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

API STANDARD 6ACRA SECOND EDITION, JANUARY 2025



Special Notes

API publications necessarily address problems of a general nature. With respect to particular circumstances, local, state, and federal laws and regulations should be reviewed. The use of API publications is voluntary. In some cases, third parties or authorities having jurisdiction may choose to incorporate API standards by reference and may mandate compliance.

Neither API nor any of API's employees, subcontractors, consultants, committees, or other assignees make any warranty or representation, either express or implied, with respect to the accuracy, completeness, or usefulness of the information contained herein, or assume any liability or responsibility for any use, or the results of such use, of any information or process disclosed in this publication. Neither API nor any of API's employees, subcontractors, consultants, or other assignees represent that use of this publication would not infringe upon privately owned rights.

API publications may be used by anyone desiring to do so. Every effort has been made by the Institute to ensure the accuracy and reliability of the data contained in them; however, the Institute makes no representation, warranty, or guarantee in connection with this publication and hereby expressly disclaims any liability or responsibility for loss or damage resulting from its use or for the violation of any authorities having jurisdiction with which this publication may conflict

API publications are published to facilitate the broad availability of proven, sound engineering and operating practices. These publications are not intended to obviate the need for applying sound engineering judgment regarding when and where these publications should be used. The formulation and publication of API publications is not intended in any way to inhibit anyone from using any other practices.

Any manufacturer marking equipment or materials in conformance with the marking requirements of an API standard is solely responsible for complying with all the applicable requirements of that standard. API does not represent, warrant, or guarantee that such products do in fact conform to the applicable API standard.

All rights reserved. No part of this work may be reproduced, translated, stored in a retrieval system, or transmitted by any means, electronic, mechanical, photocopying, recording, or otherwise, without prior written permission from the publisher. Contact the Publisher, API Publishing Services, 200 Massachusetts Avenue, NW, Suite 1100, Washington, DC 20001-5571.

Foreword

Nothing contained in any API publication is to be construed as granting any right, by implication or otherwise, for the manufacture, sale, or use of any method, apparatus, or product covered by letters patent. Neither should anything contained in the publication be construed as insuring anyone against liability for infringement of letters patent.

The verbal forms used to express the provisions in this document are as follows.

Shall: As used in a standard, "shall" denotes a minimum requirement to conform to the standard.

Should: As used in a standard, "should" denotes a recommendation or that which is advised but not required to conform to the standard.

May: As used in a standard, "may" denotes a course of action permissible within the limits of a standard.

Can: As used in a standard, "can" denotes a statement of possibility or capability.

This document was produced under API standardization procedures that ensure appropriate notification and participation in the developmental process and is designated as an API standard. Questions concerning the interpretation of the content of this publication or comments and questions concerning the procedures under which this publication was developed should be directed in writing to the Director of Standards, American Petroleum Institute, 200 Massachusetts Avenue, NW, Suite 1100, Washington, DC 20001. Requests for permission to reproduce or translate all or any part of the material published herein should also be addressed to the director.

Generally, API standards are reviewed and revised, reaffirmed, or withdrawn at least every five years. A one-time extension of up to two years may be added to this review cycle. Status of the publication can be ascertained from the API Standards Department, telephone (202) 682-8000. A catalog of API publications and materials is published annually by API, 200 Massachusetts Avenue, NW, Suite 1100, Washington, DC 20001.

Suggested revisions are invited and should be submitted to the Standards Department, API, 200 Massachusetts Avenue, NW, Suite 1100, Washington, DC 20001, standards@api.org.

Contents

		Page
1 1.1 1.2	Scope Purpose Applicability	1
2	Normative References	1
3 3.1 3.2	Terms, Definitions, and Abbreviations	2
4 4.1 4.2	Requirements for Age-hardened Nickel-based Alloys Process Control Requirements Testing Requirements	4
5	Certification	18
6	Marking	19
7	Addition of New Alloys and/or New Material Designations	19
Annex	A (informative) Reference Microstructures	20
Annex	B (normative) Addition of, or Modification to, Alloys and Material Designations in This Standard	41
Annex	C (informative) Industry Empirical Data on Hardness Conversion for Age Hardened Nickel-based Alloys.	44
Bibliog	raphy	46
Figure	s	
A.1	Acceptable Microstructure for UNS N07718; Original Magnification: 100X for Upper Photomicrograph (A); 500X for Lower Photomicrograph (B)	21
A.2	Acceptable Microstructure for UNS N07718; Original Magnification: 100X for Upper Photomicrograph (A); 500X for Lower Photomicrograph (B)	22
A.3	Acceptable Microstructure for UNS N07718 Showing Isolated Grain Boundary Precipitates; Original Magnification: 100X for Upper Photomicrograph (A); 500X for Lower Photomicrograph (B)	23
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)	24
A.5	Unacceptable Microstructure for UNS N07718 Showing Acicular Grain Boundary Precipitates; Original Magnification: 100X for Upper Photomicrograph (A); 500X for Lower Photomicrograph (B)	25
A.6	Unacceptable Microstructure for UNS N07718 Showing Acicular Grain Boundary Precipitates; Original Magnification: 100X for Upper Photomicrograph (A); 500X for Lower Photomicrograph (B)	
A.7	Unacceptable Microstructure for UNS N07718 Showing Acicular Grain Boundary Precipitates; Original Magnification: 100X for Upper Photomicrograph (A); 500X for Lower Photomicrograph (B)	
A.8	Unacceptable Microstructure for UNS N07718 Showing Acicular Grain Boundary Precipitates; Original Magnification: 100X for Upper Photomicrograph (A); 500X for Lower Photomicrograph (B)	
A.9	Magnification: 500X for Upper Priordinicograph (A), 500X for Lower Priordinicograph (B)	

Contents

		Pag
A.10	Acceptable Microstructure for UNS N06625; Original Magnification: 100X for Upper Photomicrograph (A): 500X for Lower Photomicrograph (B)	. 30
A.11	Acceptable Microstructure for UNS N06625; Original Magnification: 100X for Upper Photomicrograph (A); 500X for Lower Photomicrograph (B)	
A.12	Unacceptable Microstructure for UNS N06625 Showing Acicular Grain Boundary Precipitates; Original Magnification: 100X for Upper Photomicrograph (A); 500X for Lower Photomicrograph (B)	. 32
A.13	Acceptable Microstructure for UNS N07716, UNS N07725, UNS N09925, UNS N09935, UNS N09945, UNS N09946 and UNS N09955; Original Magnification: 100X for Upper Photomicrograph (A); 500X for	
A.14	Lower Photomicrograph (B)	
A.15	Acceptable Microstructure for UNS N07716, UNS N07725, UNS N09925, UNS N09935, UNS N09945, UNS N09946 and UNS N09955 Showing Partial Coverage of Grain Boundaries with Second Phase Particles; Original Magnification: 100X for Upper Photomicrograph (A); 500X for Lower Photomicrograph	
A.16	(B)	
A.17	(B) Unacceptable Microstructure for UNS N07716, UNS N07725, UNS N09925, UNS N09935, UNS N09945, UNS N09946 and UNS N09955 Showing Full Coverage of Grain Boundaries with Second Phase Particles; Original Magnification: 100X for Upper Photomicrograph (A); 500X for Lower Photomicrograph (B).	
A.18	Unacceptable Microstructure for UNS N07716, UNS N07725, UNS N09925, UNS N09935, UNS N09945, UNS N09946 and UNS N09955 Showing Acicular Grain Boundary Precipitates; Original Magnification: 100X for Upper Photomicrograph (A); 500X for Lower Photomicrograph (B)	
A.19	Unacceptable Microstructure for UNS N07716, UNS N07725, UNS N09925, UNS N09935, UNS N09945, UNS N09946 and UNS N09955 Due to Acicular Precipitates; Original Magnification: 100X for Upper Photomicrograph (A); 500X for Lower Photomicrograph (B)	. 39
A.20	Unacceptable Microstructure for UNS N07716, UNS N07725, UNS N09925, UNS N09935, UNS N09945, UNS N09946 and UNS N09955 Showing Grain Boundary Precipitates; Original Magnification: 500X for Photomicrograph.	.40
C.1	Rockwell C-scale (HRC) Hardness Values Versus Mean Brinell (HBW) Hardness Values for Age Hardened Nickel-based Alloys, including Empirically Derived Regression Equation	. 45
Tables		
Tables		
1 2 3 4 5 6 7	Chemical Composition (weight %). Acceptable Melt Practices	6 7 9 .10 .12
8 C.1	Hardness Requirements	
	Regression Equation	44

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 Tree Equipment.

NOTE Age-hardened and precipitation-hardened terms are interchangeably used in international standards

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

1 Scope

1.1 Purpose

This document provides material requirements, including acceptable microstructures, for age-hardened nickelbased alloys used in the manufacture of solid or hollow wrought raw materials for API 6A equipment, and other equipment where specified by the applicable Standard or Specification.

The process controls and tests requirements contained in this standard assist in achieving adequate performance in a variety of environments. These requirements have been applied since 2004 for UNS N07718 120K via API 6A718 (which was superseded by this Standard).

WARNING—Failures attributed to hydrogen-induced stress cracking (HISC) have been reported in production equipment and early in the life of subsea equipment made from age-hardened nickel-based alloys. Some age-hardened nickel-based alloys that meet the requirements of ANSI/NACE MR0175/ISO 15156 and API 6ACRA may be susceptible to HISC. Sources of hydrogen charging include but are not limited to galvanic coupling to a more active material, direct exposure to seawater with cathodic protection (CP), and decomposition of brines. The industry is evaluating alternate test methods and/or acceptance criteria to identify material with susceptibility to HISC.

1.2 Applicability

This standard is intended to apply to raw materials used in the manufacture of 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.

Thermal post-processing of API 6ACRA material designations, after the applicable final aging heat treatment specified in this standard, is outside the scope of this standard.

NOTE Examples of thermal post-processing are thermochemical surface treatments (e.g. chemical vapor deposition) and/or thermal diffusion treatments (e.g. boronizing, nitriding, nitrocarburizing), welding, and brazing.

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 Tree Equipment

ASTM A370,1 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 E8, Standard Test Methods for Tension Testing of Metallic Materials

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

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

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

ASTM E23, Standard Test Methods for Notched Bar Impact Testing 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

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

SAE² AMS2750, Pyrometry

ANSI/NACE MR0175/ISO 15156, Petroleum and natural gas industries—Materials for use in H_2 S-containing environments in oil and gas production

3 Terms, Definitions, and Abbreviations

3.1 Terms and Definitions

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

3.1.1

corrosion-resistant alloy

CRA

Nonferrous-based alloy in which any one or the sum of the specified amount of the elements titanium, nickel, cobalt, chromium, and molybdenum exceeds 50 % mass fraction.

NOTE This definition is different from that in ANSI/NACE MR0175/ISO 15156 and comes from API 6A.

SAE International, 400 Commonwealth Drive, Warrendale, Pennsylvania 15096-0001, www.sae.org.

3.1.2

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.

3.1.3

field of view

A 0.008 in. (0.203 mm) × 0.010 in. (0.254 mm) area of microstructure.

NOTE The 0.008 in. $(0.203 \text{ mm}) \times 0.010$ in. (0.254 mm) area is representative of the area shown in a 4 in. $(102 \text{ mm}) \times 5$ in. (127 mm) photomicrograph taken at 500X.

3.1.4

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.

3.1.5

retest

Additional testing when a valid test fails to meet requirements.

NOTE An invalid test for an assignable cause per the reference test standard does not constitute a failed test.

3.1.6

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.

3.2 Abbreviations

AOD

VOD

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

argon oxygen decarburization

D	tensile specimen gage diameter
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
VD	vacuum degassing
VIM	vacuum induction melting

vacuum oxygen decarburization

4 Requirements for Age-hardened Nickel-based Alloys

4.1 Process Control Requirements

4.1.1 QTC

The QTC shall be either a prolongation (full cross-section on thickest end) or sacrificial production part. When a prolongation is used, it shall remain integrally attached during all heat-treatment operation(s).

4.1.2 Chemical Composition Requirements

4.1.2.1 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.

4.1.2.2 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.

4.1.2.3 Check (Product) Analysis

When material is qualified by the use of a check (product) analysis performed on the production material, the analysis shall conform to the check (product) analysis variation specified in ASTM B880.

Table 1—Chemical Composition (weight %)

					UNS Number	ımber			
Element	N06625	N07716	N07718	N07725	N09925	N09935	N09945	N09946	N09955
z	58.0 min	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	46.5 to 48.0	52.0 to 55.0	55.0 to 60.0
ပ်	20.0 to 23.0	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	19.5 to 22.5	20.0 to 24.0
Fe a	5.0	Balance	Balance	Balance	22.0 min	Balance	Balance	Balance	Balance
qN	Ī	2.75 to 4.00	Ī	2.75 to 4.00	0.08 to 0.50	0.20 to 1.00	2.80 to 3.50	3.80 to 4.50	4.0 to 5.5
Cb(Nb) + Ta	3.30 to 4.15	I	4.87 to 5.20	I	I	1	T	I	1
Мо	8.0 to 10.0	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	3.00 to 4.00	5.5 to 7.0
F	0.40	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	0.50 to 2.50	0.50 to 1.00
Ι	0.40	0.35	0.40 to 0.60	0.35	0.10 to 0.50	0.50	0.01 to 0.70	0.01 to 0.70	0.25 to 0.80
o	0.025	0.030	0.045	0.030	0.025	0.030	0.005 to 0.040	0.005 to 0.030	0.030
ပ္ပ	1.00	1	1.00	I	1	1.00	1	I	1.00
Mn	0.50	0.20	0.35	0.35	1.00	1.00	1.00	1.00	0.50
Si	0.50	0.20	0.35	0.20	0.35	0.35	0.50	0.50	0.50
Ъ	0.015	0.015	0.010	0.015	0.020	0.025	0.020	0.020	0.015
s	0.015	0.010	0.010	0.010	0.003	0.001	0.010	0.010	0.0010
В	Ì	900.0	0.0060	ì	ı	1	I	I	0900.0
Cu	Ī	0.23	0.23	Ī	1.50 to 3.00	1.00 to 2.00	1.50 to 3.00	1.50 to 3.00	0.044
Pb		0.001	0.0010					Ī	
Se	1	1	0.0005	1	1	1	1	1	1
Ξ	1	1	0.00005	1	1	1	1	I	1
Ca b	I	Ī	0.0030	Ī	1		1	Ī	1
Mg b	Ī	Î	0.0060	Ī	I	ı	I	Ī	ı
W	I	Ī	1	I	1	1.00	I	1	1

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

To be determined if intentionally added.

Some limits are more restrictive than the UNS chemistry.

[&]quot;—" Value not required.

Value restriction.
 All single value chemical composition values are maximum limits, unless otherwise noted in the table.

4.1.3 Melt Practice Requirements

The acceptable melting practices for the alloys covered by this standard are specified in Table 2.

Table 2—Acceptable Melt Practices

Melting Order of	UNS N06625, UI UNS N		UNS	N07718	N09945, UNS N	NS N09935, UNS 109946, and UNS 19955
Operations	Process Option A	Process Option B	Process Option A	Process Option B	Process Option A	Process Option B
Step 1	EF	VIM	EF	VIM	EF	VIM
Step 2	AOD or VOD	ESR® or EFR®	AOD or VOD	ESR or EFR or VAR	AOD or VOD or VD	ESR or EFR or VAR
Step 3	VAR	VAR	VAR	ESR ^a or EFR ^a or VAR ^a	ESR or EFR or VAR	VAR ^a
Step 4	or VAR ^a VAR	_				
a This step is o	ptional for the stated	process.				

4.1.4 Forging and Hot Working Requirements

The minimum total hot work reduction ratio shall be 4.0:1 (see 3.1.6).

4.1.5 Heat Treating Requirements

4.1.5.1 Production Heat Treating Equipment

Production heat treating equipment shall be in conformance with API 6A or SAE AMS2750.

NOTE API 20H provides guidance for batch heat treatment of various materials.

4.1.5.2 Temperature Monitoring

The material temperature shall be measured by using 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 five years minimum following the date of heat treatment.

4.1.5.3 Solution Annealing and Age Hardening

The production material shall be solution annealed and age hardened in accordance with the procedures in <u>Table 3</u>.

Optional re-heat treatment steps—complete re-heat treatment i.e. re-solution annealing and age hardening per Table 3 is permitted. All requirements stated in 4.2.2, 4.2.3, 4.2.4 and 4.2.5 shall be met via testing of a QTC (see 4.1.1).

Re-aging for materials having single step aging within the parameters of <u>Table 3</u> for the original UNS and Material Designation is permitted, without re-solution annealing provided the cumulative aging time does not exceed the maximum allowable aging time. All requirements stated in <u>4.2.2</u>, <u>4.2.3</u>, <u>4.2.4</u> and <u>4.2.5</u> shall be met via testing of a QTC (see <u>4.1.1</u>).

4.1.5.4 Tolerance on Heat Treatment Times at Temperature

Rounding per ASTM E29 may be applied.

Table 3—Heat Treatment Procedures

		Solution Ar	nnealing	Age Hardening
UNS Number	Material Designation	Material Temperature	Time (hours)	Material Temperature and Time
N06625	95K	1825 °F-1875 °F (996 °C-1024 °C) °	1.0 minimum ^f	1229 °F–1247 °F (665 °C–675 °C) for 16 hours minimum ^d
N07716	120K 140K	1875 °F–1925 °F (1024 °C–1052 °C)	0.5 to 4 ^b	1310 °F –1455 °F (710 °C–791 °C) for 4–9 hours, furnace cool to 1125 °F– 1275 °F (607 °C–691 °C), and hold for total aging time of 12 hours minimum ^b
	120K	1870 °F-1925 °F (1021 °C-1052 °C)	1.0 to 2.5 ª	1425 °F–1475 °F (774 °C–802 °C) for 6–8 hours ^b
	140K	1870 °F-1925 °F (1021 °C-1052 °C)	1.0 to 2.5 a	1400 °F–1475 °F (760 °C–802 °C) for 6–8 hours ^b
N07718	150K	1870 °F–1925 °F (1021 °C–1052 °C)	1.0 to 2.5 °	1292 °F–1382 °F (700 °C–750 °C) for 8 hours minimum, furnace cool to 1112 °F –1202 °F (600 °C–650 °C), and hold for 8 hours minimum ^d
N07725	120K	1875 °F–1950 °F (1024 °C–1066 °C)	0.5 to 4 ^b	1325 °F –1425 °F (718 °C–774 °C) for 4–9 hours, furnace cool to 1125 °F –1275 °F (607 °C–691 °C), and hold for total aging time of 12 hours minimum ^b
N09925	110K	1825 °F–1925 °F (996 °C–1052 °C)	0.5 to 4 ^a	1325 °F –1400 °F (718 °C–760 °C) for 4–9 hours, furnace cool to 1125 °F– 1220 °F (607 °C–660 °C), and hold for total aging time of 12 hours minimum ^b
N09935	110K	1870 °F–1975 °F (1021 °C–1079 °C)	0.5 to 4 ^a	1345 °F -1435 °F (729 °C-779 °C) for 4–9 hours, furnace cool to 1165 °F– 1255 °F (629 °C-679 °C), and hold for total aging time of 12 hours minimum ^b
N09945	125K	1800 °F–1950 °F (982 °C–1066 °C)	0.5 to 4 ª	1250 °F–1350 °F (677 °C–732 °C) for 4–9 hours, furnace cool to 1110 °F– 1190 °F (599 °C–643 °C), and hold for total aging time of 12 hours minimum ^b
N09946	140K	1800 °F–1950 °F (982 °C–1066 °C)	0.5 to 4 ª	1250 °F–1350 °F (677 °C–732 °C) for 4–9 hours, furnace cool to 1110 °F– 1190 °F (599 °C–643 °C), and hold for total aging time of 12 hours minimum ^b

Table 3—Heat Treatment Procedures (Continued)

		Solution Ar	inealing	Age Hardening
UNS Number	Material Designation	Material Temperature	Time (hours)	Material Temperature and Time
N09946	150K	1800 °F–1925 °F (982 °C–1052 °C)	0.5 to 4 ª	1250 °F–1350 °F (677 °C–732 °C) for 4–9 hours, furnace cool to 1110 °F– 1190 °F (599 °C–643 °C), and hold fo total aging time of 12 hours minimum
N09946	160K	1825 °F–1950 °F (996 °C–1066 °C)	0.5 to 4 ^a	1250 °F–1350 °F (677 °C–732 °C) for 4–9 hours, furnace cool to 1110 °F– 1190 °F (599 °C–643 °C), and hold fo total aging time of 12 hours minimum
N09955	120K	1868 °F –1976 °F (1020 °C–1080 °C)	0.5 to 6 ^a	1292 °F–1400 °F (700 °C–760 °C) for 4–8 hours ^d
N09955	140K	1868 °F–1976 °F (1020 °C–1080 °C)	0.5 to 6 ^a	1328 °F–1436 °F (720 °C–780 °C) for 4–8 hours, air cooling to room temperature, heating at 1112 °F–1202 °F (600 °C–650 °C) and hold fo a combined total aging time for both cycles of 10 hours minimum ⁴

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. shall only be upon agreement between purchaser, manufacturer, and end user.

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.

- 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 4.

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

Air cool or faster.

d Air cool.

The temperature range for UNS N06625–95K is an annealing treatment as per ASTM B446 rather than solution annealing.
 Water quench.

Table 4—Etchants Used for Macroetch Examination

Option	Chemistry Volumetric Ratios ^{a, b}	Additional Requirements
Option A: Canada's Etchant	2 –3 parts $\mathrm{H_2SO_4}$ 2–4 parts HF 1 –2 parts HNO $_3$ 8 –10 parts H $_2$ O	Etch at 120 °F to 180 °F (49 °C to 82 °C)
Option B: Aqua Regia	2–3 parts HCI 1 part HNO ₃	_
Option C: Kalling's No. 2 (Waterless Kalling's)	1–2 parts Methanol or Ethanol 1 part HCl 0.05g CuCl _z /ml of HCl	Methanol or Ethanol shall be a minimum of 95 % pure (i.e. reagent quality).
Option D: Hydrochloric—Peroxide	1 part H_2O_2 (30 %) 2 parts HCl 1–3 parts H_2O	Remove stains with 50 % HNO ₃
Option E: Dilute Heated Aqua Regia	25 parts HCI 1–2 parts 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 3.1.4) 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/, 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 midwall 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 5.

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

Milliliters (ml) should be used as the standard unit for ratio parts.

Table 5—Etchants Used for Microstructure Examination

Option	Chemistry Volumetric Ratios ^{a, b}	Additional Requirements
Option A: Kalling's No. 2 (Waterless Kalling's)	1–2 parts Methanol or Ethanol 1 part HCl 0.05g CuCl ₂ /ml of HCl	Methanol or Ethanol shall be a minimum of 95 % pure (i.e. reagent quality).
Option B: Seven acids	10 parts HCI 2 parts HNO ₃ 2 parts H ₃ PO ₄ 1 part 40 % HF 1 part H ₂ SO ₄ 1g FeCl ₃ (anhydrous)/ml of H ₂ SO ₄ 2 parts CH ₃ COOH 10 parts H ₂ O	_
Option C: Diluted Glyceregia	1 part Glycerol 15 parts HCl 1.5 parts HNO ₃	_
Option D: Bromine-Methanol	1 %–3 % Bromine Balance % Methanol or Ethanol	Methanol or Ethanol shall be a minimum of 95 % pure (i.e. reagent quality). Clean in HCl before etching
Option E: Nitric-HCI	1 part HNO ₃ 3–6 parts HCI	_
	Option A: Kalling's No. 2 (Waterless Kalling's) Option B: Seven acids Option C: Diluted Glyceregia Option D: Bromine-Methanol Option E:	Option A: Kalling's No. 2 (Waterless Kalling's) 1-2 parts Methanol or Ethanol 1 part HCl 0.05g CuCl_/ml of HCl 10 parts HCl 2 parts HNO ₃ 2 parts H,PO ₄ 1 part H,PO ₄ 2 parts CH,POOH 10 parts H,PO ₄ 2 parts CH,POOH 10 parts H,PO ₄ 1 part Glycerol 15 parts HCl 1.5 parts HCl 1.5 parts HCl 1.5 parts HNO ₃ 1 %-3 % Bromine Balance % Methanol or Ethanol Option E:

b Milliliters (ml) should be used as the standard unit for ratio parts.

4.2.2.2 Grain Size Evaluation

The average grain size shall be determined in accordance with ASTM E112 for unimodal distribution or ASTM E1181 for random duplex grain size distribution. The ASTM average grain size shall be No. 2 or finer. In the case of random duplex grain size bimodal condition each area fraction average grain size numbers reported shall be No. 2 or finer. No topologically 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 for all alloys shall be free from continuous networks of secondary phases along grain boundaries, except for individual, isolated grains or isolated fields of view (see 3.1.3) that are not representative of the bulk microstructure. The presence of discreet, 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 may not be needed if the final melt source certifies that the material is free from Laves phase.

The reference photomicrographs in Figure A.1 through Figure A.20 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 4.1.5 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 QTC. The test frequency shall be one test per remelt ingot per heat treat lot (see 3.1.4).

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

For solid material, the test specimen shall be removed from a location at $\frac{1}{4}$ 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.

Test method, specimen and orientation shall be per the following:

- a) The testing shall be in accordance with either ASTM A370 or ASTM E8.
- b) The test specimen orientation shall be longitudinal. Alternatively, transverse test specimen orientation may be substituted for longitudinal orientation. Other test specimen orientations are only permitted by agreement with the purchaser.
- c) For test specimen, a standard size (0.500 in. or 12.5 mm gage diameter) round specimen with a gage length with minimum of four times gage diameter (4D) shall be used unless the product size or geometry does not permit for machining of such a specimen and for such cases the largest sub-size round specimen (4D minimum) possible shall be used for solid material while for hollow material either largest sub-size round specimen (4D minimum) or strip specimen shall be used. Strip test specimens are only permitted on hollow material up to 0.750 in. (19 mm) wall thickness.

4.2.3.2 Tensile Test Acceptance Criteria

The tensile properties shall meet the acceptance criteria as shown in <u>Table 6</u>. Rounding of test results to determine conformance to specification shall be in accordance with ASTM E29.

Table 6—Tensile Requirements

		QTC Cross-section	0.2% Yield	0.2% Yield	Tensile Strength	Elongation	Reduction of
UNS	Material Designation	Thickness ^a in. (mm)	Strength Min. ksi (MPa)	Strength Max. ksi (MPa)	Min. ksi (MPa)	in 4D Min. %	Area ^b Min. %
N06625	95K	All cross-section thicknesses	95 (655)	110 (758)	120 (827)	30	40
	1007	≤10 (254)	120 (827)	150 (1034)	150 (1034)	20	35
NO7746	IZUR	>10 (254)	120 (827)	150 (1034)	150 (1034)	20	25
017700	1401/	≤10 (254)	140 (965)	160 (1103)	165 (1138)	18	30
	140V	>10 (254)	140 (965)	160 (1103)	165 (1138)	18	20
	1301	<10 (254)	120 (827)	145 (1000)	150 (1034)	20	35
	IZUN	>10 (254)	120 (827)	145 (1000)	150 (1034)	20	25
NO7740	1401/	≤10 (254)	140 (965)	150 (1034)	165 (1138)	20	35
0177001	404	>10 (254)	140 (965)	150 (1034)	165 (1138)	20	25
	1501/	≤10 (254)	150 (1034)	175 (1207)	175 (1207)	20	35
	NOCI	>10 (254)	150 (1034)	175 (1207)	175 (1207)	20	25
NIOZZOE	1301/	≤10 (254)	120 (827)	150 (1034)	150 (1034)	20	35
CZ / /ON	IZUN	>10 (254)	120 (827)	150 (1034)	150 (1034)	20	25
NOOOS	1101/	≤10 (254)	110 (758)	140 (965)	140 (965)	18	25
CZEEONI	VOI:	>10 (254)	110 (758)	140 (965)	140 (965)	18	20
NOOO3E	1101/	≤10 (254)	110 (758)	140 (965)	140 (965)	18	25
CCGGON	NOIL	>10 (254)	110 (758)	140 (965)	140 (965)	18	20
NOOOAE	1257	<10 (254)	125 (862)	155 (1069)	150 (1034)	18	25
N09945	NC21	>10 (254)	125 (862)	155 (1069)	150 (1034)	18	20
	1401/	<10 (254)	140 (965)	165 (1138)	165 (1138)	18	25
	N041	>10 (254)	140 (965)	165 (1138)	165 (1138)	18	20
9700014	1501/	<10 (254)	150 (1034)	170 (1172)	170 (1172)	18	25
040807	NOCI	>10 (254)	150 (1034)	170 (1172)	170 (1172)	18	20
	1601/	≤10 (254)	160 (1103)	180 (1241)	180 (1241)	18	25
	VOO!	>10 (254)	160 (1103)	180 (1241)	180 (1241)	18	20

Table 6—Tensile Requirements (Continued)

		QTC Cross-section	0.2% Yield	0.2% Yield	Tensile Strength	Elongation	Reduction of
UNS	Material Designation	Thickness a in. (mm)	Strength Min. ksi (MPa)	Strength Max. ksi (MPa)	Min. ksi (MPa)	in 4D Min. %	Area ^b Min. %
	1001	≤10 (254)	120 (827)	145 (1000)	150 (1034)	30	20
10000	NOZI	>10 (254)	120 (827)	145 (1000)	150 (1034)	30	45
CCSSON	4407	≤10 (254)	140 (965)	155 (1069)	165 (1138)	20	40
	Z041	>10 (254)	140 (965)	155 (1069)	165 (1138)	20	35
NOTE Reduction	Area walnes are tripical	Daduction of Area value are troivelly proportional to the amount of hot work and are less influenced by cross-section thickness at time of heat treatment	of hot work and are les	s-sacro vd bearengini sa	ection thickness at time	theat treatment	

operations and, if applicable, prior to any pre-heat treatment machining. For other UNS number/materials designations, the end user may accept QTC cross-section thickness determined after all hot work operations and, if applicable, prior to any pre-heat treatment machining. QTC cross-section thickness at time of heat treatment. For UNS N07718 material designations 120K, 140K and 150K, QTC cross-section thickness may be determined after all hot work

If tested using strip specimens, then the reduction of area requirement does not apply.

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 or same end of the same piece 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 or ASTM E23. All tests shall be performed at or below –75 °F (–60 °C) regardless of API 6A temperature classification. Rounding of test results to determine conformance to specification shall be in accordance with ASTM E29.

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 QTC. The test frequency shall be one set of tests per heat per remelt ingot per heat treat lot (see 3.1.4).

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

For solid material, the test specimens shall be removed from a location at $^{1}l_{4}$ 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.

4.2.4.2 Charpy V-notch Acceptance Criteria

Charpy-V-notch acceptance criteria shall be per Table 7. 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 <u>Table 7</u> regardless of specimen size. When the smallest sub-sized specimen per API 6A cannot be extracted, Charpy testing is not required.

4.2.4.3 Retesting

If a test fails, a retest of three additional specimens (removed from the required location within the same QTC or same end of the same piece with no additional heat treatment) may be made, each of which shall exhibit an impact value equal to or exceeding the required minimum average value and a lateral expansion not less than the required minimum value.

Table 7—Charpy V-notch Impact Toughness Requirements (10 mm × 10 mm)

UNS Number	Material Designation	QTC Cross-section ^a Thickness in. (mm)	Orientation b, c	Minimum Average ft-lbs (J)	Minimum Single ft-lbs (J)	Minimum Lateral Expansion in. (mm)
		< 3 (76)	Longitudinal	74 (100)	66 (90)	0.025 (0.64)
N06625	95K	≥ 3 (76) through 10 (254)	Transverse	60 (81)	55 (75)	0.025 (0.64)
		> 10 (254)	Transverse	55 (75)	49 (67)	0.025 (0.64)
		< 3 (76)	Longitudinal	40 (54)	35 (47)	0.015 (0.38)
	120K	≥ 3 (76) through 10 (254)	Transverse	37 (50)	32 (43)	0.015 (0.38)
N07716		>10 (254)	Transverse	32 (43)	27 (37)	0.015 (0.38)
1107716		< 3 (76)	Longitudinal	40 (54)	35 (47)	0.015 (0.38)
	140K	≥ 3 (76) through 10 (254)	Transverse	30 (41)	27 (37)	0.015 (0.38)
		> 10 (254)	Transverse	30 (41)	27 (37)	0.015 (0.38)
		< 3 (76)	Longitudinal	50 (68)	45 (61)	0.015 (0.38)
	120K	≥3 (76) through 10 (254)	Transverse	35 (47)	30 (41)	0.015 (0.38)
		> 10 (254)	Transverse	30 (41)	27 (37)	0.015 (0.38)
		< 3 (76)	Longitudinal	50 (68)	45 (61)	0.015 (0.38)
N07718	140K	≥3 (76) through 10 (254)	Transverse	35 (47)	30 (41)	0.015 (0.38)
		> 10 (254)	Transverse	30 (41)	27 (37)	0.015 (0.38)
		< 3 (76)	Longitudinal	50 (68)	45 (61)	0.015 (0.38)
	150K	≥3 (76) through 10 (254)	Transverse	35 (47)	30 (41)	0.015 (0.38)
		> 10 (254)	Transverse	30 (41)	27 (37)	0.015 (0.38)
		< 3 (76)	Longitudinal	40 (54)	35 (47)	0.015 (0.38)
N07725	120K	≥ 3 (76) through 10 (254)	Transverse	37 (50)	32 (43)	0.015 (0.38)
		> 10 (254)	Transverse	32 (43)	27 (37)	0.015 (0.38)
		< 3 (76)	Longitudinal	35 (47)	32 (43)	0.015 (0.38)
N09925	110K	≥3 (76) through 10 (254)	Transverse	35 (47)	32 (43)	0.015 (0.38)
		> 10 (254)	Transverse	35 (47)	32 (43)	0.015 (0.38)
		< 3 (76)	Longitudinal	35 (47)	30 (41)	0.015 (0.38)
N09935	110K	≥ 3 (76) through 10 (254)	Transverse	30 (41)	25 (34)	0.015 (0.38)
		> 10 (254)	Transverse	25 (34)	20 (27)	0.015 (0.38)
		< 3 (76)	Longitudinal	50 (68)	45 (61)	0.015 (0.38)
N09945	125K	≥ 3 (76) through 10 (254)	Transverse	40 (54)	35 (47)	0.015 (0.38)
		> 10 (254)	Transverse	30 (41)	27 (37)	0.015 (0.38)
		< 3 (76)	Longitudinal	45 (61)	40 (54)	0.015 (0.38)
	140K	≥ 3 (76) through 10 (254)	Transverse	35 (47)	30 (41)	0.015 (0.38)
		> 10 (254)	Transverse	30 (41)	27 (37)	0.015 (0.38)
N100043		< 3 (76)	Longitudinal	45 (61)	40 (54)	0.015 (0.38)
N09946	150K	≥ 3 (76) through 10 (254)	Transverse	35 (47)	30 (41)	0.015 (0.38)
		> 10 (254)	Transverse	30 (41)	27 (37)	0.015 (0.38)
	1001/	≥ 3 (76) through 10 (254)	Transverse	27 (37)	25 (34)	0.015 (0.38)
	160K	> 10 (254)	Transverse	27 (37)	25 (34)	0.015 (0.38)

Table 7—Charpy V-notch Impact Toughness Requirements (10 mm × 10 mm) (Continued)

		QTC		Minimum	Minimum	Minimum
UNS Number	Material Designation	Cross-section ^a Thickness in. (mm)	Orientation b, c	Average ft-lbs (J)	Single ft-lbs (J)	Lateral Expansion in. (mm)
	120K	< 3 (76)	Longitudinal	80 (108)	75 (102)	0.025 (0.64)
		≥ 3 (76) through 10 (254)	Transverse	60 (81)	55 (75)	0.025 (0.64)
N09955		> 10 (254)	Transverse	55 (75)	50 (68)	0.025 (0.64)
1409955	140K	< 3 (76)	Longitudinal	60 (81)	55 (75)	0.020 (0.51)
		≥ 3 (76) through 10 (254)	Transverse	40 (54)	35 (47)	0.015 (0.38)
		> 10 (254)	Transverse	35 (47)	30 (41)	0.015 (0.38)

^a QTC cross-section thickness at time of heat treatment. For UNS N07718 material designations 120K, 140K and 150K, QTC cross-section thickness may be determined after all hot work operations and, if applicable, prior to any pre-heat treatment machining. For other UNS number/materials designations, the end user may accept QTC cross-section thickness determined after all hot work operations and, if applicable, prior to any pre-heat treatment machining.

4.2.5 Hardness Test Requirements

4.2.5.1 Test Location, Method and Frequency

4.2.5.1.1 Production Material Surface Hardness Testing

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

Alternatively, for each piece of production material, near surface hardness testing on a cross-section shall be acceptable. The near surface test location shall be 0.100 in. to 0.150 in. (2.54 mm to 3.81 mm) from the surface and shall be performed using the Rockwell C-scale test 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 8. No individual hardness number may be greater than 2 HRC units above the maximum specified hardness number. For Brinell testing, a single indentation is sufficient. For requirements on conversion and reporting of hardness measurements, see Table 8, footnote a. In case of dispute, the Rockwell C-scale shall be the referee test method and shall take precedence.

4.2.5.1.2 Qualification Test Coupon (QTC) Cross Section Hardness Testing

The QTC shall be either a prolongation (full cross-section on the 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. For solid material, the center, V_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 8. The hardness test location for the near surface test shall be 0.100 in.—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.

For QTC cross-section thickness of 0.25 in. (6.35 mm) and below, it is acceptable to test in three separate areas that approximate the locations stated above.

See 4.2.4.1 for specific requirements regarding the orientation.

^c For QTC cross-section thickness < 3 in. (76 mm) Charpy impact testing done in transverse orientation as an alternate to longitudinal orientation is acceptable if the results meet the requirements for QTC cross-section thickness ≥ 3 in. (76 mm) through 10 in. (254 mm).</p>

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 and Conversion

The hardness tests shall meet the acceptance criteria shown in Table 8.

The conversion of hardness readings to or from other scales is material-dependent. For the alloys covered by API 6ACRA, Rockwell C-scale is the preferred test method because ANSI/NACE MR0175/ISO 15156 specifies hardness in Rockwell C-scale, and compliance with ANSI/NACE MR0175/ISO 15156 is frequently required. When conversions from other hardness scales to Rockwell C-scale are required, one of two methods shall be used:

- A hardness conversion based on the empirically derived formula between Brinell and Rockwell C-scale has been specially developed for age hardened nickel-based alloys. See <u>Annex C</u> for all converted values and statistical analysis. Based on <u>Annex C</u>, the HBW conversions for each HRC value are included in <u>Table 8</u>. The HBW to HRC conversion scale described in <u>Annex C</u> is intended only for the materials listed in API 6ACRA.
- An alternative hardness conversion agreed between the purchaser, manufacturer and end user.

In the event of a discrepancy between Brinell (or alternative) hardness measurements and the Rockwell C-scale value per material designation, Rockwell C-scale shall be the referee test method. When reporting converted hardness numbers, the measured hardness and test scale shall be reported in parentheses. For example, 40 HRC (388 HBW), where 40 HRC is the converted hardness value and 388 HBW is the original measurement value and test scale.

4.2.5.3 Retests

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 and shall meet the requirements of 4.2.5.1.1. If Brinell hardness testing is used and the converted Rockwell hardness does not meet the requirements of Table 8, two additional Brinell tests may be taken in the immediate area and both individual values shall meet the converted Rockwell hardness values per material designation listed in Table 8.

At the manufacturer's discretion, for pieces where cross-sectional 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 8—Hardness Requirements

UNS Material Number Designation		Minimum Hardness HRC (HBW °)	Maximum Hardness HRC (HBW ^a)	
N06625	95K	22 (242)	30 (298)	
N07716	120K	32 (315)	43 (419)	
NU7716	140K	34 (332)	43 (419)	
	120K	32 (315)	40 (388)	
N07718	140K	34 (332)	44 (430) b	
	150K	35 (341)	45 (441)	
N07725	120K	32 (315)	43 (419)	
N09925	110K	26 (268)	38 (368)	
N09935	110K	24 (255)	34 (332)	
N09945	125K	32 (315)	42 (409)	
	140K	34 (332)	42 (409)	
N09946	150K	35 (341)	46 (452)	
	160K	36 (350)	46 (452)	
NOOGE	120K	32 (315)	40 (388)	
N09955	140K	34 (332)	42 (409)	

Converted Brinell hardness value per Annex C.

Cross-section Testing:

If any mean (average) hardness value does not meet requirements, a retest may be made by taking three additional indentations in the immediate area of the same location (see 4.2.5.1.2 for test locations). Each new indentation shall meet the requirements of Table 8. The test specimen surface may be re-grounded before performing retesting. Only one re-grind and retest is allowed for each specimen.

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 at a minimum:

- a) a statement certifying the material was produced to the specified UNS number and unique material designation (see <u>Tables 1, 2, 3, 6, 7</u>, and <u>8</u> for UNS numbers and <u>Tables 3, 6, 7</u> and <u>8</u> for material designations).
- b) chemical analysis results (see 4.1.2);
- c) melt practice used (see 4.1.3);
- d) name(s) of company and facility performing melting operations;
- e) name(s) of company and facility performing the hot working operations;

^b By agreement on the purchase order, this material designation may be manufactured to 40 HRC maximum. (See ANSI/NACE MR0175/ISO 15156 for hardness limits for specific temperature and/or elemental sulfur environments.)

- f) name(s) of company and facility performing the heat treatment;
- g) total hot-work reduction ratio (see 4.1.4);
- h) actual heat-treatment times and temperatures and cooling media (see 4.1.5);
- i) name(s) of company and facility performing testing;
- j) statement that the material complies with the requirements of the macroetch examination (see 4.2.1);
- statement describing the QTC. For example, "prolongation (full cross-section on thickest end)" or "sacrificial production part";
- for unimodal grain size distribution, average grain size (see <u>4.2.2</u>). For random duplex grain size, report as per ASTM E1181:
- m) statement of compliance with topological duplex grain size testing requirement (see 4.2.2);
- n) statement that the material complies with the requirements of the metallographic examination for deleterious phases (see 4.2.2);
- a set of legible photomicrographs (see <u>4.2.2.3</u>);
- p) tensile test results and test orientation(s) (see 4.2.3);
- q) impact test specimen size, temperature, orientation, and results (see 4.2.4); (see 4.2.4.2 for exceptions).
- r) 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 conversion in <u>Table</u>
 8 is used, the conversion method shall be reported (see 4.2.5);
- s) 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 3.1.4).

7 Addition of New Alloys and/or New Material Designations

For the requirements for adding new alloys or new material designations, see Annex B.

Annex A

(informative)

Reference Microstructures

These photomicrographs (Figures A.1 through A.20) are representative of this product's microstructure (not individual, isolated grains).

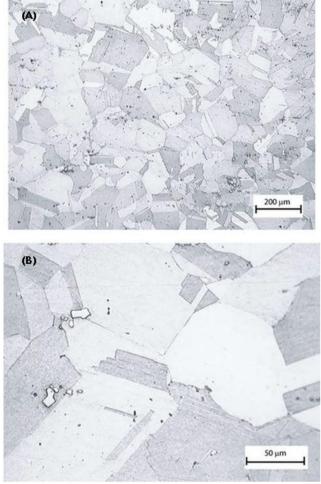
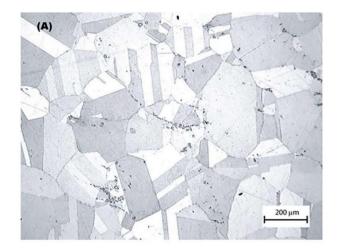


Figure A.1—Acceptable Microstructure for UNS N07718; Original Magnification: 100X for Upper Photomicrograph (A); 500X for Lower Photomicrograph (B)



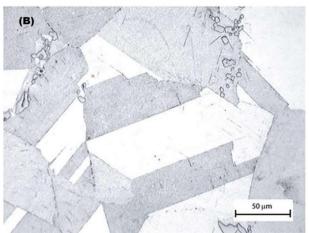


Figure A.2—Acceptable Microstructure for UNS N07718; Original Magnification: 100X for Upper Photomicrograph (A); 500X for Lower Photomicrograph (B)

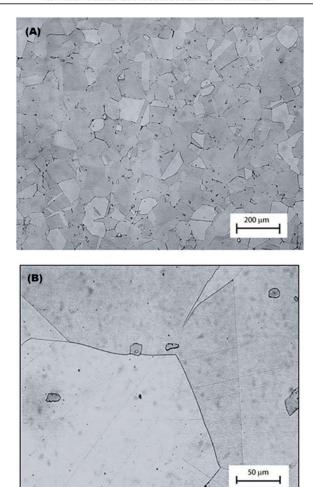


Figure A.3—Acceptable Microstructure for UNS N07718 Showing Isolated Grain Boundary Precipitates; Original Magnification: 100X for Upper Photomicrograph (A); 500X for Lower Photomicrograph (B)

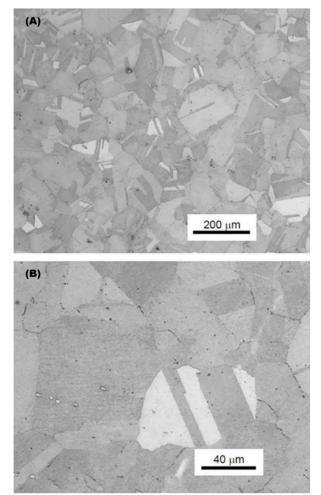


Figure A.4—Acceptable Microstructure for UNS N07718 Showing Isolated Acicular Grain Boundary Precipitates; Original Magnification: 100X for Upper Photomicrograph (A); 500X for Lower Photomicrograph (B)

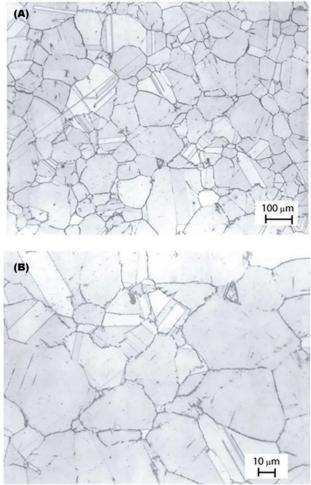


Figure A.5—Unacceptable Microstructure for UNS N07718 Showing Acicular Grain Boundary Precipitates; Original Magnification: 100X for Upper Photomicrograph (A); 500X for Lower Photomicrograph (B)

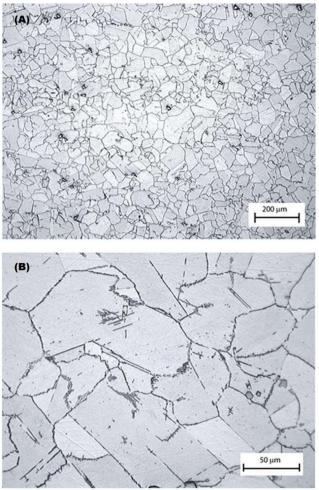


Figure A.6—Unacceptable Microstructure for UNS N07718 Showing Acicular Grain Boundary Precipitates; Original Magnification: 100X for Upper Photomicrograph (A); 500X for Lower Photomicrograph (B)

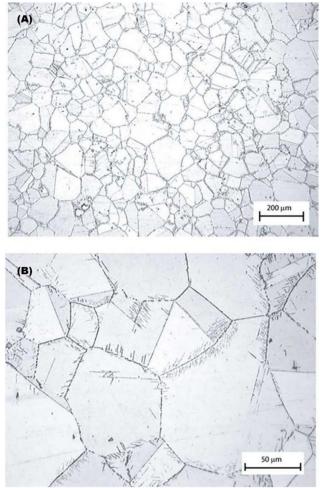


Figure A.7—Unacceptable Microstructure for UNS N07718 Showing Acicular Grain Boundary Precipitates; Original Magnification: 100X for Upper Photomicrograph (A); 500X for Lower Photomicrograph (B)

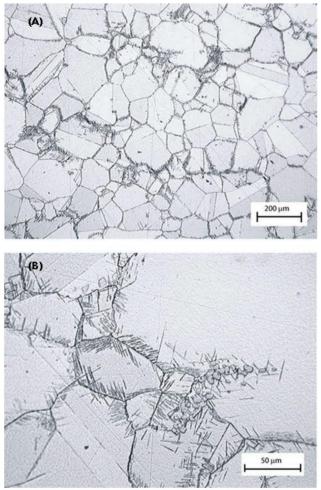


Figure A.8—Unacceptable Microstructure for UNS N07718 Showing Acicular Grain Boundary Precipitates; Original Magnification: 100X for Upper Photomicrograph (A); 500X for Lower Photomicrograph (B)

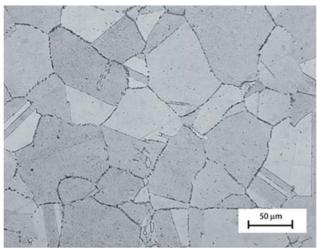


Figure A.9—Unacceptable Microstructure for UNS N07718 Showing Grain Boundary Precipitates; Original Magnification: 500X for Photomicrograph

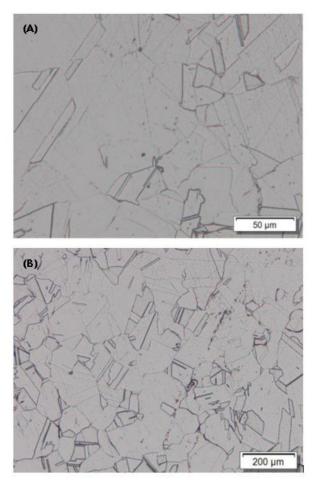


Figure A.10—Acceptable Microstructure for UNS N06625; Original Magnification: 100X for Upper Photomicrograph (A); 500X for Lower Photomicrograph (B)

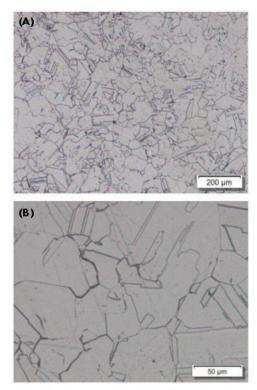


Figure A.11—Acceptable Microstructure for UNS N06625; Original Magnification: 100X for Upper Photomicrograph (A); 500X for Lower Photomicrograph (B)

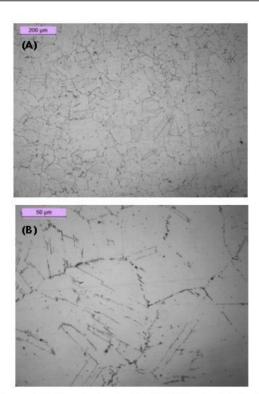


Figure A.12—Unacceptable Microstructure for UNS N06625 Showing Acicular Grain Boundary Precipitates; Original Magnification: 100X for Upper Photomicrograph (A); 500X for Lower Photomicrograph (B)

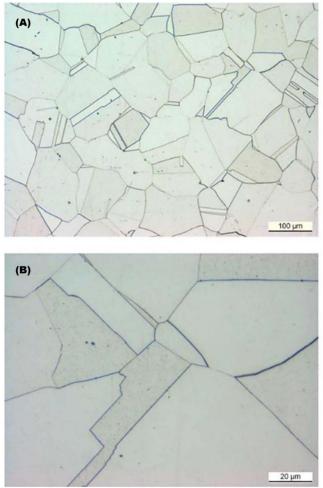


Figure A.13—Acceptable Microstructure for UNS N07716, UNS N07725, UNS N09925, UNS N09935, UNS N09945, UNS N09946 and UNS N09955; Original Magnification: 100X for Upper Photomicrograph (A); 500X for Lower Photomicrograph (B)

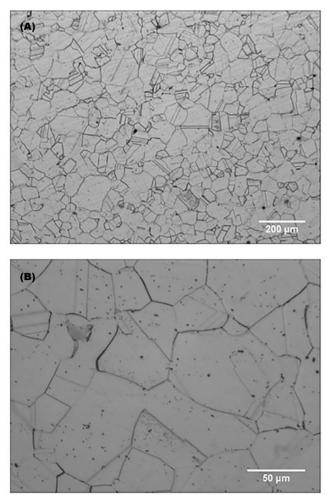


Figure A.14—Acceptable Microstructure for UNS N07716, UNS N07725, UNS N09925, UNS N09935, UNS N09945, UNS N09946 and UNS N09955 Showing Isolated Grain Boundary Precipitation; Original Magnification: 100X for Upper Photomicrograph (A); 500X for Lower Photomicrograph (B)

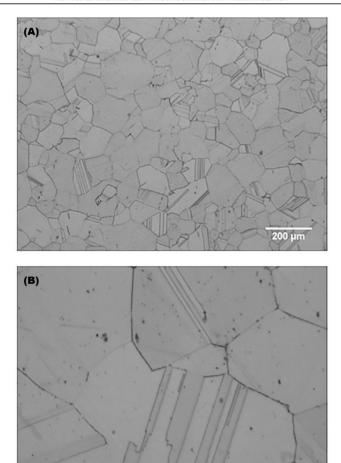
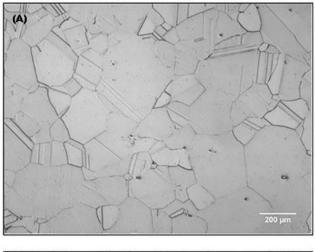


Figure A.15—Acceptable Microstructure for UNS N07716, UNS N07725, UNS N09925, UNS N09935, UNS N09945, UNS N09946 and UNS N09955 Showing Partial Coverage of Grain Boundaries with Second Phase Particles; Original Magnification: 100X for Upper Photomicrograph (A); 500X for Lower Photomicrograph (B)

50 µm



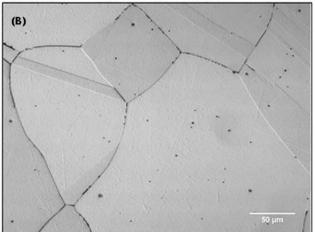


Figure A.16—Acceptable Microstructure for UNS N07716, UNS N07725, UNS N09925, UNS N09935, UNS N09945, UNS N09946 and UNS N09955 Showing Partial Coverage of Grain Boundaries with Second Phase Particles; Original Magnification: 100X for Upper Photomicrograph (A); 500X for Lower Photomicrograph (B)

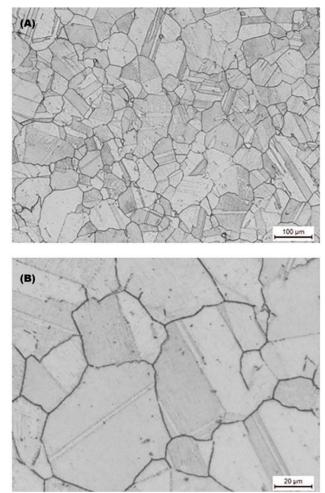
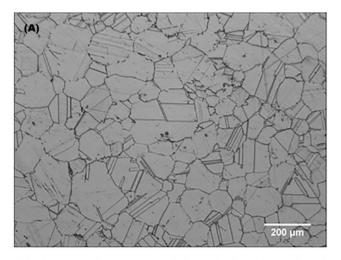


Figure A.17—Unacceptable Microstructure for UNS N07716, UNS N07725, UNS N09925, UNS N09935, UNS N09945, UNS N09946 and UNS N09955 Showing Full Coverage of Grain Boundaries with Second Phase Particles; Original Magnification: 100X for Upper Photomicrograph (A); 500X for Lower Photomicrograph (B)



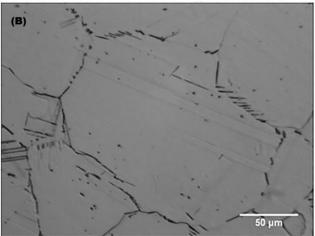


Figure A.18—Unacceptable Microstructure for UNS N07716, UNS N07725, UNS N09925, UNS N09935, UNS N09945, UNS N09946 and UNS N09955 Showing Acicular Grain Boundary Precipitates; Original Magnification: 100X for Upper Photomicrograph (A); 500X for Lower Photomicrograph (B)



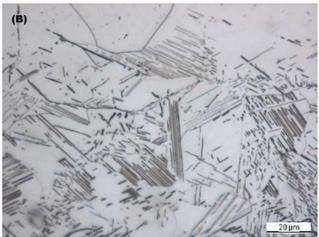


Figure A.19—Unacceptable Microstructure for UNS N07716, UNS N07725, UNS N09925, UNS N09935, UNS N09945, UNS N09946 and UNS N09955 Due to Acicular Precipitates; Original Magnification: 100X for Upper Photomicrograph (A); 500X for Lower Photomicrograph (B)

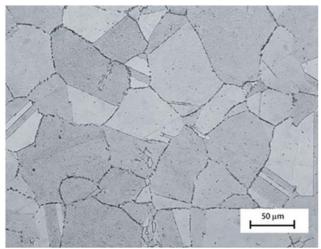


Figure A.20—Unacceptable Microstructure for UNS N07716, UNS N07725, UNS N09925, UNS N09935, UNS N09945, UNS N09946 and UNS N09955 Showing Grain Boundary Precipitates; Original Magnification: 500X for Photomicrograph

Annex B

(normative)

Addition of, or Modification to, Alloys and Material Designations in This Standard

B.1 Addition of New CRAs and/or New Material Designations

B.1.1 Acceptance Criteria

For the addition of new CRAs and/or new material designations to this standard, the following criteria shall be met and supported with documentation:

- a) The alloy shall be an age-hardened nickel-based CRA and have a published UNS number.
- b) The proposed alloy shall be in the solution annealed and age-hardened condition or the annealed and age-hardened condition (i.e. not as-cold worked or other heat treatment conditions).
- c) The alloy shall have been balloted and approved for inclusion in ANSI/NACE MR0175/ISO 15156 in the solution annealed and age-hardened condition or the annealed and age-hardened condition and shall be proposed in a condition that meets the accepted balloted metallurgical requirements.
- d) For melt practice:
 - Each proposed alloy shall have the melt practice(s) stated in the ballot approved for inclusion in ANSI/ NACE MR0175/ISO 15156.
 - Alternate melt practice(s) for the heats reported in the ANSI/NACE MR0175/ISO 15156 ballot may be proposed for Task Group evaluation.
- e) The alloy shall be evaluated in accordance with the requirements in API 6ACRA for a minimum of (3) three separately processed heats representative of commercial product.
- f) The alloy and/or alloy material designation shall have defined heat treatment hold time ranges, temperature ranges, and cooling media, with specific heat treatments of each proposed alloy and/or alloy material designation.
- g) The alloy and/or alloy material designation shall include the proposed microstructural analysis acceptance criteria, tensile test acceptance criteria, Charpy V-notch impact toughness acceptance criteria, and hardness acceptance criteria.
- h) The alloy shall conform to the volumetric NDE requirements of API 6A PSL-3, minimum, for "bodies, bonnets, end and outlet connections and clamp hub end connectors" on raw material for a minimum of (3) three separately processed heats representative of commercial product.
 - NOTE The testing in accordance with API 6ACRA requirements aids in establishing the requirements to be specified in API 6ACRA.

B.1.2 Documentation Requirements

The following documentation shall be supplied for Task Group evaluation:

- a) Requester's name, company name, company address, phone number, and email address.
- b) UNS Number and material designation.
- c) Copy of ANSI/NACE MR0175/ISO 15156 ballot and all test data, including melt practice, submitted to the NACE and ISO committees during the balloting of the alloy for inclusion in ANSI/NACE MR0175/ISO 15156, and documented evidence of ballot approval. If an alternative last melt practice is proposed that was not used for the heats tested in the NACE MR0175 ballot, then documentation of corrosion test data for a minimum three heats of commercial product using the proposed last melt practice and conducted using the same corrosion test methods and exposure conditions as used for the heats included in the NACE MR0175 ballot.
- d) Documentation of the testing in accordance with the certification requirements of <u>Section 5</u> for a minimum of (3) three separately processed heats representative of commercial product.
- e) Documentation of conformance with the API 6A PSL-3 volumetric NDE requirements for "bodies, bonnets, end and outlet connections and clamp hub end connectors" on raw material for a minimum of (3) three separately processed heats representative of commercial product.
- f) Alloy and/or material designation requirements including, but not limited to, chemical composition ranges, melt practice, heat treating time ranges, temperature ranges, and cooling media, macrostructural and microstructural etchants, macrostructural and microstructural analysis acceptance criteria, tensile test acceptance criteria, Charpy V-notch impact toughness acceptance criteria, and hardness acceptance criteria.

B.2 Modifications of Existing CRAs and/or Material Designations

B.2.1 Acceptance Criteria

For modifications to material composition, processing and/or property requirements of existing CRAs and/or material designations in this standard, the following criteria shall be met and supported with documentation:

- The modification proposed shall meet the metallurgical and manufacturing requirements set by ANSI/NACE MR0175/ISO 15156.
- b) Melt practices other than those specified in Table 2 may be proposed for Task Group evaluation.
- c) The modified alloy, or alloy processing, shall be evaluated in accordance with the requirements in API 6ACRA for a minimum of (3) three separately processed heats representative of commercial product. If the modification affects multiple material designations and/or size ranges for the same UNS number, testing shall be for each material designation and/or size range relative to acceptance criteria limits.
- d) The modified alloy and/or material designation shall conform to the volumetric NDE requirements of API 6A PSL-3, minimum, for "bodies, bonnets, end and outlet connections and clamp hub end connectors" for a minimum of (3) three separately processed heats representative of commercial product.

B.2.2 Documentation Requirements

The following documentation shall be supplied for Task Group evaluation:

- a) Requester's name, company name, company address, phone number, and email address.
- b) UNS Number and material designation.

- c) Size range or ranges affected by modification, when applicable.
- d) Documentation of the testing in accordance with the certification requirements of <u>Section 5</u> for a minimum of (3) three separately processed heats representative of commercial product.
- e) Documentation of conformance with the API 6A PSL-3 volumetric NDE requirements for "bodies, bonnets, end and outlet connections and clamp hub end connectors" on raw material for a minimum of (3) three separately processed heats representative of commercial product.
- f) Proposed modifications to requirements including, but not limited to, chemical composition ranges, melt practice, heat treatment time and/or temperature ranges (including cooling media), macrostructural and/or or microstructural etchants, macrostructural and/or microstructural analysis requirements, tensile test requirements, Charpy V-notch impact toughness requirements, or hardness requirements.

Annex C

(informative)

Industry Empirical Data on Hardness Conversion for Age Hardened Nickel-based Alloys

This annex provides justification of conversion parameters for age hardened nickel-based alloys. The data presented is based on several thousand data points from four different material manufacturers laboratories. The statistical analysis of the data resulted in an empirical equation with a regression coefficient (R²) of 0.994. Table C.1 provides the calculated HBW values for HRC based on the empirical equation generated while Figure C.1 shows the best fit equation generated from the data analysis.

Table C.1—Tabulated Hardness Conversions for Age Hardened Nickel-based Alloys Based on Empirically Derived Regression Equation

HRC	Calculated HBW	HRC	Calculated HBW
22	242	35	341
23	248	36	350
24	255	37	359
25	261	38	368
26	268	39	378
27	276	40	388
28	283	41	398
29	291	42	409
30	298	43	419
31	306	44	430
32	315	45	441
33	323	46 a	452 ª
34	332	_	_

To derive the HBW value corresponding to 46 HRC it was extrapolated based on the regression analysis.



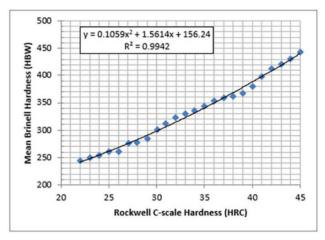


Figure C.1—Rockwell C-scale (HRC) Hardness Values Versus Mean Brinell (HBW) Hardness Values for Age Hardened Nickel-based Alloys, including Empirically Derived Regression Equation

Rockwell C-scale and Brinell hardness values (10 mm ball, 3000 kgf load) were obtained by actual measurements per ASTM E18 and ASTM E10, respectively, on the same specimen at adjacent locations. Measurements were performed on specimens removed from bar prolongations of the following age-hardened nickel-based alloy material designations: UNS N06625 95K, UNS N09935 110K, UNS N09925 110K, UNS N07718 120K, UNS N07718 140K, UNS N07716 120K and UNS N07716 140K. Between 22 and 45 HRC, hardness data were then sorted as Brinell hardness values corresponding to the same Rockwell C-scale hardness values, independent of material designation. The equivalent Brinell hardness value was statistically calculated for each Rockwell C-scale value.

For each Rockwell C-scale value in the range from 22 to 30 HRC, the 95 % confidence interval (95 % CI) of Brinell values was within 25 HBW around the corresponding mean Brinell value. From 31 HRC to 39 HRC and from 40 HRC to 45 HRC, the 95 % CI of Brinell values were within 15 HBW and 20 HBW respectively around the corresponding mean Brinell value.

For example, for $34\,\mathrm{HRC}$ there were $264\,\mathrm{pairs}$ of Rockwell C-scale and Brinell individual hardness measurements reported. The Brinell hardness equivalent corresponding to a $95\,\%$ CI of the data set for $34\,\mathrm{HRC}$ was within the range of $335\pm12\,\mathrm{HBW}$.

Bibliography

- ASTM B446, Standard Specification for Nickel-Chromium-Molybdenum-Columbium Alloy (UNS N06625), Nickel-Chromium-Molybdenum-Silicon Alloy (UNS N06219), and Nickel-Chromium-Molybdenum-Tungsten Alloy (UNS N06650) Rod and Bar
- [2] API Standard 20H, Heat Treatment Services—Batch Type for Equipment Used in the Petroleum and Natural Gas Industry



200 Massachusetts Avenue, NW Suite 1100 Washington, DC 20001-5571 USA

202-682-8000

Additional copies are available online at www.api.org/pubs

Phone Orders: 1-800-854-7179 (Toll-free in the U.S. and Canada) 303-397-7956 (Local and International)

Fax Orders: 303-397-2740

Information about API publications, programs and services is available

on the web at www.api.org.

Product No. G6ACRA2