



Designation: G209 – 14 (Reapproved 2022)

Standard Practice for Detecting mu-phase in Wrought Nickel-Rich, Chromium, Molybdenum-Bearing Alloys¹

This standard is issued under the fixed designation G209; 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 incorporates etching and metallographic examination of Wrought Nickel-Rich, Chromium, Molybdenum-Bearing Alloys such as, but not limited to, UNS N06686 and UNS N10276.

1.2 Microstructures have a strong influence on properties and successful application of metals and alloys. The presence of mu-phase in the microstructure may significantly reduce the corrosion resistance of Wrought Nickel-Rich, Chromium, and Molybdenum-Bearing Alloys.

1.3 This practice may be used to determine the presence of mu-phase in Wrought Nickel-Rich, Chromium, and Molybdenum-Bearing Alloys through comparison of microstructure observed for etched metallographic specimens to a glossary of photomicrographs displaying the presence and absence of mu-phase in the microstructure.

1.4 The values stated in SI units are to be regarded as standard. The values given in parentheses after SI units are provided for information only and are not considered standard.

1.5 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

¹ This practice is under the jurisdiction of ASTM Committee G01 on Corrosion of Metals and is the direct responsibility of Subcommittee G01.05 on Laboratory Corrosion Tests.

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2. Referenced Documents

2.1 *ASTM Standards:*²

D1193 Specification for Reagent Water

E3 Guide for Preparation of Metallographic Specimens

E7 Terminology Relating to Metallography

E1245 Practice for Determining the Inclusion or Second-Phase Constituent Content of Metals by Automatic Image Analysis

E1268 Practice for Assessing the Degree of Banding or Orientation of Microstructures

G193 Terminology and Acronyms Relating to Corrosion

3. Terminology

3.1 *Definitions:*

3.1.1 The terminology used herein, if not specifically defined otherwise, shall be in accordance with Terminology G193. Definitions provided herein and not given in Terminology G193 are limited only to this practice.

3.1.2 For metallographic definitions used in this practice, refer to Terminology E7.

3.1.3 For evaluation of inclusions, secondary phases and banding, if desired, refer to Practices E1245 and E1268.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *mu-phase* (μ), *n*—rhombohedral phase which may occur in Nickel-Rich, Chromium, Molybdenum-Bearing Alloys and may occur as coarse, irregular platelets, which form at high temperature.

4. Significance and Use

4.1 These test methods describe laboratory tests to determine the presence of mu-phase in Wrought Nickel-Rich, Chromium, and Molybdenum-Bearing Alloys through comparison of microstructure observed for etched metallographic

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.

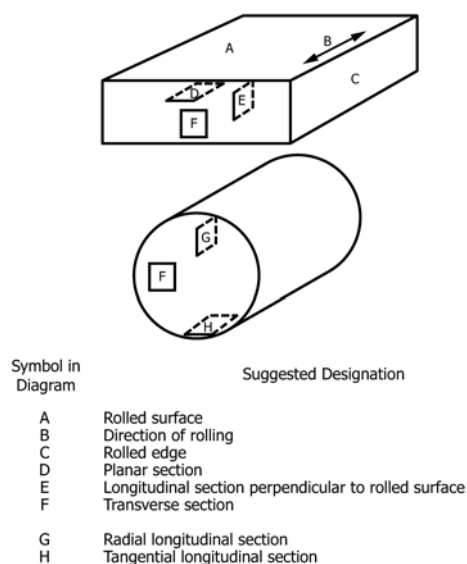


FIG. 1 Method of Designing Location of Area Shown in Photomicrograph (Guide E3)

specimens to a glossary of photomicrographs displaying the presence and absence of mu-phase in the microstructure. The presence of mu-phase in the microstructure may significantly reduce the corrosion resistance, strength, toughness and ductility of Wrought Nickel-Rich, Chromium, and Molybdenum-Bearing Alloys.

5. Sample Preparation and Etching³

5.1 Sectioning:

5.1.1 The selection of test specimens for metallographic examination is extremely important because, if their interpretation is to be of value, the specimens must be representative of the material that is being studied and shall be per location E (longitudinal section perpendicular to rolled surface) for plate and sheet and per location G (radial longitudinal section) for rod and bar per Fig. 1 (Guide E3). The intent or purpose of the metallographic examination will usually dictate the location of the specimens to be studied. For rod and bar test specimens specifically, samples are taken from location G as seen in Fig. 1. Triplicate test specimens shall be evaluated for determination of the presence of mu-phase.

5.1.2 Cut the specimen to a convenient size using any of various types of silicon carbide, diamond, boron carbide or other carbide cutoff blades. Deformation damage can be minimized by using thin cutoff wheels 0.78 mm ($\frac{1}{32}$ in.) thick as opposed to 1.58 mm ($\frac{1}{16}$ in.). Never cut dry. Use of adequate water coolant is desired to reduce the amount of disturbed metal created, in part, from frictional heat during this phase of preparation. The original microstructure of a specimen may also be radically altered, (at least superficially, on the cut surface) due to metallurgical changes if an excessive amount of frictional heat is generated.

5.2 *Coarse Grinding*—Use a 120 grit silicon carbide (SiC) wet-belt or disk grinder and light contact pressure to obtain a plane surface free from deep grooves. In addition to producing a flat surface, this procedure removes burred edges or other mechanical damage which may have occurred during sectioning.

5.3 *Mounting*—To ensure flatness, and facilitate handling, it is recommended that specimens be mounted in phenolic, acrylic or cold-setting epoxy resins. Epoxy resins involve the blending of a liquid or powder resin in a suitable hardener to initiate an exothermic reaction to promote hardening and curing at room temperature. This usually requires an overnight operation. However, an advantage of epoxy is that the mount is semitransparent and permits observation of all sides of the specimen during each phase of the preparation. (The advantages and use of acrylic mounting resin are similar to epoxy.) Compression molding techniques may be used with phenolic powders to produce the standard 31.7 mm ($1\frac{1}{4}$ in.) diameter mounts. Phenolic mounts are convenient when time constraints do not permit an overnight cold-setting operation.

5.4 *Fine Grinding and Polishing*—Rotating discs flushed with running water are recommended with successively finer grit papers of 220 grit, 320 grit, 400 grit, and 600 grit SiC. (A light to medium amount of pressure is exerted on the specimen to minimize the depth of deformation). Best results are obtained on the 600 SiC paper by grinding the specimen twice. Specimens shall be rotated 90 degrees after each step until the abrasive scratches from the preceding grit have been removed. In each step, the grinding time shall be increased to twice as long as that required to remove previous scratches. This ensures removal of disturbed metal from the previous step. Considerable care shall be used in the fine grinding stage to prevent the formation of artifacts. See Guide E3 for automated method.

5.5 *Rough Polishing*—The specimen shall be washed and, preferably, ultrasonically cleaned to ensure the complete removal of silicon carbide carryover from the fine grinding stage. A napless type cloth shall be charged with 9 μ m diamond paste, and water may be used as the lubricant. The specimen is moved counter to the direction of the rotating polishing wheel from the center to the outer periphery around the entire lapping surface. Heavy pressure is used with diamond abrasive techniques to gain the maximum cutting rate. At the conclusion of this stage, the specimen shall again be cleaned to remove any diamond polishing residue remaining in pinholes, cracks, and cavities.

5.6 Polishing:

5.6.1 Semi-final and final polishing operations on a major portion of metallographic specimens may be completed on vibratory polishing units. A nylon polishing cloth using a slurry of 30 g of 0.3 μ m alumina polishing abrasive and 500 mL of distilled or deionized water are recommended for this operation. Additional weight in the form of a stainless steel cap must be placed on the specimen. The suggested weight to achieve a satisfactory polish in 30 min to 60 min on a 31.7 mm ($1\frac{1}{4}$ in.) diameter mount is 350 g.

5.6.2 Samples shall be cleaned with a cotton swab under running water to remove the alumina particle film, placed on a

³ Manning, Paul E., Ph.D., *Metallographic Preparation of 686 Etching Specimens*, Haynes International, Inc., Kokomo, IN, 2011.