

# Materials Reliability Program: Guidelines for Thermally Treated Alloy 690 Pressure Vessel Nozzles (MRP-241)

### Materials Reliability Program: Guidelines for Thermally Treated Alloy 690 Pressure Vessel Nozzles (MRP-241)

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#### REPORT SUMMARY

This document provides guidelines for procuring and processing thick-wall thermally treated Alloy 690 seamless pipe and tube or bar material for new or replacement pressure vessel nozzles. The guidelines include Alloy 690 specifications and a discussion of the bases for these requirements.

#### **Background**

Thermal treatment has the primary objective of producing a product with a microstructure containing a desirable grain boundary carbide distribution that results in low residual stresses and demonstrates a superior resistance to stress corrosion cracking (SCC). These objectives are similar to those for Alloy 690 steam generator tubing EPRI first developed in 1991 (EPRI report NP-6743-V2) and revised in 1999 (EPRI report TR-016743-V2R1). In the case of steam generator tubing, however, a single tube fabricator is usually involved in producing all the tubing for a steam generator. In the case of pressure vessel nozzles, while a metal producer also plays a major role, the vessel manufacturer will also process the product. The vessel manufacturer could impart cold work and residual stresses during their processing. Consequently, the utility needs to take responsibility for ensuring that low residual stresses are achieved throughout the entire nozzle preparation phase.

#### **Objectives**

To help utilities make sure that the thick-wall thermally treated Alloy 690 seamless pipe and tube (SB-167), or bar (SB-166) material for new or replacement pressure vessel nozzles have optimum resistance to primary water stress corrosion cracking (PWSCC).

#### **Approach**

The project team gathered information from utilities; material suppliers; and nuclear system, supply, and service vendors to develop specifications for thick-wall thermally treated Alloy 690 materials for new or replacement pressure vessel nozzles and to detail the bases for these requirements.

#### Results

The specifications in this report can help a utility develop a procurement specification for thick walled pipe and tube or bar for new or replacement pressure vessel nozzles. When supplier specifications for Alloy 690 are used for procurement instead of utility developed specifications, the specifications and bases in this report can be used by utilities as a checklist for review of the supplier specifications.

#### **EPRI Perspective**

This Guideline and its referenced reports constitute a technical basis for setting requirements for procurement of thermally treated Alloy 690 thick walled tubing and bar material for use in the manufacture of pressure vessel nozzles. However, Revision 0 of this document, MRP-241, is not intended for implementation by plants. As such, the guidance provided in this document is not required under the implementation protocol of the Nuclear Energy Institute (NEI) 03-08.

#### **Keywords**

Nuclear Steam Generators Pressure vessel nozzles Specifications Inconel Alloys PWR Stress Corrosion Cracking

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## **1** INTRODUCTION

The objective of these guidelines is to assist utilities in ensuring that the thick-wall thermally treated Alloy 690 seamless pipe and tube (SB-167) or bar (SB-166) material for new or replacement pressure vessel nozzles has optimum resistance to primary water stress corrosion cracking (PWSCC). The thermal treatment has the primary objective of producing a product with a microstructure containing a desirable carbide distribution in the grain boundary that has demonstrated superior stress corrosion cracking (SCC) resistance and low residual stresses.

These objectives are similar to those for Alloy 690 steam generator tubing that were first developed in 1991 (NP-6743-L) [1] and revised in 1999 (TR-016743-V2R1) [2]. However, for steam generator tubing, the desired microstructure was based primarily on SCC resistance to secondary-side-related environments [3, 4]; resistance to primary water SCC was assumed and later demonstrated by long-term SCC testing [5, 6, 7].

Factors that can affect SCC resistance of Alloy 690 in pressurized water reactor (PWR) primary water continue to be studied [8]. Some of this recent work reveals that not just cold work, but also inhomogeneous deformation (that is, one-dimensional rolling or tensile straining), appears to be detrimental to crack growth rate behavior through PWSCC [8]. In addition, questions have been raised (and are yet unresolved) about the possible influence of melt practice on the PWSCC resistance of Alloy 690 [8]. The necessity of a thermally treated microstructure for PWSCC resistance has not been definitively demonstrated, and it is by analogy to Alloy 600 that the thermal treatment has been considered appropriate for Alloy 690.

For steam generator tubing, a single tube fabricator usually produces all the tubing for a steam generator. For pressure vessel nozzles, a metal producer has a major role to play, but the nozzle will likely be processed by the vessel manufacturer. During their processing, the vessel manufacturer could impart cold work and residual stresses. Consequently, the utility must take responsibility for ensuring that low residual stresses are achieved through the entire nozzle preparation phase. For example, an additional thermal treatment might be requested for a nozzle that has required extensive machining or forming during preparation, and a surface treatment during or after the attachment of the nozzle to the vessel might also be required. This type of control is beyond the scope of this document, but it can play a role in achieving the objective of optimum PWSCC resistance.

The specification contained in Section 2, Specification for Thermally Treated Alloy 690 (UNS N06690) Seamless Pipe and Tube or Bar, is considered suitable for use as a guide by a utility in the development of a procurement specification for thick-walled pipe and tube or bar for new or replacement pressure vessel nozzles. It is expected that, in many cases, Supplier specifications for Alloy 690 will be used for procurement, rather than utility-developed specifications. In such

#### Introduction

cases, the specification and bases in Sections 2 and 3, Bases for Alloy 690 Specification, respectively, can be used by utilities as a checklist for a review of the Supplier specifications.

The specific limits defined in Section 2 should be considered as bounds. In order to obtain consistent product, utilities may want to impose more restrictive limits based upon a review of recent Supplier information or specific utility goals.

## 2

## SPECIFICATION FOR THERMALLY TREATED ALLOY 690 (UNS N06690) SEAMLESS PIPE AND TUBE OR BAR

#### 2.1 Scope

This specification contains the requirements for the manufacture, quality assurance, examination, testing, and shipment of thick-wall thermally treated nickel-chromium-iron Alloy 690 (UNS N06690) seamless pipe and tube or bar with special requirements for chemical composition, heat treatment, microstructure, and quality control for manufacturing high pressure vessel nozzles.

#### 2.2 Applicable Documents and Codes

This specification incorporates the requirements for bar of ASME SB-166 Alloy UNS N06690 or for seamless pipe and tube of ASME SB-167 in accordance with ASME III NB-2000, as supplemented by Code Case N698, and incorporates additional requirements. These additional requirements are not in conflict with ASME III NB-2000, ASME II SB-166, or ASME II SB-167. If there are specific requirements in this document that appear to conflict with specific requirements of other referenced documents, these conflicts shall be referred to the Purchaser for resolution. Conflicts must be resolved prior to production.

#### 2.2.1 Code of Federal Regulations

Use the revision or issue that is in effect when the purchase order is submitted, unless the Purchaser specifies otherwise.

10CFR21 Reporting of Defects and Noncompliance

10CFR50 Appendix B Quality Assurance Criteria for Nuclear Power Plants and Fuel

Reprocessing Plants

#### 2.2.2 American Society for Testing and Materials (ASTM)

Use the revision or issue that is in effect when the purchase order is submitted, unless the Purchaser specifies otherwise.

ASTM A751	Standard Test Methods, Practices and Terminology for Chemical Analysis of Steel Products
E3	Preparation of Metallographic Specimens
E8	Test Methods for Tension Testing of Metallic Materials
E18	Rockwell Hardness and Rockwell Superficial Hardness Testing of Metallic Materials
E45	Determining the Inclusion Content of Steel
E112	Estimating the Average Grain Size of Metals
E354	Chemical Analysis of High-Temperature, Electrical, Magnetic, and Other Similar Iron, Nickel, and Cobalt-Base Alloys
E663	Practice for Flame Atomic Absorption Analysis
E930-99	Test Methods for Estimating the Largest Grain Observed in a Metallographic Section (ALA Grain Size)
E1019	Methods for Determination of Carbon, Sulfur, Nitrogen, Oxygen, and Hydrogen in Steel and in Iron, Nickel, and Cobalt Alloys
E1024	Guide for Chemical Analysis of Metals and Metal Bearing Ores by Flame Atomic Absorption
E1181	Test Methods for Characterizing Duplex Grain Sizes
E1473	Test Methods for Chemical Analysis of Nickel, Cobalt, and High- Temperature Alloys

## 2.2.3 American Society of Mechanical Engineers (ASME) Boiler and Pressure Vessel Code

Use the revision or issue that is in effect when the purchase order is submitted, unless the Purchaser specifies otherwise.

ASME NQA-1-1994	Quality Assurance Program Requirements for Nuclear Facilities
ASME SB-166	Specification for Nickel-Chromium-Iron Alloys (N06600 and N06690) Round Bar. ASME III Nuclear Power Plant Components, Division 1, Subsection NB, Class 1
ASME SB-167	Specification for Nickel-Chromium-Iron Alloys (N06600 and N06690) Seamless Pipe and Tube. ASME III Nuclear Power Plant Components, Division 1, Subsection NB, Class 1

Specification for Thermally Treated Alloy 690 (UNS N06690) Seamless Pipe and Tube or Bar

ASME Code Case N-525 Design Stress Intensities and Yield Strength Values for UNS

N06690 with a Minimum Specified Yield Strength of 30 ksi (206 MPa), Class 1 Components Section III, Division 1. December 9,

1993

ASME Code Case N-698 Design Stress Intensities and Yield Strength Values for UNS

N06690 with a Minimum Specified Yield Strength of 35 ksi (240 MPa), Class 1 Components Section III, Division 1. November 18,

2003

ASME Section V Non-Destructive Examination, Article 6

#### 2.2.4 American Society for Nondestructive Testing (ASNT)

Use the revision or issue that is in effect when the purchase order is submitted, unless the Purchaser specifies otherwise.

SNT-TC-1A 1992: Standard Practices for Nondestructive Testing as amended

by ASME Section III NB-5500

#### 2.3 Definitions

Cr and/or M(C, N) type carbide banding - Cr and/or M(C, N) type carbide banding is intragranular chromium carbide particles that are linearly distributed throughout the microstructure.

**discolorations** – Discolorations are caused by oxidation at high temperature (oxide tinting).

**fiber stress** – Fiber stress is local stress through a small area (a point or line) on a section where the stress is not uniform, as in a beam where it is under a bending load.

**final solution annealing** – Final solution annealing is a high-temperature annealing that is given a product after its reduction to final size. Solution annealing is performed for the purposes of recrystallization and dissolution of precipitates, especially carbides.

**heat -** A heat consists of the material from one single molten batch. If an ingot is remelted, then each remelt charge is a separate heat.

**heat treatment -** Heat treatment is a process that involves raising the temperature of the product significantly above room temperature [932°F (500°C) or higher] to accomplish some objective such as re-crystallization, carbide precipitation, or stress relief. Any "intermediate annealing," "final solution annealing," "stress relief," and "thermal treatment" used in this specification are heat treatments.

Specification for Thermally Treated Alloy 690 (UNS N06690) Seamless Pipe and Tube or Bar

**heat treatment lot**. A heat treatment lot is limited to product from the same heat and that meets all of the following criteria:

- Product reduced to size using the same type of equipment and reduction schedule
- Product subjected to the same solution annealing practice, including time and temperature
- Product exposed to the same thermal treatment practice
- Product straightened with essentially the same straightening equipment and equipment setup

**hot working lot -** See *heat treatment lot*.

**product material** – Product material, in this specification, should be considered as thermally treated Alloy 690 (ASME SB-166 Alloy N06690 round bar or ASME SB 167 Alloy N06690 seamless pipe and tube SB-166 or SB-167) product that may be produced in cold worked annealed, hot worked annealed, or hot finished conditions.

**solution annealing** - Solution annealing is a high-temperature annealing. It includes both intermediate annealing and final solution annealing.

**thermal treatment** - Thermal treatment is a heat treatment given to the product in its final state in order to develop a desired grain boundary carbide distribution and to reduce residual stresses to low levels. The temperature of the thermal treatment is in the carbide precipitation range around 1292°F (700°C).

#### 2.4 Technical Requirements

The product, either finished thick-wall seamless pipe and tube or bar produced to these guidelines, shall be a highly uniform and consistent product with as low variability in yield strength, grain size, and other parameters as practical. The product shall be low in residual stresses and cold work, and shall be essentially free of surface imperfections.

Achieving these goals requires close attention to each step of the production process, starting from the initial billet and carrying on to the stress relief (if applicable), inspection, and packaging. Careful attention shall be paid to anomalies discovered during preproduction runs and during production. Anomalies such as an excessive amount of surface flaws shall be evaluated for root cause, and these root causes shall be addressed to prevent a reoccurrence. A manufacturing process plan should be submitted by each Supplier, approved by the Purchaser, and any deviations from that manufacturing process plan should be reported and approved by the Purchaser.

#### 2.4.1 Product Material

The product material shall be thermally treated Alloy 690 (ASME SB-166 Alloy N06690 round bar or ASME SB 167 Alloy N06690 seamless pipe and tube, with additional requirements of ASME Code Case N-698, SB-166, or SB-167) product that may be produced in either cold worked annealed, hot worked annealed, or hot finished conditions. This guideline specifically adds a thermal treatment, as defined in Section 2.5.2.2, Thermal Treatment, with the intent to develop a desirable carbide distribution in the grain boundary (described in Section 2.4.6.2, Desirable Microstructure). The achievement of this microstructure may, by necessity, require that the product be annealed prior to the thermal treatment, thus excluding the hot finished condition from consideration. However, the Supplier, based on their production experience, shall provide a procedure for the Purchaser's approval that describes the operations through final product. The procedure shall specifically define the melting and working practices to be followed. It is recognized that annealing from different hot finishing temperatures will give different final grain sizes based on the recrystallization response to any prior hot work and that different grain sizes will yield different degrees of intergranular carbide decoration based on mass availability of carbon and chromium.

#### 2.4.2 Dimensions

Dimensions shall be in accordance with a drawing submitted with the purchase order.

#### 2.4.3 Chemical Composition

The chemistry for the ladle and product (check) analyses shall be in accordance with SB-166 or SB-167 and Table 2-1 of this document. Purchasers may wish to include more restrictive target values based on review of the Supplier's recent products to ensure that the current product is not significantly higher in residuals than recently produced products.

Table 2-1 Alloy 690 (N06690) Product Chemistry

Element	Percent
Nickel (min)	58.0
Chromium	28.5–31.0
Iron	9.0–11.0
Carbon	0.015–0.035
Silicon (max)	0.50
Manganese (max)	0.50
Cobalt (max)	0.05
Copper (max)	0.10
Sulfur (max)	0.003
Phosphorus (max)	0.015
Nitrogen (max)	0.050
Aluminum (max)	0.40
Boron (max)	0.005
Titanium (max)	0.40
Molybdenum (max)	0.20
Niobium (max)	0.10

The chemical composition of each heat shall be determined in accordance with ASTM E354 (or ASTM A751) by an analysis of ingot samples. Alternative methods may be used if approved by the Purchaser, including atomic absorption spectrophotometry per ASTM E663 ands E1024. A check analysis shall be obtained from one piece selected randomly from each lot of finished product and shall be recorded on the Certified Material Test Report (CMTR).

Chemical analyses for carbon shall be performed in accordance with ASTM E1019 or E354, or an alternative method approved by the Purchaser. The method and equipment used shall have been demonstrated to accurately determine carbon content to within  $\pm 0.002\%$ . Depending upon the product form, that is, pipe/tube or bar, the Supplier shall recommend the appropriate carbon analysis sampling to the Purchaser for approval. Care shall be taken during the removal of samples to ensure that contamination does not occur. Results for all analyses shall be reported to the same number of decimal places as specified in Table 2-1.

#### 2.4.4 Mechanical Properties

Room temperature mechanical properties of the product, following thermal treatment according to Section 2.5.2.2, Thermal Treatment, shall conform to the requirements of the appropriate minimum ultimate tensile strength, yield strength, and elongation of the SB-166 or SB-167 specification. See Section 3.4.4, Mechanical Properties, for a summary. The additional requirement of this specification is that the yield strength shall not be greater than 55 ksi (379 MPa).

A through-thickness hardness test (ASTM E18) shall be performed near the inner surface, close to mid-wall, and near the outside surface on one sample per lot. It shall be reported for information only. It is recommended that the hardness shall not exceed Rockwell 95 HRB.

#### 2.4.5 Hydrostatic Tests

Each pipe or tube shall show no evidence of leakage during hydrostatic testing at an internal hydrostatic pressure set to result in the fiber stress specified in ASME SB-167, paragraph 12.3. The water used for hydrostatic testing shall meet the requirements of Table 2-2.

Table 2-2
Demineralized Rinse Water Requirements

Element/Property	Maximum Concentration/Limit of Element/Property
Chloride ion	0.05 ppm maximum
Fluoride ion	0.05 ppm maximum
Total sulfates	0.05 ppm maximum
Sodium ion	0.05 ppm maximum
Conductivity	2 microSiemens/cm maximum
рН	5.5 to 8.0
Clarity	No turbidity, oil, or sediment
Total suspended solids	0.1 ppm maximum

#### 2.4.6 Microstructure Examination

Microstructure examinations shall be performed on one product sample per lot taken from locations near each mechanical property test location and shall include, as a minimum, specimens aligned in the longitudinal (major axis) direction, although specimens oriented in the transverse direction are also encouraged. Metallographic evaluation shall be performed on each lot of product in accordance with ASTM E3. This evaluation shall be performed on material in the final product condition, that is, after final thermal treatment, except that microcleanliness tests for inclusions may also be performed at any stage prior to final condition.

#### 2.4.6.1 Grain Size

The grain size shall be determined as described in ASTM E112. The grain size shall be reported (100X micrograph supplied of an area that is representative of where the grain size was rated) for longitudinally oriented specimens, for information only. An objective should be to have the grain size ASTM No. 3 or finer. If the microstructure is observed to have a mixture of grain sizes, the range of observed grain sizes shall be reported when determined as described in ASTM E1181.

#### 2.4.6.2 Desirable Microstructure

One sample per lot of final product shall be taken in the longitudinal direction and evaluated to verify that the desired carbide microstructure has been achieved.

A desirable microstructure shall be characterized by continuous, or nearly continuous, chromium carbide precipitation on all grain boundaries and a very low density of intragranular carbide—either Cr-type carbide or M(CN) carbide as defined in Section 2.4.6.3, Microcleanliness. Since it is difficult to avoid carbide banding completely, the level of carbide banding allowed shall be in accordance with microcleanliness requirements. There are two types of carbide banding:

- Primary (from the melt) M(C,N)-type carbonitrides that form stringers along the direction of metal deformation
- Alternating bands of grains with and without intergranular Cr carbides that are aligned along the direction of metal flow

The preferred magnification for characterizing and recording the grain boundary microstructure is 500X. A 100X magnification for characterizing and recording the grain boundary microstructure must be included for macrostructural non-uniformities to be detected. Quality micrographs shall be obtained using scanning electron microscopy, although optical microscopy can also be used.

Several metallographic etching techniques can be used and shall be agreed upon by the Purchaser and Supplier. The Purchaser and Supplier shall also agree upon a gallery of acceptable and unacceptable microstructures.

#### 2.4.6.3 Microcleanliness

Testing shall be performed according to ASTM E45 Method A for grain size and microstructural analyses. Reporting shall be according to Method A of ASTM E45, except that the method JK charts for Method D may be used. Each specimen shall be examined at least at the edge and midwall positions, using fields that are selected randomly from these areas, and shall not exceed the recommended maximum inclusion ratings shown in Table 2-3.

Table 2-3
Recommended Maximum Inclusion Ratings

			Ox	ides and S	Sulfides					
Туре	Α		В		С		D			
	Т	Н	Т	Н	Т	Н	Т	Н		
	1.0	0.5	3.0	1.0	1.0	0.5	1.5	1.0		
Ti-Carbonitrides										
	Туре		В			D				
	_	Т		Н	Т		Н			
		3.0		3.0	4.0		3.0			

Purchasers may wish to define more restrictive values and target values based on a review of recent heats from their selected Supplier to ensure that they receive product that is representative of the Supplier's recent product or to ensure the improved material's cleanliness and homogeneity.

#### 2.4.6.4 General and Intergranular Attack

If acid cleaning (pickling) is used for the removal of oxides, etc., verification of freedom from general and intergranular attack is mandatory for one piece of product from each lot. A transverse sample shall be evaluated metallographically around the full circumference on the outside and inside (if applicable) diameters (ODs and IDs). There shall be no intergranular attack (IGA) or general corrosion on these surfaces. In addition, the surfaces shall be evaluated at a magnification of 500X to verify that the sample is free from deleterious carburization, decarburization, and nitriding, as demonstrated by the material-not-exceeding amounts of these conditions shown on reference photomicrographs prepared by the Supplier and agreed to by the Purchaser.

#### 2.4.7 Surface Roughness

Surface roughness or surface finish limits shall be agreed to and defined in the purchase order. Surface roughness can vary depending on the product form, but it should be RMS 250 microinches (6.35 µm) or finer.

#### 2.5 Process Requirements

#### 2.5.1 Melting and Metal Working Practices

Melting and metal working practices that ensure a final product of high quality with respect to uniformity, homogeneity, cleanliness, cracks, galling, laps, seams, and similar quality features

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shall be used. The Supplier shall provide a procedure for Purchaser approval that describes the operations from melting through final finish working.

In the event of remelting an ingot, the Supplier is required to identify whether any reheats came from the top or bottom of the parent heat. In addition, the Supplier shall inform the Purchaser about any attempt to remelt a heat due to equipment failure, power loss, or any other event that causes a melting process to be interrupted in midstream. The Supplier shall also have a procedure to deal with such process interruptions. This procedure shall be submitted by each Supplier and approved by the Purchaser; any deviations from that manufacturing process plan should be reported to and approved by the Purchaser.

#### 2.5.2 Heat Treatment

#### 2.5.2.1 Final Solution Anneal

Subsequent to the final reduction, all final product shall be solution annealed. The minimum acceptable temperature shall be established by the Supplier based on the composition (particularly carbon content), microstructure, and mechanical requirements of this specification. The range between the hottest and coldest metal temperatures of the product in a furnace lot shall not exceed 20°C (36°F). The Supplier shall select the cooling rate with the objective of minimizing carbide precipitation. In addition, furnace temperature shall be continuously recorded.

#### 2.5.2.2 Thermal Treatment

The final product shall be heat-treated around  $1292 \pm 27^{\circ}F$  ( $700 \pm 15^{\circ}C$ ) for 10 hours minimum. The thermal treatment shall be performed after final solution annealing, straightening, and any mechanical surface treatment. Alternative thermal treatments may be proposed and used, if appropriately qualified and if Purchaser approval is obtained. The furnace atmosphere shall not result in deleterious changes in the composition of the ID or OD surfaces (decarburizing, carburizing, or nitriding) as shown by metallurgical evaluation (see Section 2.4.6, Microstructure Examination). Maximum cumulative time at temperature for thermal treatment plus stress relief shall not exceed 35 hours. Surface discolorations shall not obscure any rejectable indication and should be evaluated to understand their cause.

#### 2.5.3 Straightening, Machining, or Grinding

Any machine straightening, machining, or grinding shall be performed after solution annealing and before thermal treatment. The resultant cold work and variation in tensile properties shall be minimized and monitored during the straightening operation in order to avoid excessive residual stress or undesirable precipitation during thermal treatment. The straightening process shall not result in a total increase in yield strength of more 13 ksi (90 MPa).

Machine restraightening, machining, or grinding shall not be performed after final thermal treatment or nondestructive testing. However, these operations are permitted after thermal treatment if the operations are followed by at least an additional 2-hour period of thermal treatment and all nondestructive tests are also repeated. The residual plastic strain (cold work) introduced by grinding, machining, or straightening will not be removed by the low-temperature thermal treatment since recrystallization is required to remove plastic deformation. The goal should be to limit the magnitude of cold work introduced by these operations.

Spot hardness checks of material that has undergone any of these operations should be performed. The total time at thermal treatment temperature shall not exceed the limit of 35 hours

#### 2.5.4 Repair

The product shall be uniform in quality; sound; free from (that is, free from when evaluated with specified methods) seams, cracks, tears, laminations, and laps; and free from pitting, IGA, and other injurious defects. The product surface shall be free from deleterious manufacturing-induced damage, such as drawing marks, as evidenced by samples not having surface conditions exceeding those of the physical standards proposed by the Supplier and accepted by the Purchaser. In addition, the physical standards shall cover acceptable and unacceptable local repairs, for example, blending (if local repairs are allowed).

After final solution annealing and before thermal treatment, defects on the outside surface may be removed as long as the minimum wall thickness is maintained, surface roughness limits are met, and the area is blended uniformly. Defect removal after final thermal treatment shall be limited to Purchaser-approved methods. Removal of the defect shall be verified using the same method as originally used to find the defect. The defect removal process shall not leave embedded particles on the product's outside surface. No weld repairs shall be performed.

#### 2.5.5 Cleaning

Prohibited materials, which are defined in Section 2.5.6.1, Prohibited Materials, shall not come into contact with the product material at any time. In addition, prior to any operations involving high temperatures, the material shall be cleaned to ensure that any detrimental contaminants have been removed (detrimental materials are also defined in Section 2.5.6, Prohibited and Detrimental Materials). The cleaning processes that may be used include alkaline detergent washing and vapor degreasing, as long as these processes are followed by appropriate cleaning steps to ensure that no residual contaminants remain on the surfaces prior to any heat treatment operations. Grit blasting and acid cleaning may be used subject to the controls given in Sections 2.5.5.2, Grit Blasting, and 2.5.5.3, Acid Cleaning, respectively.

#### 2.5.5.1 Demineralized Water

The quality of the demineralized water used for any cleaning steps or pressurization testing shall be monitored continuously using conductivity meters or their equivalent and shall be checked by analysis at least once per month for compliance with Table 2-2.

#### 2.5.5.2 Grit Blasting

Grit blasting may be performed on any OD or ID surfaces; however, if grit blasting is performed on the product after reduction to final size, the surface roughness requirements of Section 2.4.7, Surface Roughness, must still be met. If performed, grit blasting shall use zirconia.

Zirconia grit blast materials shall be free of metallic particles and shall not have been previously used on non-nickel-base alloys. Grit particles shall not become embedded in the product surfaces; examination to verify freedom from embedment shall be per Section 2.5.6.3, Freedom from Grit Embedment.

No grit blasting shall be performed after final thermal treatment unless qualification tests are performed to demonstrate that residual stresses resulting from use after thermal treatment are low tensile or compressive, and Purchaser approval is obtained. Grit blasting shall not obscure surface or internal defects.

#### 2.5.5.3 Acid Cleaning

Acid cleaning may be used after reduction to final size. An acid cleaning process, which may include steps such as OD polishing or ID grit blasting performed after the acid cleaning, shall be qualified by metallurgical examination of test pieces to show that it does not result in deleterious attack as defined in Section 2.4.6.4, General and Intergranular Attack.

#### 2.5.6 Prohibited and Detrimental Materials

#### 2.5.6.1 Prohibited Materials

Red lead-graphite-mineral oil or molybdenum disulfide lubricants shall not be used, that is, these materials are prohibited from contact with the product material at any time. Metallic lead, mercury, cadmium, or other low melting temperature metals or alloys are also prohibited from contact with the material at any time.

#### 2.5.6.2 Furnace Atmosphere

During heat-treatment, if fuel or combustion gases can come into contact with the product material, the fuel shall not contain more than 0.5% sulfur by weight or 30 grains maximum per 100 ft<sup>3</sup> (2.8 m<sup>3</sup>).

#### 2.5.6.3 Freedom From Grit Embedment

If grit blasting is performed on any product, one sample per lot shall be removed from a gritblasted area and examined using stereomicroscopy at a magnification of 60X to verify the absence of embedded grit particles on any OD or ID surface.

#### 2.6 Quality Assurance Program Requirements

For all the work covered under this specification, there shall be in effect a written QA Program in compliance with the provisions of 10CFR50 Appendix B and the quality assurance requirements required by specified codes and standards. The QA program manual shall be submitted to the Purchaser for approval. After the Purchaser's approval, if significant changes to this QA Program become necessary that may affect the performance of work covered in this specification, the Supplier shall, prior to implementation, submit such changes to the Purchaser for review and approval. The Supplier's approved QA Program is subject to audit by the Purchaser's quality assurance representative to ensure compliance with applicable purchase documents.

If an ASME III QA Program Manual is submitted with the intent of complying with 10CFR50 Appendix B, the submittal shall be accompanied by a letter signed by an authorized official of the Supplier's company specifically stating this fact and identifying those parts or services, if any, not covered by the ASME III program. For those parts or services not covered by the ASME III QA program, the Supplier shall also submit, to the Purchaser, the applicable QA Program Manual in compliance with 10CFR50 Appendix B. Unless specific exceptions are noted, the ASME QA program shall be applied to all pressure-retaining parts and services furnished under the specification. The entire QA program submittal shall be subject to the Purchaser's approval.

#### 2.6.1 Identification and Control of Materials, Parts, and Components

The Supplier shall establish and maintain a system for the identification and control of materials. These measures shall ensure that identification of each item is maintained by heat number and lot number, either on the item or on records traceable to the item during shipment and use of the item. These identification and control measures shall be designed to prevent the use of incorrect or defective material.

#### 2.6.2 Supplier's Responsibilities for Subsuppliers

The Supplier shall identify, in purchase documents to his subsuppliers, all applicable quality and QA requirements imposed by the Purchaser's specification on the Supplier and shall ensure compliance thereto.

#### 2.6.3 Notification and Hold Points

The Purchaser shall have the right to establish notification points for which the Supplier shall give prior notification to the Purchaser. In addition, the Purchaser may establish temporary notification points if they deem them necessary to ensure resolution of temporary quality problems. Notification points require receipt of notification at least five working days in advance of the scheduled first time of performance. The Purchaser's quality assurance representative will witness the event or will authorize the Supplier to proceed without the Purchaser's witnessing of the event.

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Selection of notification requirements shall reflect the fact that product fabrication is a continuous process. Purchaser witnessing of notification point activities must not interfere with production work.

The Purchaser may require that activities performed without proper notification be repeated for the Purchaser's quality assurance representative to observe at the Supplier's expense.

If specified in the purchase order, the Supplier shall not carry work beyond designated hold points until released in writing by the Purchaser's representative.

## 2.6.4 Requirements for Nondestructive Examination, Visual Inspection, Dimensional Inspection, and Surface Roughness Check

#### 2.6.4.1 Test and Inspection Responsibility.

The Supplier shall be responsible and shall provide for or perform all the inspection and testing required by this guideline. The inspection and testing shall be conducted in a manner satisfactory to and subject to the approval of the Purchaser. All inspection and test reports and mill test certificates shall be submitted by the Supplier to the Purchaser.

The Purchaser, his customer, or their authorized representatives shall have access to the Supplier's and any Subcontractor's premises at all reasonable times to the extent necessary to assess compliance with the provisions of this specification. In addition, the Supplier and his Subcontractors shall make chart recordings of all production acceptance testing, such as ultrasonic testing, on a lot basis, and these shall be available to the Purchaser for assessment of compliance. Charts of the recorded heat treatment temperatures shall also be made available to the Purchaser or to his authorized representative for review and assessment of compliance.

The Purchaser or his authorized representative shall also have the right to conduct on the Supplier's premises, at his own expense, any additional inspection or testing he deems necessary above the preproduction and production testing described herein. Such additional tests or inspections will be for the information of the Purchaser to verify that the specified quality requirements are being met and will not be used to introduce requirements additional to those stated in this specification.

In the event of failure of the material or product to fully meet any inspection or test requirement specified herein, the Supplier shall notify the Purchaser and ask if he wishes to repair/rework and/or use such material or product. If the repairs/rework could potentially affect the results of tests or work previously completed, appropriate reinspection and retesting shall be conducted to the satisfaction of the Purchaser.

#### 2.6.4.2 Nondestructive Examination

#### 2.6.4.2.1 General

Nondestructive examination (NDE) of all material shall be in accordance with ASME III, subsection NB-2500, for the final product form in which the material will be used and the additional supplement requirements contained herein.

NDE procedures shall be submitted for Purchaser approval. Changes to inspection procedures shall be made only after approval of the Purchaser. If nondestructive examination procedures required by this specification have previously been submitted and approved, a statement may be submitted in lieu of the procedure that will be subject to concurrence by the Purchaser prior to implementation. The statement shall clearly identify the procedure, including revision or issue number, the date of submittal, the project, and the contract number for which it was submitted. In addition, it shall be affirmed that the procedure is exactly as previously submitted and is applicable to this specification.

Drawings for all NDE reference standards shall be submitted with the applicable NDE procedures for the Purchaser's approval.

All personnel performing nondestructive examinations shall be qualified to the American Society for Nondestructive Testing, SNT-TC-1A, as modified by ASME Section III NB-5500. Only personnel certified as Level II or III to SNT-TC-1A shall be responsible for equipment calibration and interpretation and for evaluation of the results. Only Level III personnel shall establish techniques.

#### 2.6.4.2.2 Visual and Tactile Inspection

All products shall be visually inspected with the naked eye. No cracks, linear indications, or large scratches are acceptable. Visual standards for scratches shall be mutually agreed upon between the Supplier and Purchaser. If due to product form, tactile inspection is deemed applicable, the Supplier and Purchaser shall mutually agree upon how to perform the inspection.

#### 2.6.4.2.3 Dimensional Inspection

All straight products shall be dimensionally inspected to verify compliance with purchase order requirements (see Section 2.11, Ordering Requirements).

#### 2.6.4.2.4 Surface Finish Checks

The surface finish shall be inspected to verify compliance with purchase order requirements.

#### 2.7 Documentation

#### 2.7.1 Records System

A records system shall be established and maintained to provide documentary evidence of the quality of items and activities affecting quality. The quality assurance (QA) records shall include results of reviews, inspections, tests, audits, work performance monitoring, and material analyses. Records shall, as a minimum, identify the inspector or data recorder, the date the inspection was performed, the type of observation, the procedures used, the results, the acceptability, and the action taken with any deficiencies noted. Records of relevant supporting data shall also be maintained. All quality verification records, procedures, and qualifications shall be identifiable to the item or activity involved. These records shall be retrievable and available for examination.

#### 2.7.2 Documentation Checklist

Prior to the start of fabrication, the Supplier shall prepare and submit to the Purchaser, for review and approval, a preliminary documentation checklist detailing the quality assurance documents that will be required to comply with this specification and the referenced codes and standards. This checklist shall itemize, by document type, documents that will be submitted to the Purchaser for information or approval and records that will document the results of inspections and tests. Upon completion of product fabrication, testing, and inspection, but prior to release for shipment, the checklist shall be finalized to show the procedures actually used and the records that document the results of all inspections and tests performed. The final documentation checklist shall be verified for accuracy and completeness and submitted to the quality assurance representative.

#### 2.7.3 Document Submittals

This specification requires specific documents to be formally submitted to the Purchaser for information or review and approval. If these documents are changed subsequent to submittal, the Supplier shall resubmit the revised documents to the Purchaser for information or review and approval consistent with the original requirement. Any document required by this specification that is produced by a subsupplier of the Supplier shall first be reviewed and noted as being approved by the Supplier and then submitted to the Purchaser for review and approval.

#### 2.7.4 Supplier's Documentation

QA documents are a deliverable item. The Supplier's QA organization shall approve the documents and present them to the Purchaser. The Supplier shall assemble all QA records into two identical sets. Each page of each document submitted shall be clearly identified and numbered. Each individual document shall be legible and suitable for electronic scanning or microfilming. No information shall be recorded closer than 5/8 in. (16 mm) to the binding edge or closer than 1/4 in. (6 mm) to any other edge of the paper.

Documents that have been submitted with a previous shipment on this order/contract shall not be duplicated. However, a statement shall be furnished to the Purchaser itemizing, by document, the documents previously furnished for each item of equipment and the date of that previous submittal.

#### 2.7.5 Final Inspection and Check of Records

The Supplier shall be responsible for inspecting the items and checking the applicable records, prior to shipment, to verify that all specification requirements have been complied with. Two complete sets of all documents required to comply with this specification shall be submitted to the Purchaser. Acceptance of the completed sets of records does not relieve the Supplier of responsibility for compliance with specification requirements.

After completion, but prior to submittal of these records, the Supplier shall complete and submit a Certificate of Compliance. The Certificate of Compliance, which is supplied as part of the specification, is a document that certifies that the inspections and tests required by the specification have been satisfactorily completed and that all of the requirements of this specification and applicable codes and standards have been met. The Certificate of Compliance shall be completed and signed by the Supplier's quality representative and submitted to the Purchaser, together with the other documentation applicable to the shipment.

#### 2.7.6 Shipping Release

The Supplier's completed Certificate of Compliance must be included in each shipment. This requirement also applies to shipments from the Supplier's suppliers when items are to be shipped directly to the Purchaser or its contractors.

#### 2.7.7 Record Retention

All records required by this specification, applicable regulations, codes, and standards, or generated as a result of the Supplier's QA Program shall be retained in the Supplier's file for the period specified in the ordering data after the contract requirements for manufacture have been complied with. At the expiration of this period, the Purchaser or his authorized agent shall be provided the option of receipt and/or the Supplier's continued retention of the file contents. No Quality Assurance records shall be destroyed or otherwise disposed of without written permission from the Purchaser.

## 2.8 Manufacturing Documentation Supplied by the Supplier to the Purchaser's Quality Assurance and Control Representatives with the Shipment

#### 2.8.1 NDE Reports

An inspection report shall be prepared by the Supplier for each required NDE and shall include the following in addition to the information required by ASME III:

- Purchaser's purchase order number
- Name of part
- Method or technique

#### 2.8.2 Certified Materials Test Report

A Certified Materials Test Report in accordance with ASME Section III shall be furnished for each lot of material supplied and shall include at least the following:

- Material chemistry and heat number
- Mechanical properties
  - Yield strength
  - Ultimate tensile strength
  - Elongation
  - Hardness (for information)
- Metallographic evaluation results
  - Grain size
  - Desirable microstructure (photomicrographs shall be provided and shall identify the alloy, heat number, lot number, hot working temperature, final solution anneal, thermal and stress-relief heat-treatment temperatures and times, etchant, and magnification)
  - Product microcleanliness (inclusion rating)
  - Check for IGA (if required)
  - Check for fabrication defects
- Hydrostatic test results (if required)
- Results of nondestructive tests
  - Ultrasonic
  - PT (if required)
  - Eddy current (if required)

#### 2.8.3 Heat-Treatment Documents

The Supplier shall keep a record of the product processing and make it available upon request of the Purchaser, including:

• Actual time at or above a minimum required temperature during final solution annealing and thermal treatment

For continuous furnace heat treatments, this information shall include the periodic metal temperature measurements required by Section 2.5.2.1, Final Solution Anneal, together with the recorded furnace temperature.

# 2.8.4 Certificate of Compliance

The Supplier shall provide a Certificate of Compliance as stated in Section 2.7.5, Final Inspection and Check of Records.

# 2.9 Purchaser Witnessing of Inspections

The Purchaser or his authorized representative shall have the right to witness any inspections or tests. Purchaser notification shall be handled as stated in Section 2.6.3, Notification and Hold Points.

# 2.10 Shipment

The product shall be delivered clean, free from organic material or films (oil, grease, paint, crayon), moisture, chemical residue, preservative, chips, or foreign matter. The packing and marking material must conform to the prohibited material requirements of Section 2.5.6.1, Prohibited Materials, of this specification.

Each piece shall be tagged with a unique identification number that is traceable to all other pertinent records, such as purchase order number, CMTR, etc. The shipment package shall be clearly identified as to alloy type and purchase order number.

# 2.11 Ordering Requirements

Purchasing documents shall specify the following:

- 1. Title and date of this specification
- 2. Whether check analyses of chemistry are required for each lot (see Section 2.4.3, Chemical Composition)
- 3. Product dimensions and tolerances (see Section 2.4.2, Dimensions.)
  - OD
  - ID (if applicable)

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- Wall thickness (if applicable)
- Total length (if straight)
- Straightness
- 4. Quantity
- 5. Compliance with 10CFR21
- 6. Whether any hold points are required and, if so, at what stage of manufacture (see Section 2.6.3, Notification and Hold Points)
- 7. Length of record retention in accordance with Section 2.7.7, Record Retention

# **3**BASES FOR ALLOY 690 SPECIFICATION

This section describes the bases for the requirements in Section 2 for the Alloy 690 specification.

Many of the bases were derived from an Alloy 690 guideline for steam generator tubing first developed in 1991 (NP-6743-L) [1] and revised in 1999 (TR-016743-V2R1) [2]. Although some of the requirements for thin wall steam generator tubing may not apply to pipe and thick wall tube or bar, many others do. The philosophy contained in the steam generator tubing guidelines, that is, to produce a quality product with favorable mechanical properties, microstructure, and reduced residual stresses, is carried on in the development of material requirements used for nuclear pressure vessel nozzles.

# 3.1 Scope

This section does not contain requirements, but it serves to provide justification and reasons for the requirements in Section 2.

# 3.2 Applicable Documents and Codes

# 3.2.1 Code of Federal Regulations

This section invokes U.S. Nuclear Regulatory Commission (NRC) required reporting (10CFR21) and quality assurance requirements (10CFR50 Appendix B). These requirements are mandatory for U.S. nuclear plants.

# 3.2.2 American Society for Testing and Materials (ASTM)

This section lists ASTM specifications commonly used for nickel alloys. The specifications are invoked by specific reference elsewhere in the Alloy 690 specification. It should be noted that Section 2.4.3, Chemical Composition, allows the use of methods for chemistry analysis in addition to those covered by the specifications listed in Section 2.2.2, American Society for Testing and Materials, as long as the methods are qualified.

# 3.2.3 American Society of Mechanical Engineers (ASME) Boiler and Pressure Vessel Code

This section invokes ASME SB-166 (bar), SB-167 (pipe and tube), and section NB-2000 of ASME Section III. These portions of the ASME Code are the applicable ones for nickel alloy cold-worked annealed, hot-worked annealed, and hot-finished conditions. Since the NRC invokes the ASME Code, these sections are mandatory.

# 3.2.4 American Society for Nondestructive Testing (ASNT)

This section invokes SNT-TC-1A: 1984, Standard Practices for Nondestructive Testing as amended by ASME Section III NB-5500.

#### 3.3 Definitions

The definitions are intended to ensure that terms used in the specification such as "lot" and "thermal treatment" are understandable in an unambiguous way. See Section 2.3, Definitions, for a list of definitions.

# 3.4 Technical Requirements

A preamble or introductory statement is included to emphasize the need for the product to be as homogeneous as practical and as free of surface imperfections, cold work, and residual stresses as practical.

#### 3.4.1 Product Material

This section invokes the applicable ASME specification (SB-166 or SB-167) and Section III of the ASME Code. In addition, it prohibits use of intermediate circumferential welds. This requirement is included because SB-167 requires tubes to be seamless, that is, to not have longitudinal seam welds, but it does not specifically prohibit intermediate circumferential welds. Intermediate circumferential welds are undesirable because they increase the probability of material defects being present and also increase the possibility of corrosion problems arising during service.

#### 3.4.2 Dimensions

Standard ordering requirements apply to the dimensions.

# 3.4.3 Chemical Composition

The EPRI Materials Reliability Program- (MRP-) specified material chemistry limits are given in Table 3-1, in comparison with those of SB-166/167. When compared to SB-166/167, the MRP limits are somewhat more restrictive and definitive. The reasons for the changes are described in the paragraphs following the table. In addition, wording was adopted from the steam generator specification indicating that the utility may want to include more restrictive target values based on a review of the supplier's past product to ensure that the current product does not deviate significantly from this past product or to provide improved cleanliness and homogeneity.

Table 3-1 Comparison of SB-166/167 Chemistry to the MRP Guidelines Chemistry Limits

Element	% - SB-166/167	% - 2007 MRP Guidelines
Nickel (min)	58.0	58.0
Chromium	27–31.0	28.5–31.0
Iron	7.0–11.0	9.0–11.0
Carbon	0.05 max.	0.015–0.035
Silicon (max)	0.50	0.50
Manganese (max)	0.50	0.50
Cobalt (max.)		0.050
Copper (max)	0.5	0.10
Sulfur (max)	0.015	0.003
Phosphorous (max)		0.015
Nitrogen (max)		0.050
Aluminum (max)		0.40
Boron (max)		0.005
Titanium(max)		0.40
Molybdenum (max)		0.2
Niobium (max)		0.1

The general reason for changing the chemistry limits was to tighten limits where industry experience indicates that it is practical to do so with the objective of ensuring reduced variability. Comments on changes to specific elements are as follows:

- Chromium: The minimum chromium level of 28.5% reflects industry practice because the higher chromium is in the direction of improved corrosion performance. This requirement serves to tighten the composition range of 27.0–31.0 contained in SB-166/167.
- Iron: The minimum iron level of 9.0% reflects much of industry's practice to minimize concerns about possible long-range ordering reactions that might lead to embrittlement after a long time at temperature [9]. This requirement serves to tighten the composition range of 7.0–11.0 contained in SB-166/167.
- Carbon. The carbon content range of 0.015–0.035 is within the SB-166/167 maximum of 0.05. However, a minimum value of 0.015 was adopted from the steam generator tubing specification to provide adequate carbon to ensure uniform grain boundary carbide precipitation during thermal treatment. In contrast to cold-worked annealed steam generator tubing, the maximum carbon content was increased from 0.025 to 0.035 to increase the chances of achieving the minimum yield strength in thicker hot-worked annealed sections. One reviewer wanted a carbon maximum value of 0.03. Since more restrictive values are allowed, the 0.035 maximum value is retained.
- Silicon. Silicon content is consistent with the SB-166/167 requirements.
- Manganese. Manganese content is consistent with the SB-166/167 requirements.
- Cobalt: Cobalt is not specified in SB-166/167. A cobalt maximum requirement of 0.020% was used in the steam generator specifications where its origin was related to the concern to reduce cobalt release and subsequent activation from the large steam generator surface area. Although the amount of nozzle surface area is minuscule compared to the steam generator surface area, the industry has little problem in achieving a maximum level of 0.05% in nuclear material.
- Copper: The maximum value for copper is again a carryover from the steam generator specification. The value of 0.1% serves to tighten the requirement of 0.5 maximum in SB-166/167.
- Sulfur: The maximum value for sulfur is again a carryover from the steam generator specification. The value of 0.003 % serves to significantly tighten the requirement of 0.015 maximum contained in SB-166/167. Since sulfur is known to be deleterious to the hot workability of nickel-based alloys and since the industry appears to have little difficulty in achieving this requirement, a maximum level of 0.003% is considered appropriate.
- Phosphorous. SB-166/167 has no requirement for phosphorous. The maximum phosphorous content is a carryover from the steam generator specification and reflects the industry practice to achieve this requirement.
- Nitrogen. SB-166/167 has no requirement for nitrogen. The maximum nitrogen content is a carryover from the steam generator specification and reflects the industry practice to achieve this requirement.

- Aluminum: SB-166/167 has no requirement for aluminum. The maximum aluminum content is a carryover from the steam generator specification and reflects the industry practice to achieve this requirement. If weldability is an issue, the suggested range for aluminum is 0.015–0.040 wt%, instead of a maximum.
- Boron: SB-166/167 has no requirement for boron. The maximum boron content is a carryover from the steam generator specification and reflects the industry practice to achieve this requirement.
- Titanium: SB-166/167 has no requirement for titanium. The maximum titanium content is a carryover from the steam generator specification and reflects the industry practice to achieve this requirement. For weldability purposes and ensuring strength, the suggested range for Titanium is 0.015–0.045 wt%, instead of a maximum.
- Molybdenum. SB-166/167 has no requirement for molybdenum. The maximum molybdenum content is a carryover from the steam generator specification and reflects the industry practice to achieve this requirement.
- Niobium. SB-166/167 has no requirement for niobium. The maximum niobium content is a carryover from the steam generator specification and reflects the industry practice to achieve this requirement.

Material chemistry is required to be determined for each heat from the ladle or remelted ingot. In addition, one check analysis is required for each heat using material from the final product. This serves to verify that nothing in the processing resulted in deleterious changes in the chemistry. In general, chemistry variations from lot to lot are expected to be small and to not warrant chemical analysis for each lot. However, the option is provided for the Purchaser to require the chemistry to be determined for each lot.

The option has been provided for the use of "alternate qualified methods" when performing chemical analyses since a variety of methods are being used with satisfactory results, in addition to those listed in early draft EPRI specifications [1, 2]. The intent is that the Supplier will have test results available to verify that the methods used are, in fact, qualified.

### 3.4.4 Mechanical Properties

The minimum room temperature mechanical properties are adopted from the appropriate SB-166 or SB-167 ASME specification and Code Case N-698 (previously N-474-2, annulled 1/1/00). Assigning a maximum yield strength of 55 ksi (379 MPa) is a carryover from the philosophy established in the Steam Generator Tubing Guidelines [1], where, based on the experience with Alloy 600, it was considered desirable from an SCC-resistance standpoint to restrict the maximum yield strength. Restricting the range of yield strength is also viewed as a means of developing uniformity of properties and controlling cold work. The minimum room temperature ultimate tensile strength (UTS) and yield strength values of the SB-166 and SB-167 ASME Specification vary according to working practice and product size. Table 3-2 is a summary of the requirements in each.

Table 3-2
Mechanical Property Requirements

ASME Specification	Min. Tensile Strength ksi (MPa)	Min. Yield Strength ksi (MPa)	Min. Elongation %
SB-166 Rod, Bar, and Wire:			
All Sizes			
CW Ann or HW Ann	85 (586)	35 (240)	30
SB-167 Seamless Pipe and Tube:			
< 5" (12.7 cm) OD			
HW or HW Ann	85 (586)	30 (205)	35
CW Ann	85 (586)	35 (240)	30
>5" (12.7 cm) OD	00 (000)	33 (2.10)	33
HW or HW Ann	75 (515)	25 (170)	35
CW Ann	85 (586)	30 (205)	35

Generally, the ASME minimum mechanical properties are 85 ksi (586 MPa) tensile strength, 35 ksi (240 MPa) yield strength, and 35 percent elongation. However, depending upon size, and whether the product has been hot worked (HW) or cold worked (CW) and annealed (Ann), the minimum tensile strength may be allowed to fall to 75 ksi (515 MPa). The minimum yield strength for the 75 ksi tensile strength level is as low as 25 ksi (170 MPa). These lower values should be acceptable from an SCC perspective since it is generally accepted that SCC resistance increases with lower mechanical properties.

Consequently, it is the control of a yield strength maximum at 55 ksi (379 MPa) that is considered most important in ensuring stress corrosion resistance. One reviewer wanted the maximum yield strength set at 50 ksi, and said that their experience had shown that suppliers have not had any problems in providing material with this limit. Since there is no definitive data to imply that 50 ksi is better than 55 ksi, the 55 ksi value is retained. This maximum yield strength value will apply to all conditions covered by SB-166 or SB-167.

Hardness requirements are not included but are requested for information only. This is based on the position taken in the steam generator guidelines that the consensus of people working on Alloy 690 were of the opinion that taking hardness measurements did not serve a useful purpose [1]. Assurance that the product has the desired mechanical properties is provided by the specified tensile yield and ultimate property tests.

# 3.4.5 Hydrostatic Testing

Standard ASME Section II hydrostatic tests have been adopted.

#### 3.4.6 Microstructure Examination

Metallographic evaluation has been a standard requirement for Alloy 600 and Alloy 690 steam generator tubing for a number of years [1, 2]. Since thermo-mechanical processing can strongly affect carbide precipitation, metallographic evaluation is performed on the product in the final thermally treated condition to record the grain size, ensure that a favorable grain boundary carbide distribution has been achieved, and ensure quality material by assessing the microcleanliness.

#### 3.4.6.1 Grain Size

No specific requirements on grain size have been established since the required yield strength range is considered adequate to achieve the objective of optimum resistance to PWSCC. An upper limit on grain size (ASTM 3) is suggested as an objective because very large grains are undesirable from a standpoint of resistance to fatigue crack initiation. In addition, if the grain size approaches that of the wall thickness, concerns arise that the grains could result in excessive anisotropy and that the grain boundaries could present a relatively easy through-thickness path for corrosion attack. Large grains are also undesirable since they could make ultrasonic inspection difficult.

No lower limit on grain size has been established. In this regard, it should be recognized that very small grains are undesirable because they indicate that the solution anneal temperature was not high enough to dissolve all carbides; this would make it probable that insufficient carbon was put into solution by the solution anneal to properly decorate grain boundaries during thermal treatment. However, such a condition would make it difficult to stay below the imposed upper limit on yield strength of 55 ksi (379 MPa). In addition, microstructural checks to verify proper carbide decoration of grain boundaries provide further protection against such conditions.

#### 3.4.6.2 Desirable Microstructure

A requirement to perform a microstructural evaluation is included since extensive testing has shown that microstructure, particularly the distribution of carbide phase along the grain boundaries, has a strong influence on the SCC resistance of Alloy 600 and Alloy 690. This has been specifically demonstrated for Alloy 690 steam generator tubing in caustic environments [3, 4]. However, there is a scarcity of data for Alloy 690 which demonstrates that a thermally treated (TT) microstructure is required for improved primary water cracking resistance. To date, long-term (approximately 90,000 hours) tests in primary water have been performed only on Alloy 690TT with no apparent susceptibility to cracking [5, 6, 8]. The equivalent testing of Alloy 690 mill annealed (MA) has not been performed. Consequently, it is by analogy with Alloy 600 that the thermal treatment is considered beneficial also to Alloy 690 in primary water.

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It would be useful for future testing to evaluate non-thermally treated Alloy 690 in primary water since every welded Alloy 690TT nozzle will have heat-affected zones where the thermally treated microstructure has been transformed to an annealed structure with little or no grain boundary carbide or a grain boundary that has depleted chromium content.

A requirement that the fields used for the evaluation of microstructure must be randomly selected was added in the 1999 Steam Generator Tubing Guidelines [2] to ensure that the fields evaluated are representative of the material and are not biased by selection of fields with better than average microstructure.

A requirement was also added in 1999 to evaluate the microstructure at the OD surface to verify that the surface layer of recrystallized grains is minimized. A fine grained surface layer can be caused during thermal treatment by the recrystallization of a cold worked surface. It is likely that these grains do not have optimum grain boundary carbide decoration. The typical thickness of this layer observed on steam generator tubing is about 3–10  $\mu$ m (0.118–0.394 mils) [2]. The intent of this paragraph is to limit the depth of the layer so that possible adverse effects on susceptibility to SCC are minimized, although authoritative data for primary water are lacking.

It was requested by utility advisers that these guidelines contain a definitive gallery of acceptable and unacceptable microstructures. At this time, EPRI does not have in its possession examples of good versus poor microstructure, particularly as it relates to SCC resistance in primary water<sup>1</sup>. Consequently, the Purchaser and Supplier must establish an agreement on this issue based on the Supplier's production experience.

#### 3.4.6.3 Microcleanliness

Requiring low inclusion contents in high-nickel alloys has become standard practice, although there is very little definitive correlation between inclusions (that is, microcleanliness) and corrosion performance. From very early pitting studies of steam generator tubing, areas where titanium carbonitrides were located were thought to be preferred sites for pits. With regard to SCC performance, there has been one experience at Comanche Peak 1 where sulfide inclusions in excess of the required rating may have caused the secondary-side SCC cracking of solution annealed Alloy 600 steam generator tubing [10]. The inclusion limits for Alloy 690 go back to the 1991 Steam Generator Tubing Guidelines [1] where the testing method (Method A) was based on INCO recommendations [11] and industry input [12]. In the 1999 Steam Generator Tubing Guidelines [2] the limits on titanium carbonitride inclusions were tightened to reflect current commercial practice and also in response to utility desires to have as uniform and clean a product as practical. The limits for oxides and sulfides were not changed.

The sampling requirement stated in the current specification differs from previous steam generator guidelines. Those earlier guidelines require testing of samples corresponding to the top and bottom position of one ingot per heat. Sampling at those locations is still a very good idea,

<sup>&</sup>lt;sup>1</sup> EPRI does not currently have examples of acceptable and unacceptable Alloy 690TT microstructures and requests that a Supplier make these examples available.

particularly for the Suppliers as process controls. However, the current position is to require testing of the final product using the same longitudinal metallographic samples used for grain size and microstructural analyses. These metallographic samples might even come from the grip section of tensile specimens. The evaluation of inclusions to be performed using fields that are selected randomly in the designated areas (edge and mid radius) was added in the 1999 Steam Generator tubing Guidelines [2] and is intended to ensure that the fields evaluated are representative of the material and are not biased by the selection of fields with better than average inclusions. This requirement is retained.

A reviewer wanted the microcleanliness requirement removed altogether. Their position was that there is no evidence that this requirement affects primary-side SCC and that most suppliers have not been providing inclusion analysis. This may be true. However, the origin of this requirement was intended to answer the question: What is good ("clean") Inconel? As indicated above, that definition was established in 1991 by INCO and published by EPRI in the Steam Generator Tubing Guidelines [1]. Unless a better definition of what clean Inconel looks like can be developed, the requirements contained in Section 2.4.6.3, Microcleanliness, will be retained.

# 3.4.6.4 General and Intergranular Attack

This metallographic examination is required if acid cleaning (pickling) is used. Transverse sections of each lot of product are to be examined to verify freedom from evidence of general corrosion or IGA. Experience has shown that properly made Alloy 690 product does not show evidence of such attack, principally because of its high chromium content.

Some steam generator tube suppliers suggest that transverse samples be required only for preproduction lots, and not for production lots [2]. However after a review of the issue, the position taken in the Alloy 690 Steam Generator Tubing Guidelines was still to test each lot as a quality control check [2]. The same position is taken here for pipe, tube, and bar, especially since the carbon content has been allowed to increase up to 0.035%, which might increase the chance of sensitization, that is, grain boundary chromium depletion, developing during processing, which could make the product vulnerable to IGA during an acid cleaning step.

# 3.4.7 Surface Roughness

Steam generator tubing surface finishes were typically required to be RMS 63 microinches (1.60  $\mu$ m) or finer. Reviewers pointed out that reactor vessel nozzles were typically machined by the component supplier to at least a RMS 250 microinch (6.35  $\mu$ m) finish, which is adopted here.

# 3.5 Process Requirements

# 3.5.1 Melting and Metal Working Practices

The selection of melting and metal working practices is left to the Supplier, but the Supplier is required to document the practices and to obtain the Purchaser's approval. This approach was

taken to provide the Supplier with the flexibility needed to optimize their practices, while still giving the Purchaser sufficient control to ensure the needed quality.

For the purposes of this specification, there is no distinction made as to what melting practice should be used. Common melting practices can be electric furnace melting utilizing argonoxygen decarburization (AOD), vacuum induction melting (VIM), or air induction melting (AIM) without remelting or additionally with either of these followed by remelting under either vacuum arc remelting (VAR) or electroslag remelting (ESR).

No test data are available to imply whether the melting or remelting practice plays any role in affecting the resistance of Alloy 690 to SCC in primary water. ESR is considered to result in lower impurities levels, particularly sulfur, but if the composition and microcleanliness requirements can be achieved, the additional remelting may not be justified. Recently, however, the Bettis Laboratory presented information at an EPRI meeting (Expert Panel meeting, Oct 31, 2006) that the crack growth rate in high-temperature water of cold worked Alloy 690 melted by AOD +ESR was slower than cold worked Alloy 690 melted by VIM+ ESR [8]. Crack growth did not occur in annealed material; however, VIM+ESR material with 12% cold work (CW) showed SCC susceptibility. Annealed material had yield strengths in the mid-40 ksi (~310 MPa), while the 12% CW conditions had yield strengths in excess of 80 ksi (552 MPa) [8]. Consequently, the yield strength restriction of 55 ksi (379 MPa) in this guideline should minimize—if not eliminate—this SCC susceptibility. At present, no explanation for the difference has been offered, except that the difference might be explained by the inherent scatter of crack growth testing. Until resolution of this apparent issue, the position with regard to melting practices will remain unchanged.

#### 3.5.2 Heat Treatment

#### 3.5.2.1 Final Solution Anneal

A minimum temperature of 1958°F (1070°C) for the final solution anneal was used for cold drawn and annealed steam generator tubing [2]. However, for the various materials covered by this guideline, a single minimum annealing temperature is unrealistic. Consequently, the choice of temperature will be left to the discretion of the Supplier, based upon their experience.

The furnace gas conditions are required to result in bright annealing with no significant surface discoloration, carburizing, etc. The use of reference photomicrographs for judging the acceptability of carburization, decarburization, and nitriding is considered necessary to provide enforceable standards. Since there are no industry-accepted reference photomicrographs, the Supplier is required to develop them for the Purchaser's acceptance.

The temperature recording requirements are directed at providing a record that demonstrates that uniform solution annealing temperatures were obtained. Cooling rate requirements are not specified for either SB-166 or SB-167 type product. However, since the rate of cooling from the annealing temperature could become an issue for achieving the microstructural requirement, it is specified here to be considered by the Supplier.

#### 3.5.2.2 Thermal Treatment

The minimum thermal treatment time was increased in the 1999 Steam Generator Tubing Guidelines [2] from 5 to 10 hours. The current temperature of 700°C (1292°F) was adopted as a result of an MHI comment that their normal procurement is to 700°C (1292°F) for 15 hours minimum. Either time-temperature combination is considered adequate and reflects current industry practice. There certainly are no specific data to imply that either time-temperature combination is preferred for resistance to PWSCC. The MHI comment is believed to reflect their position that the longer time is in the right direction for increasing resistance to IGSCC in caustic tests.

The maximum time at thermal treatment temperatures of 35 hours is a carryover from steam generator production experience. The time was established to ensure that excessive thermal treatment plus stress relief does not place the product in an unqualified condition, that is, a condition that has not been thoroughly qualified by test.

# 3.5.3 Straightening, Machining, or Grinding

The limit on increase in yield stress from straightening ensures that excessive cold work is not introduced by straightening. Cold work can be introduced by machining and grinding. If excessive, this cold work can cause the formation of very fine surface grains due to recrystallization of the cold worked region during the thermal treatment or undesirable carbide precipitation along dislocations. To date, there is no definitive evidence that either of these conditions is deleterious to primary water cracking resistance, but the conservative position that they should be avoided is taken.

On the subject of the increase in yield stress from straightening, in the 1999 Steam Generator Tubing Guidelines [2], the limit was increased from 11.6 ksi (80 MPa) to 13.1 ksi (90 MPa). Increasing the allowed amount of strengthening from 11.6 to 13.1 ksi (80 to 90 MPa) reflected the fact that the higher solution annealing temperatures being used resulted in lower as-annealed yield strengths, and thus a small amount of additional strengthening was sometimes desirable. In addition, there is no known deleterious effect of strengthening at the 13.6 ksi (90 MPa) level as opposed to the 11.6 ksi (80 MPa) level. A requirement was also added in 1999 Steam Generator Tubing (Alloy 690) Guidelines to check the amount of strengthening at least once per lot. This frequency, together with the requirements to check each time the equipment is adjusted, seemed sufficient to ensure that excessive strengthening was not occurring.

A requirement was added in the 1999 Steam Generator Tubing Guidelines [2] that residual stresses associated with grinding be shown to not result in stresses higher than 20 ksi (138 MPa) tensile, measured before thermal treatment. This requirement was added to take advantage of the position taken by vendors that properly controlled grinding results in compressive or low tensile stresses. It also ensures that grinding stresses will be negligible after thermal treatment since thermal treatment is expected to significantly reduce the stresses caused by the grinding. An equivalent control of cold work and residual stresses is carried forth in this guideline.

No grinding or machine straightening is permitted after the final thermal treatment to ensure that residual stresses and cold work in the final product are minimized.

### 3.5.4 Repair

Defect removal after the final thermal treatment shall be limited to the Purchaser's approved methods. Defect removal has been permitted in all steam generator guidance but has been limited to hand polishing using abrasive materials (for example, abrasive paper or Scotch Brite pads), unless alternative methods are qualified in order to ensure that residual stresses associated with defect removal are low. For reactor vessel nozzles, MHI has encouraged that qualified alternative local repair methods be permitted, followed by operations that will reduce the surface tensile stresses.

### 3.5.5 Cleaning

The requirements in this section ensure that harmful contaminants (for example, sulfur) are not incorporated into the material during processing. This subject is further discussed in Section 3.5.6, Prohibited and Detrimental Materials.

#### 3.5.5.1 Demineralized Water

Water quality requirements are a carryover from steam generator requirements and were based on information provided by Duke Energy [2]. At the time, the water quality requirements were considered to be consistent with standard industrial practice. It is likely that the product related to this guideline will serve as the starting (raw) material for a vessel part that will be fabricated from starting material. Consequently, the concerns for steam generator tubing cleanliness are different than vessel nozzle raw material product. However, the position taken in this guideline is that if any of the cleaning steps or pressurization tests involves water, the equivalent water quality should be maintained.

#### 3.5.5.2 Grit Blasting

Grit blasting is included, and grit blasting is permitted prior to thermal treatment as long as the surface roughness (Section 2.4.7, Surface Roughness) of RMS 250 microinches (6.35  $\mu$ m) or finer is achieved. Grit blasting is prohibited after thermal treatment, unless special qualification tests are performed to show that it results in low tensile stresses since it could introduce undesirable stresses or superficial cold work.

Use of aluminum oxide grit was prohibited in the EPRI Alloy 600 specification [13] and SGOG Alloy 690 specification [1, 2]. It is understood that the reason for this prohibition is concern that embedded grit particles could be left in the tubes and that they would be more deleterious in the reactor coolant system if they were aluminum oxide than if they were zirconia. Several mills have indicated [14, 15] that zirconia performs satisfactorily and can be used if desired.

#### 3.5.5.3 Acid Cleaning

Acid cleaning is permitted prior to final reduction to size since acid cleaning is generally necessary after hot working steps to remove scale. Acid cleaning is not permitted after final reduction to size because of concerns that it could lead to grain boundary ditching, which would be deleterious in regard to corrosion resistance.

Selection of the acid cleaning solution is left to the Supplier, but is subject to qualification to ensure that it does not result in grain boundary ditching.

#### 3.5.6 Prohibited and Detrimental Material

#### 3.5.6.1 Prohibited Materials

The prohibited materials are commonly prohibited from contact with nickel alloys because of concerns that they can lead to SCC or other deleterious corrosive attack.

# 3.5.6.2 Furnace Atmosphere

The limit on the sulfur content of fuel is based on the known aggressiveness of sulfur toward nickel-base alloys at high temperatures. In 1999 [2], the prohibition against the use of high-sulfur fuel was clarified to indicate that it applies only if fuel or combustion gases can come into contact with the material. There is no technical reason to limit the sulfur content if the fuel or combustion gases do not come into contact with the tube material.

#### 3.5.6.3 Freedom from Grit Embedment

The requirement to inspect a product sample at a magnification of 60X for grit embedment is included to ensure that the prohibition against grit embedment in Section 2.5.5.2, Grit Blasting, is met.

# 3.6 Quality Assurance Program Requirements

The requirements in this section are standard quality assurance requirements, with minor modifications.

Both ASME SB-166 and SB-167 specifications require inspection of the material. In this bases section, issues relating to Identification and Control of Materials, Parts, and Components (Section 2.6.1), Supplier's Responsibilities for Subsuppliers (Section 2.6.2), and Notification and Hold Points (Section 2.6.3) are not addressed since these are standard QA requirements and have been addressed in Section 2.6, Quality Assurance Program Requirements. In addition, a detailed description of ultrasonic, eddy current, and liquid penetrant examinations have been addressed in

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Section 2.6.4.2, Nondestructive Examination. Only visual and tactile, dimensional, and surface finish checks are addressed in this bases section

# 3.6.1 Visual and Tactile Inspection

These requirements are intended to ensure that all product passes a visual examination. Tactile examination depends upon the product form and was usefully performed for small diameter steam generator tubing. It may not be as useful on a larger diameter product.

#### 3.6.2 Dimensional Inspection

The requirements are to be defined in the purchase order.

# 3.6.3 Surface Roughness Check

The intent is that the observed surface be compared to physical standards that have been agreed upon between the Supplier and Purchaser to ensure that conditions such as excessive scratching, etc., are not present.

#### 3.7 Documentation

Practices from the EPRI Alloy 690 Steam Generator Tubing Guidelines should be utilized [2].

# 3.8 Manufacturing Documentation Supplied by the Supplier to the Purchaser's Quality Assurance and Control Representatives with the Shipment

Practices from the EPRI Alloy 690 Steam Generator Tubing Guidelines should be utilized [2].

# 3.9 Purchaser Witnessing of Inspections

Practices from the EPRI Alloy 690 Steam Generator Tubing Guidelines should be utilized [2].

# 3.10 Shipment

Practices from the EPRI Alloy 690 Steam Generator Tubing Guidelines should be utilized [2].

# 3.11 Ordering Requirements

The requirements in this section are standard ones and are taken from earlier Alloy 690 specifications [1, 2], with minor changes to reflect other modifications in the specification.

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