

GAS NITRIDING AND HEAT TREATMENT OF LOW-ALLOY STEEL PARTS

1. SCOPE: This document specifies the procedure and requirements for heat treating Nitralloy 135M, Nitralloy EZ, Nitralloy N, 4140, 4340, and H11 steels and for the subsequent gas nitriding of these alloys by the use of raw or dissociated ammonia.
 - 1.1 Purpose: Primarily for producing a wear resistant and fatigue resistant surface on steel parts.
 - 1.2 Application: The nitriding process, described herein, is applicable to the above low-alloy and tool steels. Other processes, such as salt bath nitriding, ion nitriding, and nitriding of other materials, are not included.
 - 1.3 Classification: Processes covered by this specification are classified as follows:
 - 1.3.1 Class 1: Two-stage nitriding with first stage at 940° - 1040°F (505° - 560°C) and second stage at 975° - 1050°F (525° - 565°C).
 - 1.3.2 Class 2: One-stage nitriding at 940° - 1040°F (505° - 560°C).
 - 1.3.3 If no class is specified, either Class 1 or Class 2 may be used.
2. APPLICABLE DOCUMENTS: The following publications form a part of this specification to the extent specified herein. The latest issue of Aerospace Material Specifications and Aerospace Recommended Practices shall apply. The applicable issue of other documents shall be as specified in AMS 2350.
 - 2.1 SAE Publications: Available from SAE, 400 Commonwealth Drive, Warrendale, PA 15096.

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2.1.1 Aerospace Material Specifications:

- AMS 2350 - Standards and Test Methods
- AMS 2418 - Copper Plating
- AMS 2429 - Bronze Plate Masking
- AMS 2759 - Heat Treatment of Steel, General Requirements
- AMS 2759/1 - Heat Treatment of Carbon and Low Alloy Steel Parts, Minimum Tensile Strength up to 220,000 psi (1515 MPa)
- AMS 2759/2 - Heat Treatment of Low-Alloy Steel Parts, Minimum Tensile Strength 220,000 psi (1515 MPa) and Higher

2.1.2 Aerospace Recommended Practices:

ARP 1820 - Chord Method of Evaluating Surface Characteristics

2.2 ASTM Publications: Available from American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.

2.2.1 ASTM Specifications:

- ASTM E18 - Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials
- ASTM E92 - Vickers Hardness of Metallic Materials
- ASTM E140 - Standard Hardness Conversion Tables for Metals (Relationship Between Brinell Hardness, Vickers Hardness, Rockwell Hardness, Rockwell Superficial Hardness, and Knoop Hardness)
- ASTM E384 - Microhardness of Materials

2.3 ANSI Publications: Available from American National Standards Institute Inc., 1430 Broadway, New York, NY 10018.

ANSI B46.1 - Surface Texture

2.4 ASM Publications: Available from American Society for Metals, Metals Park, OH 44073.

ASM Metals Handbook, Volume 7

3. TECHNICAL REQUIREMENTS:

3.1 Heat Treatment: Shall conform to AMS 2759 and to AMS 2759/1 or AMS 2759/2, as applicable, and the requirements specified herein.

3.2 Equipment: Shall conform to AMS 2759. Furnace temperature uniformity requirements for normalizing, hardening, straightening, stress relieving, and baking shall be $+25^{\circ}\text{F}$ ($+15^{\circ}\text{C}$), for tempering, $+15^{\circ}\text{F}$ ($+8^{\circ}\text{C}$), and for nitriding, $+10^{\circ}\text{F}$ ($+5^{\circ}\text{C}$).

3.2.1 Nitriding Atmosphere: Equipment shall be available for introducing ammonia gas into the furnace at a controlled rate. A separate system for ammonia dissociation is recommended.

3.2.2 Atmosphere Control: Equipment shall be available to measure and maintain the dissociation of the expelled gases within $\pm 5\%$ of the selected percent dissociation throughout the nitriding cycle.

3.3 Pre-Nitriding Requirements:

3.3.1 Decarburization: Surfaces to be nitrided shall be free from decarburization.

3.3.2 Hardening: All parts shall be hardened and tempered in accordance with Table I prior to nitriding. The tempering temperature should be at least 50°F (30°C) above the highest nitriding temperature to be used.

3.3.3 Subcritical Annealing and Normalizing: When these thermal treatments are required, they shall be carried out in accordance with Table I.

3.3.4 Stress Relieving: Unless otherwise specified, parts which have been ground, straightened, or otherwise mechanically worked, shall be stress relieved prior to nitriding. The stress relieving temperature shall be 50°F (30°C) below the tempering temperature.

3.3.5 Contamination: Parts to be nitrided shall be free from grease, oil, scale, and other contaminants. Care shall be exercised after cleaning to prevent recontamination.

3.3.6 Selective Nitriding: Selective nitriding shall be accomplished by masking surfaces not required to be nitrided. Alternatively, parts may be nitrided on all surfaces and the case ground off those surfaces not required to be nitrided.

3.3.6.1 Nitriding is prohibited on surfaces not designated to be nitrided except where optional nitriding is permitted.

3.3.6.2 Maskant: Shall be fine grained copper plate, not less than 0.001 in. (0.025 mm) in thickness, applied in accordance with AMS 2418 or shall be bronze plated not less than 0.0005 in. (12.5 μ m) in thickness, applied in accordance with AMS 2429. Other maskants may be used if approved by the cognizant engineering organization.

3.3.6.3 Loading: Parts shall be placed and supported to prevent distortion and to ensure free circulation of the nitriding gas to all surfaces. Test specimen(s) shall be placed at appropriate location(s) in the working zone in the same location as the parts they represent.

3.4 Nitriding:

- 3.4.1 Temperature Range: Shall be 940° - 1040°F (505° - 560°C) for Class 2 and for the first stage of Class 1. The temperature for the second stage of Class 1 shall be 975° - 1050°F (525° - 565°C).
- 3.4.2 Ammonia Dissociation: The ammonia gas dissociation shall be 15 - 35% for Class 2 and for the first stage of Class 1 (approximately 20% of total nitriding time). Ammonia dissociation for the second stage of Class 1 shall be 65 - 88%.
- 3.4.2.1 Measurement: Percent dissociation should be determined by infrared analysis (preferred) but other methods (e.g., burette) are acceptable.
- 3.4.3 Nitriding Procedure: The load shall be held at nitriding temperature(s) in ammonia atmosphere(s) for sufficient time to produce the specified depth of case. Subsequently, the load shall be cooled in either the nitriding atmosphere or in an inert atmosphere to 300°F (150°C) or below. The furnace or retort should be flushed with inert gas before opening.
- 3.5 Post-Nitriding Operations:
- 3.5.1 Maskant Stripping: Shall be accomplished by use of a non-embrittling stripper.
- 3.5.2 Removal of White Layer: Unless otherwise specified, white layer shall be removed by tapping, honing, grinding, or etching with maximum stock removal of 0.002 in. (0.05 mm). If grinding is used, the parts shall be stress relieved in accordance with 3.5.3.
- 3.5.2.1 Limitation on Grinding: At no time shall the grinding operation reduce the effective case depth to below specification requirements.
- 3.5.3 Stress Relieving of Ground Parts: Except as noted, parts shall be stress relieved at 900°F + 10° (480°C + 5°) for not less than 1 hr, plus one additional hour for each inch (25 mm) of thickness, or fraction thereof greater than 1 in. (25 mm), followed by cooling in air. Lightly ground parts, subject to approval of the cognizant engineering organization, are not required to be stress relieved.
- 3.5.4 Embrittlement Relief: Pickled, plated, or electrolytically cleaned parts shall be baked to remove hydrogen as specified in the document controlling the embrittling process (e.g., AMS 2400). Where pickling or electrolytic cleaning is performed as an integral part of a plating operation, baking is not required after each process but only the final one.
- 3.6 Core Hardness and Strength Properties: Shall conform to the requirements of the drawing.

3.7 Microstructure: Metallographic examination shall be made at not lower than 400X magnification. The finished case shall exhibit a uniform distribution of nitrides diminishing gradually from the surface to the core. There shall be no evidence of a nitride network. Unless otherwise specified when white layer is permitted, the maximum depth of the white layer shall be 0.0005 in. (12.5 μ m) for Class 1 and 0.001 in. (25 μ m) for Class 2. Core microstructure shall be uniform tempered martensite.

3.7.1 Reference Photomicrographs: Case and core microstructures are illustrated in photographs 528, 529, 531, 532 and 534 of the ASM Metals Handbook, Volume 7, 8th Edition.

3.8 Case Depth: Shall conform to drawing requirements.

3.8.1 When effective case depth is specified, it shall be determined in accordance with ASTM E384. It shall be the depth below the surface at which the following minimum Rockwell hardness values are met.

Specification	Alloy	Minimum Hardness (HRC) or Equivalent*
AMS 6470, AMS 6471, AMS 6472	Nitralloy 135 Mod	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

* Conversions shall be in accordance with ASTM E140.

3.8.2 When total case depth, as distinct from effective case depth, is specified, it shall be the depth of the dark aging band, determined metallographically using suitable etch procedures.

- 3.8.3 Surface Hardness: Shall meet the following requirements, determined in accordance with ASTM E18 or ASTM E92.

Alloy	Minimum Hardness (HR15N) or Equivalent*
Nitralloy 135 Mod	92.5
Nitralloy EZ	92.5
Nitralloy N	92.5
4140, 4340	85.5
H11	92.1

* Conversions shall be in accordance with ASTM E140.

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 shall be as specified in AMS 2759 and as specified in 4.1.1, 4.1.2, and 4.1.3.
- 4.1.1 Acceptance Tests: In addition to the tests specified in 2759, tests to determine conformance to the requirements of 3.6, 3.7, 3.8 and 3.8.3 shall be performed on each lot.
- 4.1.2 Periodic Tests: Shall be as specified in the applicable requirements of AMS 2759.
- 4.1.3 Preproduction Tests: In addition to the tests specified in AMS 2759, tests to determine conformance to the requirements of 3.6, 3.7, 3.8, and 3.8.3 shall be performed prior to initial production nitriding in each furnace.
- 4.2 Sampling: A lot shall be all parts of the same alloy and part number, heat treated to the same property requirements in the same furnace(s) at the same time, and presented for vendor's inspection at one time.
- 4.2.1 Process Control Specimens: Each lot of parts nitrided in each furnace load shall be accompanied by at least one process control specimen of the same alloy. Specimen(s) shall be identified for correlation with the furnace load lot(s) and the parts. Specimen(s) shall have been hardened by the same treatment as the parts and shall have been appropriately masked, if the parts are masked, along with the parts.

Exception: When more than five lots of the same alloy are included in a single furnace load, five specimens may be used to represent all lots.

- 4.2.2 Specimen Description: Specimens shall have a thickness not less than 0.125 in. (3.2 mm), be free of any surface imperfections such as decarburization, and have a surface texture not rougher than 32 microin. (0.8 μm) determined in accordance with ANSI B46.1. Alternatively, parts may be used in lieu of a specimen(s).
- 4.2.3 Metallographic Examination: Shall be made at not less than 400X magnification on polished and etched specimens. Specimens may be plated for edge retention prior to mounting. The chord method, described in ARP 1820, or equivalent, shall be used to assess the surface features (e.g., white layer) of the case. If a part is used in lieu of a specimen, the surface shall be sliced at an angle to magnify the surface layer by a factor of at least 3. The portion to be sectioned shall have a surface texture not rougher than 32 microin. (0.8 μm), determined in accordance with ANSI B46.1.
- 4.2.4 Core Hardness: Each part shall be hardness tested in accordance with AMS 2759, prior to nitriding, except when statistical sampling is authorized by the cognizant quality assurance organization.
- 4.2.5 Case Hardness: Unless otherwise specified, 5% of each lot of nitrided parts shall be tested for case hardness. This is in addition to hardness testing of control test specimens.
- 4.2.6 Effectiveness of Maskant: Unless otherwise specified, masked control specimens and 5% of each lot of nitrided parts containing surfaces on which nitriding is not permitted shall be tested to ensure that the hardness of masked surfaces conforms to the core hardness specified.
5. PREPARATION FOR DELIVERY: Shall be in accordance with AMS 2759.
6. ACKNOWLEDGMENT: The heat treating processor shall mention this specification number in all quotations and when acknowledging purchase orders.
7. REJECTIONS: Parts not heat treated in accordance with this specification or modifications authorized by the cognizant engineering organization will be subject to rejection and shall be submitted for disposition in accordance with purchaser's procedures for nonconformance.
8. NOTES: Shall be in accordance with AMS 2759 and as follows:
- 8.1 Other steels (e.g. 17-4PH, 17-7PH, 300 and 400 series corrosion-resistant) can also be nitrided using proprietary additives to the atmosphere.
- 8.2 Allowance should be made for dimensional growth during nitriding. Depending upon the base material, growth will generally be 2 to 10% of the effective case depth per surface.
- 8.3 Case depths up to 0.035 in. (0.88 mm) can be obtained by nitriding, depending on the base material, the nitriding process, and the time (12 to 100 hr) at the nitriding temperature.