SPECIFICATION FOR 110K YIELD NICKEL BASE PH GRADE 925, WITH MICROSTRUCTURAL REQUIREMENTS, AND MEETING THE LATEST API REQUIREMENTS

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Summary:

This document provides the requirements for Nickel Base Alloy 925 (UNS N09925) with SMYS of 110K for critical service applications. Meeting API 6ACRA, latest edition, is a requirement for this specification. In addition, this specification for Alloy 925 requires approved sources for raw material.

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Abbreviations	
AOD	Argon Oxygen Decarburization
EF	Electric Furnace
EFR	Electroflux Remelting (same as ESR)
ER	Equivalent Round
ESR	Electro-slag Remelting
QTC	Qualification Test Coupon
VAR	Vacuum Arc Remelting
VIM	Vacuum Induction Melting
VOD	Vacuum Oxygen Decarburization

1.0 Scope

This document provides a summary of the requirements for 110K SMYS Nickel Base Alloy 925 (UNS N09925) for critical service applications. Such applications include (but may not be limited to) fasteners, stems and associated equipment where the potential embrittlement by deleterious phases should be avoided.

This specification includes requirements pertaining to detailed process control requirements and detailed testing requirements

API 6ACRA (bar and forgings), latest edition, is a requirement for this specification.

2.0 Approved Vendors

The following vendors are approved for melting, forging and heat treatment, except as noted:

- Foroni SpA
- Special Metals Corp (part of PCC)
- Italfond
- VDM Metals USA (formerly Thyssen Krupp VDM)
- WASA (Western Australia Specialty Alloys--part of PCC) -Melting only
- Bohler Edelstahl
- Carpenter Technologies
- ATI Specialty Materials, LTD (formerly AIT Allvac LTD)
- ATI Allvac
- Alcoa Glossup (formerly Firth Rixon Superalloys)--melting only

Material that is to be acquired or processed by any source not listed in section 2.0 shall be pre-approved by FMC.

The melt source must be one of those specifically listed in 2.0, No Exceptions. All other forge and heat treat facilities used must be an approved FMCTI supplier, listed on the

FMCTI GQSL or GSPSL, and whose scope of work includes processing nickel base alloys.

Due to maximization of efficiencies, it is permissible for the melting mills listed to use the forging, rolling or heat treating capabilities of the other approved companies listed in this section.

3.0 Reference Specifications

Documents	Descriptions
API Spec 6A	Specification for Wellhead and Christmas Tree Equipment
API Spec 6ACRA	Age-hardened Nickel-based Alloys for Oil and Gas Drilling and Production Equipment
ASTM A 370	Standard Test Methods and Definitions for Mechanical Testing of Steel Products
ASTM A 604	Standard Test Method for Macroetch Testing of Consumable Electrode Remelted Steel Bars and Billets
ASTM B 880	General Requirements for Chemical Check Analysis Limits for Nickel, Nickel Alloys and Cobalt Alloys
ASTM E 10	Standard Test Method for Brinell Hardness Test of Metallic Materials
ASTM E 18	Standard Test Methods for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials
ASTM E110	Standard Test Method for Rockwell and Brinell Hardness of Metallic Materials by Portable Hardness Testers
ASTM E 112	Standard Test Methods for Determining Average Grain Size
ASTM E 354	Test Methods for Chemical Analysis of High-Temperature Electrical, Magnetic, and Other Similar Iron, Nickel and Cobalt Alloys
ASTM E 1181	Standard Test Methods for Characterizing Duplex Grain Sizes
ASTM E 1473	Test Methods for Chemical Analysis of Nickel, Cobalt, and High-Temperature Alloys
ASTM E1823	Standard Terminology Relating to Fatigue and Fracture Tests
SAE AMS 2750	Pyrometry
M40187	Specification for 110k yield nickel base PH grade 925, with microstructural requirements, and meeting the latest API requirements

4.0 Process Control Requirements

4.1 Chemical Composition Requirements

4.1.1 Chemical Composition Limits

The chemical composition shall conform to the table 1.

Table 1: Alloy 925 composition. Values are maximums unless otherwise noted.

Ni	42.0-46.0	**Ti**	1.90-2.40	**Si**	0.35	**Pb**	--
Cr	19.5-22.5	**Al**	0.10-0.50	**P**	0.020	**Se**	--
Fe*	22 minimum	**C**	0.025	**S**	0.003	**Bi**	--
Nb(Cb) + Ta	0.08 - 0.50	**Co**	--	**B**	--	**Ca**	--
Mo	2.50-3.50	**Mn**	1.00	**Cu**	1.50-3.00	**Mg**	--

Notes:

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

4.2 Melt Practice Requirements

4.2.1 Acceptable Melt Practices

The alloy shall be melted by one of the following processes (A or B):

A.	B.
Step 1-Basic electric furnace (EF).	Step 1-Vacuum induction melting (VIM).
Step 2-Either argon oxygen decarburization (AOD), vacuum oxygen decarburization (VOD), or vacuum degassing.	Step 2-Either electroslag remelting (ESR) or electroflux remelting (EFR) or vacuum arc remelting (VAR).
Step 3-Either electroslag remelting (ESR), electroflux remelting (EFR) or vacuum arc remelting (VAR).	Optional Step 3- VAR
Optional Step 4-Additional VAR.	

4.3 Forging and Hot Working Requirements

4.3.1 Hot Work Reduction Ratio

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

4.4 Heat Treating Requirements

4.4.1 Solution Annealing and Age Hardening

Material shall be heat treated in accordance with the following procedure:

Solution anneal: 1825°F – 1925°F (996°C – 1052°C) for one half hour minimum to 4 hours maximum.

Cool in air, water, polymer, oil or inert gas to ambient temperature. Air or inert gas cooling of section sizes greater than 3 inches shall only be used upon agreement between purchaser, manufacturer and user.

Age harden: 1325°F – 1400°F (718°C – 760°C) for 4 to 9 hours; then furnace cool to 1125ºF - 1220ºF (607ºC - 660ºC) and hold for a total ageing time of 12 hours minimum.

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

4.4.2 Temperature Monitoring

The material temperature shall be measured by use of either a contact surface thermo-couple (TC) or a heat sink as described in API Spec 6A. The hold time shall not commence until the contact surface TC or 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 TC or heat sink for 5 years minimum following the date of heat treatment.

4.5 Testing Requirements

4.5.1 Macroetch Requirements

4.5.1.1 Test Location, Method and Frequency

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

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

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

The acceptable macro etchants are as follows:

Option A	Canada's Etchant	100 ml $H2SO_4$, 100 ml HF, 50 ml HNO_3, 400 ml H_2O Etch at 160º F - 180º F (71º C - 82º C)
Option B	Aqua Regia	200 ml HCl, 100 ml HNO_3
Option C	Kalling's No. 2 or Waterless Kallings	200 ml Methanol, 200 ml HCl, 10 g $CuCl_2$
Option D	Hydrochloric --Peroxide	H_2O_2 (30%) 100 ml, HCl 200 ml, H_2O 300 ml Remove stains with 50% HNO_3
Option E	Dilute Heated Aqua Regia	250 ml HCl, 10 - 20 ml HNO_3 Etch at 140º F - 165º F (60º C - 74º C)

4.5.1.2 Macroetch Examination and Acceptance Criteria

The macrostructure of the slice shall be examined and rated to all four classes in ASTM A 604. The acceptance criteria are as follows:

Class 1 (Freckles) - Severity A or better

Class 2 (White Spots) - Severity A or better

Class 3 (Radial Segregation) - Severity A or better

Class 4 (Ring Pattern) - Severity A or better

4.5.2 Microstructural Analysis Requirement

4.5.2.1 Test Location, Method and Frequency

Material with the same shape and equivalent round from each remelt ingot per heat treat lot shall be sampled and subjected to a microstructural analysis. Samples taken for evaluation shall be a minimum ¼ in. (6 mm) square and oriented longitudinally to the

primary axis of grain flow. In the event the product cross section is less than ¼ in. (6 mm), the sample(s) shall be full cross section. The microstructural analysis shall be performed after final heat treatment. Test locations shall be a minimum of 1.25 in. (32 mm) from a heat treated end surface.

The center, ¼ thickness and surface locations for solid shapes shall be evaluated. The mid-wall location and both the inner and outer surfaces or hollow shapes shall be evaluated. The acceptable etchants for microstructure evaluation are listed below:

Option A	Kalling's No. 2 or Waterless Kalling's	200 ml Methanol, 200 ml HCl, 10 g $CuCl_2$
Option B	Seven acids	300 ml HCl, 60 ml HNO_3, 60 ml $H3PO_4$, 30 ml 40% HF, 30 ml $H2SO_4$, 30 g $FeCl_3$ (anhydrous), 60 ml CH3COOH, 300 ml H_2O
Option C	Diluted Glyceregia	10 ml glycerol, 150 ml HCl. 15 ml HNO_3
Option D	Bromine-Methanol	Clean in HCl before etching in immersed 1-3% Bromine, Methanol
Option E	Nitric-HCl	10 ml HNO_3, 60 ml HCl

4.5.2.2 Grain Size Evaluation

4.5.2.2.1 Grain Size

The average grain size shall be determined in accordance with ASTM E 112. The ASTM average grain size shall be No.2 or finer.

4.5.2.2.2 Duplex Grain Size

No topological duplex grain size as defined and measured per ASTM E 1181 is permitted.

4.5.2.3 Metallographic Examination for Deleterious Phases

The microstructural samples shall be examined at 100 and 500X for deleterious phases, using optical light microscopy. Microstructural acceptance criteria:

a. The microstructure shall be free from continuous networks of secondary phases along grain boundaries, except for isolated grains or isolated fields of view that are not representative of the bulk of the microstructure. The presence of isolated particles of secondary phases or carbides is acceptable.

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

c. There shall be no laves phase.

NOTE

Examination of the microstructural samples for laves phase is not required if the original melt source certifies that the material is free from laves phase.

The Reference Photomicrographs in figure A.1 through A.17 in Annex A in API6ACRA 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 4.4 and re-examined. In the event a heat treat lot is rejected, other pieces within the rejected heat treat lot may be qualified on a per-piece basis. Material containing rejectable locations based on microstructural features may be accepted if the rejectable locations are removed by machining and no longer contained in the finished part.

The material manufacturer or material supplier shall retain the metallurgical specimen mounts for 5 years minimum following the date of examination. They shall be available for examination by purchasers of this material, during this time period.

4.5.3 Tensile and Impact Property Requirements

4.5.3.1 Test Location, Method and Frequency

One tensile test and one set of three Charpy Impact tests shall be performed for each tested QTC. The test frequency shall be one test per remelt ingot per heat treat lot (batch) (as defined in API Spec 6A) for material of the same size.

The QTC shall be either a prolongation (full cross section on thickest end) or sacrificial production part. For solid material, the test specimen shall be removed from a location at ¼ 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 specimens and test methods shall be in accordance with ASTM A 370. Rounding of test results to determine conformance to specification shall be in accordance with ASTM E29.

All Charpy tests shall be performed at or below –75°F (–60°C) regardless of API Spec 6A Temperature Classification or other FMC specification referenced on part DBI. Specimens shall be oriented transverse to the primary direction of grain flow unless the size or geometry of the QTC prevents the usage of transverse specimens (material less than 3 in. [76 mm] in cross section). Longitudinal specimens shall be used for such cases. 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.

If a tensile test or Charpy test fails, retesting shall meet the requirements of API 6ACRA.

4.5.3.2 Tensile Test Acceptance Criteria

The tensile properties shall meet the acceptance criteria as shown in Table 2.

Table 2-Material Property Requirements

Material Designation	QTC Cross Section Thickness in. (cm)	0.2% Yield Strength min. psi (MPa)	0.2% Yield Strength max. psi (MPa)	Tensile Strength min. psi (MPa)	Elongation in 4D min. %	Reduction of Area min. %
110K	≤ 10 (25.4)	110,000 (758)	140,000 (965)	140,000 (965)	18	25
	> 10(25.4)	110,000 (758)	140,000 (965)	140,000 (965)	18	20

4.5.3.3 Charpy V-notch Acceptance Criteria

The average energy value for a set of three specimens shall meet or exceed the specified average. No more than one of the specimens shall have an energy value below the specified average. No specimens shall have a lateral expansion below the specified value. The adjustment factors for sub-size impact specimens in API Spec 6A shall apply to the absorbed energy values. Lateral expansion shall meet the requirements in Table 3 regardless of specimen size.

Table 3-Charpy V-notch Impact Toughness Requirements (10 mm X 10 mm)

Temperature		QTC Cross Section Thickness in. (cm)	Minimum Impact Value PSL 1, 2, 3, and 4					
			Transverse Direction			Longitudinal Direction		
API Spec 6A Temperature Classification	Test Temperature °F(°C)		Average Value Ft-lbf (J)	Single Minimum Value Ft-lbf (J)	Lateral Expansion in. (mm)	Average Value Ft-lbf (J)	Single Minimum Value Ft-lbf (J)	Lateral Expansion in. (mm)
All	–75 (–60) or below	< 3 (7.6)	Not applicable	Not applicable	Not applicable	35 (47)	32 (43)	0.015 (0.38)
		≥ 3 (7.6) ≤ 10 (25.4)	35 (47)	32 (43)	0.015 (0.38)	Not applicable	Not applicable	Not applicable
		> 10 (25.4)	35 (47)	32 (43)	0.015 (0.38)	Not applicable	Not applicable	Not applicable

4.5.4 Hardness Testing

4.5.4.1 Test Location, Method and Frequency

4.5.4.1.1 Production Material

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

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

For Rockwell C scale testing, three adjacent indentations shall be made, averaged to calculate the mean, and the mean shall meet the hardness limits in Table 4. No individual hardness number may be greater than 2 HRC units above the maximum specified number. For Brinell testing, one indentation is sufficient. For requirements on hardness conversion, see table 4. When a conflict exists between the Rockwell C scale hardness numbers and the Brinell hardness numbers, the Rockwell C scale shall be the referee method.

4.5.4.1.2 Qualification Test Coupon (QTC)

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

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

4.5.4.2 Hardness Test Acceptance Criteria

The hardness tests shall meet the requirements of Table 4.

Table 4-Hardness Requirements

Material Designation	Minimum Hardness HBW (HRC)	Maximum Hardness HBW (HRC)
110K	275 (26)	364 (38)

Note: Brinell conversions are based on industry produced empirical data.

4.6 Nondestructive Examination

At a minimum, the nondestructive examination requirements in API Spec 6A shall apply as required for the specified type and PSL specified on the purchase order. Additional NDE may be specified on the FMC Part Report (DBI).

5.0 Certification

The material supplier shall provide a certified test report containing the following information as a minimum:

- A statement certifying that the material was produced to the specified UNS number and material designation (i.e. 110K)
- Chemical analysis results

- Melt practice utilized
- Name of melt source and facility performing the melting operation
- Name of company and facility performing the hot working operations
- Name of company and facility performing the heat treatment
- Total hot work reduction ratio
- Actual heat treatment times and temperatures and cooling media
- Name of company and facility performing testing
- Statement that the material complies with the requirements of the macroetch examination
- Average grain size
- Statement of compliance with topological duplex grain size testing requirement
- Statement that the material complies with the requirements of the metallographic examination for deleterious phases. A set of legible photographs taken at 100X and 500X at the specified locations shall be included.
- Tensile test results
- Impact test temperature, specimen size, orientation, and results
- Hardness test results, reported by test location in the same scale as used for the measurements and the converted value, if converted. Report hardness conversion method.
- NDE results

6.0 Marking

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

7.0 Documentation

Documentation that is in compliance with EN 10204 type 3.1 inspection certificates shall be supplied provided to SARA with each shipment of material. This type of certificate requires the supplier to provide test results for all requirements listed in specifications that are attached to the Part Report.