

Automated Gaseous Nitriding Controlled by Nitriding Potential

RATIONALE

AMS 2759/10A is a Five Year Review and update of this specification.

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 8.5.1.

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 8.4).

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.

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2. APPLICABLE DOCUMENTS

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 724-776-4970 (outside USA), or www.sae.org.

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

| | |
|------------|--|
| ASTM A 370 | Standard Test Methods and Definitions for Mechanical Testing of Steel Products |
| ASTM E 92 | Vickers Hardness of Metallic Materials |
| ASTM E 140 | Hardness Conversion Tables for Metals |
| ASTM E 384 | Microhardness of Materials |

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 8.5.3).

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 (12.7 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 8.5.2.

3.5.3.1 Nitriding Potential K_N

(See 8.5.3.) Shall be set, based on alloy and given application. Recommended ranges for typical alloys are given in 8.5.4.

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.

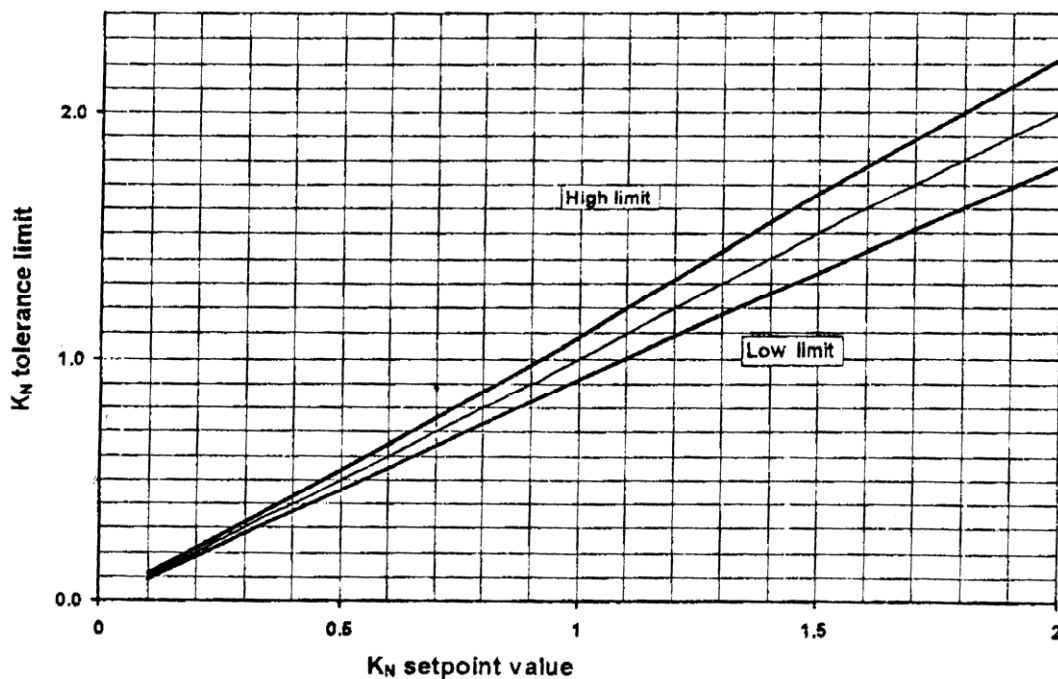


FIGURE 1 - TOLERANCE LIMITS OF ACTUAL K_N VALUE VS. SETPOINT VALUE FOR LOW NITRIDING POTENTIAL RANGE (SEE 3.5.3.2)

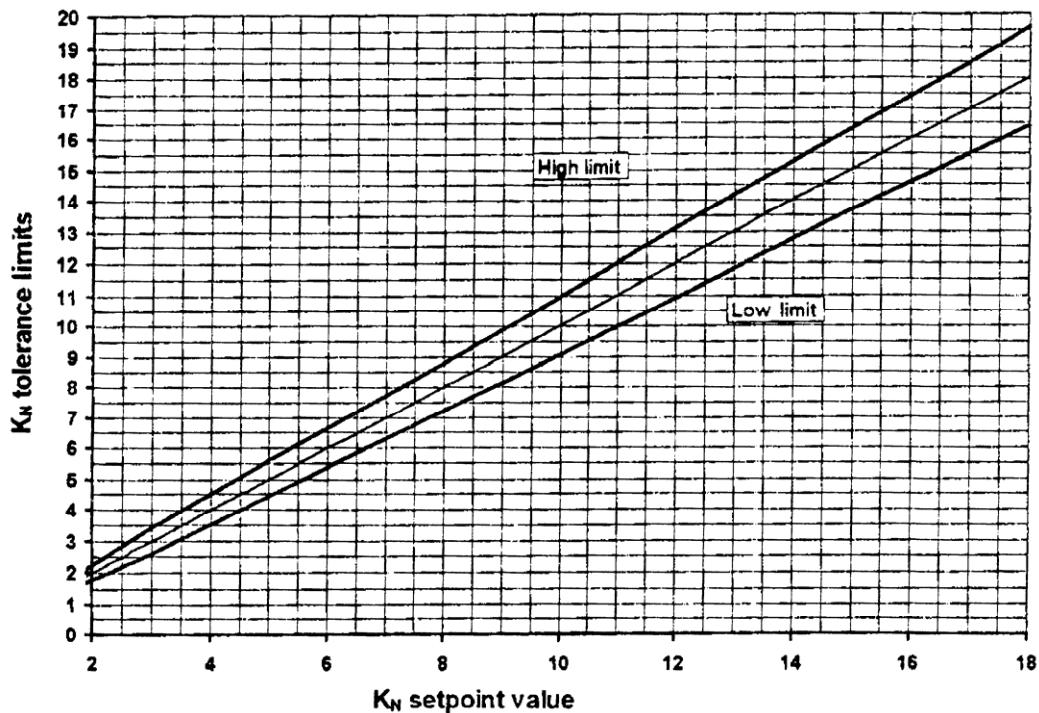


FIGURE 2 - TOLERANCE LIMITS OF ACTUAL KN VALUE VS. SETPOINT VALUE FOR HIGH NITRIDING POTENTIAL RANGE (SEE 3.5.3.2)

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 A 370, ASTM E 92 or ASTM E 384. For conversion between scales and correlation with strength (See Notes 8.1 and 8.2).

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 (See 8.2) |
|-----------------|--|
| Nitralloy 135 M | 92.5 |
| Nitralloy EZ | 92.5 |
| Nitralloy N | 92.5 |
| 4140, 4340 | 85.5 |
| D6, D6AC | 85.5 |
| H11 | 92.1 |
| Other alloys | Per drawing requirements |

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 (12.7 μm) for Class 1 and 0.001 inch (25.4 μm) for Class 2, for steels specified in Table 1. For stainless steels, the maximum thickness of white layer shall be 0.0005 inch (12.7 μm), 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 (± 25.4 μm) or 10%, whichever is greater, of the case depth on the engineering drawing.

TABLE 2 – HARDNESS AT EFFECTIVE CASE DEPTH

| Specification | Alloy | Minimum Hardness (HRC Converted)(See 8.2) |
|---------------------------------|-----------------|---|
| AMS 6470, AMS 6471. AMS 6472 | Nitralloy 135 M | 50 |
| AMS 6475 | Nitralloy EZ | 50 |
| AMS 6382; AMS 6414; AMS 6415 | Nitralloy N | 50 |
| AMS 6431; AMS 6439 | 4140; 4340 | 50 |
| AMS 6485, AMS 6487, AMS 6488 | D6; D6AC | 50 |
| | H11 | 60 |
| | Other grades | Per drawing requirements |

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 and its revision letter 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 Tensile strength to hardness conversions are presented in ASTM A 370.

8.2 Hardness conversion tables for metals are presented in ASTM E 140.

8.3 On austenitic and precipitation-hardening stainless steels, the diffusion layer may form a sharp border with the underlying matrix, exhibiting no transition zone from case to core. On such steels, the case depth is defined as the visual, dark etching zone.

8.4 Limitations on Grinding

The grinding operation, if applicable, should not reduce the effective case depth to below specification requirements. As guidance, the expected growth from side to side, e.g., across a diameter, is equal to approximately 70% of the thickness of the white layer.

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

8.5.1 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".

8.5.2 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.

8.5.3 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.

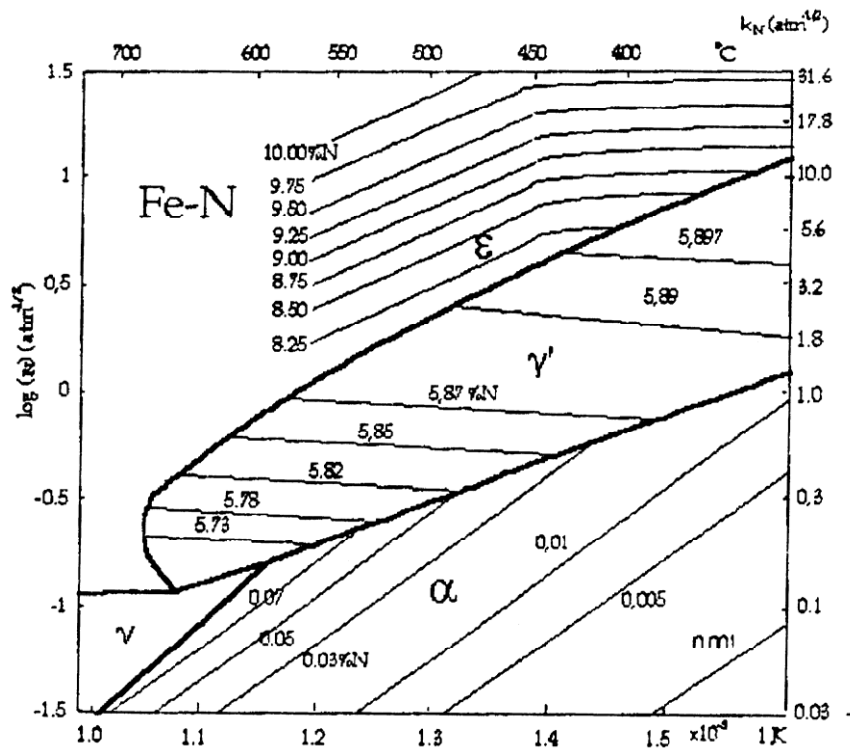


FIGURE 3 - THE LEHRER DIAGRAM WITH ADSORPTION ISOTHERMS FOR IRON

8.5.4 Nitriding Potential Values

Recommended ranges of nitriding potential values are given in Table 3.

TABLE 3 - RECOMMENDED RANGES OF NITRIDING POTENTIAL

| Alloy | Class 0 | Class 0 | Class 1 | Class 1 | Class 2 | 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 | 0.25 - 0.7 | 4 - 15 | 0.6 - 2.6 | 4 - 15 | 1.2 - 4.5 |
| D6, D6AC | 4 - 12 | 0.25 - 0.7 | 4 - 15 | 0.6 - 2.6 | 4 - 15 | 1.2 - 4.5 |
| H11 | 5 - 15 | 0.3 - 0.8 | 5 - 15 | 0.4 - 0.9 | 5 - 15 | 2.2 - 5.5 |
| Stainless | 5 - 15 | 0.2 - 0.7 | 5 - 15 | 0.4 - 0.9 | Not recommended | |
| Carbon steels | N/A | N/A | 5 - 12 | 0.8 - 2.6 | 1.2 - 4.0 | N/A |

8.6 Dimensions and properties in inch/pound units and the Fahrenheit temperatures are primary; dimensions and properties in SI units and the Celsius temperatures are shown as the approximate equivalents of the primary units and are presented only for information.