Designation: ~~G209 – 12~~ G209 – 12a

Standard Practice for
Detecting mu-phase in Wrought Nickel-Rich, Chromium, Molybdenum-Bearing Alloys[1]

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~~ 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 the standard. Other units are given in parentheses for information only.

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

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*~~—rhomohedral~~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 specimens to a

---

[1] This test method 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.

Current edition approved ~~May 1, 2012~~Nov. 15, 2012. Published ~~May 2012~~December 2012. Originally approved in 2012. Last previous edition approved in 2012 as G209–12. DOI: ~~10.1520/G0209–12.~~10.1520/G0209–12A.

[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.

***A Summary of Changes section appears at the end of this standard**

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 Etching[3]

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 ¼-diameter per 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 (1⁄32 in.) thick as opposed to 1.58 mm (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.

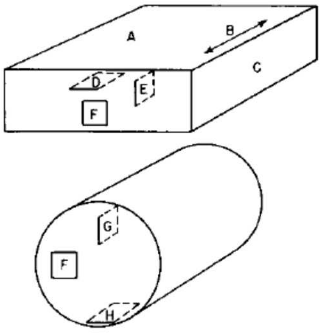
5.2 *Coarse Grinding*—Use a 120 grit silicon carbide (SiC) wet-belt or disk grinder and light contact pressure to obtain a plane surface free from deep grooves. In addition to producing a flat surface, this procedure removes burred edges or other mechanical damage which may have occurred during sectioning.

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 (1¼-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, 320, 400, 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.

---

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



Symbol in Diagram	Suggested Designation
A	Rolled surface
B	Direction of rolling
C	Rolled edge
D	Planar section
E	Longitudinal section perpendicular to rolled surface
F	Transverse section
G	Radial longitudinal section
H	Tangential longitudinal section

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

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-µm 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 *Vibrator 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 µm 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 30-60 min on a 31.7 mm (1¼-in.) diameter mount is 350 g.

5.6.2 Other methods of final polishing may be utilized, for example using a manual or automatic polishing wheel with fine (3 µm) abrasive polishing compound.

NOTE 1—For a more extensive description of various metallographic techniques, refer to Samuels, Petzow, and VanderVoort.[4]

5.6.3 Samples shall be cleaned with a cotton swab under running water to remove the alumina particle film, placed on a short nap micro-cloth with a slurry of 30 g of 0.05 µm alumina abrasive and 500 mL of distilled water, and polished until a scratch-free surface is obtained. Again a 350-g weight is used to augment polishing. Specimens usually require 25 to 30 min to produce a satisfactory final polish. The specimen can usually be polished an additional 10 to 15 min without producing harmful over-polishing effects, but too much time may create relief on samples which are narrow across the polished surface.

5.7 *Surface Preparation:*

5.7.1 The surface, prior to etching, shall:

5.7.1.1 Be free from scratches, stains, and other imperfections which mar the surface,

5.7.1.2 Retain all non-metallic inclusions intact, and

5.7.1.3 Not exhibit any appreciable relief effect between micro-constituents.

5.8 *Electrolytic Etching Procedures:*

5.8.1 Structural components of an alloy are revealed during etching by a preferential attack or staining of the various constituents by the reagents. This is due to differences in the chemical composition of the phases and attending rates of solution. Immediately prior to etching, specimens shall be lightly polished (using 0.05 µm or equivalent substitute) and swabbed with cotton under running water to remove any air-formed oxide film, to reduce chances of staining.

5.8.2 Place the specimen immersed face up in the etching reagent. The cathode is placed approximately one inch from the specimen, and the anode is put in contact with the sample. During etching, the cathode is moved to assure a uniform action of the etching reagent on the specimen. The sample is then washed and repolished lightly, if needed, to remove any traces of disturbed metal on the surface, and then re-etched.

5.8.3 *Etchant:*

5.8.3.1 *Option A*—10 % chromic acid in Specification D1193 water.

5.8.3.2 *Option B*—5 g oxalic acid mixed with 95 mL HCl (reagent grade).

5.8.4 *Etching Parameters:*

5.8.4.1 Electrolytic—6 volts DC.

5.8.4.2 Cathode, Carbon or Stainless Steel may be used.

5.8.4.3 Stainless anode probe.

5.8.5 *Etching Time:*

5.8.5.1 *Option A*—1 to 5 s, depending on heat treated condition and size of sample.

5.8.5.2 *Option B*—20 to 25 s, depending on heat treated condition and size of sample.

5.8.6 *Sample Polishing:*

5.8.6.1 The sample must have a fresh polish. If the surface has been dry, even for a few seconds, give the sample 6 to 10 laps on soft nylon-type cloth with 0.05 µm alumina final polishing compound, then place directly under running water and swab with a cotton pad. The sample surface must be kept wet.

5.8.6.2 Place sample face up in etchant. With good overhead light to visually see sample surface: make contact at end or corner of sample with anode probe or wire lead tacked to the back of the specimen, dip carbon cathode into etchant, watch to see any surface change, and break contact when finished. Before removing sample from etchant, agitate it to remove any film on surface. Pull sample and put it under running water. Rinse with methanol, then place sample under forced hot air dryer until it is thoroughly dry.

5.8.6.3 If etch is too light and needs to be heavier, do not take sample back to running water and then into etchant. Instead, it must go back to the final cloth for 6 to 10 laps making sure that no part of surface dries; failure to do this can, and most likely

---

[4] Samuels, L. E., *Metallographic Polishing by Mechanical Methods*, American Society for Metals (ASM), Metals Park, OH, 3rd Ed., 1982; Petzow, G., *Metallographic Etching*, ASM, 1978; and VanderVoort, G., *Metallography: Principles and Practice*, McGraw Hill, NY, 2nd Ed., 1999.

will, result in staining. If the sample does stain do not try to remove stain on final cloth. Rather, go back to the papers (at least to the 400 and 600 grit), then 5 to 9 µm diamond and then to 0.05 µm alumina, again, keeping sample surface wet. Repeat as described before.

NOTE 2—The use of either 3 µm diamond or 0.3 µm alumina in the polishing procedure in 5.8.6.3 may result in scratches remaining on the polished surface of the mounted specimen.

~~5.9 *Immersion Etching:*~~

~~5.9.1 The preferred etchant for this family of alloys is chrome-regia (1 part chromic acid to 3 parts reagent grade HCl). Stock chromic acid is made by mixing 300 g chromic acid with 300 mL of hot (32 to 60°C (90 to 140°F)) Specification D1193 water.~~

~~5.9.2 For immersion etching, it is equally important to work with a wet, freshly polished surface (that is, follow step 5.8.3.1 above for electrolytic etching). The wet sample is then immersed face up into the chrome regia for 1 to 3 s, depending on heat treated condition and sample size. Then pull sample, put under running water, rinse with methanol, and blow dry. If etch is too light, follow procedure described in step 5.8.6.3 above.~~

6. Examination and Evaluation

6.1 A visual examination and photographic reproduction of specimen surface is compared to photomicrographs in Fig. 2 (*a to n*) and Fig. 3 (*a to j*) for microstructures exhibiting the absence and presence, respectively, of significant mu phase. A magnification of 200× shall be used for metallographic evaluation. If any of the evaluated triplicate test specimens are considered rejectable for the presence of mu phase, the tested material shall be considered rejectable.

6.2 Microstructures shown in Fig. 2 (*a to n*) are considered *Acceptable*, reflecting the absence of significant mu phase.

6.3 Microstructures shown in Fig. 3 (*a to j*) are considered *Rejectable*, reflecting the presence of significant mu phase.

7. Report

7.1 The specimen size, source, and identification.

7.2 The test sample orientation per Fig. 1.

7.3 The etching procedure: Electrochemical (Option A or ~~B) or Immersion Etching.~~B).

7.4 The 200× magnification used for metallographic evaluation.

7.5 Identify deviations from this practice.

7.6 The photomicrograph(s) in Fig. 2 or Fig. 3, which most closely represent the evaluated test specimen.

7.7 *Acceptable* or *Rejectable* microstructure, based on comparison to photomicrographs in Fig. 2 or Fig. 3.

8. Glossary of Acceptable and Rejectable Microstructures

8.1 See Fig. 2 and Fig. 3.

9. Keywords

9.1 corrosion; ferric chloride test solution; localized corrosion; microstructure; mu-phase; nickel–base alloys; pitting; properties; stainless steels