AEROSPACE MATERIAL SPECIFICATION

AMS2759/6	REV. B

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Superseding AMS2759/6A

Gas Nitriding and Heat Treatment of Low-Alloy Steel Parts

RATIONALE

AMS2759/6B has been reaffirmed to comply with the SAE five-year review policy.

1. SCOPE:

This document specifies the procedure and requirements for heat treating low-alloy and tool steels and for their subsequent gas nitriding by the use of raw or dissociated ammonia. Specific process parameters are given for Nitralloy 135M, Nitralloy EZ, Nitralloy N, 4140, 4340, D6AC and H11 steels. Additional alloys and steel families (e.g., stainless steels) can also be nitrided using parameters acceptable to the cognizant engineering organization.

1.1 Application:

The nitriding process described herein has been used typically for producing a wear resistant and fatigue resistant surface on steel parts but usage is not limited to such applications. This process only applies to gas nitriding. Other processes, such as salt bath nitriding, or ion nitriding, are not included.

1.2 Classification:

Processes covered by this specification are classified as follows:

1.2.1 Class 1: Two-stage nitriding with first stage at 940 to 1050 °F (504 to 566 °C) and second stage at 975 to 1050 °F (524 to 566 °C).

Class 2: One-stage nitriding at 940 to 1050 °F (504 to 566 °C).

1.2.2 If no class is specified, either Class 1 or Class 2 may be used.

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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 canceled and no superseding document has been specified, the last published issue of that document shall apply.

2.1 SAE Publications:

Available from SAE, 400 Commonwealth Drive, Warrendale, PA 15096-0001 or www.sae.org.

AMS 2418	Plating, Copper
AMS 2429	Masking, Bronze Plate, Nitriding Stop-Off
AMS 2759	Heat Treatment of Steel Parts, General Requirements
AMS 2759/1	Heat Treatment of Carbon and Low-Alloy Steel Parts, Minimum Tensile Strength Below 220 ksi (1517 MPa)
AMS 2759/2	Heat Treatment of Low-Alloy Steel Parts, Minimum Tensile Strength 220 ksi (1517 MPa) and Higher
AMS 2759/9	Hydrogen Embrittlement Relief (Baking) of Steel Parts
ARP1820	Chord Method of Evaluating Surface Microstructural Characteristics

2.2 ASTM Publications:

Available from ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959 or www.astm.org.

ASTM E 18	Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials
ASTM E 92	Vickers Hardness of Metallic Materials
ASTM E 384	Microindentation Hardness of Materials

2.3 ANSI Publications:

Available from ANSI, 25 West 43rd Street, New York, NY 10036.

ANSI B46.1 Surface Texture

2.4 ASM Publications:

Available from ASM International, Materials Park, OH 44073-0002.

ASM Metals Handbook, Volume 7, Eighth Edition
ASM Metals Handbook, Volume 9, Ninth Edition

3. TECHNICAL REQUIREMENTS:

3.1 Heat Treatment:

Shall be conducted in accordance with AMS 2759 and to AMS 2759/1 or AMS 2759/2, as applicable, and the requirements specified herein. In case of conflict, the requirements of this publication shall apply.

3.2 Equipment:

Shall conform to AMS 2759. Furnace temperature uniformity requirements for normalizing, hardening, straightening, stress relieving, and baking shall be ±25 F (±15 C) degrees, for tempering, ±15 F (±8 C) degrees, and for nitriding ±15 F (±8 C) degrees.

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 process atmosphere in the retort within ±5 percent 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: Unless otherwise specified, all parts shall be hardened and tempered in accordance with Table 1 for applicable alloys prior to nitriding.

3.3.3 Subcritical Annealing and Normalizing: When these thermal treatments are required, they shall be carried out in accordance with Table 1.

3.3.4 Stress Relieving: Unless otherwise specified, parts which have been ground, straightened, or otherwise mechanically worked after hardening, shall be stress relieved prior to nitriding. The stress relieving temperature shall not be higher than 50 °F (28 °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 inch (25 µm) in thickness, applied in accordance with AMS 2418, or shall be bronze plate, not less than 0.0005 inch (12.7 µm) in thickness, applied in accordance with AMS 2429. Other maskants may be used if acceptable to the cognizant engineering organization (See 8.10.1).

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 specimens, or parts to be destructively evaluated, shall be placed in the working zone in the same location or locations as the parts they represent.

TABLE 1 - Pre-Nitriding Heat Treating Requirements for Nitralloy 135 Mod, Nitralloy EZ, Nitralloy N, 4140, 4340, D6AC and H11(1)

Steel	Temperatures °F (°C) Subcritical Anneal Before Hardening	Temperatures °F (°C) Normalize	Temperatures °F (°C) Austenitize	Approximate Tempering Temperature(2) °F (°C)	Required Minimum(4) HRC	Strength Ranges ksi	Strength Ranges Mpa	Quench Method
Nitralloy 135 Mod	1275 (690)	1800 (982)	1725 (941)	1000 (538) 1100 (593) 1200 (649) 1225 (663)	40 36 30 28	180-200 160-180 135-155 130-150	1241-1379 1103-1241 931-1069 896-1034	Oil, Polymer
Nitralloy EZ	1275 (690)	1800 (982)	1725 (941)	1100 (593) 1200 (649)	31 23	140-160 125-145	965-1103 862-1000	Oil, Polymer
Nitralloy N	1275 (690)	1750 (954)	1650 (899)	1200 (649)	28	130-150(5)	896-1034	Oil, Polymer
4140	1250 (677)	1650 (899)	1575 (857)	1025 (552) 1100 (593) 1200 (649)	34 31 27	150-170 140-160 125-145	1034-1172 965-1103 862-1000	Oil, Polymer
4340	1250 (677)	1650 (899)	1500 (816)	1000 (538) 1050 (566) 1100 (593)	36 34 31	160-180 150-170 140-160	1103-1241 1034-1172 965-1103	Oil, Polymer
D6AC	1250 (677)	1725 (941)	1625 (885) (3)	1025 (552)	46	220-240	1517-1655	Oil, Polymer
H11	1250 (677)		1850 (1010)	1025 (552) 1100 (593) 1150 (621)	46 43 40	220-240 200-220 180-200	1517-1655 1379-1517 1241-1379	Oil, Polymer or Air

(1) Preheat parts 36 HRC [160 ksi (1105 MPa)] or higher and parts which include complicated shapes, sharp reentrant angles, and drastic transitions in thickness by soaking at 1100 to 1250 °F (593 to 677 °C).
(2) The temperature will vary from the listed typical temperatures depending on the specified core hardness or strength, the hardenability of each heat of steel, and the severity of the quench, thus the as-quench hardness.
(3) 1700 °F (927 °C) is permitted for D6AC parts when approved by the cognizant engineering organization.
(4) These values apply to the “as hardened” condition.
(5) Strength level will increase after nitriding due to precipitation hardening at the nitriding temperature. Final strength level is dependent upon choice of nitriding temperature. A typical strength level of 190 ksi (1310 MPa) is developed after nitriding at 975 °F.

3.4 Nitriding:

3.4.1 Temperature Range: Shall be 940 to 1050 °F (504 to 566 °C).

3.4.1.1 Nitriding Temperature: Should be not higher than 50 °F (28 °C) below the tempering temperature and shall not exceed the tempering temperature.

3.4.2 Ammonia Dissociation: The ammonia gas dissociation shall be 15 to 35 percent for Class 2 and for the first stage of Class 1 (approximately 20 percent of total nitriding time). Ammonia dissociation for the second stage of Class 1 shall be 65 to 88 percent. Raw ammonia may be used when permitted by the purchaser (See 8.10.2).

3.4.2.1 Measurement: Equipment used for measuring shall be checked periodically for accuracy. Water absorption techniques (burette) are acceptable, but infrared analysis or in-situ probes are preferred.

3.4.3 Nitriding Procedure: The load shall be held at nitriding temperature(s) in partially dissociated ammonia atmosphere for sufficient time to produce the specified depth of case. Subsequently, the load shall be cooled from the nitriding temperature in a suitable atmosphere to 300 °F (149 °C) or below. The furnace or retort should be flushed with inert gas before opening.

3.5 Postnitriding Operations:

3.5.1 Maskant Stripping: Shall be accomplished by use of a non-embrittling stripper and shall not pit or etch the part.

3.5.2 Removal of White Layer: Unless otherwise specified, white layer shall be removed to meet the requirements of paragraph 3.7 by lapping, honing, grinding, or etching with maximum stock removal of 0.002 inch (0.05 mm). If grinding is used, the parts shall be stress relieved after grinding in accordance with 3.5.2.1. If etching is used, parts shall be embrittlement relieved after etching in accordance with 3.5.3.

3.5.2.1 Stress Relieving of Ground Parts: Parts shall be stress relieved at 800 °F ± 15 (427 °C ± 8) for not less than 1 hour, plus one additional hour for each inch (25 mm) of thickness, or fraction thereof greater than 1 inch (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.

3.5.2.2 Limitation on Metal Removal: When any metal removal is performed, it shall not reduce the effective case depth or surface hardness to below specification requirements.

3.5.3 Embrittlement Relief: Pickled, plated, etched (to remove white layer), or electrolytically cleaned parts shall be baked to remove hydrogen as specified in AMS 2759/9. 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 provided not more than 4 hours elapse between such operations.

3.6 Core Hardness and Strength Properties:

Shall conform to the requirements of the drawing.

3.7 Microstructure:

The finished case of low alloy steels shall exhibit a uniform distribution of nitrides diminishing gradually from the surface to the core. Corrosion resistant steels and highly alloyed steels (e.g. tool steels) may exhibit one or two metallographically distinct zones which may end abruptly. There shall be no evidence of a 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 µm) for Class 2. Core microstructures shall be uniform tempered martensite. (See 8.11.)

3.8 Total Case Depth:

Shall conform to drawing requirements. The total case depth shall be the depth of the continuous etching subsurface zone, determined metallographically on the as-nitrided part or specimen prior to machining. On those alloys which do not respond by darker etching, the 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, conducted in accordance with ASTM E 384.

3.8.1 Effective Case Depth: Where specified, it shall be in accordance with drawing requirements. It shall be the depth, after finish machining, at which the values specified in Table 2, converted from microhardness, are obtained in accordance with ASTM E 384 (See 8.9).

TABLE 2 - Hardness at Effective Case Depth

Specification	Alloy	Hardness HRC or Equivalent (See 8.9)
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 6431, AMS 6438	D6AC	50
AMS 6485, AMS 6487, AMS 6488	H11	60
Other alloys		As specified

3.8.1.1 For gear and splines, case depth applies at the pitch line of the gear or spline. For internal bores, case depth applies in all areas.

3.9 Surface Hardness:

Shall meet the requirements of Table 3 or equivalent, as determined in accordance with ASTM E 18, ASTM E 92 or ASTM E 384. In case of dispute, Rockwell superficial hardness or microhardness tests, as appropriate, shall govern.

TABLE 3 - Minimum Surface Hardness Requirements

Alloy	Hardness (HR15N) or Equivalent (See 8.9)
Nitralloy 135 modified	92.5
Nitralloy EZ	92.5
Nitralloy N	92.5
AISI 4140, AISI 4340	85.5
D6AC	85.5
H11	92.1
Other alloys	As Specified

4. QUALITY ASSURANCE PROVISIONS:

4.1 Responsibility for Inspection:

Shall be in accordance with AMS 2759. Where parts are required for destructive test, these shall be provided by the purchaser.

4.2 Classification of Tests:

4.2.1 Acceptance Tests: In addition to the tests specified in AMS 2759, core hardness (3.6), microstructure (3.7), case depth (3.8) and surface hardness (3.9) are acceptance tests and shall be performed on each lot.

4.2.2 Periodic Tests: Shall be as specified in the applicable requirements of AMS 2759.

4.2.3 Preproduction Test: In addition to the tests specified in AMS 2759, all tests are preproduction tests and shall be performed prior to initial production nitriding in each furnace.

4.3 Sampling and Testing:

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, nitrided in the same furnace at the same time, and presented for vendor's inspection at one time.

4.3.1 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. Actual parts, or sections of parts, produced in the same machining and heat treating lot as the parts to be nitrided, are preferred. When test specimens are used, they shall conform to 4.3.2. Specimens shall be identified for correlation with the furnace load lots and the parts. Specimens or test parts 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.

4.3.1.1 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.

4.3.2 Specimen Description: Specimens of the same alloy as the actual parts shall have a thickness not less than 0.125 inch (3.2 mm), be free of any surface imperfections such as decarburization, and have a surface texture not rougher than 32 microinches (0.0008 mm) in the areas to be tested, determined in accordance with ANSI B46.1. Alternatively, parts may be used in lieu of a specimen (See 4.2.1). Specimen preparation shall be done in the same manner as the parts they represent.

4.3.3 Metallographic Examination: Shall be made on polished and etched specimens. Specimens may be plated for edge retention prior to mounting. If plating is used, it shall be by a method that does not remove material from the surface of the specimen. The chord method, described in ARP1820, or equivalent, may 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 should be sliced at an angle to magnify the surface by a factor of at least three.

4.3.4 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. Test specimen(s) processed with the load through heat treating and nitriding shall meet the core hardness requirement.

4.3.5 Surface Hardness: Unless otherwise specified, 5% of each lot of nitrided parts shall be tested for surface hardness after completion of white layer removal. This is in addition to hardness testing of control test specimens.

4.3.6 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. Additionally, maskants shall be checked visually for evidence of blistering after nitriding but before subsequent stripping and etching.

4.4 Reports:

In addition to the requirements of AMS 2759, the processor shall furnish with each shipment of parts a report showing the results of tests made on parts or test specimens, as appropriate, to ensure conformance to specification requirements as follows:

4.4.1 For the Heat Treater: The report shall show actual hardness test results, and state that hardening and tempering conforms to the other specified requirements.

4.4.2 For the Nitrider: The report shall show the results of tests for core hardness, microstructure, as-nitrided case depth and surface hardness, and state that nitriding conformed to the other specified requirements.

4.4.3 For the Finish Machining Facility: The report shall show maximum and minimum stock removal, and effective case depth, depth of white layer if present, and surface hardness after machining, and state that processing conforms to the other specified requirements.