SAE Aerospace
An SAE International Group

AEROSPACE MATERIAL SPECIFICATION

AMS 2759/12	
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Gaseous Nitrocarburizing, Automatically Controlled by Potentials

RATIONALE

AMS 2759/12 is a new specification to cover gaseous nitrocarburizing processing that is automatically controlled by carburizing and nitriding potentials.

1. SCOPE

1.1 Purpose

This specification covers the requirements for producing a continuous compound layer with controlled phase composition by means of a gaseous process, automatically controlled to maintain set values of the nitriding and carburizing potentials that determine properties of the nitrocarburized surface. Automatic control is intended to ensure repeatability of nitrogen and carbon content of the compound layer which establishes properties such as wear and corrosion resistance, ductility and fatigue strength.

1.2 Application

1.2.1 The nitrocarburizing process has been used typically for enhancement of wear resistance and fatigue strength, as well as corrosion resistance in ferrous alloys but usage is not limited to such applications. Nitrocarburizing can be selectively applied to only those areas that require nitrocarburizing.

1.2.2 Specifically, this process is recommended for those applications where control of phase composition is required.

1.3 Classification

The nitrocarburizing process described herein is classified as follows:

1.3.1 Class 1 - Porosity not exceeding 15% of the thickness of the compound (white) layer

1.3.2 Class 2 - Porosity above 10% but not exceeding 50% of the thickness of the white layer

1.3.3 If no class is specified, no limitations shall be placed on the degree of porosity

1.4 Safety - Hazardous Materials

While the materials, methods, applications, and processes described or referenced in this specification may involve the use of hazardous materials, this specification does not address the hazards which may be involved in such use. It is the responsibility of the user to ensure familiarity with the safe and proper use of any hazardous materials and to take necessary precautionary measures to ensure the health and safety of all personnel involved.

TO PLACE A DOCUMENT ORDER: **Tel: 877-606-7323 (inside USA and Canada)**
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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), www.sae.org.

AMS 2418	Plating, Copper
AMS 2429	Masking, Bronze Plate, Nitriding Stop-Off, 90Cu-10Sn
AMS 2750	Pyrometry
AMS 2759	Heat Treatment of Steel Parts, General Requirements
ARP1820	Chord Method of Evaluating Surface Microstructural Characteristics
SAE J423	Methods of Measuring Case Depth
SAE J864	Surface Hardness Testing With Files

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, www.astm.org.

ASTM D 1193	Reagent Water
ASTM E 384	Microhardness of Materials

2.3 ASM Publications

Available from ASM International, 9639 Kinsman Road, Materials Park, OH 44073-0002, Tel: 800-336-5152 (inside U.S. and Canada), 440-338-5151 (outside USA), www.asm-intl.org.

ASM Handbook, 9th Edition, Metallography and Microstructures

3. TECHNICAL REQUIREMENTS

3.1 Processing Equipment

Parts to be nitrocarburized shall be processed in a sealed retort type furnace with forced atmosphere circulation or a furnace with a sealed chamber and may be equipped with an integral quench facility conforming to AMS 2759. The furnaces shall be equipped with gas analyzing equipment capable of measuring the concentration of reactive gases in the retort, necessary for determination of the nitriding and carburizing potentials, based on reactions referenced in 8.5. The furnace temperature uniformity requirements shall be ± 15 °F (± 8 °C).

3.1.1 Atmosphere

The nitrocarburizing atmosphere shall consist of a mixture of gases supplying nitrogen, and supplying carbon. Any mixture of gases is acceptable, as long as it enables effective control of the nitriding and carburizing potentials.

3.1.2 Thermocouples

Load thermocouples, if used, shall be sheath-protected to prevent deterioration due to furnace atmospheres. Pyrometry shall conform to AMS 2750.

3.1.3 Shutdowns 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 to prevent any damage to parts being processed.

3.2 Pretreatment

3.2.1 Parts requiring hardening to a specified strength level shall be heat treated in accordance with AMS 2759 or as specified by drawing or cognizant engineering organization, to the required core hardness prior to nitrocarburizing. In cases of discrepancy, the requirements specified on the engineering drawing shall take precedence over those on the purchase order. Unless otherwise approved by the cognizant engineering organization, tempering temperature shall be at least 50 °F (28 °C) above the nitrocarburizing temperature.

3.2.2 Surface Condition

Parts shall be clean and free from grease, oil, and other contaminants. Parts shall be dry.

3.2.3 Stress Relieving

Unless otherwise specified, parts that have been ground, straightened, or otherwise mechanically worked after hardening, shall be stress relieved prior to nitrocarburizing, in accordance with AMS 2759/11. The stress relieving temperature shall not be higher than 50 °F (28 °C) below the tempering temperature. Surfaces to be nitrocarburized shall be cleaned by any effective method subsequent to stress relieving and prior to nitrocarburizing, to meet the requirements of 3.2.2.

3.2.4 Selective Nitrocarburizing

Surfaces that require protection from nitrocarburizing shall be masked by a suitable maskant, e.g., copper plating (AMS 2418), bronze plating (AMS 2429) or by alternative methods, acceptable to the cognizant engineering organization. Copper plate shall be fine-grained and nonporous, 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 (0.0125 mm) in thickness.

3.2.4.1 Alternative to Masking

If authorized by the cognizant engineering organization, parts may be nitrided all over and subsequently the case removed from areas not to be nitrocarburized.

3.3 Procedure

3.3.1 Loading

Parts shall be placed on racks or suspended, so as to minimize distortion, and to allow all surfaces to be nitrocarburized. If used, test coupons representing parts fabricated from the same alloy, with the same surface finish and in the same heat treatment condition, shall be placed in the working zone of the furnace, in a location adjacent to the parts they represent, as close as possible to the lowest and highest load temperatures based on the most recent temperature uniformity survey.

3.3.2 Preheating

Parts may be preheated in air to a temperature not higher than 850 °F (454 °C)

3.4 Nitrocarburizing of parts.

Nitrocarburizing shall be carried out in an automated mode, maintaining set parameters of temperature and nitriding and carburizing potentials, selected from Table 1. For any intermediate temperatures potential values may be interpolated graphically. See 8.8. The two potentials shall be selected in pairs from within the specified limit ranges.

TABLE 1 - PROCESS PARAMETERS ENSURING THE FORMATION OF AN EPSILON-TYPE COMPOUND LAYER WITH TWO RANGES OF POROSITY LEVELS, IN ACCORDANCE WITH 1.3

	Process Temperature		Process Time	Class 1 Not exceeding 15% of thickness of white layer				Class 2 Above 10% but not exceeding 40% of thickness of white layer			
				K_N		K_C		K_N		K_C	
Material	°F	°C	hr	min	max	min	max	min	max	min	max
Group 1*	1040	560	3 - 6	2.13	2.41	0.57	0.69	2.48	2.68	0.49	0.54
	1075	579	2 - 5	1.50	1.60	1.10	1.22	1.68	1.78	0.86	0.94
Group 2**	980	527	6 - 30	4.51	5.55	0.16	0.24	6.03	7.10	0.09	0.13
Group 3***	1060	571	3 - 10	1.82	2.10	0.76	0.99	2.22	2.64	0.48	0.68

For recommended ranges of nitriding and carburizing potentials at other temperatures, see 8.8. Temperatures shown are not firm requirements, however, once a temperature selected, both potential values shall be within specified limits for the given temperatures.

* Group 1 - HSLA, carbon steels
** Group 2 - 4140, 4340, Nitralloy 135M
*** Group 3 - Cast iron

3.5 Cooling

Following nitrocarburizing treatment, depending on the available equipment, parts shall be either furnace cooled or quenched in oil, water or soluble oil solutions, polymer quenchants, inert or nitrocarburizing atmospheres, or air.

3.6 Properties of Nitrocarburized Layers

Gaseous nitrocarburized components shall meet the following requirements, determined on either components or test specimens made from the same material and in the same heat treatment condition and processed in the same nitrocarburizing cycle.

3.6.1 Thickness of compound layers shall conform to limits shown in Table 2, determined in accordance with the procedure of SAE J423 or ARP1820 microscopic methods, at magnification of min. 500X.

TABLE 2 - COMPOUND LAYER THICKNESS

	Thickness (inch)		Thickness (mm)	
	min	max	min	max
Carbon and HSLA steel	0.0002	0.0010	0.0051	0.025
Low Alloy Steel	0.0002	0.0010	0.0051	0.025
Tool Steel	0.0001	0.0006	0.0025	0.015
Cast Iron	0.0002	0.0010	0.0051	0.025

3.6.2 Presence of Compound Layer

The presence of compound layer on non-austenitic steels and cast irons shall be determined by a chemical spot test. The test shall be performed by applying a drop of copper sulfate solution complying with 3.6.3 or copper ammonium chloride solution complying with 3.6.4 to a clean nitrocarburized surface. If, after 15 seconds, the spot to which the drop was applied turns red, due to the deposition of copper, compound layer is not present and the part is subject to rejection.

3.6.3 Preparation of Copper Sulfate Solution

Dissolve approximately 40 grams copper sulfate ($CuSO_4 \cdot 5H_2O$) in 1 liter of reagent water (See ASTM D 1193 Type II). A wetting agent (e.g., 5 mL glycerin) may be added. The pH shall be 3.5 to 4.1.

3.6.4 Preparation of Ammonium Chloride Solution

Dissolve approximately 100 grams of cupric chloride ($CuCl_2 \bullet 2H_2O$) in reagent water to make 1 liter. Add sufficient ammonium hydroxide solution (NH_4OH) to form a brilliant blue copper ammonium chloride complex solution.

3.6.5 Compound Layer Structure

At least three fields of view shall be examined, using magnification of minimum 500X. Porosity shall not exceed 15% of the total thickness of the compound layer for Class 1 and shall be above 10% but not exceeding 40% of the total thickness of the compound layer for Class 2. Porosity shall be concentrated in the external (upper) half of the white layer. Porosity in the lower quarter of the compound layer, at the interface with the substrate shall not exceed 5% for all steel grades, and 15% for cast irons. Extent of porosity shall be measured. See 8.7 for an acceptable method.

3.6.6 Compound layer hardness shall be measured in accordance with SAE J864 test for file hardness and shall be equivalent of 60 HRC or higher. Alternatively, surface hardness may be measured by a Vickers or a Knoop tester under a light test load, if specified by the cognizant engineering organization. In such cases, the surface hardness shall meet requirements of the engineering drawing or purchase order.

3.6.7 Diffusion zone depth. If specified by the cognizant engineering organization, diffusion case depth shall be measured and recorded. For carbon steels, diffusion case depth is determined visually with the aid of a metallurgical microscope, as the distance from the surface to the depth at which needle-like nitride precipitations are still visible. On rapidly cooled carbon steels, where needle precipitations are not visible, diffusion depth can be determined by appropriate application of etchants in addition to Nital. For alloy steels, case depth is determined by a microhardness traverse, defined as effective case depth to a specified hardness or total case depth to where the hardness drops to 50 HK or 50 HV above core hardness. If the engineering drawing specifies a case depth, but does not specifically state that it is to be effective at a given hardness level, total case depth shall apply. Where "etched case depth" or “visual case depth” is specified, it shall be the distance measured from the surface in the direction of the core to the location where the zone darkened by etching ends.

3.6.8 Diffusion Zone (Case) Hardness

If required by the cognizant engineering organization, shall be determined by a microhardness measurement, performed at a depth of 0.002 inch (0.05 mm) from the nitrocarburized surface, in accordance with ASTM E 384 or by the chord method of ARP 1820, on a mounted and polished cross-section of the nitrocarburized case. The Knoop or Vickers indenter may be used, as specified by the cognizant engineering organization.

TABLE 3 - DIFFUSION ZONE MINIMUM HARDNESS

Material	Hardness HK100	Hardness HV100
Carbon Steel	300	290
Low Alloy Steel	460	440
Tool Steels	600	565
Cast Iron	300	290

3.7 Records

A record (written or archived) shall be kept for each furnace load. The information on the combination of documents shall include: equipment identification, approved personnel identification, date, part number or product identification, number of parts, alloy, purchase order, lot identification, reference to this specification and/or other applicable specifications, actual times and temperatures used, as well as nitriding and carburizing potentials. When applicable, other atmosphere control parameters, quench delay, quenchant type, polymer concentration and quenchant temperature shall also be recorded. The maximum thickness, when process parameters are based on thickness, shall be recorded and shall be taken as the minimum dimension of the heaviest section of the part. Log data shall be recorded in accordance with documented procedures.

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for inspection

Purchaser of treated parts shall supply all necessary test coupons. The processor shall be responsible for the performance of all required tests. The cognizant engineering organization reserves the right to sample and to perform any confirmatory testing deemed necessary to ensure that the parts conform to specified requirements.

4.2 Classification of Tests

4.2.1 Acceptance Tests

Measurement of the thickness of compound layer (3.6.1), and, where specified, test for the presence of compound layer (3.6.2), case depth and/or diffusion zone depth (3.6.7), and compound layer hardness measurements (3.6.6) are acceptance tests and shall be performed on each processed lot.

4.2.2 Periodic Tests

Compound layer microstructure (3.6.5) and measurement of diffusion zone hardness (3.6.8) are periodic tests and shall be performed at a frequency established by the processor and approved the cognizant engineering organization.

4.2.3 Preproduction Tests

All property verification tests are preproduction tests and shall be performed prior to production and when the cognizant engineering organization requires verification testing.

4.3 Sampling for testing shall be not less than the following:

The processor shall process a sufficient number of parts and/or test coupons of the same material and heat treatment condition with each furnace load to permit performance of acceptance tests for each requirement by both the processor and the purchaser. A lot shall be all parts of the same part number processed in the same furnace load. The number of parts or coupons required for testing shall be as established by the cognizant engineering organization.

4.4 Approval

4.4.1 The process and control factors or a preproduction part, or both, whichever is specified, shall be approved by the cognizant engineering organization before production parts are supplied.

4.4.2 If the processor makes a significant change to any material, process, or control factor from that which was used for process approval, all preproduction tests shall be performed and the results submitted to the purchaser for process reapproval unless the change is approved by the cognizant engineering organization. A significant change is one which, in the judgment of the cognizant engineering organization, could affect the properties or performance of the parts.

4.5 Reports

The processor shall furnish with each shipment a report stating that the parts have been processed and tested in accordance with the specified requirements and that they conform to the acceptance test requirements. The report shall include purchase order number, AMS 2759/12, part number, heat lot and any other agreed control numbers, and quantity.

4.5.1 Resampling and Retesting

If any acceptance test result fails to meet specified test requirements, three additional parts or test coupons for each nonconformant test result may be acceptance tested. Failure of any retest specimen to meet all requirements shall be cause for rejection of the shipment. Results of all testing shall be reported.

5. PREPARATION FOR DELIVERY

5.1 Identification

Each container of processed parts shall be legibly identified with AMS 2759/12, processor's identification, purchase order number, part number, lot number and quantity.

5.2 Packaging

5.2.1 Nitrocarburized parts shall be packaged such as to ensure protection against damage during shipment by handling and/or exposure to environment.

5.2.2 Packages of nitrocarburized parts shall be prepared for shipment in accordance with commercial practice and in compliance with applicable rules and regulations pertaining to handling, packaging, and transportation, to ensure carrier acceptance and safe delivery.

6. ACKNOWLEDGEMENT

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

7. REJECTIONS

Parts that do not conform to this specification or to modifications authorized by the cognizant engineering organization will be subject to rejection.

8. NOTES

8.1 The change bar (|) located in the left margin is for convenience of the user in locating areas where technical revisions, not editorial changes, have been made to the previous issue of this specification. An (R) symbol to the left of the document title indicates a complete revision of the specification including technical revisions. Change bars and (R) are not used in original publications, nor in specifications that contain editorial changes only.

8.2 It is recommended that samples for metallurgical evaluation be prepared with adequate edge retention, such as can be obtained with nickel plate, for reliable readings of surface properties.

8.3 The content of nitrogen and carbon is related to the nitriding and carburizing potentials that are automatically controlled to be maintained within set parameters.

8.4 The nitriding potential is defined as $K_N = pNH_3/(pH_2)^{3/2}$ where:

pNH_3 is the partial pressure of ammonia in the outgoing atmosphere and pH_2 is the partial pressure of hydrogen in the outgoing atmosphere.

8.5 The carburizing potential can be defined based on one or another of two reactions:

1. The Boudouard reaction: $2CO = CO_2 + C$ for which $K_C = p^2CO/pCO_2$
2. The water gas shift reaction: $CO + H_2O = CO_2 + H_2$ for which $K_C = pCO*pH_2/pH_2O$

8.6 Range of concentrations

Values of nitriding and carburizing potentials are interrelated. Values of K_N and K_C given in Table 1 constitute limits within which process parameters must be set at a given temperature. If higher values of K_N are selected within any given range, selection of a higher K_C value from within the corresponding range will be conducive to limiting the extent of porosity. For any given fixed content of the two elements, values of K_N and K_C vary in opposite directions with temperature. For temperatures other than those shown in Table 1, interpolation or extrapolation may be used.

8.7 Extent of porosity shall be measured as the distance between the surface and the end of the dense porous zone, as revealed by etching with 2% Nital. Where observed, the deeper situated “spikes” of porosity, jutting out in the direction of the core, formed by transformation of the ε nitride into γ′ nitride during cooling, shall not be taken into account, in accordance with Fig. 1.

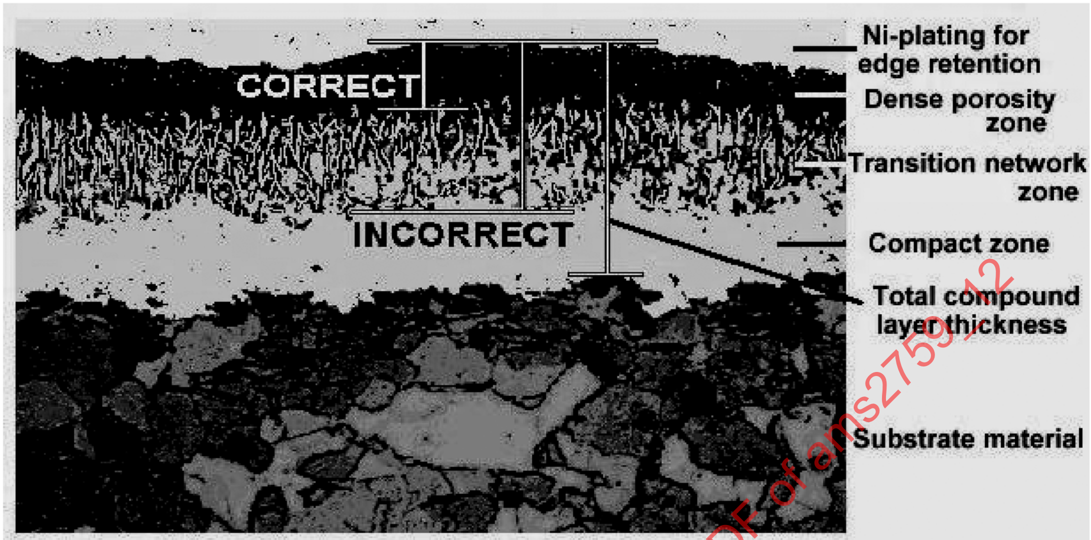

FIGURE 1 - EVALUATION OF EXTENT OF POROSITY IN COMPOUND LAYER ON NON-ALLOYED STEEL

8.8 Examples of microstructures showing porosity complying with Class 1 and Class 2.

Porosity on nitrided HSLA steel is shown in Fig. 2. Porosity on nitrided 4140 steel is shown in Fig. 3.

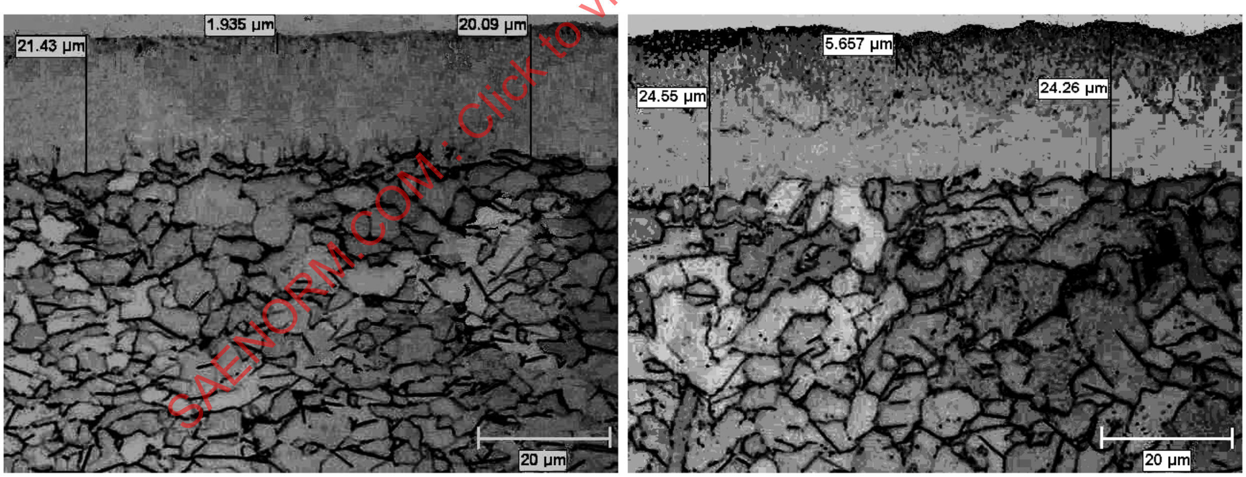

FIGURE 2 - NITRIDED COMPOUND LAYER ON HSLA STEEL. LEFT COMPLIES WITH CLASS 1 POROSITY; RIGHT: COMPLIES WITH CLASS 2 POROSITY