

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

API STANDARD 6ACRA
FIRST EDITION, AUGUST 2015

ADDENDUM 3, FEBRUARY 2019
ADDENDUM 2, SEPTEMBER 2018
ADDENDUM 1, SEPTEMBER 2017
ERRATA 3, JULY 2022
ERRATA 2, FEBRUARY 2018
ERRATA 1, OCTOBER 2015



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.

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 assure 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 utilized. 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.

Classified areas may vary depending on the location, conditions, equipment, and substances involved in any given situation. Users of this Standard should consult with the appropriate authorities having jurisdiction.

Users of this Standard should not rely exclusively on the information contained in this document. Sound business, scientific, engineering, and safety judgment should be used in employing the information contained herein.

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, Washington, DC 20001.

Copyright © 2015 American Petroleum Institute

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.

Shall: As used in a standard, “shall” denotes a minimum requirement in order to conform to the specification.

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

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, 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, Washington, DC 20001.

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

Contents

	Page
1 Scope	1
1.1 Purpose	1
1.2 Applicability	1
2 Normative References	1
3 Terms, Definitions, and Abbreviated Terms	2
3.1 Terms and Definitions	2
3.2 Abbreviated Terms	3
4 Requirements for Age-hardened Nickel-based Alloys	3
4.1 Process Control Requirements	3
4.2 Testing Requirements	9
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) Criteria for Consideration of a New Alloy and/or Alloy Material Designation	38

Figures

A.1 Acceptable Microstructure for UNS N07718	21
A.2 Acceptable Microstructure for UNS N07718	22
A.3 Acceptable Microstructure for UNS N07718 Showing Isolated Grain Boundary Precipitates	23
A.4 Acceptable Microstructure for UNS N07718 Showing Isolated Acicular Grain Boundary Precipitates ..	24
A.5 Unacceptable Microstructure for UNS N07718 Showing Acicular Grain Boundary Precipitates	25
A.6 Unacceptable Microstructure for UNS N07718 Showing Acicular Grain Boundary Precipitates	26
A.7 Unacceptable Microstructure for UNS N07718 Showing Acicular Grain Boundary Precipitates	27
A.8 Unacceptable Microstructure for UNS N07718 Showing Acicular Grain Boundary Precipitates	28
A.9 Unacceptable Microstructure for UNS N07718 Showing Grain Boundary Precipitates	29
A.10 Acceptable Microstructure for UNS N09925, UNS N09935, UNS N09945, UNS N09946, UNS N09955, UNS N07716, and UNS N07725	30
A.11 Acceptable Microstructure for UNS N09925, UNS N09935, UNS N09945, UNS N09946, UNS N09955, UNS N07716, and UNS N07725 Showing Isolated Grain Boundary Precipitation	31
A.12 Acceptable Microstructure for UNS N09925, UNS N09935, UNS N09945, UNS N09946, UNS N09955, UNS N07716, and UNS N07725 Showing Partial Coverage of Grain Boundaries with Second Phase Particles	32
A.13 Acceptable Microstructure for UNS N09925, UNS N09935, UNS N09945, UNS N09946, UNS N09955, UNS N07716, and UNS N07725 Showing Partial Coverage of Grain Boundaries with Second Phase Particles	33
A.14 Unacceptable Microstructure for UNS N09925, UNS N09935, UNS N09945, UNS N09946, UNS N09955, UNS N07716, and UNS N07725 Showing Full Coverage of Grain Boundaries with Second Phase Particles	34
A.15 Unacceptable Microstructure for UNS N09925, UNS N09935, UNS N09945, UNS N09946, UNS N09955, UNS N07716, and UNS N07725 Showing Acicular Grain Boundary Precipitates	35

Contents

Page

A.16 Unacceptable Microstructure for UNS N09925, UNS N09935, UNS N09945, UNS N09946, UNS N09955, UNS N07716, and UNS N07725 Due To Acicular Precipitates	36
A.17 Unacceptable Microstructure for UNS N09925, UNS N09935, UNS N09945, UNS N09946, UNS N09955, UNS N07716, and UNS N07725 Showing Grain Boundary Precipitates	37

Tables

1 Chemical Composition	5
2 Heat Treatment Procedures	8
3 Etchants Used for Macroetch Examination	9
4 Etchants Used for Microstructure Examination	10
5 Tensile Requirements	13
6 Charpy V-notch Impact Toughness Requirements (10 mm × 10 mm)	15
7 Hardness Requirements	17

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.

NOTE 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-base alloys. Some age-hardened nickel-base alloys that meet the requirements of NACE MR0175 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 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*

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

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*

SAE AMS 2750², *Pyrometry*

ANSI/NACE MR0175³, *Petroleum and natural gas industries—Materials for use in H₂S-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

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 NACE MR0175 and comes from API 6A.

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 the microstructure.

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.

NOTE The 0.008 in. (0.203 mm) $\times$ 0.010 in. (0.254 mm) area is representative of the area shown in a 4 in. (102 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

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 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 ^{c,d}

Element	UNS Number							
	N07716	N07718	N07725	N09925	N09935	N09945	N09946	N09955
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	46.5 to 48.0	52.0 to 55.0	55.0 to 60.0
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	19.5 to 22.5	20.0 to 24.0
Fe ^a	Balance	Balance	Balance	22 min	Balance	Balance	Balance	Balance
Nb	2.75 to 4.00	—	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	—	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	3.00 to 4.00	5.5 to 7.0
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	0.50 to 2.50	0.50 to 1.00
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	0.01 to 0.70	0.25 to 0.80
C	0.030 max	0.045 max	0.030 max	0.025 max	0.030 max	0.005 to 0.040	0.005 to 0.030	0.030 max
Co	—	1.00 max	—	—	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	1.00 max	0.50 max
Si	0.20 max	0.35 max	0.20 max	0.35 max	0.35 max	0.50 max	0.50 max	0.50 max
P	0.015 max	0.010 max	0.015 max	0.020 max	0.025 max	0.020 max	0.020 max	0.015 max
S	0.010 max	0.010 max	0.010 max	0.003 max	0.001 max	0.010 max	0.010 max	0.0010 max
B	0.006 max	0.0060 max	—	—	—	—	—	0.0060 max
Cu	0.23 max	0.23 max	—	1.50 to 3.00	1.00 to 2.00	1.50 to 3.00	1.50 to 3.00	0.044 max
Pb	0.001 max	0.0010 max	—	—	—	—	—	—
Se	—	0.0005 max	—	—	—	—	—	—
Bi	—	0.00005 max	—	—	—	—	—	—
Ca ^b	—	0.0030 max	—	—	—	—	—	—
Mg ^b	—	0.0060 max	—	—	—	—	—	—
W	—	—	—	—	1.00 max	—	—	—

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

^b To be determined if intentionally added.

^c Some limits are more restrictive than the UNS chemistry.

^d “—” 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, UNS N09945, UNS N09946, and UNS N09955

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.