Age-hardened Nickel-based Alloys for Oil and Gas Drilling and Production Equipment

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Introduction

This standard for Age-Hardened Nickel-Based Alloys was formulated by the API Committee on Standardization of Oilfield Equipment and Materials (CSOEM), Subcommittee on Valves and Wellhead Equipment (SC6), Materials Task Group. It is based on the conclusions of a task group evaluation of requirements needed for Age-Hardened Nickel-Based Alloys to supplement the existing requirements of API Specification 6A, *Specification for Wellhead and Christmas Tree Equipment*.

Age-hardened Nickel-based Alloys for Oil and Gas Drilling and Production Equipment

1 Scope

1.1 Purpose

This document provides requirements for age-hardened nickel-base alloys that are intended to supplement the existing requirements of API 6A. For downhole applications refer to API 5CRA.

These additional requirements include detailed process control requirements and detailed testing requirements. The purpose of these additional requirements is to ensure that the age-hardened nickel-base alloys used in the manufacture of API 6A pressure-containing and pressure-controlling components are not embrittled by the presence of an excessive level of deleterious phases and meet the minimum metallurgical quality requirements.

1.2 Applicability

This standard is intended to apply to pressure-containing and pressure-controlling components as defined in API 6A. Requirements of this standard may be applied by voluntary conformance by a manufacturer, normative reference in API 6A or other product specification(s), or by contractual agreement.

2 Normative References

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

API Specification 6A, *Specification for Wellhead and Christmas Tree Equipment*

ASTM A370 [1], *Standard Test Methods and Definitions for Mechanical Testing of Steel Products*

ASTM A604, *Standard Practice for Macroetch Testing of Consumable Electrode Remelted Steel Bars and Billets*

ASTM B880, *Standard Specification for General Requirements for Chemical Check Analysis Limits for Nickel, Nickel Alloys and Cobalt Alloys*

ASTM E10, *Standard Test Method for Brinell Hardness Test of Metallic Materials*

ASTM E18, *Standard Test Methods for Rockwell Hardness of Metallic Materials*

ASTM E29*, Standard Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications*

ASTM E110*, Standard Test Method for Rockwell and Brinell Hardness of Metallic Materials by Portable Hardness Testers*

ASTM E112, *Standard Test Methods for Determining Average Grain Size*

ASTM E354, Standard *Test Methods for Chemical Analysis of High-Temperature Electrical, Magnetic, and Other Similar Iron, Nickel, and Cobalt Alloys*

[1] ASTM International, 100 Barr Harbor Drive, West Conshohocken, Pennsylvania 19428-2959. www.astm.org

ASTM E1019, *Standard Test Methods for Determination of Carbon, Sulfur, Nitrogen, and Oxygen in Steel, Iron, Nickel, and Cobalt Alloys by Various Combustion and Fusion Techniques*

ASTM E1086, *Standard Test Method for Analysis of Austenitic Stainless Steel by Spark Atomic Emission Spectrometry*

ASTM E1181, *Standard Test Methods for Characterizing Duplex Grain Sizes*

ASTM E1473, *Standard Test Methods for Chemical Analysis of Nickel, Cobalt, and High-Temperature Alloys*

ASTM E1823, *Standard Terminology Relating to Fatigue and Fracture Testing*

ASTM E2465, *Standard Test Method for Analysis of Ni-Base Alloys by Wavelength Dispersive X-Ray Fluorescence Spectrometry*

SAE AMS 2750 [2], *Pyrometry*

ANSI/NACE *MR0175* [3], *Petroleum and natural gas industries—Materials for use in H_2S-containing environments in oil and gas production*

3 Terms, Definitions, and Abbreviated Terms

3.1 Terms and Definitions

For the purposes of this document, the following definitions apply.

3.1.1
deleterious phases
Phases present in the microstructure of an alloy that have a negative effect on the desired mechanical properties, toughness, or corrosion resistance of the alloy.

3.1.2
field of view
A 0.008 in. (0.203 mm) × 0.010 in. (0.254 mm) area of the microstructure.

NOTE The 0.008 in. (0.203 mm) × 0.010 in. (0.254 mm) area is representative of the area shown in a 4 in. (102 mm) × 5 in. (127 mm) photomicrograph taken at 500X.

3.1.3
heat treat lot
Material from the same remelt ingot; processed together using the same procedures and hot/cold work equipment; same nominal dimensions (excluding length); and heat treated in the same furnace loads, including solution annealing, aging and, if applicable, re-heat treatment.

3.1.4
total hot work reduction ratio
The product of the individual reduction ratios achieved at each step in the hot work operation from ingot cross-section to final hot work cross-section, where the ingot cross-section shall be the cross-section of the ingot obtained after the last remelt step and any ingot grinding or surface preparation prior to hot working.

[2] SAE International (formerly the Society of Automotive Engineers), 400 Commonwealth Drive, Warrendale, Pennsylvania 15096-0001, www.sae.org.

[3] NACE International (formerly the National Association of Corrosion Engineers), 1440 South Creek Drive, Houston, Texas 77084-4906, www.nace.org.

3.2 Abbreviated Terms

For the purpose of this standard, the following abbreviated terms apply:

AOD	argon oxygen decarburization
EF	electric furnace (or electronic arc furnace)
EFR	electroflux remelting (same as ESR)
ESR	electro-slag remelting
HBW	Brinell hardness
HRC	Rockwell hardness scale C
PSL	product specification level
QTC	qualification test coupon
VAR	vacuum arc remelting
VIM	vacuum induction melting
VOD	vacuum oxygen decarburization

4 Requirements for Age-hardened Nickel-based Alloys

4.1 Process Control Requirements

4.1.1 Chemical Composition Requirements

4.1.1.1 Chemical Composition Limits

The chemical composition for the alloys covered by this International Standard shall conform to the weight percent requirements for the applicable alloy as identified in Table 1. Rounding of chemical composition for acceptance purposes shall be in accordance with ASTM E29.

4.1.1.2 Chemical Composition Frequency and Test Methods

The chemical composition shall be tested on a remelt ingot basis on product representative of a remelt ingot per ASTM E354, ASTM E1019, ASTM E1086, ASTM E1473, and ASTM E2465 or a nationally or internationally recognized industry standard.

4.1.1.3 Check (Product) Analysis

When material is qualified by the use of a check (product) analysis performed on either the qualification test coupon (QTC) or a non-critical area of the production material, the analysis shall conform to the check (product) analysis variation specified in ASTM B880.

4.1.2 Melt Practice Requirements

4.1.2.1 Acceptable Melt Practices

4.1.2.1.1 General

The acceptable melting practices for the alloys covered by this standard are specified in 4.1.2.1.2, 4.1.2.1.3, and 4.1.2.1.4.

Table 1—Chemical Composition [d,e]

Element	UNS Number					
	N07716	N07718	N07725	N09925	N09935	N09945 [c]
Ni	59.0 to 63.0	50.0 to 55.0	55.0 to 59.0	42.0 to 46.0	35.0 to 38.0	45.0 to 55.0
Cr	19.0 to 22.0	17.0 to 21.0	19.0 to 22.5	19.5 to 22.5	19.5 to 22.0	19.5 to 23.0
Fe [a]	Balance	Balance	Balance	22 min	Balance	Balance
Nb	2.75 to 4.00	—	2.75 to 4.00	0.08 to 0.50	0.20 to 1.00	2.50 to 4.50
Cb(Nb) + Ta	—	4.87 to 5.20	—	—	—	—
Mo	7.00 to 9.50	2.80 to 3.30	7.00 to 9.50	2.50 to 3.50	3.00 to 5.00	3.00 to 4.00
Ti	1.00 to 1.60	0.80 to 1.15	1.00 to 1.70	1.90 to 2.40	1.80 to 2.50	0.50 to 2.50
Al	0.35 max	0.40 to 0.60	0.35 max	0.10 to 0.50	0.50 max	0.01 to 0.70
C	0.030 max	0.045 max	0.030 max	0.025 max	0.030 max	0.005 to 0.040
Co	—	1.00 max	—	—	1.00 max	—
Mn	0.20 max	0.35 max	0.35 max	1.00 max	1.00 max	1.00 max
Si	0.20 max	0.35 max	0.20 max	0.35 max	0.35 max	0.50 max
P	0.015 max	0.010 max	0.015 max	0.020 max	0.025 max	0.030 max
S	0.010 max	0.010 max	0.010 max	0.003 max	0.001 max	0.010 max
B	0.006 max	0.0060 (60 ppm) max	—	—	—	—
Cu	0.23 max	0.23 max	—	1.50 to 3.00	1.00 to 2.00	1.50 to 3.00
Pb	0.001 max	0.0010 (10 ppm) max	—	—	—	—
Se	—	0.0005 (5 ppm) max	—	—	—	—
Bi	—	0.00005 (0.5 ppm) max	—	—	—	—
Ca [b]	—	0.0030 (30 ppm) max	—	—	—	—
Mg [b]	—	0.0060 (60 ppm) max	—	—	—	—
W	—	—	—	—	1.00 max	—

[a] Shall be determined arithmetically by difference or by direct measurement.

[b] To be determined if intentionally added.

[c] For UNS N09945, material designation 125K, it is recommended that the chemistry be modified to 46.5 to 48.0 Ni and 2.80 to 3.50 Nb. For UNS N09945 material designation 140K, it is recommended that the chemistry be modified to 52.0 to 55.0 Ni and 3.80 to 4.50 Nb.

[d] Some limits are more restrictive than the UNS chemistry limits to suppress formation of deleterious phases.

[e] "—" Value not required.

4.1.2.1.2 UNS N07718

One of the following sequences of processes shall be utilized.

a) Step 1—Basic electric furnace (EF).

   Step 2—Either argon oxygen decarburization (AOD) or vacuum oxygen decarburization (VOD).

   Step 3—Vacuum arc remelting (VAR).

   Step 4—VAR.

or:

b) Step 1—Vacuum induction melting (VIM).

   Step 2—Either electroslag remelting (ESR), electroflux remelting (EFR), or VAR.

   Optional Step 3—ESR, EFR, or VAR.

4.1.2.1.3 UNS N09925, UNS N09935, and UNS N09945

One of the following sequences of processes shall be utilized.

a) Step 1—Basic EF.

   Step 2—Either AOD, VOD, or vacuum degassing.

   Step 3—Either ESR, EFR, or VAR.

   Optional Step 4—VAR

or:

b) Step 1—VIM.

   Step 2—Either ESR, EFR, or VAR.

   Optional Step 3—VAR

4.1.2.1.4 UNS N07716 and UNS N07725

One of the following sequences of processes shall be utilized.

a) Step 1—Basic EF.

   Step 2—Either AOD or VOD.

   Step 3—VAR.

   Optional Step 4—VAR.

or:

b) Step 1—VIM.

Optional Step 2—ESR or EFR.

Step 3—VAR.

Optional Step 4—VAR.

4.1.3 Forging and Hot Working Requirements

The minimum total hot work reduction ratio shall be 4.0:1.

4.1.4 Heat Treating Requirements

4.1.4.1 Heat Treating Equipment Qualification and Calibration

The practice for qualification of heat-treating equipment shall be per the API 6A annex for qualification of heat-treating equipment, AMS 2750, or another internationally recognized standard. Furnace instrumentation shall be calibrated at least every 3 months with acceptable grace period as defined in AMS 2750 or other internationally recognized standard. Furnaces shall be surveyed no less than once a year. When a furnace is moved, repaired or rebuilt refer to API 6A for survey requirements. When the API 6A annex for qualification of heat-treating equipment is selected as the basis for furnace calibration, the requirements of the API 6A annex shall be treated as normative.

4.1.4.2 Temperature Monitoring

The material temperature shall be measured by use of either a contact surface thermocouple or a heat sink as described in API 6A. The hold time shall not commence until the contact surface thermocouple or a heat sink reaches at least the minimum required material temperature.

The material manufacturer or material supplier shall maintain copies of the heat treating charts showing the material temperature as measured by the contact surface thermocouple or heat sink for 5 years minimum following the date of heat treatment.

4.1.4.3 Solution Annealing and Age Hardening

The production material and QTC(s) shall be solution annealed and age hardened in accordance with the procedures in Table 2.

Optional re-heat treatment steps—complete re-heat treatment is permitted. Alternatively, re-aging within the parameters of Table 2 is permitted, provided the cumulative aging time does not exceed the maximum allowable aging time. All other requirements shall be met by material that has been subjected to either of these optional steps.

If the material is re-aged without re-solution annealing, the testing required by 4.2.2, 4.2.3, 4.2.4, and 4.2.5 may be performed on remnants of the original QTC, provided the remnants are re-aged with the production material they represent.

Table 2—Heat Treatment Procedures

UNS	Material Designation	Solution Annealing		Age Hardening [b]
		Material Temperature	Time (hours)	Material Temperature and Time
N07716	120K 140K	1875 °F to 1925 °F (1024 °C to 1052 °C)	0.5 to 4 [b]	1310 °F to 1455 °F (710 °C to 790 °C) for 4-9 hours, furnace cool to 1125 °F to 1275 °F (607 °C to 690 °C) and hold for total aging time of 12 hours minimum
N07718	120K	1870 °F to 1925 °F (1021 °C to 1052 °C)	1 to 2.5 [a]	1425 °F to 1475 °F (774 °C to 802 °C) for 6 to 8 hours
	140K	1870 °F to 1925 °F (1021 °C to 1052 °C)	1 to 2.5 [a]	1400 °F to 1475 °F (760 °C to 802 °C) for 6 to 8 hours
N07725	120K	1875 °F to 1950 °F (1024 °C to 1065 °C)	0.5 to 4 [b]	1325 °F -1425 °F (720 °C to 774 °C) for 4 to 9 hours, furnace cool to 1125 °F to 1275 °F (607 °C to 690 °C) and hold for total aging time of 12 hours minimum
N09925	110K	1825 °F to 1925 °F (996 °C to 1052 °C)	0.5 to 4 [a]	1325 °F -1400 °F (720 °C to 760 °C) for 4 to 9 hours, furnace cool to 1125 °F to 1220 °F (607 °C to 660 °C) and hold for total aging time of 12 hours minimum
N09935	110K	1870 °F to 1975 °F (1021 °C to 1080 °C)	0.5 to 4 [a]	1345 °F to 1435 °F (730 °C to 780 °C) for 4 to 9 hours, furnace cool to 1165 °F to 1255 °F (630 °C to 680 °C) and hold for total aging time of 12 hours minimum
N09945	125K 140K	1800 °F to 1950 °F (982 °C to 1065 °C)	0.5 to 4 [a]	1250 °F to 1350 °F (677 °C to 732 °C) for 4 to 9 hours furnace cool to 1110 °F to 1190 °F (599 °C to 643 °C) and hold for total aging time of 12 hours minimum

[a] Cool in air, inert gas, water, polymer, or oil to ambient temperature. Air cooling or inert gas cooling of section thickness greater than 3 in. (76 mm) shall only be upon agreement between purchaser, manufacturer and end user.

[b] Cool in air, inert gas, water, polymer or oil to ambient temperature.

4.2 Testing Requirements

4.2.1 Macroetch Requirements

4.2.1.1 Test Location, Method, and Frequency

A macroetch examination shall be performed. The macroetch examination shall be performed on either (a) or (b) as shown below.

a) Full transverse cross-section slices representative of the top and bottom of each final remelt ingot or product thereof.

b) For product not tested by the mill and not identified as to its relative location within the ingot, the macroetch testing shall be performed on a per billet, bar or other raw material product form basis. A full transverse cross-section slice shall be examined from each end.

The full cross-section slices shall be etched for examination. The acceptable etchants are as shown in Table 3.

Table 3—Etchants Used for Macroetch Examination

Option	Composition
Option A: Canada's Etchant	100 ml H_2SO_4, 100 ml HF, 50 ml HNO_3, 400 ml H_2O Etch at 160 °F to 180 °F (71 °C to 82 °C)
Option B: Aqua Regia	200 ml HCl, 100 ml HNO_3
Option C: Kalling's No. 2 (Waterless Kalling's)	200 ml Methanol, 200 ml HCl, 10 g $CuCl_2$
Option D: Hydrochloric—Peroxide	H_2O_2 (30 %) 100 ml, HCl 200 ml, H_2O 300 ml Remove stains with 50 % HNO_3
Option E: Dilute Heated Aqua Regia	250 ml HCl, 10 to 20 ml HNO_3 Etch at 140 °F to 165 °F (60 °C to 74 °C)

4.2.1.2 Macroetch Examination and Acceptance Criteria

The macrostructure of the slice shall be examined and rated to all four classes in ASTM A604. The acceptance criteria are as follows:

— Class 1 (freckles)—no worse than Severity A;

— Class 2 (white spots)—no worse than Severity A;

— Class 3 (radial segregation)—no worse than Severity A;

— Class 4 (ring pattern)—no worse than Severity A.

4.2.2 Microstructural Analysis Requirement

4.2.2.1 Test Location, Method and Frequency

Sample(s) shall be from material with the same shape and equivalent round (ER) from each remelt ingot per heat treat lot (see 3.1.3) and shall be subjected to a microstructural analysis. The sample shall be either a prolongation (full cross-section on thickest end) or sacrificial production part.

The sample(s) to be examined shall be a minimum $^1/_4$ in. (6 mm) square and oriented longitudinally to the primary axis of grain flow. If the cross-section of the material is less than $^1/_4$ in. (6 mm), then the sample(s) shall be full cross-section. The microstructural analysis shall be performed on material in the final heat treatment condition. Test locations shall be a minimum of 1.25 in. (32 mm) from a heat treated end surface.

For solid material, the center, $^1/_4$ thickness and surface locations shall be evaluated. For hollow material, the mid-wall location and both the inner and outer surfaces shall be evaluated.

The microstructural samples shall be etched for examination. The acceptable etchants are as shown in Table 4.

The material manufacturer or material supplier shall retain the metallographic specimen mounts for 5 years minimum following the date of the examination.

Table 4—Etchants Used for Microstructure Examination

UNS Number	Option	Composition
All UNS Numbers	Option A: Kalling's No. 2 (Waterless Kalling's)	200 ml Methanol, 200 ml HCl, 10 g $CuCl_2$
All UNS Numbers	Option B: Seven acids	300 ml HCl, 60 ml HNO_3, 60 ml H_3PO_4, 30 ml 40% HF, 30 ml H_2SO_4, 30 g $FeCl_3$ (anhydrous), 60 ml CH_3COOH, 300 ml H_2O
All UNS Numbers	Option C: Diluted Glyceregia	10 ml glycerol, 150 ml HCl, 15 ml HNO_3
UNS N09925, UNS N09935, UNS N07725, UNS N07716	Option D: Bromine-Methanol	Clean in HCl before etching in immersed 1 % to 3 % Bromine, Methanol
UNS N09925	Option E: Nitric-HCl	10 ml HNO_3, 60 ml HCl

4.2.2.2 Grain Size Evaluation

4.2.2.2.1 Grain Size

The average grain size shall be determined in accordance with ASTM E112. The ASTM average grain size shall be No. 2 or finer.

4.2.2.2.2 Duplex Grain Size

No topological duplex grain size, as defined and measured per ASTM E1181, is allowed.

4.2.2.3 Metallographic Examination for Deleterious Phases

The microstructural samples shall be examined for deleterious phases. The microstructural samples shall be examined at 100X and 500X using light microscopy.

The acceptance criteria are as follows.

a) The microstructure for all alloys shall be free from acicular phases except in individual, isolated grains that are not representative of the bulk microstructure. In no case shall any individual grain be surrounded with acicular phases.

b) The microstructures shall be free from continuous networks of secondary phases along grain boundaries, except for individual, isolated grains or isolated fields of view (see 3.1.2) that are not representative of the bulk microstructure. The presence of discrete, isolated particles of secondary phases or carbides is acceptable.

c) There shall be no Laves phase.

NOTE Examination of the microstructural samples for Laves phase is not required if the final melt source certifies that the material is free from Laves phase.

The reference photomicrographs in Figure A.1 through Figure A.17 in Annex A are examples of acceptable and unacceptable microstructures.

Material that is rejected for unacceptable microstructural features may be fully re-heat treated (solution annealed and age hardened) in accordance with 4.1.4 and re-examined.

If a heat treat lot is rejected, then the other pieces within the heat treat lot may be qualified on an individual piece basis if both ends are examined and meet the microstructural requirements. Material containing rejectable metallographic locations may be accepted if both ends are examined and if it is demonstrated that the rejectable locations will be removed with final machining.

4.2.3 Tensile Property Requirements

4.2.3.1 Test Location, Method, and Frequency

One tensile test shall be performed for each tested QTC. The test frequency shall be one test per remelt ingot per heat treat lot (see 3.1.3).

The QTC shall be either a prolongation (full cross-section on thickest end) or sacrificial production part.

For solid material, the test specimen shall be removed from a location at $^1/_4$ thickness or deeper from the side or outer diameter and at least 1.25 in. (32 mm) from the end. For hollow material, the test specimen shall be removed from a mid-wall location and at least 1.25 in. (32 mm) from the end.

The test specimen and test method shall be in accordance with ASTM A370. Rounding of test results to determine conformance to specification shall be in accordance with ASTM E29.

4.2.3.2 Tensile Test Acceptance Criteria

The tensile properties shall meet the acceptance criteria as shown in Table 5.

4.2.3.3 Retesting

If the results of the tensile test(s) do not satisfy the applicable requirements, two additional tests on two additional specimens (removed from the same QTC with no additional heat treatment) may be performed in an effort to qualify the material. The results of each of these tests shall satisfy the applicable requirements.

4.2.4 Impact Toughness Requirements

4.2.4.1 Test Location, Method, and Frequency

Charpy V-notch impact testing shall be performed on all material in accordance with ASTM A370. All tests shall be performed at or below –75 °F (–60 °C) regardless of API 6A temperature classification.

Impact testing shall be performed on a set of three specimens. Specimens oriented transverse to the primary direction of grain flow shall be used unless the size or geometry of the QTC prevents the usage of transverse specimens (material less than 3 in. [76 mm] in cross-section). For transverse specimens, the orientation shall be either C-L or T-L and for longitudinal specimens, the orientation shall be either L-C or L-T. See ASTM E1823 for specimen orientation.

One set of Charpy V-notch impact tests shall be performed for each tested QTC. The test frequency shall be one set of tests per heat per remelt ingot per heat treat lot (see 3.1.3).

The QTC shall be either a prolongation (full cross-section on thickest end) or sacrificial production part.

For solid material, the test specimens shall be removed from a location at $^1/_4$ thickness or deeper from the side or outer diameter and at least 1.25 in. (32 mm) from the end. For hollow material, the test specimens shall be removed from a mid-wall location from the side and at least 1.25 in. (32 mm) from the end.

Table 5—Tensile Requirements

UNS number	**Material Designation**	**QTC Cross-Section Thickness *** in. (mm)	**0.2 % Yield Strength Min.** ksi (MPa)	**0.2 % Yield Strength Max.** ksi (MPa)	**Tensile Strength Min.** ksi (MPa)	**Elongation in 4D Min.** %	**Reduction of Area Min** %
N07716	120K	≤10 (254)	120 (827)	150 (1034)	150 (1034)	20	35
		>10 (254)	120 (827)	150 (1034)	150 (1034)	20	25
	140K	≤10 (254)	140 (965)	160 (1103)	165 (1138)	18	30
		>10 (254)	140 (965)	160 (1103)	165 (1138)	18	20
N07718	120K	≤10 (254)	120 (827)	145 (1000)	150 (1034)	20	35
		>10 (254)	120 (827)	145 (1000)	150 (1034)	20	25
	140K	≤10 (254)	140 (965)	150 (1034)	165 (1138)	20	35
		>10 (254)	140 (965)	150 (1034)	160 (1103)	20	25
N07725	120K	≤10 (254)	120 (827)	150 (1034)	150 (1034)	20	35
		>10 (254)	120 (827)	150 (1034)	150 (1034)	20	25
N09925	110K	≤10 (254)	110 (758)	140 (965)	140 (965)	18	25
		>10 (254)	110 (758)	140 (965)	140 (965)	18	20
N09935	110K	≤10 (254)	110 (758)	140 (965)	140 (965)	18	25
		>10 (254)	110 (758)	140 (965)	140 (965)	18	20
N09945	125K	≤10 (254)	125 (862)	155 (1068)	150 (1034)	18	25
		>10 (254)	125 (862)	155 (1068)	150 (1034)	18	20
N09945	140K	≤10 (254)	140 (965)	165 (1138)	165 (1138)	18	25
		>10 (254)	140 (965)	165 (1138)	165 (1138)	18	20

* QTC cross-section thickness at time of heat treatment.

The test specimens and test method shall be in accordance with ASTM A370. Rounding of test results to determine conformance to specification shall be in accordance with ASTM E29.

4.2.4.2 Charpy V-notch Acceptance Criteria

The average energy value for a set of three specimens shall meet or exceed the specified average. No more than one of the specimens shall have an energy value below the specified average and it shall not be below the specified single minimum. No specimens shall have a lateral expansion below the specified value.

The adjustment factors for sub-size impact specimens in API 6A shall apply to the absorbed energy values for all product specification levels (PSLs). Lateral expansion shall be as stated in the Table 6 regardless of specimen size.

4.2.4.3 Retesting

If a test fails, then a retest of three additional specimens removed from the same QTC with no additional heat treatment may be made, each of which shall exhibit an impact value equal to or exceeding the minimum average value.

Table 6—Charpy V-notch Impact Toughness Requirements (10 mm × 10 mm)

UNS number	Material Designation	QTC Cross Section [a] Thickness in. (mm)	Orientation [b]	Minimum Average ft-lbs (J)	Minimum Single ft-lbs (J)	Lateral Expansion in. (mm)
N07716	120K	<3 (76)	Longitudinal	40 (54)	35 (47)	0.015 (0.38)
		≥3 (76) to 10 (254)	Transverse	37 (50)	32 (43)	0.015 (0.38)
		>10 (254)	Transverse	32 (43)	27 (37)	0.015 (0.38)
	140K	<3 (76)	Longitudinal	40 (54)	35 (47)	0.015 (0.38)
		≥3 (76) to 10 (254)	Transverse	30 (41)	27 (37)	0.015 (0.38)
		>10 (254)	Transverse	30 (41)	27 (37)	0.015 (0.38)
N07718	120K	<3 (76)	Longitudinal	50 (68)	45 (61)	0.015 (0.38)
		≥3 (76) to 10 (254)	Transverse	35 (47)	30 (41)	0.015 (0.38)
		>10 (254)	Transverse	30 (41)	27 (37)	0.015 (0.38)
	140K	<3 (76)	Longitudinal	50 (68)	45 (61)	0.015 (0.38)
		≥3 (76) to 10 (254)	Transverse	35 (47)	30 (41)	0.015 (0.38)
		>10 (254)	Transverse	30 (41)	27 (37)	0.015 (0.38)
N07725	120K	<3 (76)	Longitudinal	40 (54)	35 (47)	0.015 (0.38)
		≥3 (76) to 10 (254)	Transverse	37 (50)	32 (43)	0.015 (0.38)
		>10 (254)	Transverse	32 (43)	27 (37)	0.015 (0.38)
N09925	110K	<3 (76)	Longitudinal	35 (47)	32 (43)	0.015 (0.38)
		≥3 (76) to 10 (254)	Transverse	35 (47)	32 (43)	0.015 (0.38)
		>10 (254)	Transverse	35 (47)	32 (43)	0.015 (0.38)
N09935	110K	<3 (76)	Longitudinal	35 (47)	30 (41)	0.015 (0.38)
		≥3 (76) to 10 (254)	Transverse	30 (41)	25 (34)	0.015 (0.38)
		>10 (254)	Transverse	25 (34)	20 (27)	0.015 (0.38)
N09945	125K	<3 (76)	Longitudinal	50 (68)	45 (61)	0.015 (0.38)
		≥3 (76) to 10 (254)	Transverse	40 (54)	35 (47)	0.015 (0.38)
		>10 (254)	Transverse	30 (41)	27 (37)	0.015 (0.38)
	140K	<3 (76)	Longitudinal	45 (61)	40 (54)	0.015 (0.38)
		≥3 (76) to 10 (254)	Transverse	35 (47)	30 (41)	0.015 (0.38)
		>10 (254)	Transverse	30 (41)	27 (37)	0.015 (0.38)

[a] QTC cross-section thickness at time of heat treatment.

[b] See 4.2.4.1 for specific requirements regarding the orientation.

4.2.5 Hardness Test Requirements

4.2.5.1 Test Location, Method and Frequency

4.2.5.1.1 Production Material

Each piece of production material shall be hardness tested on or near the surface using the Rockwell C scale method per ASTM E18 or ASTM E110, or the Brinell (10 mm ball, 3000 kgf) method per ASTM E10 or ASTM E110 after the final heat treatment cycle. The surface may be prepared using light grinding. When light grinding is used it shall be to a maximum depth of 0.125 in. (3.18 mm).

Alternatively, for each piece of production material, near surface hardness testing on a cross-section shall be acceptable. Near surface test shall be 0.100 to 0.150 in. (2.54 mm to 3.81 mm) from the surface, and shall be performed using the Rockwell C scale method per ASTM E18.

For Rockwell C-scale testing, three adjacent indentations shall be made, averaged to calculate the mean, and the mean shall meet the hardness limits in Table 7. No individual hardness number may be greater than 2 HRC units above the maximum specified hardness number. For Brinell testing, one indentation is sufficient. For requirements on conversion and reporting of hardness measurements, see Table 7, footnote a. In case of dispute, the Rockwell C-scale shall be the referee method and shall take precedence.

4.2.5.1.2 Qualification Test Coupon (QTC)

The QTC shall be either a prolongation (full cross-section on thickest end) or sacrificial production part.

Cross-section hardness testing shall be performed on each QTC using the Rockwell C scale method per ASTM E18 or ASTM E110. For solid material, the center, 1/4 thickness and near surface locations shall be evaluated. For hollow material, the mid-wall location and both near the inner and outer surfaces shall be evaluated. At each location three adjacent indentations shall be performed, and the mean hardness number from each location shall meet the requirements in Table 7. The hardness test location for the near surface test shall be 0.100 - 0.150 in. (2.54 mm - 3.81 mm) from the surface. All hardness indentations shall be reported. No individual hardness indentation may be greater than 2 HRC units above the maximum specified hardness number.

4.2.5.1.3 Rounding

Rounding of test results to determine conformance to specification shall be in accordance with ASTM E29.

4.2.5.2 Hardness Test Acceptance Criteria

The hardness tests shall meet the acceptance criteria shown in Table 7.

4.2.5.3 Retests

4.2.5.3.1 Production Material Surface Testing

If any Rockwell hardness requirements are not met, three additional Rockwell C scale indentations shall be made in the immediate area to determine new hardness numbers. If any Brinell hardness requirements are not met, additional Brinell tests may be taken in the immediate area. If one or more of the re-test indentations do not meet hardness requirements, then the piece is rejected.

At the manufacturer's option, for pieces where the cross-section hardness testing was not previously performed, the rejected piece may be qualified by performing cross-section hardness tests in accordance with 4.2.5.1.2.

Table 7—Hardness Requirements

UNS number	Material Designation	Minimum Hardness HRC (HBW)	Maximum Hardness HRC (HBW)
N07716	120K	32 ([a])	43 [c] ([a])
	140K	34 ([a])	43 [c] ([a])
N07718	120K	32 ([a])	40 ([a])[b]
	140K	34 ([a])	40 ([a])[b]
N07725	120K	32 ([a])	43 [c] ([a])
N09925	110K	26 ([a])	38 ([a])[b]
N09935	110K	24 ([a])	34 ([a])[b]
N09945	125K	32 ([a])	42 ([a])[b]
	140K	34 ([a])	42 ([a])[b]

[a] The conversion of hardness readings to or from other scales is material-dependent. Rockwell C scale is the preferred hardness method for hardness testing the family of alloys covered by API 6ACRA since compliance with ANSI/NACE MR0175 is frequently required, and ANSI/NACE MR0175 specifies the maximum acceptable hardness limits using the Rockwell C scale. When conversions from other hardness scales to the Rockwell C scale are required or vice versa, one of two methods shall be used:

— a hardness conversion agreed to by the equipment manufacturer and the end user;

— ASTM E140 conversion.

When a conflict exists between Rockwell C scale hardness numbers and Brinell hardness numbers, the Rockwell C scale shall be the referee method.

When a conversion other than the ASTM E140 conversion is utilized, the conversion method shall be documented and traceable to test results.

In accordance with ASTM E140, when reporting converted hardness numbers, the measured hardness and test scale shall be reported in parentheses. For example, 20.0 HRC (228 HBW), where 20.0 HRC is the converted hardness value and 228 HBW is the original measurement value and test scale.

[b] Maximum hardness limits shall be in accordance with ANSI/NACE MR0175.

[c] The maximum hardness limits presented in Table 7 represent the lowest maximum hardness values specified in ANSI/NACE MR0175 for the individual alloys. See ANSI/NACE MR0175 for hardness limits for specific temperature and/or elemental sulfur environments. These may be used in lieu of the values in the table within the ANSI/NACE MR0175 environmental limits.

4.2.5.3.2 Cross-section Testing

If any hardness requirements are not met, three additional indentations shall be made in the immediate area to determine new hardness indentations, and new mean hardness numbers for each location. If each new hardness indentation meets the requirements of this International Standard, then the piece is qualified.

If one or more of the indentations do not meet hardness requirements, then the piece is rejected.

4.2.6 Nondestructive Examination

The nondestructive examination requirements in API 6A shall apply as required for the specified component type and PSL specified on the purchase order.

5 Certification

The material supplier shall provide a certified test report to the equipment manufacturer containing the following information as a minimum.

a) Chemical analysis results (see 4.1.1).

b) Melt practice utilized (see 4.1.2).

c) Name(s) of company and facility performing melting operations.

d) Name(s) of company and facility performing the hot working operations.

e) Name(s) of company and facility performing the heat treatment.

f) Total hot work reduction ratio (see 4.1.3).

g) Actual heat treatment times and temperatures and cooling media (see 4.1.4).

h) Name(s) of company and facility performing testing.

i) Statement that the material complies with the requirements of the macroetch examination (see 4.2.1).

j) Statement describing the QTC. For example, “prolongation (full cross section on thickest end)” or “sacrificial production part”.

k) Average grain size (see 4.2.2).

l) Statement of compliance with topological duplex grain size testing requirement (see 4.2.2).

m) Statement that the material complies with the requirements of the metallographic examination for deleterious phases (see 4.2.2).

n) A set of legible photomicrographs (see 4.2.2.3).

o) Tensile test results (see 4.2.3).

p) Impact test specimen size, temperature, orientation, and results (see 4.2.4).

q) Hardness test results, reported by the test location in the same scale as used for the measurements and the converted value and scale, if converted. Additionally, when a conversion other than the ASTM E140 conversion is utilized, the conversion method shall be reported (see 4.2.5).

r) NDE results, if performed (see 4.2.6).

6 Marking

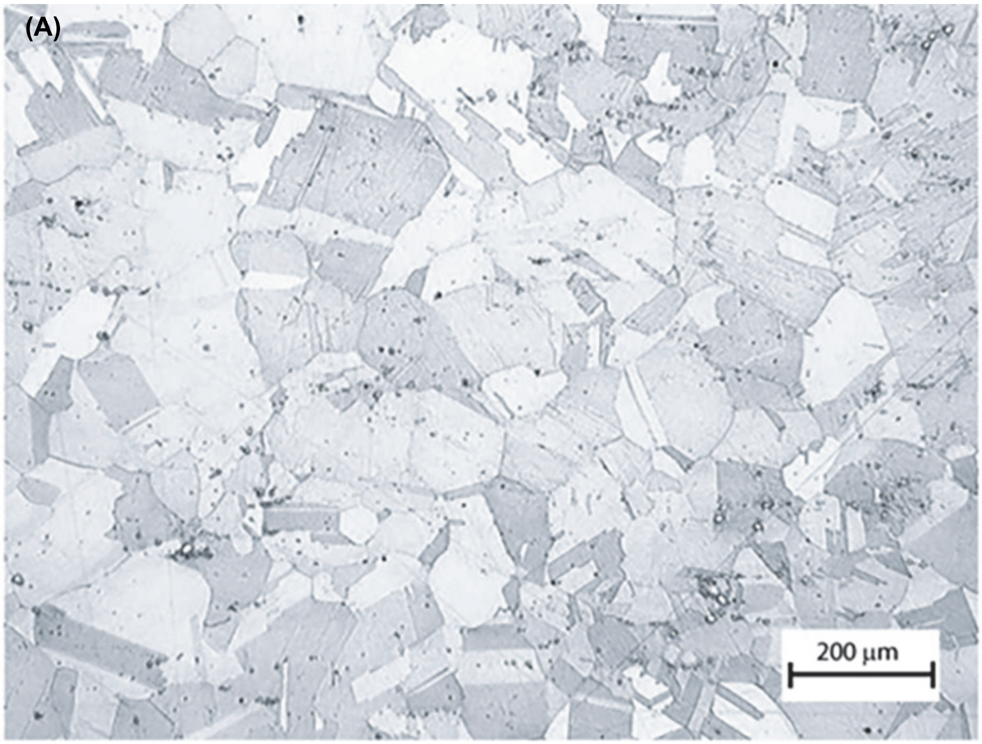
The raw material shall be marked or tagged with identification traceable to the certification for the remelt ingot and heat treat lot (see 3.1.3).

Annex A
(informative)

Reference Microstructures

These photomicrographs are representative of this product’s microstructure (not individual, isolated grains).

Original Magnification: 100X for Upper Photomicrograph (A); 500X for Lower Photomicrograph (B).



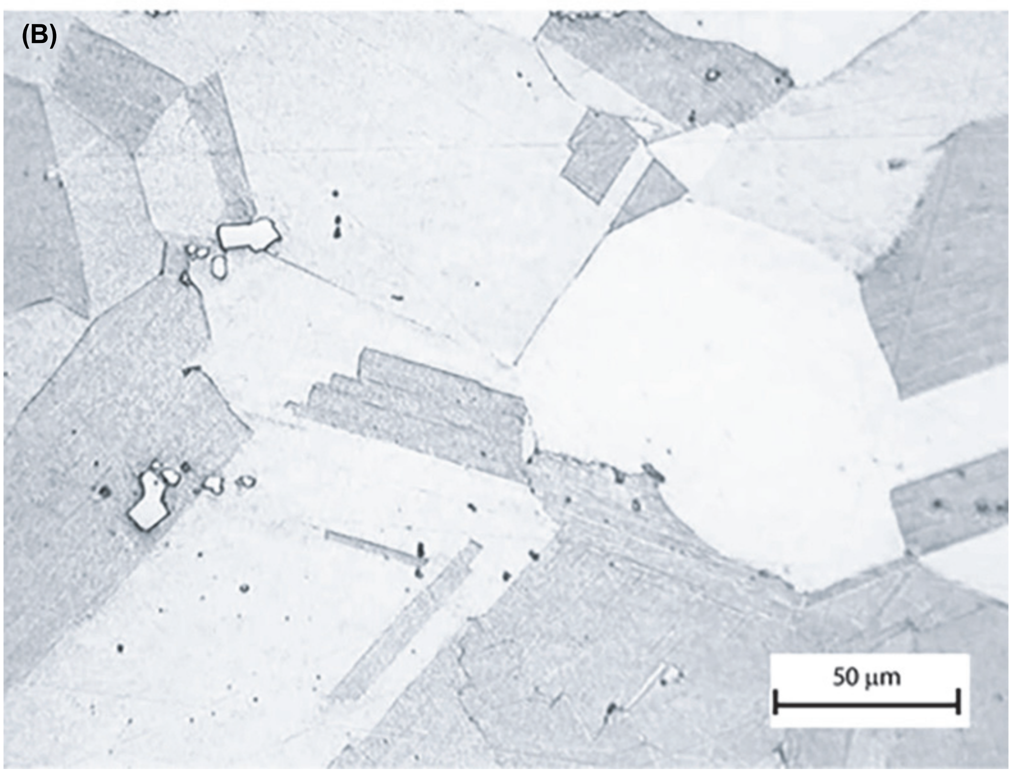


Figure A.1—Acceptable Microstructure for UNS N07718

Original Magnification: 100X for Upper Photomicrograph (A); 500X for Lower Photomicrograph (B).

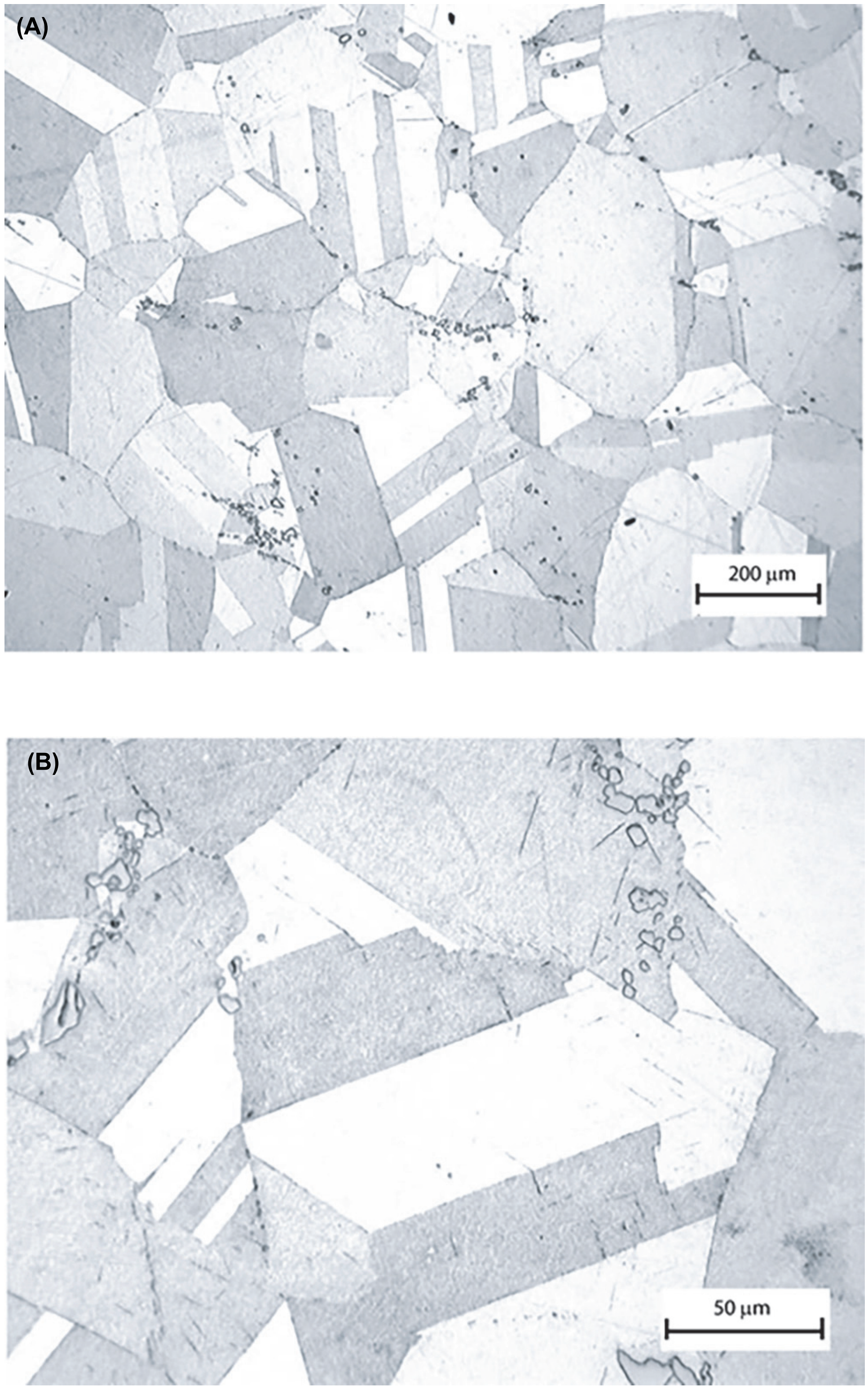


Figure A.2—Acceptable Microstructure for UNS N07718

Original Magnification: 100X for Upper Photomicrograph (A); 500X for Lower Photomicrograph (B).

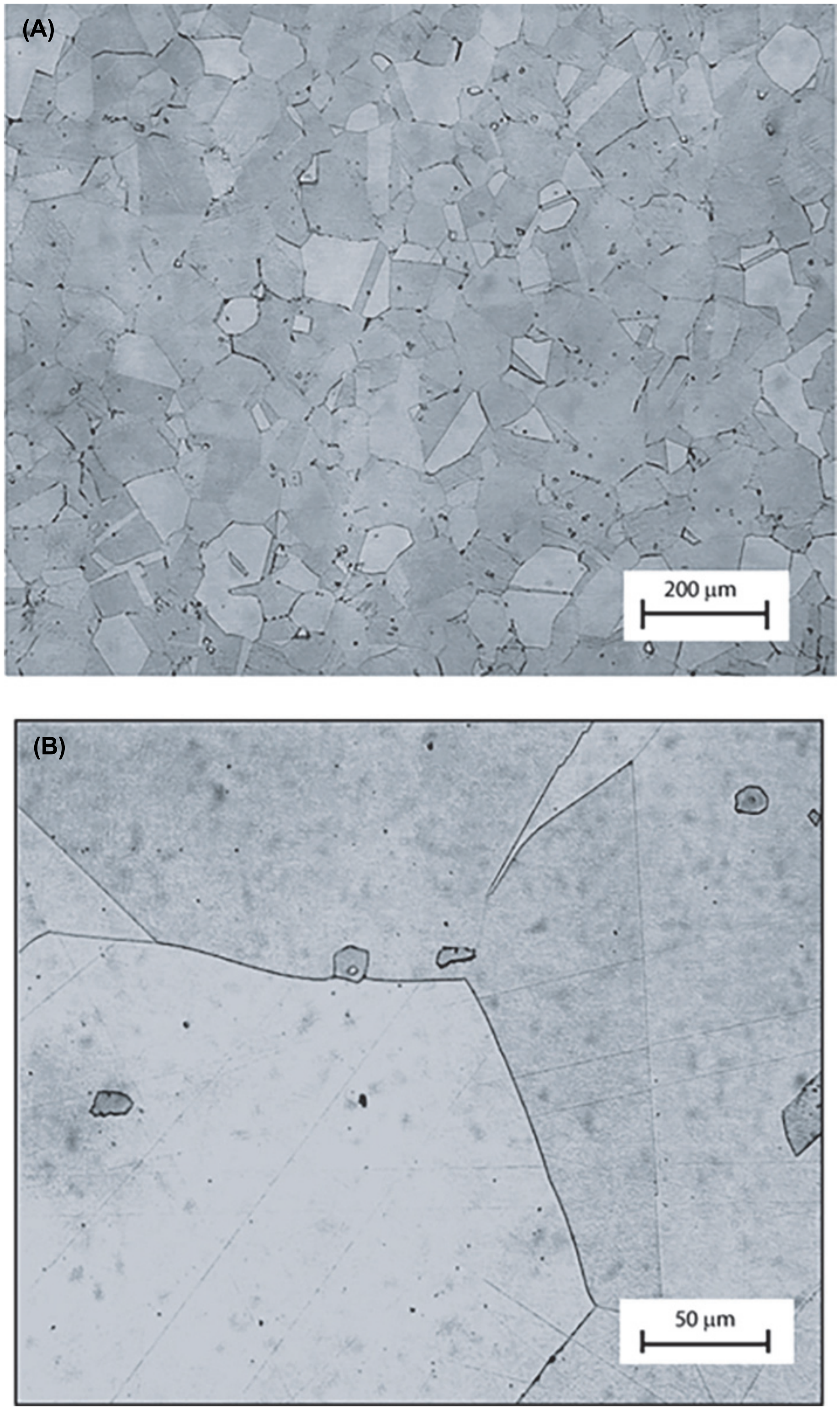


Figure A.3—Acceptable Microstructure for UNS N07718 Showing Isolated Grain Boundary Precipitates

Original Magnification: 100X for Upper Photomicrograph (A); 500X for Lower Photomicrograph (B).

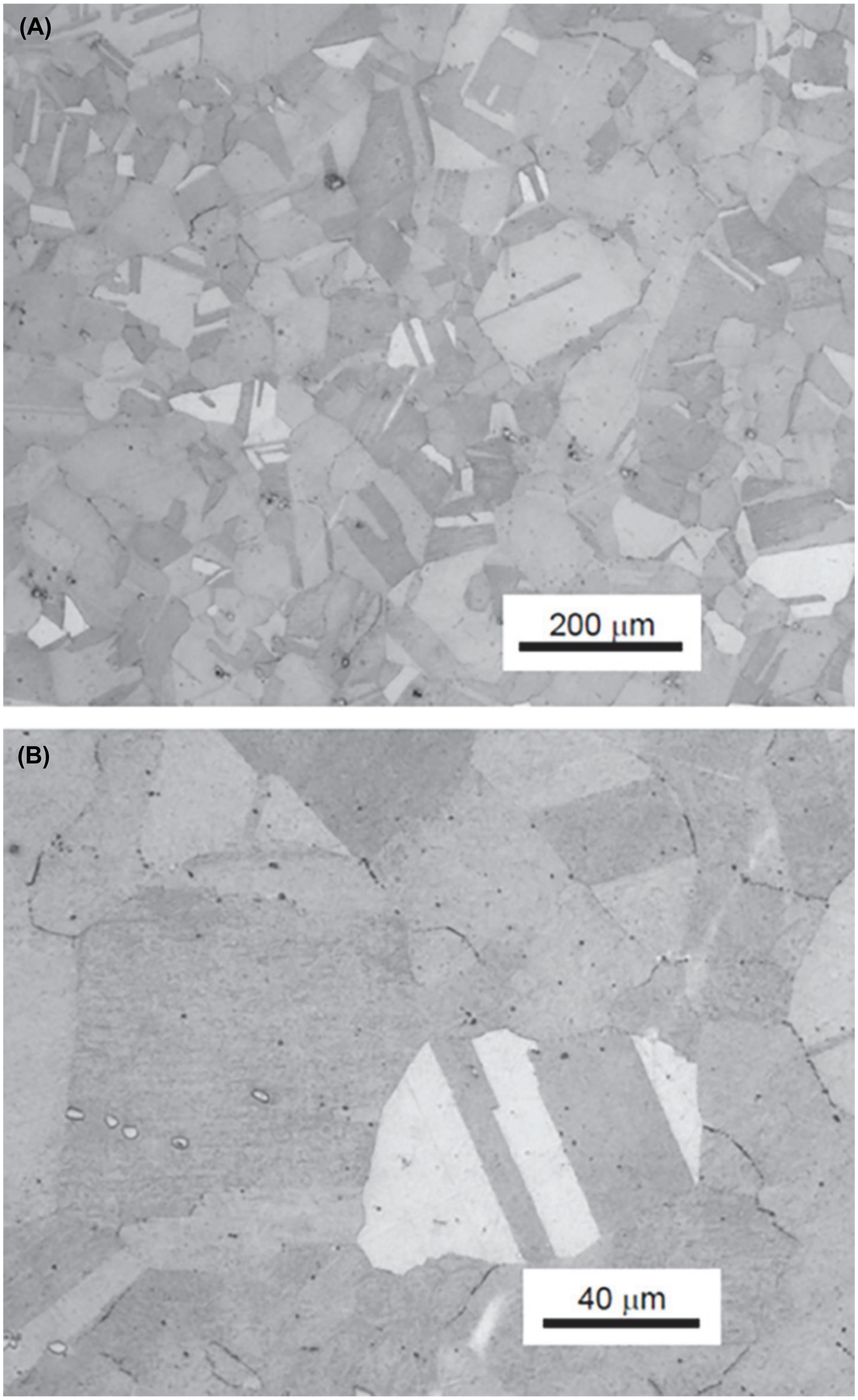


Figure A.4—Acceptable Microstructure for UNS N07718 Showing Isolated Acicular Grain Boundary Precipitates

Original Magnification: 100X for Upper Photomicrograph (A); 500X for Lower Photomicrograph (B).

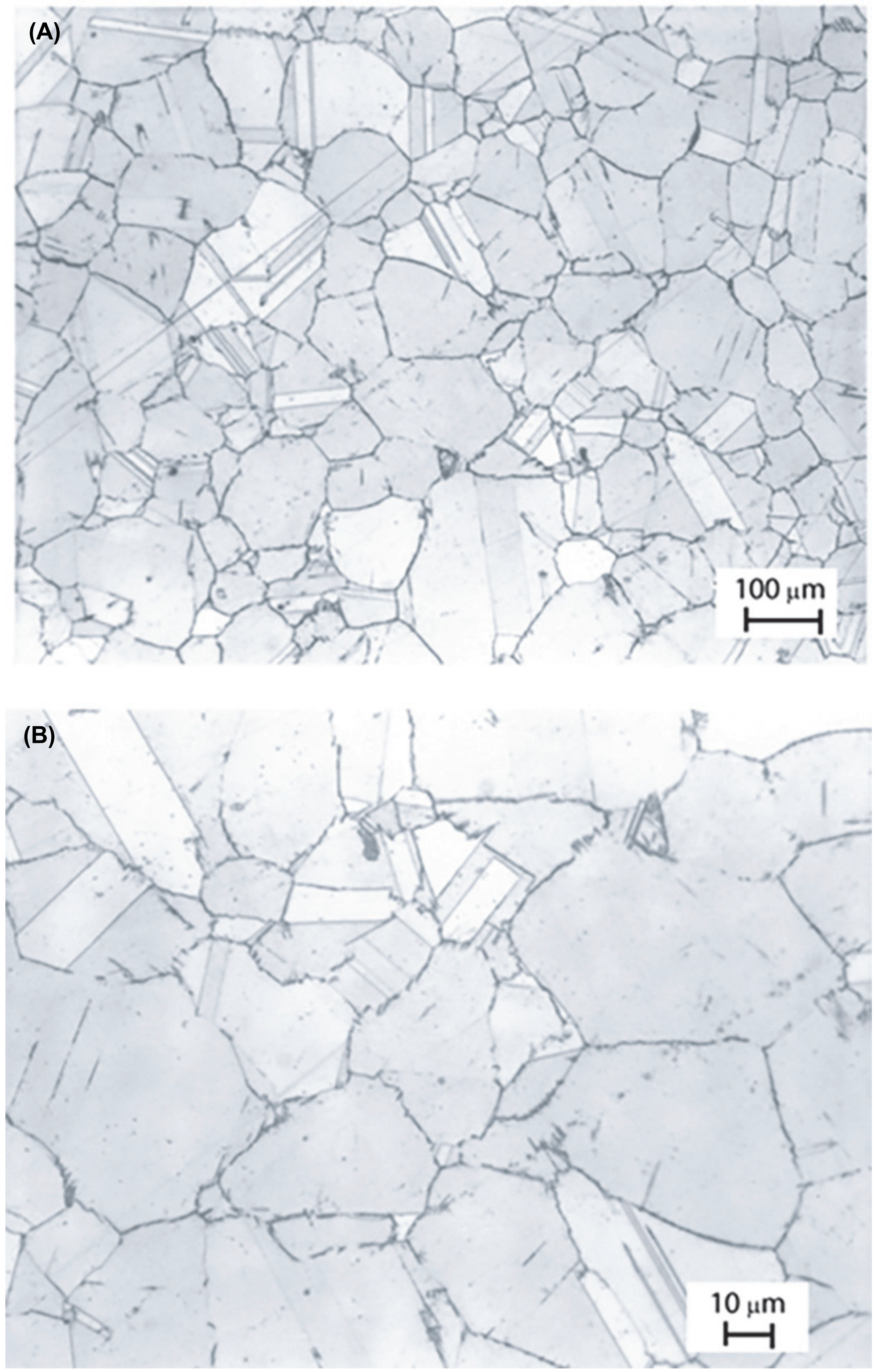


Figure A.5—Unacceptable Microstructure for UNS N07718 Showing Acicular Grain Boundary Precipitates

Original Magnification; 100X for Upper Photomicrograph (A); 500X for Lower Photomicrograph (B).

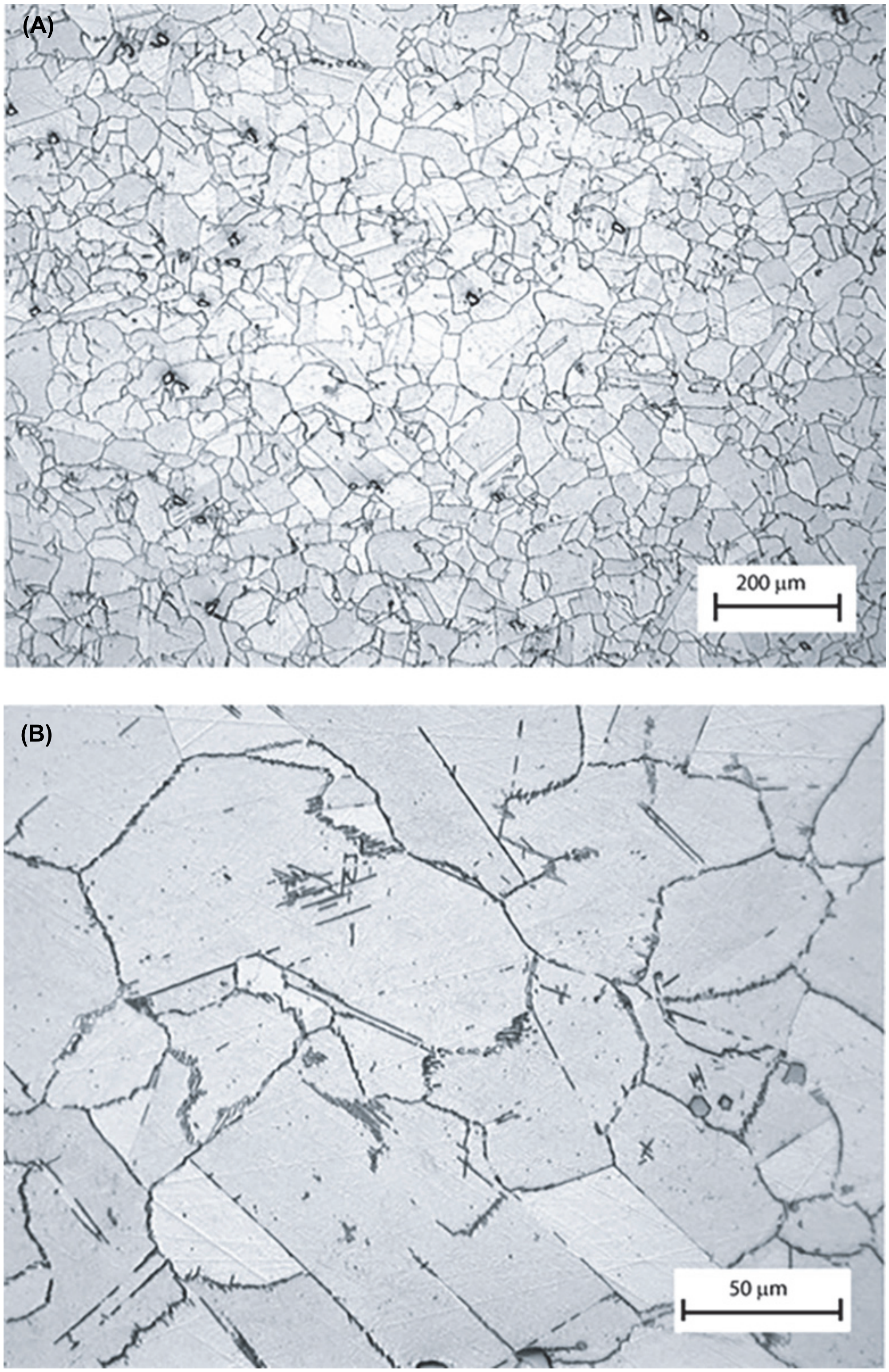


Figure A.6—Unacceptable Microstructure for UNS N07718 Showing Acicular Grain Boundary Precipitates

Original Magnification: 100X for Upper Photomicrograph (A); 500X for Lower Photomicrograph (B).

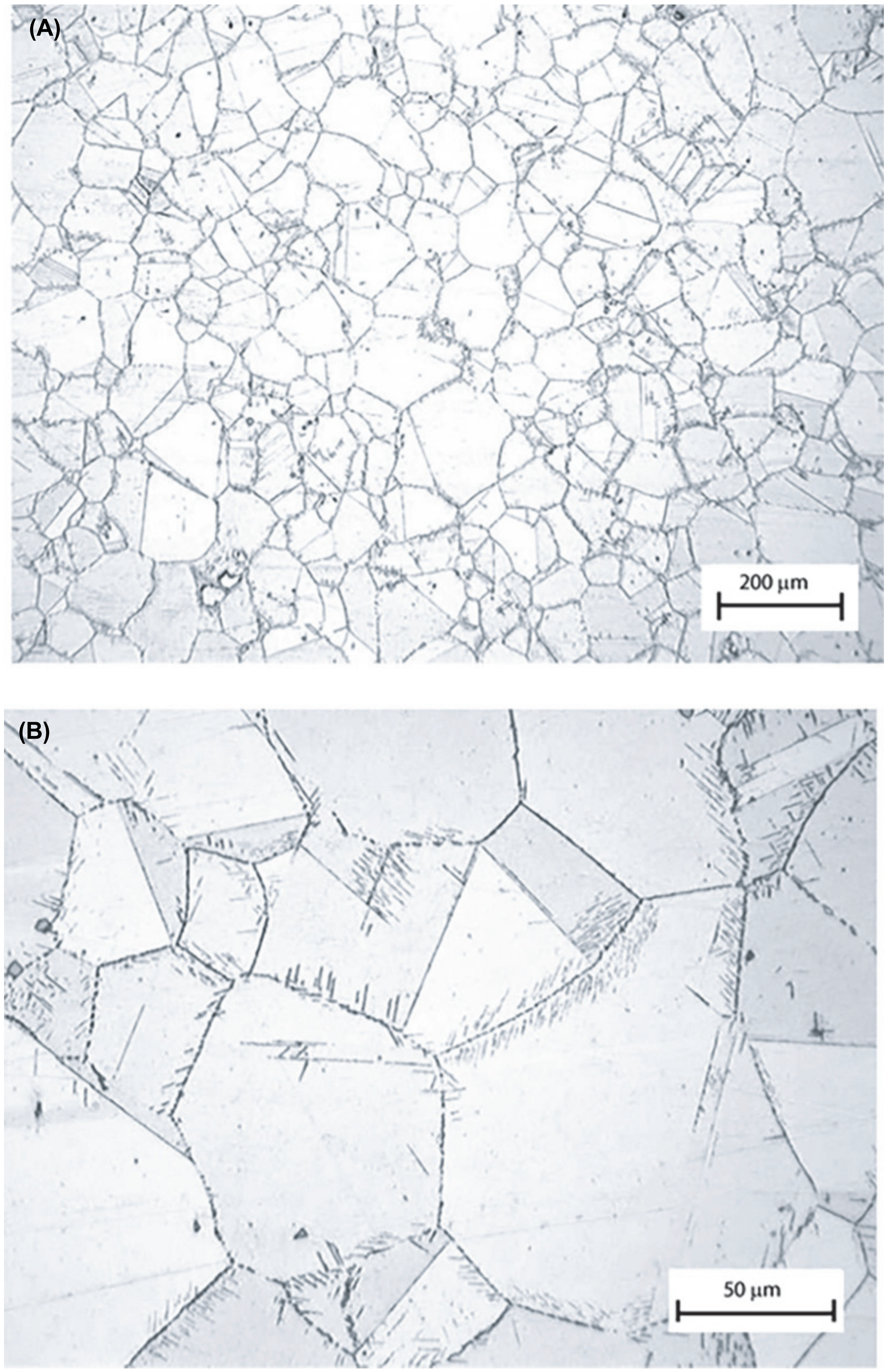


Figure A.7—Unacceptable Microstructure for UNS N07718 Showing Acicular Grain Boundary Precipitates

Original Magnification: 100X for Upper Photomicrograph (A); 500X for Lower Photomicrograph (B).

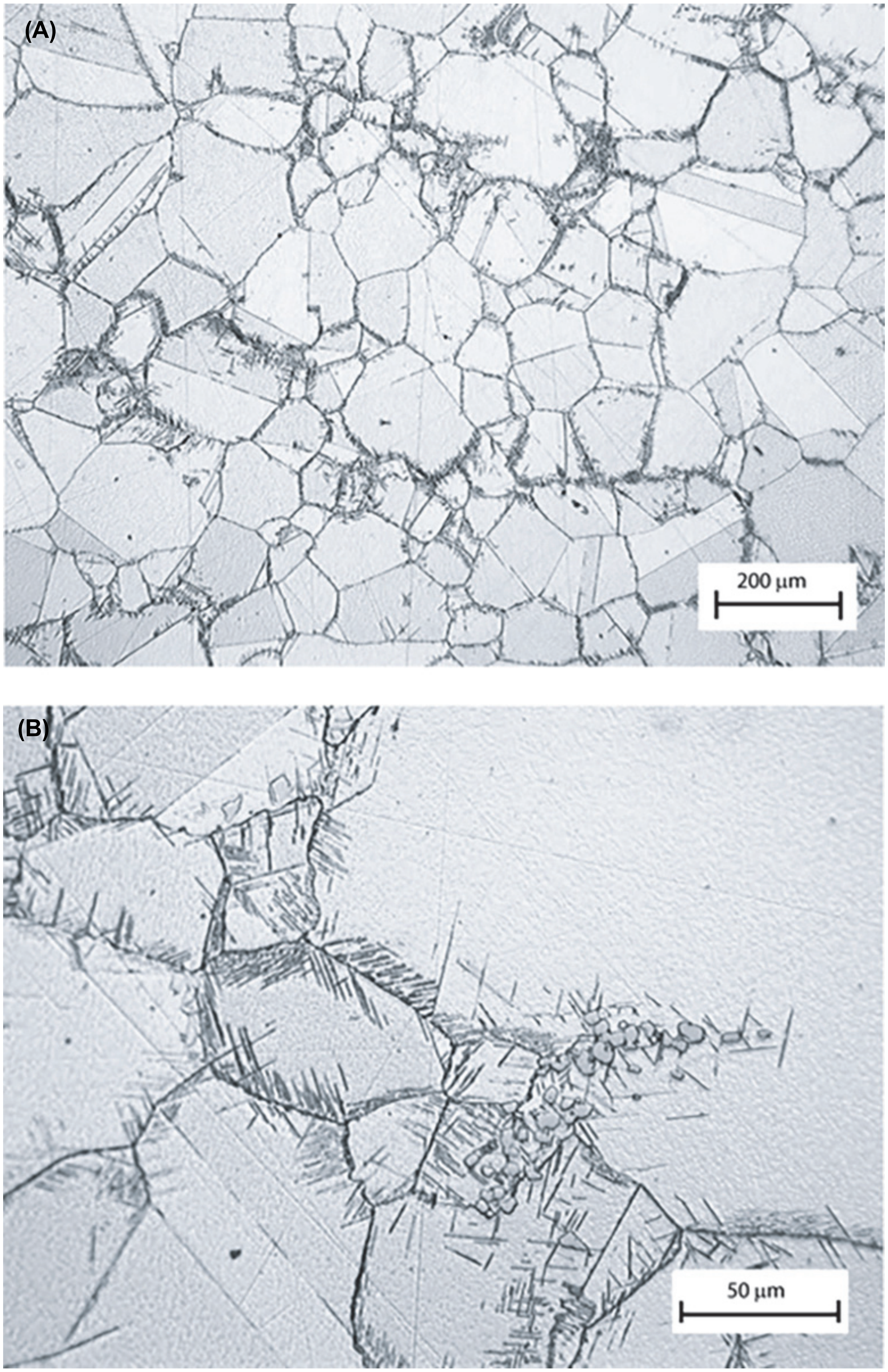


Figure A.8—Unacceptable Microstructure for UNS N07718 Showing Acicular Grain Boundary Precipitates

Original Magnification: 500X for Photomicrograph.

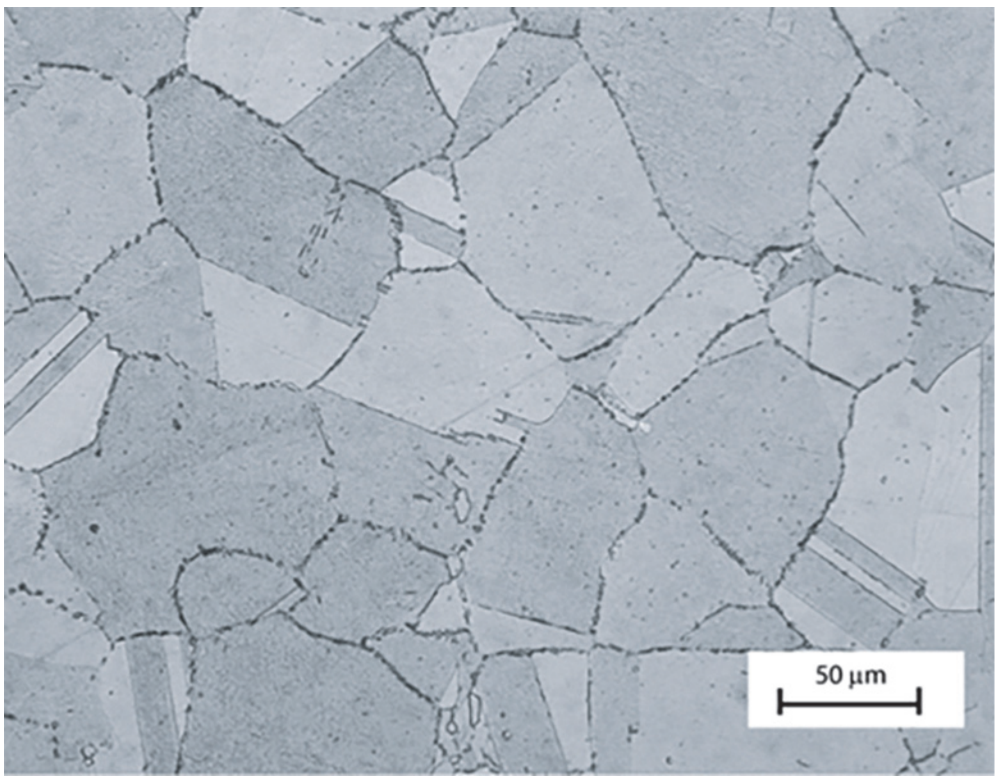


Figure A.9—Unacceptable Microstructure for UNS N07718 Showing Grain Boundary Precipitates

Original Magnification: 100X for Upper Photomicrograph (A); 500X for Lower Photomicrograph (B).

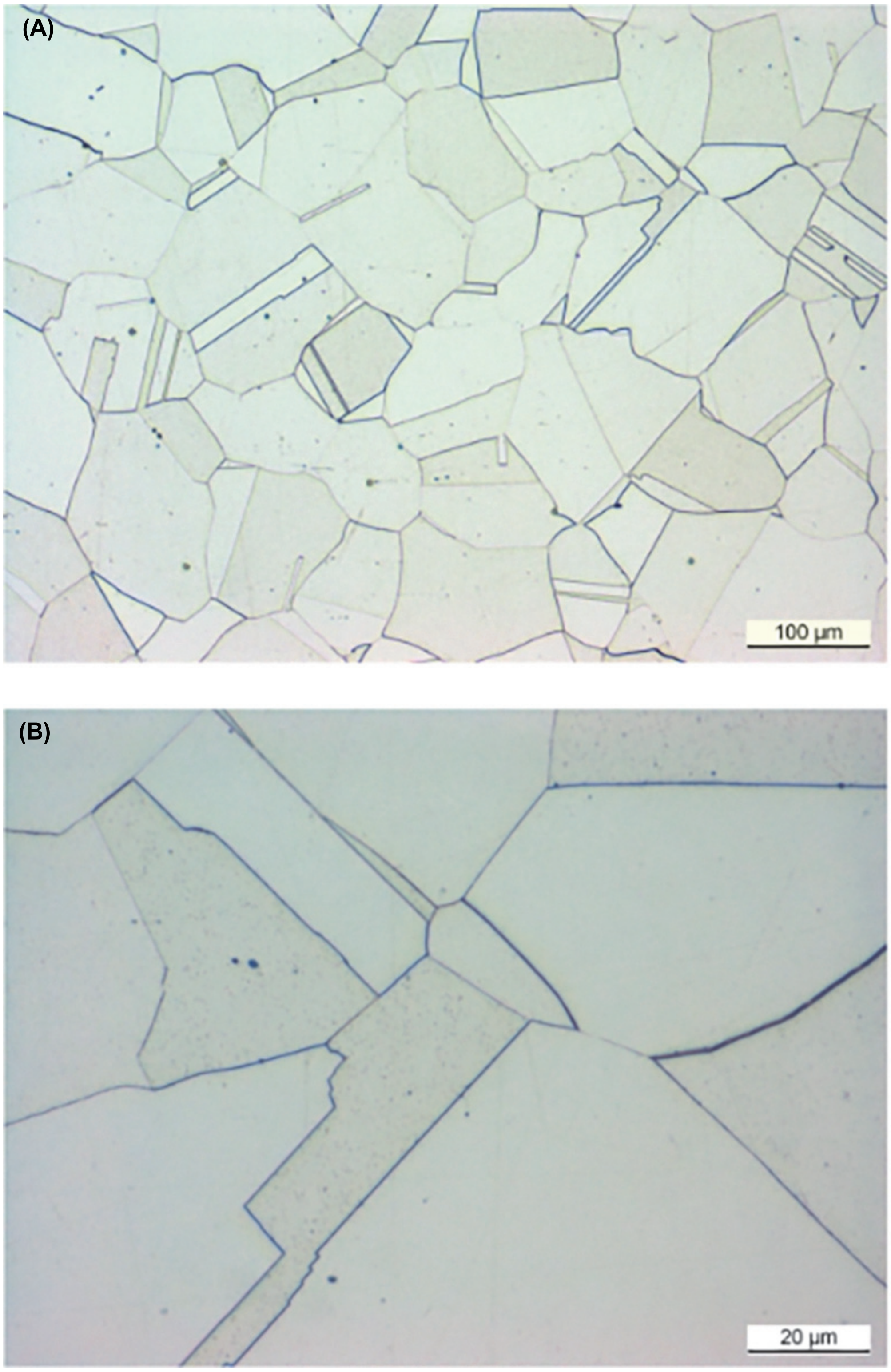
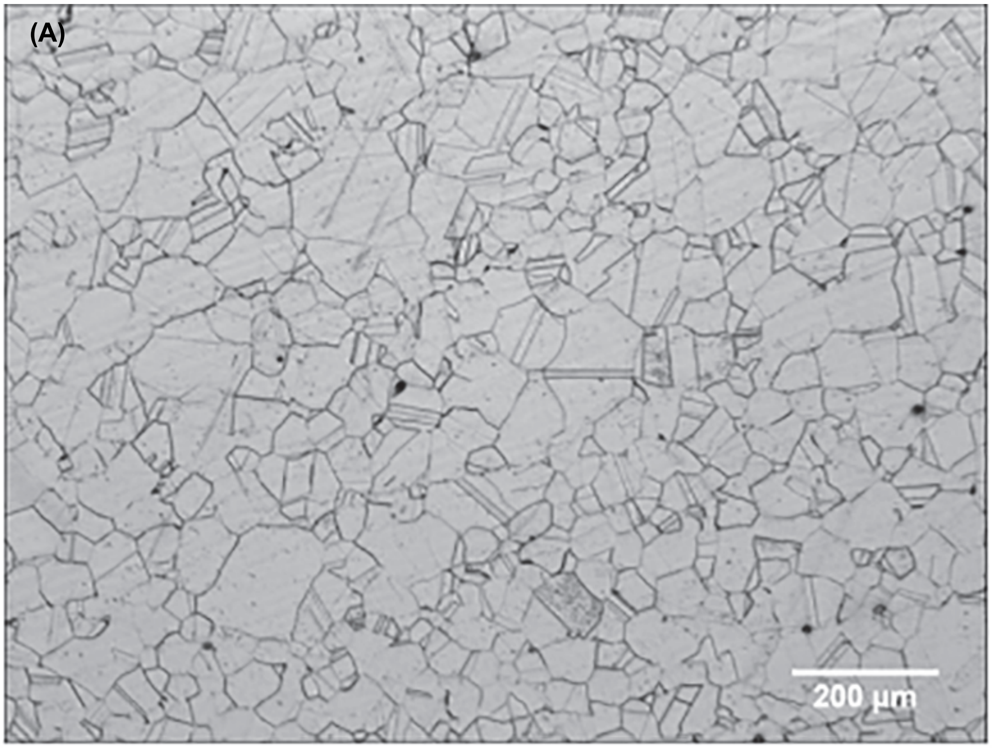


Figure A.10—Acceptable Microstructure for UNS N09925, UNS N09935, UNS N09945, UNS N07716, and UNS N07725

Original Magnification: 100X for Upper Photomicrograph (A); 500X for Lower Photomicrograph (B).



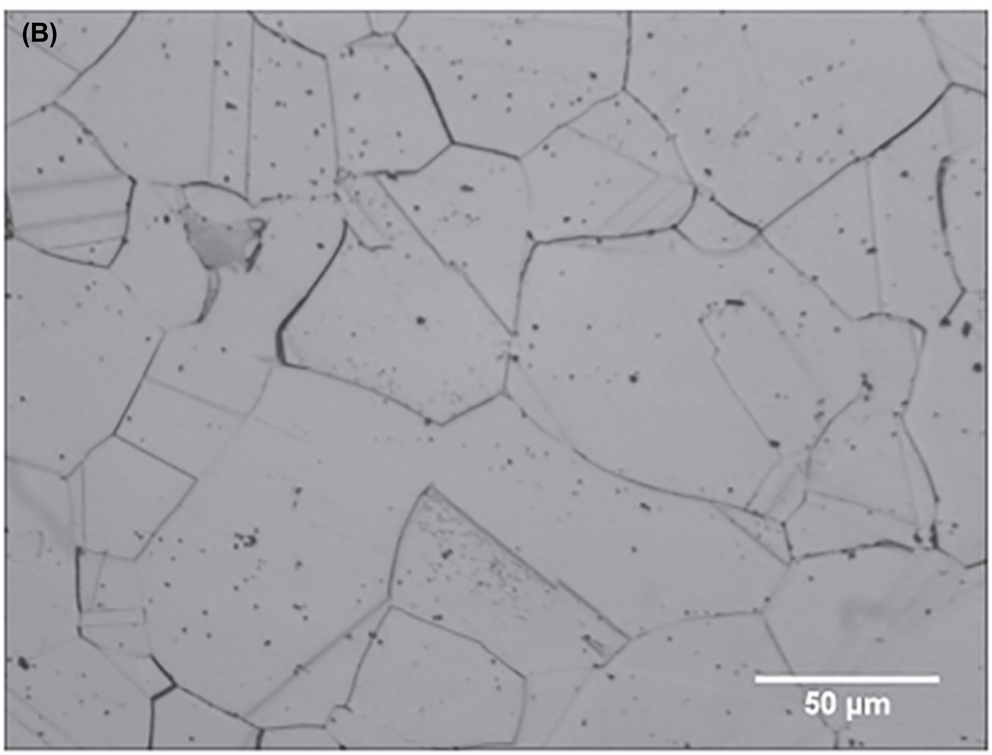


Figure A.11—Acceptable Microstructure for UNS N09925, UNS N09935, UNS N09945, UNS N07716, and UNS N07725 Showing Isolated Grain Boundary Precipitation

Original Magnification: 100X for Upper Photomicrograph (A); 500X for Lower Photomicrograph (B).

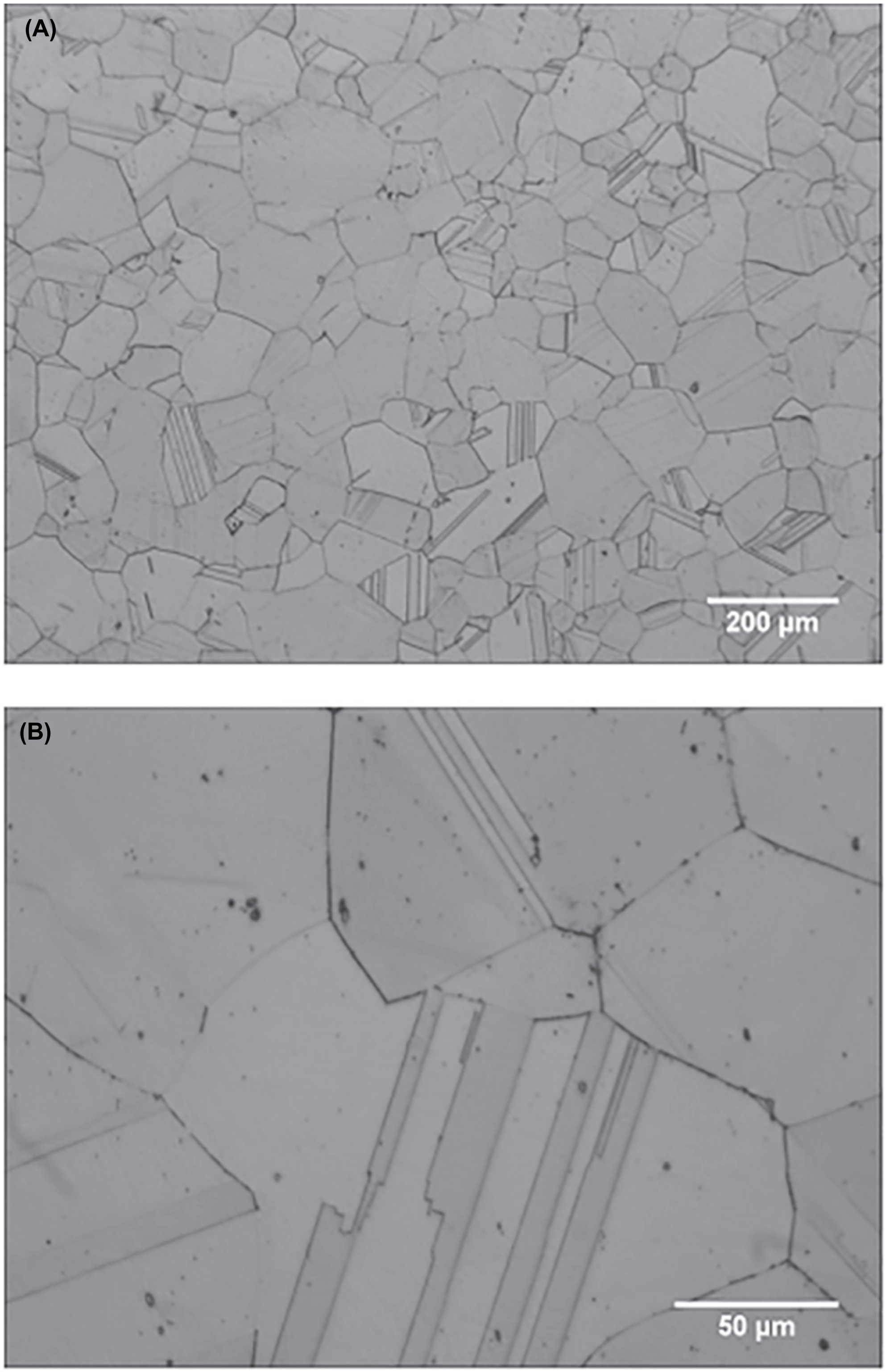


Figure A.12—Acceptable Microstructure for UNS N09925, UNS N09935, UNS N09945, UNS N07716, and UNS N07725 Showing Partial Coverage of Grain Boundaries with Second Phase Particles

Original Magnification: 100X for Upper Photomicrograph (A); 500X for Lower Photomicrograph (B).

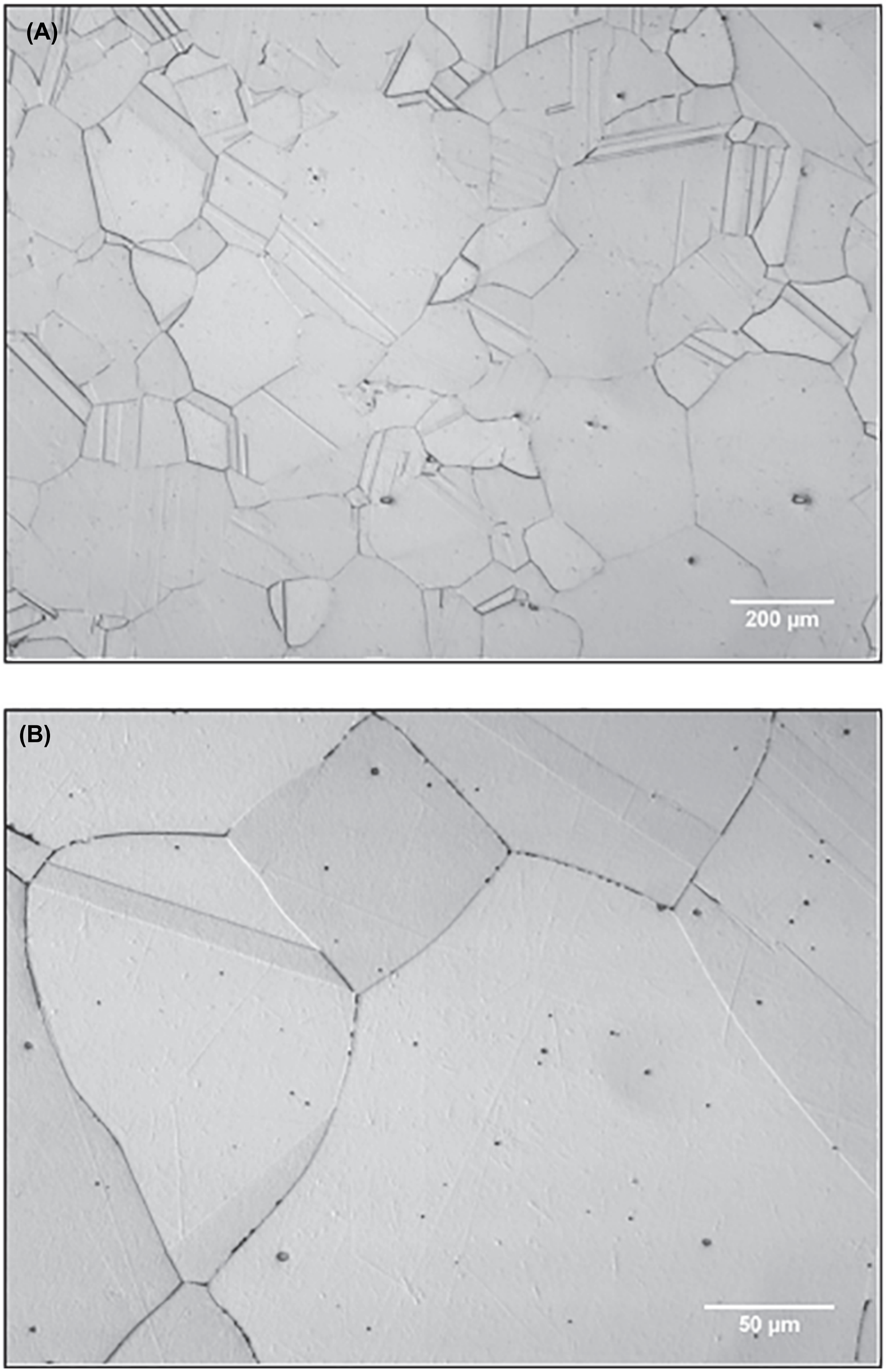


Figure A.13—Acceptable Microstructure for UNS N09925, UNS N09935, UNS N09945, UNS N07716, and UNS N07725 Showing Partial Coverage of Grain Boundaries with Second Phase Particles

Original Magnification: 100X for Upper Photomicrograph (A); 500X for Lower Photomicrograph (B).

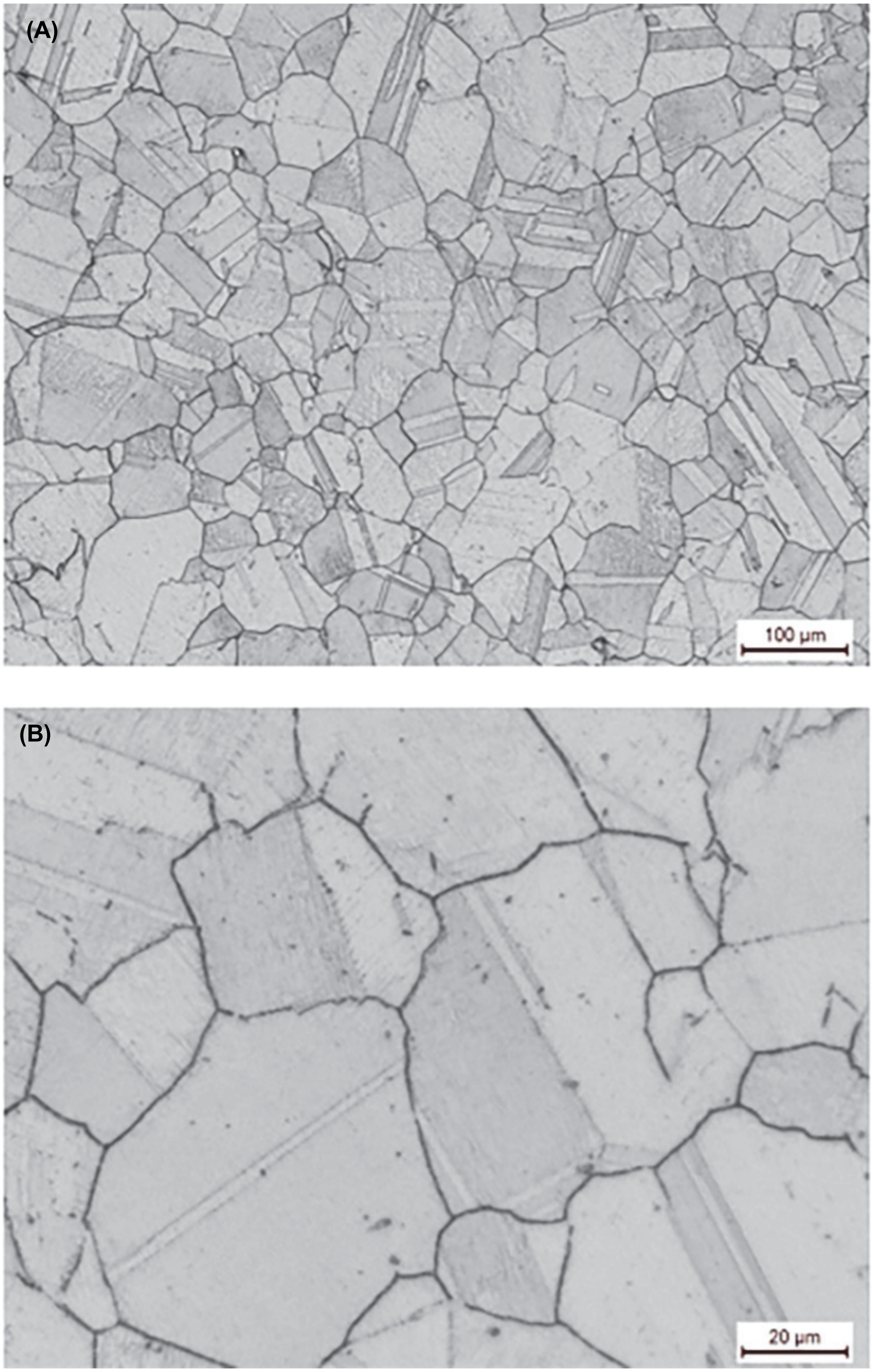


Figure A.14—Unacceptable Microstructure for UNS N09925, UNS N09935, UNS N09945, UNS N07716, and UNS N07725 Showing Full Coverage of Grain Boundaries with Second Phase Particles

Original Magnification: 100X for Upper Photomicrograph (A); 500X for Lower Photomicrograph (B).

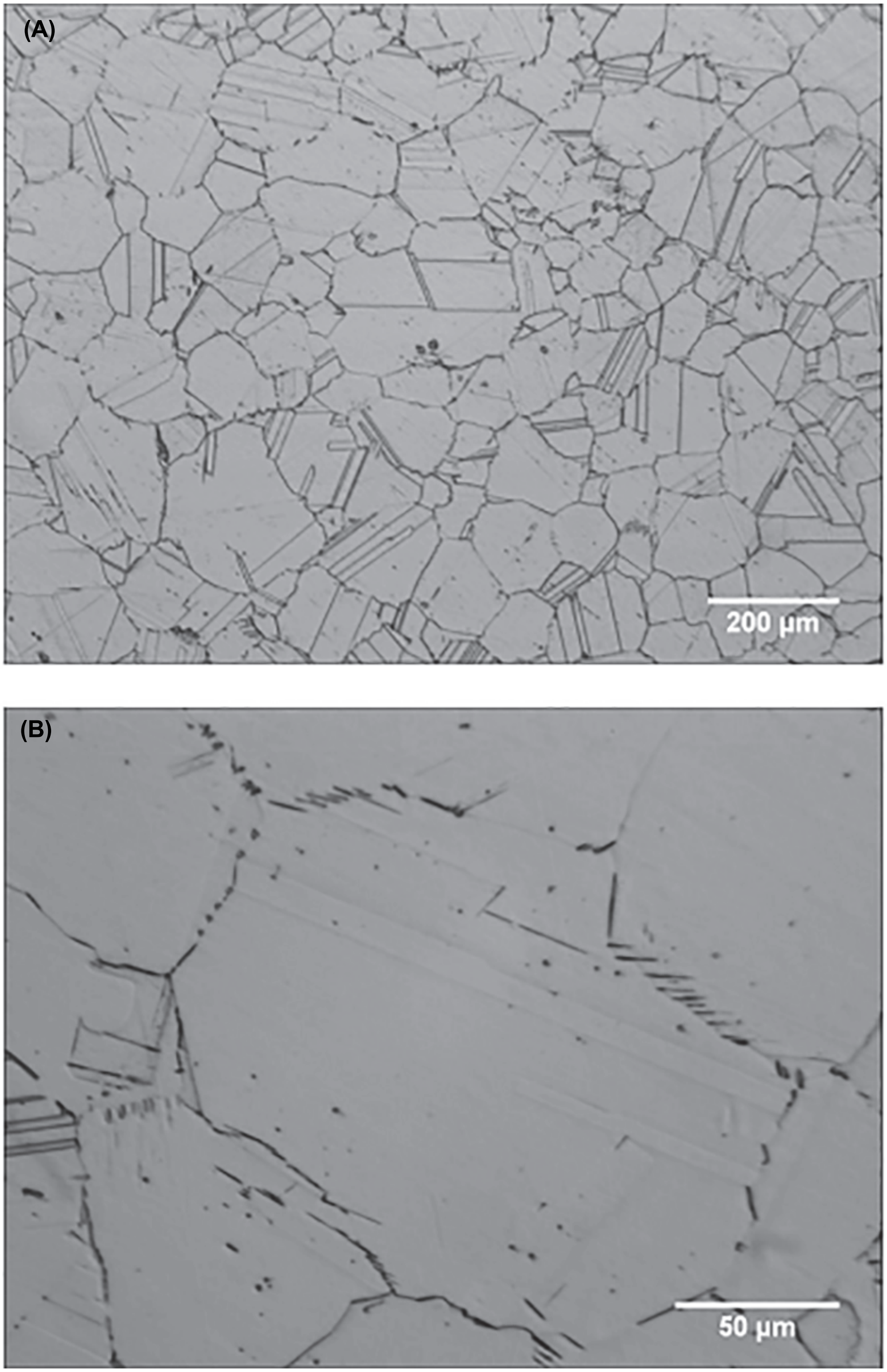


Figure A.15—Unacceptable Microstructure for UNS N09925, UNS N09935, UNS N09945, UNS N07716, and UNS N07725 Showing Acicular Grain Boundary Precipitates

Original Magnification; 100X for Upper Photomicrograph (A); 500X for Lower Photomicrograph (B).

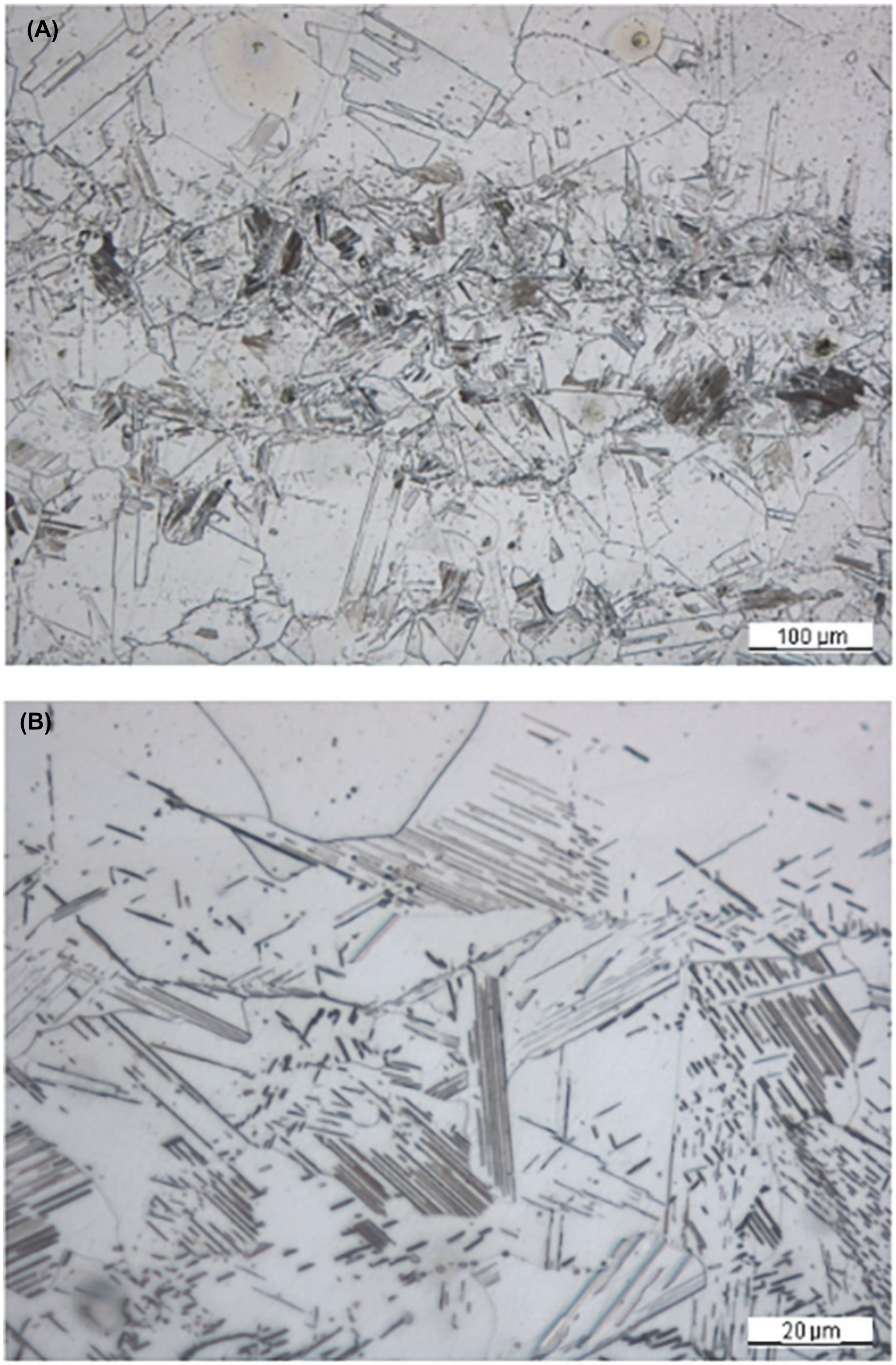


Figure A.16—Unacceptable Microstructure for UNS N09925, UNS N09935, UNS N09945, UNS N07716, and UNS N07725 Due To Acicular Precipitates

Original Magnification: 500X for Photomicrograph.

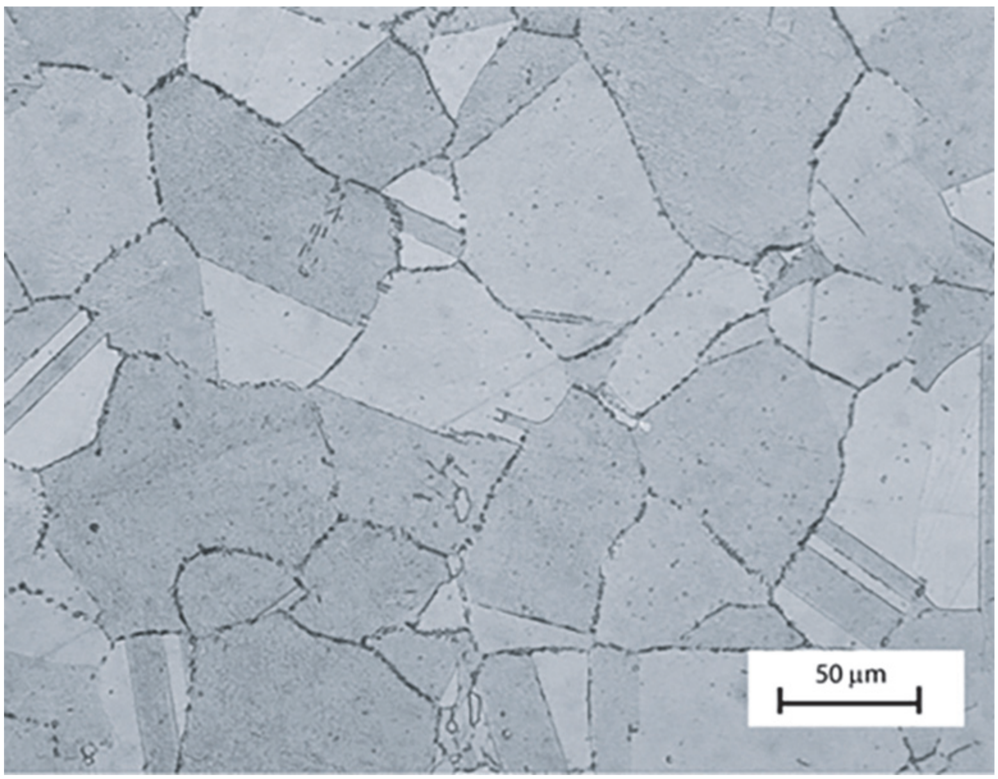


Figure A.17—Unacceptable Microstructure for UNS N09925, UNS N09935, UNS N09945, UNS N07716, and UNS N07725 Showing Grain Boundary Precipitates

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