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INTERNATIONAL
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AEROSPACE MATERIAL SPECIFICATION

Submitted for recognition as an American National Standard

SAE

AMS 2759/8

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ION NITRIDING

1. SCOPE:

1.1 Purpose:

This specification establishes the requirements and procedures for producing a hard, wear resistant ion nitrided surface on steel parts.

1.2 Application:

The nitriding process, described herein, is applicable to alloy steels, tool steels, nitriding steels, corrosion resistant steels, and other ferrous alloys.

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.

2.1 SAE Publications:

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

AMS 2418	Copper Plating
AMS 2759	Heat Treatment of Steel Parts, General Requirements
AMS 2759/6	Gas Nitriding and Heat Treatment of Low-Alloy Steel Parts
ARP1820	Chord Method of Evaluating Surface Characteristics

Printed in U.S.A.
91-O942/ EG

2.2 ASTM Publications:

Available from ASTM, 1916 Race Street, Philadelphia, PA 19103-1187.

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

3. TECHNICAL REQUIREMENTS:

3.1 Heat Treatment Prior to Nitriding:

Shall conform to AMS 2759 and its applicable detail specification and to the requirements specified herein for the particular grade of material treated. For steels not covered by AMS 2759, heat treatment shall be in accordance with steel supplier recommendations and process controls shall be in accordance with AMS 2759.

3.2 Pyrometric equipment shall conform to AMS 2759 except that temperature uniformity shall be ±15 °F (±8 °C) with the glow discharge power on.

3.2.1 Thermocouples: Shall be protected to prevent deterioration due to contamination by furnace atmospheres. Thermocouples shall be isolated electrically from both the anode and cathode to prevent heating of the thermocouple by the glow discharge.

3.2.2 Vacuum (Pressure) Controls: Equipment shall be available for measuring, controlling, and recording vacuum (pressure) levels in the vessel during heating and at any time the power is on for glow discharge. The control equipment shall be capable of maintaining the vacuum (pressure) level at any setpoint within the equipment operating range with sufficient accuracy to meet the processing requirements of the parts being treated.

3.2.3 Nitriding Atmosphere: The type of surface layer is influenced by gas composition. The nitriding gas composition shall be selected to produce a monophase compound layer or a pure diffusion zone as specified by the cognizant engineering organization. Guidelines on gas composition effects on compound layer formation are as follows:

3.2.3.1 Effects of Nitriding Atmosphere on Type of Compound Layer Produced: The type of nitride layer produced is mainly influenced by the composition and substrate material. Steels with high carbon content, greater than 1% by weight, readily form the epsilon iron nitride phase as carbon is soluble in and stabilizes this phase. Carbon in the form of methane or CO_2 reacts to stabilize the epsilon phase. This phase is desired only for plain carbon steels and cast irons. Presence of oxygen may also lead to an epsilon phase. Treatments to produce an epsilon phase compound layer are referred to as nitrocarburizing treatments and are not covered by this specification. Nitrogen is necessary for compound layer formation. The following minimum levels of nitrogen in the ingoing gas are given as a guideline:

3.2.3.2 Low-alloy steels (containing up to 5% nominal alloying elements by weight) - 5% nitrogen by volume, to ensure at least a diffusion zone.

3.2.3.3 High alloy, corrosion resistant, and tool steels (steels with more than 5% nominal alloying elements by weight) - 15% nitrogen by volume.

3.2.3.4 When a compound layer is required, a minimum of 25% nitrogen by volume is suggested, unless otherwise determined by preproduction tests.

3.2.4 Atmosphere Control: Equipment shall be available for mixing the nitrogen and hydrogen gases in variable ratios so that the process gas may be varied from 0% nitrogen (pure hydrogen) to 100% nitrogen, and for introducing the mixture of these into the vacuum chamber at a controlled rate. Alternatively, a premixed gas of certified composition can be used. The atmosphere equipment shall be capable of providing a measured value of the components of the mixed gas within ±1% by volume of the selected value. For example, if the gas contains 25% nitrogen, the concentration can range from 24 to 26%.

3.2.5 Leak Rate: The furnace leak rate, expressed as microns per hour shall be measured before each treatment cycle. The leak rate shall not exceed 10 microns per hour. Leak rate shall be determined, before starting the heat cycle, after loading and closing the vessel, and evacuating to 75 microns (10 Pa) or lower, closing all valves to the vessel chamber. The initial pressure is recorded and a second reading taken at least 15 minutes after the first reading. Leak rate shall be calculated by subtracting the first reading from the second, dividing the result by the time interval, and multiplying the resultant number by 60.

3.2.6 Initial Evacuation of Vessel: At the start of each cycle, before admitting nitriding gas and before turning on power for heating, the vessel shall be evacuated to a pressure of 75 microns (10 Pa) or lower.

3.3 Nitriding Requirements:

3.3.1 Surfaces to be nitrided shall be free from decarburization.

3.3.2 All heat treatable steels to be nitrided shall be hardened and tempered prior to nitriding. The nitriding temperatures shall be no higher than 25 °F (14 °C) below the part tempering temperature.

3.3.3 Parts which have been ground, straightened, etc before nitriding may be stress relieved prior to nitriding. The stress relieving temperature shall be in accordance with AMS 2759 and its applicable detail specification. For materials, such as tool steels, which are not covered by AMS 2759, stress relieving temperature shall be not less than 20 °F (11°C) above the nitriding temperature. Surfaces to be nitrided shall be mechanically or chemically cleaned subsequent to stress relieving and prior to nitriding.

3.3.4 Parts to be nitrided shall be free of grease, oil, and other contaminants. Equipment shall be available to suitably degrease and otherwise clean areas of the part to be nitrided. (Cleaning agents shall be free of water or compounds which would inhibit nitriding).

3.3.5 Ion nitriding may cause sputtering of commonly used masking materials. As a result, the preferred method of masking is by mechanical means such as plates or other covers which act as barriers between the glow discharge and the part surfaces. Other masking materials, such as copper plate masks or copper paste, may be used if approved by the cognizant engineering organization. When copper plating is used, it shall be applied in accordance with AMS 2418. Tin-bearing masking material shall not be used. When copper plate or paint is used for masking, ion power voltage shall be limited to 600 volts. As an alternative to masking, parts may be nitrided all over and the case ground from the surface where a nitride case is not desired.

3.4 Procedure:

3.4.1 Test specimens as in 4.3 shall be placed at locations representing the extremes of work temperature established in preproduction testing.

3.4.2 Nitriding: Parts shall be processed in ion nitriding equipment in the selected gas environment at a temperature and for a time sufficient to produce the specified depth of case and compound layer. After completion of the nitriding cycle and prior to removal from the furnace, parts and test specimens shall be cooled to 300 °F (149 °C) or below in either the nitriding atmosphere, a non-reactive atmosphere, or vacuum. Removal of parts at higher temperatures shall be approved by the cognizant engineering organization.

3.5 Post Nitriding Operations:

3.5.1 Compound Layer Removal: When removal of compound layer generated during nitriding is required, it should be accomplished by lapping or honing with maximum stock removal of 0.001 inch (0.025 mm). If grinding is used, the parts shall be stress relieved as in 3.5.2, after grinding. Alternatively, compound layer may be removed by chemical means if approved by the cognizant engineering organization. When remaining compound layer is permitted, the depth of the compound layer shall be no greater than 0.0005 inch (0.0127 mm), unless otherwise permitted by the cognizant engineering organization.

3.5.2 Stress Relief: Parts ground after nitriding shall be stress relieved by heating to a temperature within the range 325 - 650 °F (163 - 343 °C), holding at the selected temperature within ±15 °F (±8 °C) for not less than 2 hours, and cooling in air.

3.5.3 Stripping of Masking Materials: Shall be accomplished by use of a non-embrittling stripper.

3.6 Properties:

Shall be as follows, determined on parts or on test specimens.

3.6.1 Compound Layer and Nitride Distribution: The compound layer, when permitted, shall be single phase.

3.6.1.1 Compound Layer Identification: After qualification of the procedures in preproduction testing, one or both of the etching techniques described in 8.9 and 8.10 may be used to detect and identify compound layer. Alternatively, the type of compound layer may be identified by x-ray diffraction.

3.6.1.2 Nitride Distribution: The finished case shall show a uniform distribution of nitrides, diminishing gradually from the surface to the core. There shall be no evidence of continuous nitride-networks. Examination shall be made on specimens polished, etched, and examined at not lower than 400X magnification.

3.6.2 Case Depth: Shall be as specified by the cognizant engineering organization. The effective and total case depths shall be determined by microhardness surveys in accordance with ASTM E 384, on metallographically prepared specimens or parts, or in accordance with ARP1820. Case depth shall be defined as in 8.3 and 8.4.

3.6.3 Surface Hardness: Shall be as specified by the cognizant engineering organization or as follows:

3.6.3.1 For nitriding grade steels, the surface hardness shall be as specified in AMS 2759/6, determined in accordance with ASTM E 18 or ASTM E 92, as applicable.

3.6.4 Evidence of arcing on the part surfaces shall be cause for rejection.

4. QUALITY ASSURANCE PROVISIONS:

Shall be as follows:

4.1 Responsibility for Inspection:

Shall be as specified in AMS 2759.

4.2 Classification of Tests:

Shall be as specified in AMS 2759 and as follows:

4.2.1 Acceptance Tests: In addition to the tests specified in AMS 2759, the tests in 3.6 shall be performed on each furnace load.

4.2.2 Preproduction Tests: In addition to the tests specified in AMS 2759, the following shall be performed prior to any production nitriding for each piece of equipment to be used.

4.2.2.1 Certification of leak rate and accuracy of vacuum measuring and controlling instrumentation acceptable to the cognizant quality assurance organization.

4.2.2.2 Certification of gas mix accuracy acceptable to the cognizant quality assurance organization.

4.2.2.3 Certification of pressure vs. temperature program for avoidance of hollow cathodes, for each part number to be processed, by a method acceptable to the cognizant quality assurance organization.

4.2.2.4 Establishment of Processing Procedure: For each part number, a loading, operating, and testing practice shall be established. This shall include, for each furnace in which the parts are to be processed, the following:

4.2.2.4.1 Loading diagram showing the location and spacing of each identical part number.

4.2.2.4.2 Minimum and maximum number of parts which can be run with the same practice.

4.2.2.4.3 Other part numbers or geometries which can be run in the same load.

4.2.2.4.4 Location of work thermocouples and method of attaching to the load.
4.2.2.4.5 Procedure of masking.
4.2.2.4.6 Description of test specimens and their location in the load.
4.2.2.4.7 Specification of fixturing used including auxiliary anodes or cathodes.

4.2.2.4.8 Program for pressure vs. temperature and gas mixture to be used throughout the nitriding cycle.

4.2.2.4.9 Verification of temperature uniformity for the load and for the range of acceptable variations of the loading parameters.

4.2.2.4.10 Limits of gas composition to be used to produce the specified compound layer.

4.3 Sampling:

Shall be in accordance with AMS 2759 and as follows:

4.3.1 Each specific production part number or lot of parts nitrided in one furnace charge shall be accompanied by at least two test specimens as in 4.3.1.3 or 4.3.3.1.

4.3.1.1 In the case of furnace lots containing more than one steel, one suitable specimen for each steel shall be used.

4.3.1.2 Test specimens shall accompany the parts through all processing operations, including hardening, tempering, masking, nitriding, mask removal, etc.

4.3.1.2.1 Each test specimen shall be identified for correlation of the test specimen to furnace lot of parts.

4.3.1.3 The preferred test specimen shall be a finished part of each part number in the load.

4.3.1.3.1 As an alternative, a specimen having the same processing history as the parts can be used if approved by the cognizant quality assurance organization. The specimen shall be fabricated from the same steel as the parts and shall have hardness in the same range as the parts. The test specimen shall be similar in dimensions, tolerances, and geometry as the part which it represents. It shall have been verified to be representative in preproduction tests as in 4.2.2.4.

4.3.2 When a re-nitriding cycle is required, one half of all the original coupons plus a new set of coupons shall be included in the rerun. Alternatively, a cut face from the original specimen may be used to verify the nitriding parameters provided that the new specimen created meets the requirements of 4.3.1. Where the likelihood of retreatment is high, the specimens can be partially masked during the first treatment. If a second treatment is required, the masking is removed and the same test pieces rerun. The use of either of these alternative procedures shall be approved in advance by the cognizant quality assurance organization.

4.4 Approval:

4.4.1 Vendors (Subcontractors): Approval of a facility shall be in accordance with the applicable requirements of AMS 2759 and 4.2.2.

4.4.2 Personnel: Qualification of personnel shall be in accordance with the applicable requirements of AMS 2759.

4.5 Furnace Log Entries and Recorder Chart Entries:

Shall be in accordance with AMS 2759, and in addition, shall include the following for each furnace load:

Ion voltage at each temperature and pressure used
Ion current at each temperature and pressure used
Furnace pressure before start of heating and at each temperature used
Gas mixture
Leak test results prior to starting of cycle
Calibration of gas mixture
Sketch, diagram or photograph of load arrangement and work thermocouple locations, indicating location of control point
Type and description of masking used

4.6 Records:

Shall be in accordance with AMS 2759.

4.7 Reports:

Shall be in accordance with AMS 2759.

5. PREPARATION FOR DELIVERY:

Identification, protective treatment, and packaging shall be in accordance with AMS 2759.

6. ACKNOWLEDGMENT:

A vendor shall mention this specification number in all quotations and when acknowledging purchase orders.

7. REJECTIONS:

Parts on which the ion nitriding does not conform to this specification, or to modifications authorized by purchaser, will be subject to rejection.

8. NOTES:

8.1 Definition of Ion Nitriding:

Ion nitriding is a vacuum process for producing a nitrided case on ferrous materials using a glow discharge. Various terms have been used to describe the process, including plasma, plasma/ion nitriding, and ionitriding.

8.1.1 The workpieces to be ion nitrided are suspended or placed in a vacuum furnace in such a way that effective electrical isolation is provided. Together with the vacuum pump, the gas distribution system enables the furnace to be evacuated, filled with the appropriate treatment gas, and maintained at the required vacuum, usually between 133 to 1333 Pa (1 to 10 Torr).

8.1.2 Voltage, which can be set to any value from 300 volts to approximately 1000 volts, is applied between the workpiece to be ion nitrided and the wall of the furnace, the former being connected as the cathode and the latter as the anode. Under this potential, the molecules and atoms of the treatment gas are excited and ionized, producing the typical luminous phenomenon known as glow discharge around the exposed surfaces of the workpieces.

8.1.3 Ion nitriding can be carried out using either pure nitrogen or mixtures of nitrogen with hydrogen or suitable hydrocarbons. Gas composition determines the type of surface layer to be produced for the particular steel processed. Treatments using hydrocarbons are considered to be nitrocarburizing.

8.1.4 The temperature of the workpiece is measured with the aid of thermocouples and regulated by means of a controller varying the power output of the electric unit. The treatment temperature is, in most cases, set between 750 and 1110 °F (399 and 599 °C), depending on the composition and structure of the steel to be treated, as well as the stress which the workpiece will have to resist.

8.2 Types of Surface Layers:

In ion nitriding, as in other nitriding processes, two types of surfaces may be produced.

8.2.1 Diffusion Zone: In the steel itself, nitrogen diffuses according to classical principles, producing a hardened zone by precipitation and solid solution hardening. This is known as the diffusion zone and is detected as a dark etching region below the surface varying in hardness from the surface to the core. This is the region measured as the case in nitrided steels and may be as deep as 0.035 inch (0.89 mm) depending upon the steel, time, and temperature.

8.2.2 Compound 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 commonly called white layer in gas nitriding. When the layer is very thick or contains a mixture of the two dominant nitride phases predicted by the iron-nitrogen phase diagram, this layer is very brittle and shall be removed.

8.3 Total case depth is defined as the maximum depth below the surface at which a Rockwell C hardness, or equivalent, of five HRC units above core hardness is achieved.

8.4 Effective case depth is defined as the maximum depth below the surface at which a hardness of 50 HRC, or equivalent, is achieved.

8.5 Allowance should be made for dimensional growth during nitriding. Growth varies with steel and specific treatment and should be determined by preproduction testing. In the absence of data, a growth of 0.0001 inch per 0.005 inch (0.0025 mm per 0.127 mm) diffusion depth may be considered typical.

8.6 Ion nitriding can be used to process a wide variety of steels. Table 1 shows typical results which can be achieved for various steels.

8.7 Hollow Cathode:

An effect created by gaps, holes, or cavities in parts resulting in an intense localized glow discharge. The intensity of the localized glow discharge is a function of temperature, pressure, gas composition, and length (depth) of the cavity divided by its width (or diameter in case of holes). Hollow cathode may result in localized overheating which can be detected by hardness tests as soft areas near susceptible areas.

8.8 Sharp corners or edges on parts should be avoided wherever possible. Growth takes place on all surfaces and combines at the corners to form projections containing a high nitrogen content which are very brittle and likely to chip.