographers, each of which may produce acceptable replicas.

1. Scope

1.1 This practice covers recognized methods for the preparation and evaluation of cellulose acetate or plastic film replicas which have been obtained from metallographically prepared surfaces. It is designed for the evaluation of replicas to ensure that all significant features of a metallographically prepared surface have been duplicated and preserved on the replica with sufficient detail to permit both LM and SEM examination with optimum resolution and sensitivity.

1.2 This practice may be used as a controlling document in commercial situations.

1.3 The values stated in SI units are to be regarded as the standard. Inch-pound units given in parentheses are for information only.

1.4 *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.*

2. Referenced Documents

2.1 *ASTM Standards:*

A 335/A 335M Specification for Seamless Ferritic Alloy-Steel Pipe for High-Temperature Service[2]

E 3 Guide for Preparation of Metallographic Specimens[3]

E 7 Terminology Relating to Metallography[3]

E 407 Practice for Microetching Metals and Alloys[3]

3. Terminology

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

4. Significance and Use

4.1 Replication is a nondestructive sampling procedure that records and preserves the topography of a metallographically prepared surface as a negative relief on a plastic film (replica). The replica permits the examination and analysis of the metallographically prepared surface on the LM or SEM.

4.2 Enhancement procedures for improving replica contrast for microscopic examination are utilized and sometimes necessary (see 8.1).

NOTE 1—It is recommended that the purchaser of a field replication service specify that each replicator demonstrate proficiency by providing field prepared replica metallography and direct LM and SEM comparison to laboratory prepared samples of an identical material by grade and service exposure.

[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 01.01.

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

5. Evaluation Methods

5.1 A suitable replica should accurately reproduce all the microstructural features present on the surface that was replicated.

5.2 No visible loss of resolution is permitted over the normal range of magnifications on the LM as shown in Figs. 1-3.

5.3 The resolution of the structural detail in the replica should exceed 0.1 µm to permit SEM examination at high magnifications (up to 5000×). See Figs. 4-6.

6. Metal Surface Preparation

6.1 If magnetic particle testing was previously used on the work-piece, demagnetize the piece before beginning surface preparation.

6.2 Surface preparation may be accomplished using manual, mechanical, or electrolytic polishing methods.

NOTE 2—Electrolytic preparation always carries the risk of pitting, and of enlarging existing voids such as creep cavities and porosity.

6.3 Prepare the surface to be replicated using the methods suggested in Methods E 3 modified for field use, as appropriate, in such a way as to obtain a surface free of deformation, scratches, polishing defects, etch pits, and other artifacts which may obscure the true microstructural features.

NOTE 3—The presence of decarburization can be detected with a portable hardness tester during the grinding steps. Further grinding to reach a surface free of decarburization can be monitored with the hardness tester. A replica may also be made on the decarburized surface, if it serves the purpose of the investigation.

6.4 Do not remove any precipitates, carbides, nonmetallic inclusions such as oxides and sulfides during the polishing or etching operations.

6.5 Etching procedures for surface metallographic examination should be performed in accordance with Practice E 407.

6.6 The quality of the surface preparation should be controlled by the use of a portable field microscope.

6.7 To prevent possible contamination of any components, the etched area should be prepared carefully and thoroughly washed after replication.

7. Replication Technique

7.1 In general, a replicated area of 12 by 18 mm (0.5 by 0.75 in.) is satisfactory.

7.2 A replica is produced by one of the two methods described below. All methods produce acceptable replicas.

7.2.1 A replica may be produced by wetting one side of a sheet of plastic film with a suitable solvent, such as acetone or methyl acetate, and applying the wetted side of the film to the prepared metal surface.

7.2.2 Alternatively, a replica may be produced by wetting the prepared metallic surface with a suitable solvent, such as acetone or methyl acetate, and applying the strip of plastic film (usually cellulose acetate) to the wet surface. The film is pressed against the surface for several seconds to ensure adherance.

7.2.3 Apply a rubber-based replicating compound to the prepared metal surface. Cover with the material's carrier paper, then use a roller to spread the compound into a uniformly thin layer under the paper.

7.3 The replica shall be prepared as soon as possible after specimen preparation of the original surface is completed, to minimize transfer of post-preparation oxidation and contamination to the replicating film.

7.4 After the film has dried, remove the replica and permanently mount on a rigid slide to facilitate analysis of the replica and to protect it from damage during subsequent transport and storage. The mounting may be accomplished using a double-sided adhesive tape, either applied to the back side of the dried replica film while it remains on the prepared surface or applied

FIG. 1 Example of Replica Microstructure at 100× LM. Material: See Specification A 335/A 335M, Grade P22. Etchant: 2 % Nital

FIG. 2 Example of Replica Microstructure at 400× LM. Material: See Specification A 335/A 335M, Grade P22. Etchant: 2 % Nital

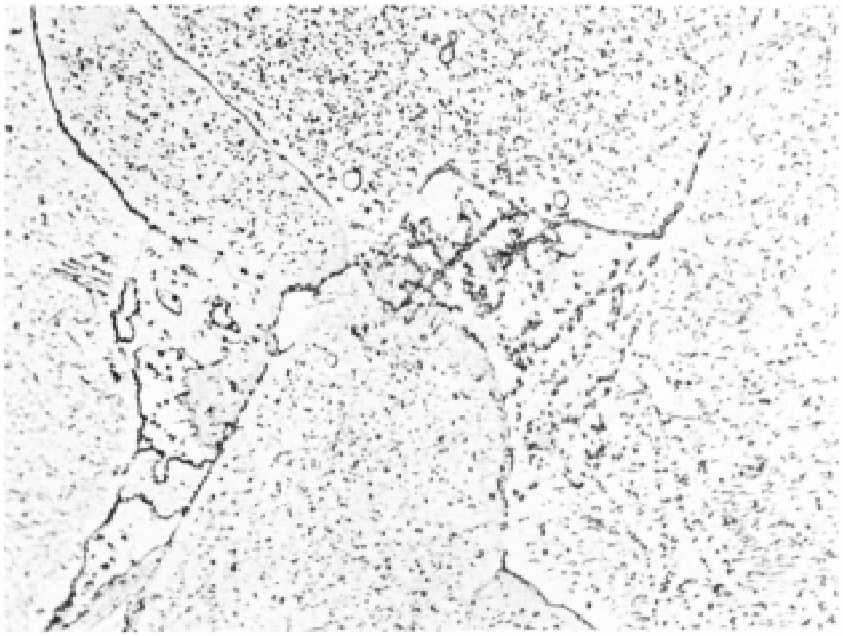

FIG. 3 Example of Replica Microstructure at 1000× LM. Material: See Specification A 335/A 335M, Grade P22. Etchant: 2 % Nital

to the slide before transferring the replica on the tape surface. Using the rounded end of a glass rod to apply the replica on the tape is usually beneficial in reducing air bubbles and ensuring a flat replica. Some metallographers prefer to coat the back side of the replica with an opaque substance such as black paint or ink prior to applying tape to improve the contrast when the replica is subsequently examined.

7.5 Place identification on the slide, and store it in a durable slide container for safe, contamination-free transfer to the examination laboratory.

8. Replica Examination

8.1 To enhance contrast of the replica for microscopic examination at low magnifications on the LM, the replica may be placed on an underlying polished surface, such as a mirror, which acts as a reflector. In addition, using a coating of black ink or paint on the back side of the replica can improve the contrast. In some cases, the use of interference illumination will also improve contrast; however, optimum contrast for both LM and SEM can be achieved by coating the replica surface

FIG. 4 Example of Replica Microstructure at 1000× SEM. Material: See Specification A 335/A 335M, Grade P22. Etchant: 2 % Nital

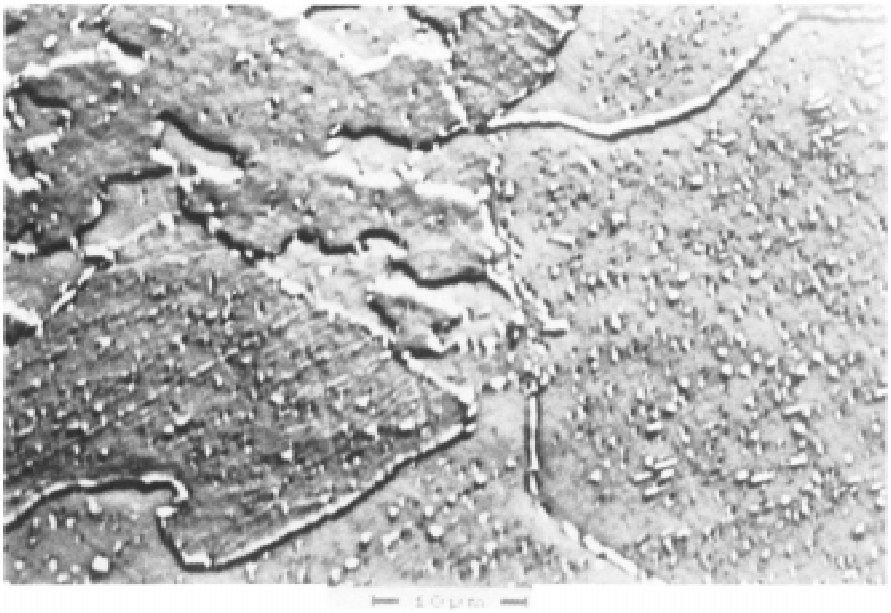

FIG. 5 Example of Replica Microstructure at 2000× SEM. Material: See Specification A 335/A 335M, Grade P22. Etchant: 2 % Nital

with a thin, highly reflective metallic layer material normally deposited in a vacuum coating unit. Aluminum, carbon, and chromium have been found to be satisfactory coatings for replicas to be evaluated by LM. If the replica is to be examined in the SEM, however, gold, which yields optimum contrast, is preferable. ASTM STP 547 is the suggested guide for electron metallographic techniques.[4]

8.2 The coating is generally applied at a 45° angle to provide a light to shadow length of 1 to 1. The coating thickness should be no greater than that required to provide full contrast in the replicated microstructure when examined in the LM and to prevent charging of the film when examined in the SEM. Documentation of the structural features of interest should then be made at the appropriate magnifications with the LM or SEM.

[4] *Manual on Electron Metallography Techniques, ASTM STP 547,* ASTM, 1973.

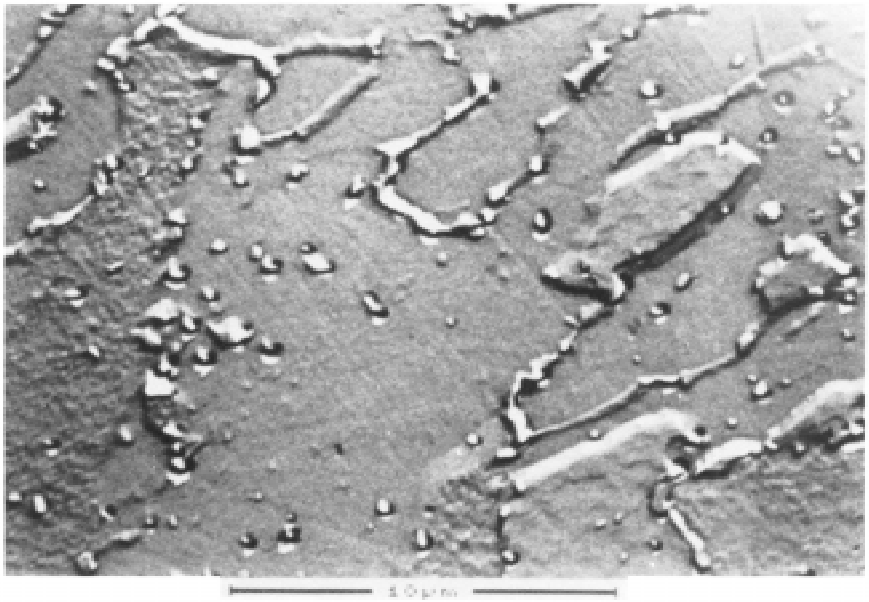

FIG. 6 Example of Replica Microstructure at 5000× SEM. Material: See Specification A 335/A 335M, Grade P22. Etchant: 2 % Nital

9. Replica Surface[5]

9.1 High quality replicas must meet the criteria listed in Section 5.

9.2 No folds or permanent deformation shall be allowed to develop in the replica film during processing.

9.3 Microstructural features should be clearly developed over an area of at least 6 mm (0.25 in.) in diameter.

9.4 Replicas taken at weldments should accurately record the base metal, weld metal, and heat-affected-zone microstructure along the fusion line for a distance of at least 13 mm (0.5 in.).

9.5 All microstructural features shall be accurately rendered and photographic documentation shall be obtainable over the range of magnifications normally used for replica evaluation: 50 to 1000X for LM and 500 to 5000X for SEM.

9.6 All grain boundaries, grain boundary precipitates, cracks and cavities should be easily identified.

9.7 Precipitates and inclusions contained in the material that are greater than 0.1 µm should be accurately recorded on the replica. In addition, neither precipitates, such as primary and secondary carbides and carbonitrides, nor nonmetallic inclusions, such as oxides and sulfides, shall be dislodged during polishing or etching regardless of their size.

10. Documentation

10.1 Photomicrographic documentation of microstructural features shall be obtainable over the normal range of magnification: 50 to 1000X LM and 500 to 5000X SEM.

10.2 Each replica be adequately identified. Minimum identification should include the job or other identification number, the name, the presence and characteristics of any coating applied to either side of the replica, and the name of the preparer.

10.3 A statement shall be made on the conformance or non-conformance of the replica to this practice.

10.4 Replicas shall remain attached to their carrier slides, and shall be stored in dust-free biological slide cases, with proper indexing to permit retrieval where required for record.

11. Precision and Bias

11.1 It is not possible to specify the precision or bias of this practice because no quantitative results are produced. However, all quantitative results produced through the examination of replicas can be influenced by the replication process.

12. Keywords

12.1 replica; replication

[5] Examples of replicated microstructures, (Specification A 335/A 335M steel, Grade P22 shown, with 2 % Nital etch. This material exhibits a wide range of microstructures, not all of which are shown here.)