

AEROSPACE MATERIAL SPECIFICATION

Submitted for recognition as an American National Standard

Automated Gaseous Nitriding Controlled by Nitriding Potential

1. SCOPE:

1.1 Purpose:

This specification establishes requirements and procedures for generating nitrided surfaces on steel parts using a process controlled by the nitriding potential. See definition of compound layer in 8.1.1.

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. Applicable, allowance should be made for growth. See 8.3.

1.2.1 Specifically, the nitriding process described herein is recommended for those nitriding applications where engineering drawings do not permit a white layer or only a limited white layer thickness.

1.3 Classification:

The nitriding processes described herein, are classified as follows:

1.3.1 Class 0: No white layer permitted. (Not recommended for stainless steels.)

1.3.2 Class 1: 0.0005 inch (0.013 mm) maximum white layer thickness permitted.

1.3.3 Class 2: 0.001 inch (0.025 mm) maximum white layer thickness permitted.

1.3.4 If no class is specified, Class 2 applies. Class 2 is not recommended for corrosion resistant steels.

2. APPLICABLE DOCUMENTS:

The following publications form a part of this specification to the extent specified herein. The latest issue of SAE publications shall apply. The applicable issue of other publications shall be the issue in effect on the date of the purchase order.

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2.1 SAE Publications:

Available from SAE, 400 Commonwealth Drive, Warrendale, PA 15096-0001.

AMS 2418 Plating Copper
AMS 2750 Pyrometry
AMS 2759 Heat Treatment of Steel Parts, General Requirements
AMS 2429 Bronze Plate Masking

ARP1820 Chord Method of Evaluating Surface Characteristics

2.2 ASTM Publications:

Available from ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959.

ASTM E 18 Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials
ASTM E 92 Vickers Hardness of Metallic Materials
ASTM E 140 Hardness Conversion Tables for Metals (Relationship between Vickers and Rockwell Hardness)
ASTM E 384 Microhardness of Materials

2.3 ANSI Publications:

Available from ANSI, 11 West 42nd Street, New York, NY 10036-8002.

ANSI B46.1 Surface Texture

3. TECHNICAL REQUIREMENTS:

3.1 Equipment:

3.1.1 Heating Equipment: Shall conform to the requirements of AMS 2759.

3.1.2 Gases: Gases used for the nitriding process (anhydrous ammonia and nitrogen) shall be of the high purity grade (99.98%) with dew point -54 °F (-48 °C) or lower.

3.1.3 Controls:

3.1.3.1 Pressure Controls: The control equipment shall maintain pressure above atmospheric in the furnace retort throughout the process.

3.1.3.2 Nitriding Atmosphere: The composition of the ingoing gas mixture and the nitriding potential for each stage of the process shall be automatically controlled, maintained and recorded (See 8.1.3).

3.1.3.3 Atmosphere Flow Rate Controls: Devices for accurate control of flow rates of the individual gases shall operate automatically in response to signals from the atmosphere analyzer.

3.1.3.4 Shut-Downs and Alarms: The system shall be capable of automatically and safely shutting down the process in the event of any malfunction of the equipment such as power failure, interruption of gas flow, or any parameters exceeding their tolerance limits before damage occurs to the parts being processed.

3.2 Heat Treatment Prior to Nitriding:

3.2.1 Heat Treatment Condition: Steels hardenable by heat treatment shall be in the quenched and tempered, or solution treated and aged condition. Unless otherwise specified, heat treatment procedure shall conform to AMS 2759. For materials not covered by AMS 2759, heat treatment parameters shall be as specified by the cognizant engineering organization and process controls shall be in accordance with AMS 2759. The highest nitriding temperature shall be at least 50 °F (30 °C) below the lowest tempering or aging temperature.

3.2.2 Surface Condition: Surfaces to be nitrided shall be free from decarburization, as determined in accordance with ARP1820 or equivalent microhardness techniques. Decarburized zone is defined as the material having a hardness 20HK points or more softer than that of the core.

3.3 Pyrometry:

In accordance with AMS 2750.

3.3.1 Load Thermocouples: If used, shall be sheathed and sealed.

3.4 Preparation for Nitriding:

3.4.1 Unless otherwise specified, parts which have been ground, straightened or otherwise mechanically worked after heat treatment shall be stress relieved prior to nitriding. The stress-relief temperature shall be in accordance with AMS 2759.

3.4.2 Part surfaces to be nitrided shall be free from grease, oil, oxidation, and other contaminants.

3.4.3 Part surfaces which are not to be nitrided shall be masked by a suitable maskant, e.g., copper (AMS 2418), bronze (AMS 2429), or by alternative methods like nickel, or other copper or bronze plating, pastes or paints, acceptable to the cognizant engineering organization. Copper plate shall be fine-grained and non-porous, not less than 0.001 inch (0.025 mm) in thickness. If bronze plating is used, it shall be not less than 0.0005 inch (12.5 μm) thick.

3.4.3.1 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.

3.5 Procedure:

- 3.5.1 Loading: Parts shall be placed on racks or suspended, so as to minimize distortion, and to allow all surfaces to be nitrided. If used, test specimens representing parts fabricated from the same alloy, and in same heat treatment condition, shall be placed in the working zone in a location adjacent to parts they represent as close as possible to the lowest and highest temperatures based on latest temperature uniformity tests.
- 3.5.2 If parts are masked, the specimen(s) shall be similarly masked on a portion of the surface.
- 3.5.3 Nitriding of Parts: Nitriding shall be accomplished in a process comprising one or more stages. The definition of a process stage is given in 8.1.2.
- 3.5.3.1 Nitriding Potential K_N : (See 8.1.3.) Shall be set, based on alloy and given application. Recommended ranges for typical alloys are given in 8.1.4.
- 3.5.3.2 Nitriding Potential Control: For any nitriding potential setpoint value, the average K_N value shall be maintained within limits shown in Figure 1 (for low potential range) and Figure 2 (for high potential range), after stabilization of the atmosphere. Stabilization for each stage of the cycle shall be reached such that at least 60% of the stage time shall be conducted under stable conditions for short-duration processes (up to 6 hours), and at least 85% of the stage time shall be conducted under stable conditions for longer processes. Sample reading shall be taken from the outgoing atmosphere. The frequency of readings shall be not less than once every 30 seconds.

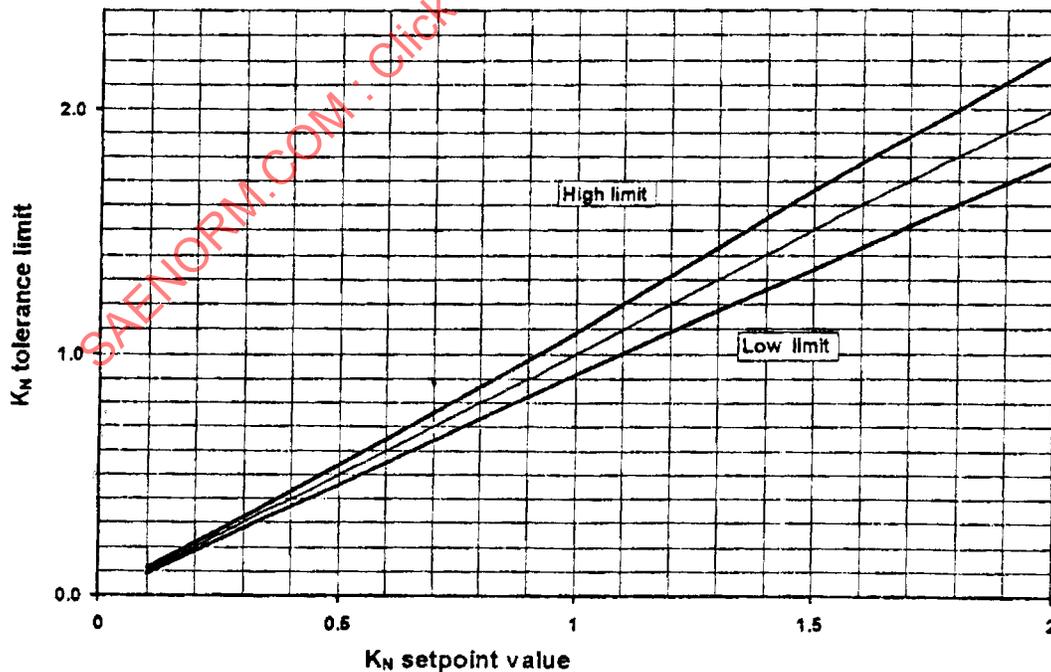


FIGURE 1 - Tolerance Limits of Actual K_N Value Vs. Setpoint Value for Low Nitriding Potential Range (See 3.5.3.2)

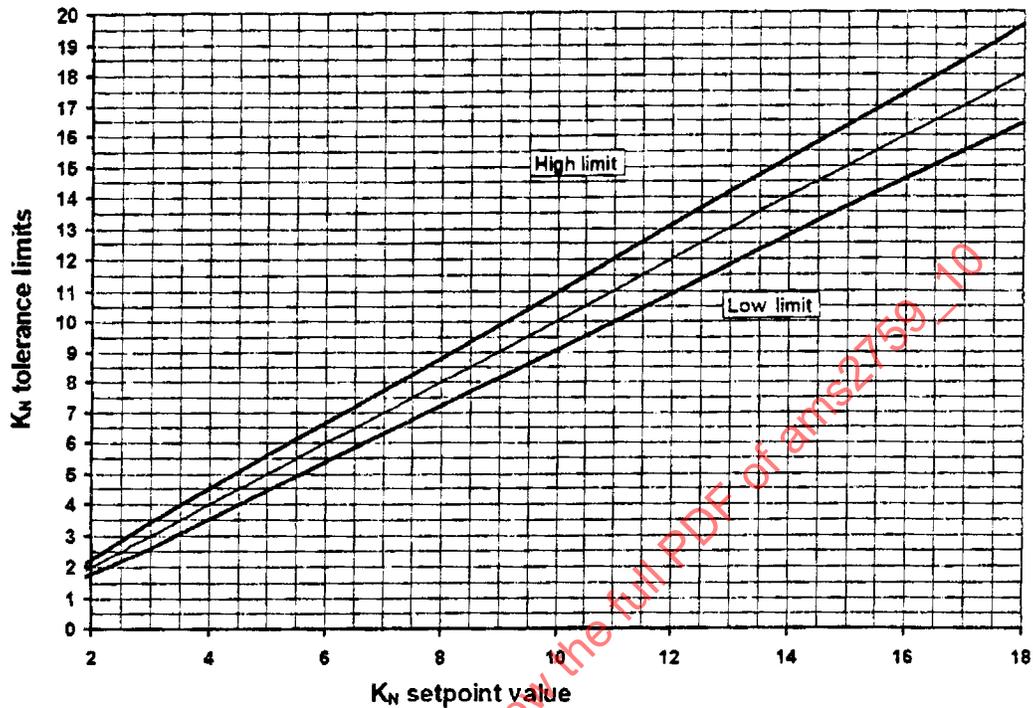


FIGURE 2 - Tolerance Limits of Actual K_N Value Vs. Setpoint Value for High Nitriding Potential Range (See 3.5.3.2)

3.5.3.3 Temperature: Shall not exceed 1200 °F (649 °C). The recommended range is 915 to 1095 °F (490 to 590 °C).

3.5.3.4 Cooling: The load shall be cooled from the nitriding temperature in a suitable protective atmosphere to a temperature not exceeding 300 °F (149 °C).

3.6 Post-Nitriding Operations:

3.6.1 Maskant Stripping: Maskant materials shall be removed by non-embrittling and non-pitting methods.

3.7 Properties:

Parts shall conform to the following property requirements unless otherwise specified by the cognizant engineering organization.

3.7.1 Hardness: Testing shall be performed in accordance with ASTM E 18, ASTM E 92 or ASTM E 384. Conversion between scales and correlation with strength shall be in accordance with ASTM E 140.

3.7.1.1 Core Hardness: In accordance with the requirements of the engineering drawing.

- 3.7.1.2 Surface Hardness: Shall meet the requirements shown in Table 1. Minimum surface hardness for alloys not shown in Table 1 shall be in accordance with engineering drawing requirements.

TABLE 1 - Minimum Surface Hardness

Alloy	Minimum Hardness (HR15N) or Equivalent ¹
Nitralloy 135 M	92.5
Nitralloy EZ	92.5
Nitralloy N	92.5
4140, 4340	85.5
H11	92.1
Other alloys	Per drawing

¹Conversions shall be in accordance with ASTM E 140.

- 3.7.2 Microstructure: Metallographic examination shall be made at a magnification not lower than 400X. The case shall exhibit a uniform distribution of nitrides, diminishing gradually, from surface to core. There shall be no evidence of continuous nitride network in grain boundaries. Unless otherwise specified, when white layer is permitted, its maximum thickness shall be 0.0005 inch (12.5 μ m) for Class 1 and 0.001 inch (25 μ m) for Class 2, for steels specified in Table 1. For stainless steels, the maximum thickness of white layer shall be 0.0005 inch (12.5 μ m), regardless of class.
- 3.7.3 Case Depth: In accordance with drawing requirements. It shall be the depth of the dark etching subsurface zone, determined metallographically. On those alloys which do not respond by darker etching, case depth shall be the depth below the surface at which microhardness is 10% higher than that of the core, as determined by a Knoop or a Vickers hardness traverse, in accordance with ASTM E 384.
- 3.7.3.1 Effective Case Depth: Where specified, it shall be in accordance with drawing requirements. It shall be the depth at which the hardness values specified in Table 2, converted from microhardness, are obtained in accordance with ASTM E 384. Effective case depth for alloys not shown in Table 2 shall be in accordance with drawing requirements. Where no ranges or tolerance limits are specified, the acceptance limits shall be ± 0.001 inch or 10%, whichever is greater, of the case depth on the engineering drawing.

TABLE 2 - Minimum Case Hardness

Specification	Alloy	Minimum Hardness (HRC Converted) ¹
AMS 6470; AMS 6471; AMS 6472	Nitralloy 135 M	50
	Nitralloy EZ	50
AMS 6475	Nitralloy N	50
AMS 6382; AMS 6414; AMS 6415	4140, 4340	40
AMS 6485; AMS 6487; AMS 6488	H11	60
	Other grades	Per drawing requirements

¹Conversions shall be made in accordance with ASTM E 140.

4. QUALITY ASSURANCE PROVISIONS:

The responsibility for inspection, classification of tests, sampling, approval, entries, records, and reports shall be in accordance with AMS 2759 and as specified in 4.1.

4.1 Classification of Tests:

The classification of acceptance, periodic, and preproduction tests, as well as sampling, shall be in accordance with AMS 2759.

4.1.1 Acceptance Tests: In addition to tests specified in AMS 2759, tests to determine conformance to requirements of 3.7.1, 3.7.2 and 3.7.3 shall be performed on parts from each nitrided lot, or on process control specimens, supplied by the purchaser. The specimens shall be processed with the parts they represent.

4.2 Sampling and Testing:

Shall be in accordance with AMS 2759.

5. PREPARATION FOR DELIVERY:

Shall be in accordance with AMS 2759.

6. ACKNOWLEDGMENT:

The process vendor shall mention this specification number in all quotations, when acknowledging purchase orders and on certifications.

7. REJECTIONS:

Parts not processed in accordance with this specification or modifications authorized by the cognizant engineering organization, and parts not meeting specified requirements, shall be subject to rejection and shall be submitted for disposition in accordance with purchaser's procedures of nonconformance.

8. NOTES:

Also see AMS 2759.

8.1 Definitions:

Terms used in this AMS are clarified in ARP1917 and as follows:

- 8.1.1 Compound Layer: External, non-etching portion of nitrided layer, composed of compact epsilon or gamma prime nitrides or a combination thereof, commonly referred to as "white layer".
- 8.1.2 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 the above parameters, other than time, to be recognized as a stage.
- 8.1.3 Nitriding Potential: The nitriding potential is a measure of the nitriding capability of the nitriding atmosphere, controlling surface nitrogen concentration in steel at a given temperature, and is described by Equation 1:

$$K_N = \frac{P_{\text{NH}_3}}{(P_{\text{H}_2})^{3/2}} \quad (\text{Eq. 1})$$

Where:

P_{NH_3} = the partial pressure of ammonia in the outgoing atmosphere, and
 P_{H_2} = the partial pressure of hydrogen in the outgoing atmosphere, the hydrogen being a product of dissociated ammonia.

The correlation between the nitriding potential and nitrogen concentration in pure iron is shown in Figure 3.