AEROSPACE MATERIAL SPECIFICATION

AMS2759™/10 REV. D

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Superseding AMS2759/10C

Automated Gaseous Nitriding Controlled by Nitriding Potential

RATIONALE

AMS2759/10D corrects an error in Composition (see 3.3.1.1), where the minimum hydrogen composition has been changed from 99.985% to 99.95%.

NOTICE

ORDERING INFORMATION: In addition to that listed in AMS2759, the purchaser shall supply the following information to the nitriding processor:

- AMS2759/10D
- Engineering drawing or detailed operation information
- Any critical dimensions to be held through nitriding (see 3.4.1.2)
- Prior heat-treatment information, including actual tempering/aging temperature and hardness (see 3.4.1.1)
- Any prior stress relieving performed, including specifics, and if stress relief is to be performed by the nitride processor (see 3.4.1.2)
- Class of compound zone (white layer) depth (thickness) (see 3.1)
- Areas to be nitrided, areas to be masked, areas to be optional (see 3.4.2)
- Total case depth, if applicable (see 3.5.3.2)
- Effective case depth, if applicable (see 3.5.3.3)
- Case (surface) hardness (see 3.5.2)
- Core hardness, if specified (see 3.5.1)
- Areas to be ground after nitride, if applicable (see 3.4.2.3)

- If parts are to be used for testing (see 4.2.1)
- Post-nitride surface removal requirements, if applicable (see 3.5.3)
- Grind stock allowances to be removed in subsequent grinding, polishing, etc., if applicable (see 3.5.5.2)

1. SCOPE

1.1 Purpose

This document specifies the procedure and requirements for nitriding steel parts in a process automatically controlled by the nitriding potential (see 2.3.6).

1.2 Application

The nitriding process described herein is used typically to produce a nitride case on carbon and alloy steels, tool steels, nitriding steels, corrosion-resistant steels, and other ferrous alloys using a process controlled automatically by the nitriding potential. Specifically, the nitriding process described herein is recommended for those applications where white layers are not permitted or are allowed to a limited thickness.

2. APPLICABLE DOCUMENTS

In addition to those listed in AMS2759, the issue of the following documents in effect on the date of the purchase order forms a part of this specification to the extent specified herein. The supplier may work to a subsequent revision of a document unless a specific document issue is specified. When the referenced document has been cancelled and no superseding document has been specified, the last published issue of that document shall apply.

2.1 SAE Publications

Available from SAE International, 400 Commonwealth Drive, Warrendale, PA 15096-0001, Tel: 877-606-7323 (inside USA and Canada) or +1 724-776-4970 (outside USA), www.sae.org.

AMS2418 Plating, Copper

AMS2429 Plating, Bronze, Nitriding Stop-off, 90Cu - 10Sn

AMS2759 Heat Treatment of Steel Parts, General Requirements

AMS2759/9 Hydrogen Embrittlement Relief (Baking) of Steel Parts

AMS2759/11 Stress Relief of Steel Parts

AS7766 Terms Used in Aerospace Metals Specifications

2.2 ASTM Publications

Available from ASTM International, 100 Barr Harbor Drive, P.O. Box C700, West Conshohocken, PA 19428-2959, Tel: 610-832-9585, www.astm.org.

ASTM E18 Standard Test Methods for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials

ASTM E140 Standard Hardness Conversion Tables for Metals Relationship Among Brinell Hardness, Vickers Hardness, Rockwell Hardness, Superficial Hardness, Knoop Hardness, Scleroscope Hardness, and Leeb Hardness

ASTM E384 Standard Test Method for Microindentation Hardness of Materials

2.3 Definitions

Terms used in AMS2759/10 are defined in AMS2759, AS7766, and as follows:

2.3.1 COMPOUND ZONE LAYER (WHITE LAYER)

The surface of the steel may be converted to one or more compounds of essentially pure nitrides of metal, primarily iron. The layer formed, usually less than 0.001 inch (0.025 mm) in thickness, remains unetched when a metallographic sample is etched with Nital etchant and appears white in the microscope. For this reason, the layer is also called white layer.

2.3.2 CORE HARDNESS

The hardness used in determination of case depth. The hardness below the nitride case, as determined by a Knoop or a Vickers hardness traverse, where the average measured hardness does not change. A minimum of three hardness readings taken at a minimum distance of two times the case depth usually corresponds to this hardness.

2.3.3 PART HARDNESS

The overall hardness of the part as determined by the heat treatment used prior to any surface hardening and/or after surface hardening on a non-treated surface.

2.3.4 CASE HARDNESS

Case hardness and surface hardness are synonymous terms as they relate to determining the hardness of the nitrided area of the part.

2.3.5 PROCESS STAGE

A stage is a part of a process cycle, defined by temperature, time, atmosphere composition, and nitriding potential. It shall differ from other stages by at least one of these parameters, except time, to be recognized as a stage.

2.3.6 NITRIDING POTENTIAL

The nitriding potential is a measure of the nitriding capability of the nitriding atmosphere, which determines the surface nitrogen concentration in steel at a given temperature, and is described by Equation 1:

$$K_n = P_{NH_3}/\sqrt{P_{H_2}}^{3/2} \quad \text{(Eq. 1)}$$

where:

P_{NH_3} = partial pressure of ammonia in the outgoing atmosphere

P_{H_2} = partial pressure of hydrogen in the outgoing atmosphere, the hydrogen being a product of dissociated ammonia

2.3.7 CORRELATION WITH NITROGEN CONCENTRATION

The correlation between the nitriding potential and nitrogen concentration in pure iron is shown in Figure 1.

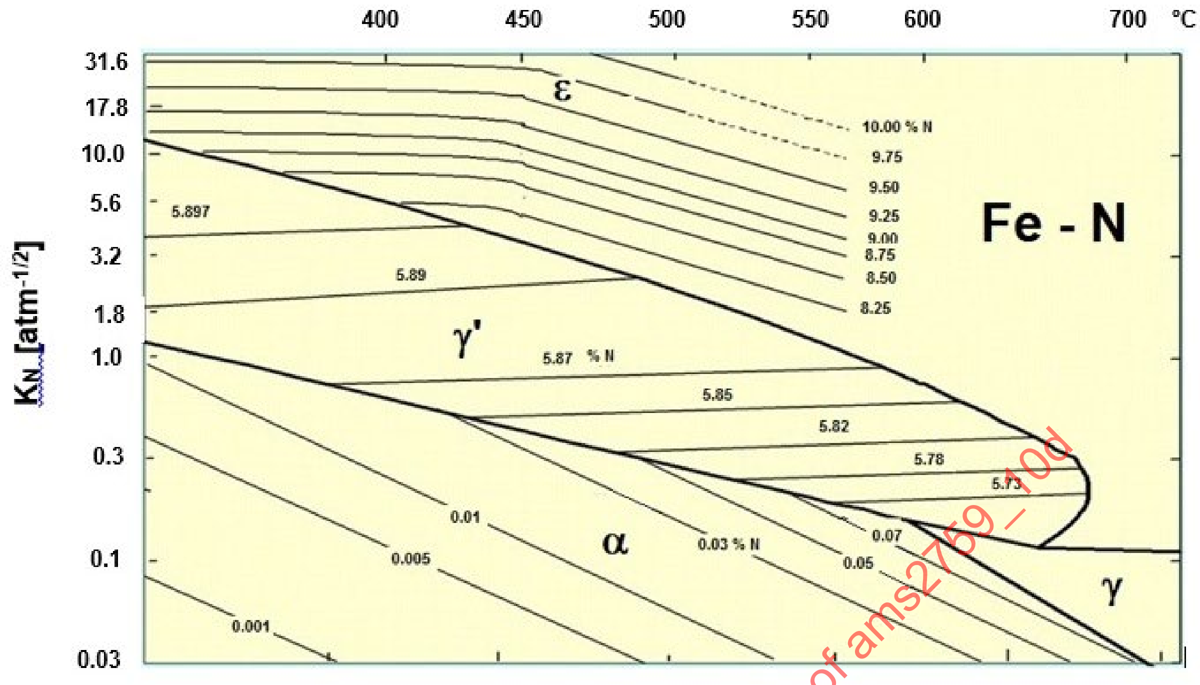

Figure 1 - The Lehrer diagram with adsorption isotherms for iron

3. TECHNICAL REQUIREMENTS

Shall be in accordance with AMS2759 and as follows:

3.1 Classification

The nitriding processes described herein are classified by the result they produce, as follows:

Class 0: No compound zone (white layer) permitted.

Class 1: 0.0005-inch (0.013-mm) maximum compound zone (white layer) thickness permitted.

Class 2: 0.001-inch (0.025-mm) maximum compound zone (white layer) thickness permitted.

If no class is specified, Class 2 applies.

3.2 Furnace Equipment

Shall be in accordance with AMS2759. Nitriding furnaces shall be Class 3 or better.

3.2.1 Shutdowns and Alarms

The furnace system shall be capable of automatically and safely shutting down the process in the event of a malfunction of the equipment such as power failure, interruption of gas flow, or any parameters exceeding their tolerance limits.

3.2.2 Atmosphere Control

Equipment shall be capable of introducing ammonia and dilution gases into the furnace at a controlled rate.

3.2.3 Auxiliary Equipment

Shall be in accordance with AMS2759.

3.3 Atmosphere

3.3.1 Composition

3.3.1.1 Gases used for nitriding shall be as follows:

Anhydrous Ammonia

99.98% minimum ammonia

35 ppm maximum water content

2 ppm maximum oil content

Nitrogen

99.998% minimum nitrogen

Hydrogen

99.95% minimum hydrogen

3.3.2 Control

The composition of the ingoing gas mixture, outgoing gaseous atmosphere, and the nitriding potential for each stage of the process shall be automatically controlled, maintained, and recorded (see 4.3). Devices for accurate control of flow rates of the individual gases shall operate automatically in response to signals from the atmosphere analyzer.

3.3.3 Nitriding Potential

Shall be set based on alloy and given application. Recommended ranges for typical alloys are given in Table 1.

Table 1 - Recommended ranges of nitriding potential

Alloy	Class 0		Class 1		Class 2	
	Stage 1	Stage 2	Stage 1	Stage 2	Stage 1	Stage 2
Nitralloy 135M	4 - 12	0.3 - 0.8	4 - 12	0.6 - 1.8	6 - 15	1.2 - 2.6
Nitralloy EZ	4 - 12	0.3 - 0.8	4 - 12	0.6 - 1.8	6 - 15	1.2 - 2.6
Nitralloy N	4 - 12	0.3 - 0.8	4 - 12	0.6 - 1.8	6 - 15	1.2 - 2.6
4140, 4340	4 - 12	0.2 - 0.7	4 - 15	0.6 - 2.6	4 - 15	1.2 - 4.5
D6, D6AC	4 - 12	0.2 - 0.7	4 - 15	0.6 - 2.6	4 - 15	1.2 - 4.5
H11, H13	5 - 15	0.3 - 0.8	5 - 15	0.4 - 0.9	5 - 15	2.2 - 5.5
Stainless Steels	0.5 - 1.0	N/A	5 - 15	0.4 - 0.9	5 - 15	0.4 - 1.2
Carbon steels	N/A	N/A	5 - 12	0.8 - 2.6	1.2 - 4.0	N/A

3.3.4 Pressure

The gas pressure shall be above atmospheric in the furnace throughout the process.

3.4 Procedure

3.4.1 Pre-Nitriding Operations

3.4.1.1 Prior Heat Treatment

3.4.1.1.1 If any specified heat treatment has already been performed, information regarding that heat treatment shall have been provided. Heat-treatment certifications are recommended but not required. The hardness of heat-treated parts should be verified by the processor prior to nitriding by testing at least one part in accordance with ASTM E18. The part hardness may be determined on a surface of the part where no nitriding is required (such as on a surface that is to be masked) or may be determined at a designated position on a cross section of a specimen or part.

3.4.1.1.2 If any specified heat treatment is to be performed by the nitriding processor, it shall be performed prior to nitriding. If a heat treatment is specified but a control document is not referred to, then the heat treatment shall be controlled and tested in accordance with AMS2759 and applicable AMS2759 slash specifications.

3.4.1.2 Stress Relieving

Unless otherwise specified, parts that have critical dimensions to be held through nitride that have been rough ground, straightened, machined, or otherwise mechanically worked after heat treatment shall be stress relieved prior to nitriding in accordance with AMS2759/11.

3.4.1.3 Cleaning

Parts shall be cleaned in accordance with AMS2759 such that sufficient and uniform nitriding can take place. Care shall be exercised after cleaning to prevent recontamination. After stress relieving and prior to nitriding, it is permissible for surfaces to be mechanically or chemically cleaned.

3.4.2 Masking

Part surfaces that are not to be nitrided shall be masked.

3.4.2.1 Maskant Application

Masking shall be copper plate per AMS2418 or bronze plate per AMS2429. Alternative methods, such as masking paste, may be used if acceptable to the cognizant engineering organization. Copper plate shall be fine-grained and nonporous, not less than 0.001 inch (0.025 mm) in thickness. Bronze plate shall be not less than 0.0005-inch (0.0127-mm) thick.

3.4.2.2 Masking of Specimens

Specimens, if used, shall be masked on a portion of the surface the same as the parts (see 4.2.1).

3.4.2.2.1 By Purchaser

If the purchaser is applying masking to the parts, they shall also apply masking to the specimens if used for testing.

3.4.2.2.2 By Processor

If the processor is applying masking to the parts, they shall also apply masking to the specimens if used for testing.

3.4.2.3 Alternative to Masking

When specified as an alternative to masking, the nitrided case may be removed by grinding from surfaces specified to be free from nitriding.

3.4.2.4 Inspection

Prior to nitriding, masking shall be visually inspected without magnification for blistering, peeling, or porosity. Parts with masking exhibiting blistering, peeling, or porosity shall be rejected.

3.4.3 Racking

Rack or support parts to prevent distortion and to ensure free circulation of the nitriding gas to all surfaces. Specimens, if used, are to be placed as close to the parts as possible.

3.4.4 Nitriding

Nitriding shall be accomplished in a process comprising one or more stages as described in 3.4.4.2.

3.4.4.1 Nitriding Temperature Range

The recommended nitriding temperature range is 915 to 1095 °F (491 to 591 °C). Lower temperatures can be used when needed. The nitriding temperature shall not exceed 1200 °F (649 °C). The nitriding temperature shall not be higher than 50 °F (28 °C) below the final tempering or processing temperature unless the part hardness can be verified on an actual or representative part. The nitriding temperature shall not exceed the tempering or aging temperature.

3.4.4.2 Nitriding Potential

For any nitriding potential (K_n) set point value, the K_n value shall be maintained within the tolerances shown in Table 2 after stabilization of the atmosphere.

3.4.4.2.1 For processing stages of up to 6 hours, stabilization shall be reached such that at least 60% of the stage time shall be conducted within respective tolerances.

3.4.4.2.2 For processing stages greater than 6 hours, stabilization shall be reached such that at least 85% of the stage time shall be conducted within respective tolerances.

3.4.4.2.3 For processing stages less than 2 hours, the tolerances are recommendations.

3.4.4.2.4 Sample readings shall be taken from the outgoing atmosphere. The frequency of readings shall be not less than once every 30 seconds.

Table 2 - Tolerance limits for nitriding potential control

K_N Range	Tolerance
< 0.5	±0.1
> 0.5 to 1	±0.2
> 1 to 2	±0.3
> 2 to 3	±0.4
> 3 to 6	±1.0
> 6 to 18	±2
>18	±4

3.4.4.3 Cooling

The parts shall be cooled from the nitriding temperature in a suitable nonoxidizing atmosphere to a temperature not exceeding 300 °F (149 °C). The furnace or retort shall be purged with inert gas before opening.

3.4.5 Post-Nitriding Operations

3.4.5.1 Inspection

Masking on parts (see 3.4.2) shall be visually inspected without magnification for blistering, peeling, or porosity. Parts with masking exhibiting these shall be rejected.

3.4.5.2 Maskant Stripping

Maskant materials shall be removed by methods that do not cause embrittlement, pitting, or damage to the part.

3.4.5.3 Embrittlement Relief

Parts that have been pickled or etched in the course of plating removal after nitriding shall be baked to remove hydrogen as specified in AMS2759/9.

3.5 Properties

3.5.1 Core Hardness

If specified, the post-nitriding core hardness shall meet requirements. Post-nitriding core hardness shall be the hardness at a minimum of two times the specified case depth. It shall be the average of at least three hardness readings taken in accordance with ASTM E384. Core hardness readings can also be taken in accordance with ASTM E18.

3.5.2 Case Hardness (Surface Hardness)

Specified case hardness (see 2.3.4) shall be determined using Vickers, Knoop, or HR15N in accordance with ASTM E18 or ASTM E384 as applicable. Other methods shall be acceptable to the cognizant engineering organization. Minimum hardness requirements are shown in Table 3. Hardness for alloys not shown in Table 3 shall be specified.

Table 3 - Minimum case hardness (surface hardness) requirements

Alloy	Hardness (HR15N) or Equivalent (see 8.2)
Nitralloy 135 Modified	92.5
Nitralloy EZ	92.5
Nitralloy N	92.5
AISI 4140, AISI 4340	85.5
D6AC	85.5
H11, H13	92.1
GKP, GKPW, GKPYW	90.3
GKHYW	90.3
Other alloys	As specified

3.5.3 Case Depth

The case depth shall meet the specified requirements. If the case depth is specified but does not specifically state that it is to be an effective case depth, total case depth, as defined below, shall apply. In cases where post-nitride surface removal is specified (see Ordering Information), the reported case depth shall include the amount to be removed.

3.5.3.1 Visual Case Depth

For carbon steels and cast iron, case depth shall be determined visually with the aid of a metallurgical microscope as the distance from the surface to the depth at which needle-like nitride precipitates are still visible. When such precipitates are not visible, diffusion depth can be determined by application of etchants in addition to or in lieu of Nital. On such steels, when “etched case depth” or “visual case depth” is specified, it shall be the distance measured from the surface in the direction of the core to the location where the needle zone or zone darkened by etching ends.