SAE INTERNATIONAL

AEROSPACE STANDARD

AS6414™ REV. A

Issued 2016-08
Revised 2017-06

Superseding AS6414

Manufacturing Processing Requirements for Molded Elastomer Components Used in Aerospace Applications

RATIONALE

This revision changes the CERTIFICATE OF CONFORMANCE requirement to CERTIFICATE OF ANALYSIS, clarifies blended batch requirements and updates references.

1. SCOPE

1.1 Application

This specification is applicable to the processes that are used in the manufacture of aerospace elastomer parts utilizing materials conforming to AMS7XXX series specifications, user specifications, or print on a Purchase Order (PO) that calls out this document for aerospace applications. The elastomer parts are in the form of O-rings, plate seals and other compression seals.

1.2 Safety-Hazardous Materials

While the use of materials, applications and processes described or referenced in this specification may involve hazardous materials, this specification does not address the hazards involved in their use. It is the responsibility of the manufacturer to provide the purchaser with the safe and proper use of materials and processes and to take necessary precautionary measures and training of personnel to ensure the health and safety of all personnel involved. However, it is not addressed in this document.

1.3 Order of Precedence

This specification is in addition to and in no way limiting, superseding, or abrogating any contractual obligation as required by the applicable procurement document. In the event of conflict in requirements, the order of precedence shall be:

1. Procurement Document or Contractual Agreement (excluding this document)
2. Applicable purchaser's drawing
3. Specifications referenced on the drawing
4. This document
5. All specifications referenced in this document

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SAE WEB ADDRESS: **http://www.sae.org**

2. REFERENCES

2.1 APPLICABLE DOCUMENTS

The following publications form a part of this document 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. In the event of conflict between the text of this document and references cited herein, the text of this document takes precedence. Nothing in this document, however, supersedes applicable laws and regulations unless a specific exemption has been obtained.

2.1.1 SAE Publications

Available from SAE International, 400 Commonwealth Drive, Warrendale, PA 15096-0001, Tel: 877-606-7323 (inside USA and Canada) or +1 724-776-4970 (outside USA), www.sae.org.

AMS2750 Pyrometry

AMS2817 Packaging and Identification, Preformed Packings

ARP5316 Storage of Elastomer Seals and Seal Assemblies Which Include an Elastomer Element Prior to Hardware Assembly

AS568 Aerospace Size Standard for O-Rings

AS5752 Aerospace - Visual Inspection Standard for Elastomeric Sealing Elements Other than O-Rings

AS9100 Quality Management Systems - Requirements for Aviation, Space and Defense Organizations

2.1.2 ANSI Accredited Publications

Copies of these documents are available online at http://webstore.ansi.org/.

ANSI/ASQ Z1.4 Sampling Procedures and Tables for Inspection by Attributes

2.1.3 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 D1414 Standard Test Methods for Rubber O-Rings

ASTM D2240 Standard Test Method for Rubber Property-Durometer Hardness

ASTM D7969 Standard Specification for Gravity Convection and Forced Ventilation Ovens

ASTM E145 Standard Specification for Gravity-Convection and Forced-Ventilation Ovens

2.1.4 ISO Publications

Copies of these documents are available online at http://webstore.ansi.org/.

ISO 3601-1	Fluid Power Systems – O-Rings – Part 1: Inside diameter, cross sections, tolerances and size identification codes
ISO 3601-3	Fluid Power Systems – O-Rings – Part 3: Quality Acceptance Criteria
ISO 9001	Quality management systems — Requirements
ISO 17025	General requirements for the competence of testing and calibration laboratories
ISO 27996	Aerospace Fluid Systems – Elastomer Seals – Storage and Shelf Life

2.1.5 PRI Publications

Available from Performance Review Institute, 161 Thorn Hill Road, Warrendale, PA 15086-7527, Tel: 724-772-1616, www.pri-network.org.

AC7115	Nadcap Audit Criteria for Manufacture of Elastomer Seals
AC7122-P	Nadcap Audit Criteria for Non Metallic Materials - Testing Laboratories P - Part Manufacturers Captive Laboratories
AC7122/1	Nadcap Audit Criteria for Non Metallic Materials - Testing Laboratories - Mechanical Testing
AC7122/2	Nadcap Audit Criteria for Non Metallic Materials - Testing Laboratories - Physical Testing
AC7122/3	Nadcap Audit Criteria for Non Metallic Materials - Testing Laboratories - Chemical Testing
AC7122/4	Nadcap Audit Criteria for Non Metallic Materials - Testing Laboratories - Thermal Analysis
PD2102	Aerospace Quality Assurance Product Standards, Qualification Procedure, Elastomeric Seal

2.2 Definitions

BATCH: The quantity of a compound run through a mill or mixer at one time. Mixing of batches of previously compounded material restricted to the requirements of this document.

BATCH TESTING (BATCH ACCEPTANCE TESTING or BATCH CONFORMANCE TESTING): Material property testing on a batch of compound through identified industry test methods and utilizing the same operating parameters of time, temperature, and pressure that lead to the same state of cure as the test specimens used to generate qualification data. The batch testing or acceptance tests and methods are identified within the applicable specification. In the event no specimen type or test method is identified then durometer (ASTM D2240, Type A), tensile and elongation (ASTM D1414), and specific gravity (ASTM D1414) shall be utilized.

BLENDED: The combination of the same formulation of multiple batches of uncured, conforming material to create a new batch of material of the same formulation.

CAPTIVE COMPOUNDER: A rubber mixing operation owned by or otherwise integrated into the same organization or management structure as the manufacturer.

CAPTIVE LABORATORY: A rubber testing laboratory owned by or otherwise integrated into the same organization or management structure as the manufacturer.

CERTIFICATE OF ANALYSIS (CofA): A document that displays the requirements and results with a statement of conformance for the compounded material or a finished part.

CHEMICAL MANUFACTURER: A company that produces compounding ingredients.

COMPOUND: A general term used to describe a particular formula/formulation that is mixed, uncured rubber material.

COMPOUNDER: A company that combines raw materials into the form of an uncured product (can be the same as the manufacturer).

COMPOUNDING INGREDIENT*: Any material incorporated with an elastomer to form a compound or mix.

DEFLASHING: The removal excess rubber at the parting line on molded rubber parts.

EXTERNAL COMPOUNDER: An independent rubber mixing operation that is not owned by the manufacturer or otherwise integrated into the manufacturer's organization or management structure.

FORMULA/FORMULATION: A list containing the identity of the ingredients to be included in a compound.

INITIAL QUALIFICATION (QPL): Refer to the PD2102 document for the amount of testing required where a full specification testing is based on one batch of data and a second set of verification data is also required.

LOT: A quantity of one size of product processed as one entity from a single batch.

LOT TESTING: Testing on lot samples with the specimen type and test methods as identified within the applicable specification.

MANUFACTURER: A company that receives an uncured rubber compound and produces finished product (can be the same as the compounder).

MASTER BATCH: The first pass of a multiple pass mix that does not include curatives. This is not tested.

PROCEDURE: An established, documented, implemented and maintained way to carry out an activity or a process.

PROCESS: Any activity or set of activities that uses resources to transform inputs into outputs.

PURCHASER: A company that procures product directly from a particular specification.

RECIPE: Based on the formula/formulation wherein the concentration of each ingredient to be present is specified with tolerances.

REQUALIFICATION (QPL): Refer to the PD2102 for the amount of testing required.

REWORK: The act of reprocessing noncomplying, in-process materials or finished parts, through the use of original or equivalent processing, in a manner that assures full compliance of the final part with applicable drawings or specifications.

SUBCONTRACTOR: A company that is hired to perform any function requested by a compounder or manufacturer.

WORK INSTRUCTION: A detailed document outlining tasks within a procedure.

WORK ORDER: A document that provides all processing conditions for a specific part that accompanies the part through the manufacture process which should include at a minimum reference to the part or drawing being manufactured, the quantity of material being produced, material identification, material lot identification, material cure date, and reference to the manufacturing process.

(*Definitions taken from Fundamentals of Rubber Technology)

3. COMPOUNDER REQUIREMENTS

3.1 Compounder

3.1.1 If an external compounder is used, they shall be approved by the manufacturer based upon:

1. Being currently registered to ISO 9001, or

2. If the compounder is not currently registered to ISO 9001 then the manufacturer shall annually approve the compounder by on-site audit.

The compounder's quality record shall be current and on-file at the manufacturer's facility.

3.1.2 A captive compounder shall be qualified as part of the manufacturer's AS9100 registration.

3.2 Control of Compounding Ingredients

3.2.1 The compounder shall have a process for receiving and storing compound ingredients until they are needed. All compounding ingredients shall be stored according to the chemical manufacturer's recommendations and in a work environment per AS9100 to ensure conformity of ingredient requirements.

3.2.2 The compounder shall have a process to identify and maintain initial shelf life of compounding ingredients not to exceed the chemical manufacturer's expiration date.

3.2.3 If an extension of shelf life is required, the compounder shall have a process to verify the extension of all compounding ingredients' shelf lives. Extended shelf life of any ingredient shall be documented along with the verification data per the manufacturer's standard of work.

3.3 Process for Combination of Compounding Ingredients

3.3.1 Formulation Control

3.3.1.1 The compounder shall have a controlled recipe that specifies formulation or the compounder shall use the manufacturer's supplied formulation.

3.3.1.2 The compounder shall not change any ingredient in the formulation without the prior written approval of the manufacturer who may require verification testing up to and including full qualification testing prior to approving a change.

3.3.1.3 If required by the customer contract or purchase order, the manufacturer shall notify the customer of any formulation change.

3.3.2 Mixing

3.3.2.1 The Compounder shall have a documented procedure for the mixing of the compound and shall have evidence that will include but are not limited to:

COMPOUND INGREDIENTS AND PROPORTIONS THEREOF WITHIN ESTABLISHED LIMITS
SEQUENCE OF MIXING COMPOUND INGREDIENTS
TYPE OF MIXING EQUIPMENT (OPEN MILL OR INTERNAL MIXER)

The Compounder shall have evidence that the procedure has been followed and verified.

The supplier shall identify those changes which require customer acceptance in accordance with contractual requirements prior to making any change.

Changes affecting processes, production equipment, tools and programs shall be documented. Documented procedures shall be available to control their implementation.

The results of changes to production processes shall be assessed to confirm that the desired effect has been achieved without adverse effects to product quality.

3.3.3 Testing at the Compounder

3.3.3.1 The compounder shall perform the appropriate tests required by the manufacturer to confirm that the manufacturer's requirements for each batch have been met and the results shall be documented and available upon request by the manufacturer. If required by the manufacturer, the compounder shall also prepare a duplicate series of test samples for the manufacturer.

3.3.4 Rework at the Compounder

3.3.4.1 Material that fails to meet the initial manufacturer's PO requirements, may be reworked up to a maximum of three (3) times and retested after each rework prior to informing the manufacturer. If the reworked compound meets the initial manufacturer's PO requirements for acceptance, then the compound can be shipped with provisional approval pending the manufacturer's review of the compounder's data and the manufacturer's acceptance testing.

3.3.5 Rejected Compound

3.3.5.1 Compound batches that fail the compounder's quality control requirements or those rejected by the manufacturer shall be clearly identified and excluded from use in products requiring AMS7XXX series specifications, user specifications, or print on a Purchase Order (PO) that calls out this document for aerospace applications.

4. MANUFACTURING REQUIREMENTS

4.1 General Requirements

4.1.1 The manufacturer shall have a procedure for identification of the status of all batches of compounded material at the production facility (for example, "Hold for Testing", "Approved", "Rejected").

4.1.2 The manufacturer shall have a documented acceptance procedure for approving compound before it is released to production.

4.1.3 Storage and Identification

4.1.3.1 All compounded material shall bear a unique identification batch number for traceability of test results.

4.1.3.2 Manufacturer shall have a procedure for storage of compounded material and maintaining shelf life control.

4.1.3.2.1 For AMS7XXX series specifications, user specifications, or print on a Purchase Order (PO) that calls out this document for aerospace applications, prior to use, the manufacturer shall run batch acceptance tests on stored compound if it has exceeded its initial shelf life per the applicable specification. If all the values are within the acceptance testing requirements per the applicable specification, then the material is still approved for use. This process shall not exceed a maximum of 1 year for all specifications and if beyond 1 year the manufacturer shall have a process to run a batch/acceptance test prior to extending the shelf life for an additional shelf life cycle per the applicable specification.

4.1.3.3 If testing indicates that the compound is outside the allowable limits, it cannot be reworked and must be clearly identified and segregated and excluded from use in products requiring AMS7XXX series specifications, user specifications, or print on a Purchase Order (PO) that calls out this document for aerospace applications.

4.2 Blending of Compound

4.2.1.1 If the practice is observed for blending batches, the compounder/manufacturer shall have a material approval process, which shall include these minimum requirements. These minimum requirements are applicable to all materials conforming to AMS7XXX series specifications, user specifications, or print on a Purchase Order (PO) that calls out this document for aerospace applications.

4.2.1.1.1 The original batch numbers and weights utilized in the blended batch shall be documented and easily accessible.

4.2.1.1.2 The original batches used shall be the same formulation and the same mix location.

4.2.1.1.3 The original batches used in creating a blended batch shall meet all of the acceptance, lot testing, and shelf life requirements of 4.1.3.2.1 prior to blending.

4.2.1.1.4 The new, blended batch shall be tested for batch and lot acceptance testing prior to shipment.

4.2.1.1.5 The new, blended batch shelf life shall be based on the oldest original batch date of manufacture and shall follow the requirements of 4.1.3.2.1 for shelf life extension.

4.2.1.1.6 Blended material cannot be blended a second time.

4.3 Preforms

4.3.1 Preform type and machine operating conditions shall be defined, documented in the work order and available to the operator. Documentation shall be available to verify that preforms were properly made.

4.3.2 The manufacturer shall have a process for managing the shelf life of preforms. If the shelf life cannot be determined or is not identified, then it shall default to the expiration date of the compound.

4.4 Mold Approval

4.4.1 The manufacturer shall have a mold approval process.

4.4.1.1 Molds should be verified prior to the first production run but in those cases where this is not possible they shall be verified prior to shipment of parts. The manufacturer shall have a procedure for verifying the mold. Any additional purchase document, drawing or specification requirements. (e.g., first article process, etc.) shall be complied with. The mold approval documentation shall be recorded and stored per the manufacturer's record retention policy.

4.4.1.2 The manufacturer shall have a procedure to segregate parts made in a mold that has not been verified and the parts shall remain segregated in production until the mold has been verified.

4.4.1.3 Having nonconforming cavities in a mold does not disqualify a mold from use if the manufacturer has a policy in place to clearly identify nonconforming parts produced in nonconforming cavities to isolate and prevent shipping nonconforming parts to the customer.

4.4.1.4 If a mold is required to be repaired, the manufacturer shall have a process to validate conformance.

4.5 Set up

4.5.1 The press and mold are set up per the manufacturer's process instructions specific to the method of manufacture such as compression, transfer or injection molding. Set up includes temperature, pressure and time. All controls shall be calibrated.

4.5.2 Confirmation that the set up was completed successfully shall be documented per manufacturer's procedures prior to beginning the production run.

4.6 Molding

4.6.1 Mold parts per process instructions specified on the manufacturer's Work Order.

4.6.2 Time and temperature shall be recorded, monitored, and controlled per the manufacturer's Work Order.

4.7 Deflash

4.7.1 Manufacturer shall have process instructions on the Work Order specific to the method of deflash for the parts such as cryogenic, hand, etc.

4.8 Post Cure (If Required)

4.8.1 The Production oven shall be calibrated in accordance with one of the following:

1. The Temperature Uniformity Survey (TUS) procedure outlined in Appendix A, or
2. ASTM D7969 Standard Specification for Gravity Convection and Forced Ventilation Ovens, or
3. ASTM E145 Standard Specification for Gravity-Convection and Forced-Ventilation Ovens, or
4. AMS2750 Pyrometry.

4.8.1.1 Parts shall be placed in a post cure oven and separated as appropriate, by lot/geometry to maintain traceability.

4.8.1.2 The post cure procedure shall require that the parts are placed to allow even distribution of heat during the post cure process.

NOTE: Fluorosilicone and silicone parts shall not be post cured in the same oven as parts molded from other polymers as the off gassing products from fluorosilicone and silicone parts can adversely affect the parts molded from other polymers.

4.8.1.3 Time and temperature shall be controlled per the process posted on the Work Order. Time and temperature shall be monitored and recorded.

5. INSPECTION

The manufacturer shall have a procedure to document all parts being inspected with the inspection criteria per the Part Standard or the Purchase Order. If no visual requirement is specified use ISO 3601-3, Grade S or AS5752 Type II depending on the geometry.

5.1.1 Visual discrepancies outside the allowable limits shall be a cause for rejection.

5.1.2 Exceptions to any customer requirement shall be noted and the customer's written acceptance or acknowledgement of each exception shall be kept and stored in accordance with the manufacturer's record retention policy.

5.1.3 Dimensional verification shall be conducted as specified per Contract, Purchase Order, Part Standard or Print. If not specified then the default requirement sampling plan shall be in accordance with the ANSI/ASQ Z.1.4, C=0, AQL 2.5. Parts that do not meet the dimensional requirements shall be discarded. Both AS568 and ISO 3601-1 establish standard O-ring Sizes and Tolerances.

5.2 Lot testing shall be conducted per applicable material standard or purchase order requirement using calibrated instrumentation and equipment.

5.2.1 If the product meets the lot testing requirements, the test results shall be documented and the parts can proceed to the next step in the manufacturing process (i.e., Packaging and Shipping).

5.2.2 If the product does not meet the testing requirements, the lot shall be either scrapped or quarantined for customer acceptance or rejection. If the customer chooses to accept the parts, this will be acknowledged in writing and the acknowledgement shall be kept and stored in accordance with the manufacturer's record retention policy.

6. STORAGE, PACKAGING AND SHIPPING

6.1 Parts shall be packaged per the Procurement Document, Contractual Agreement, or applicable purchaser's drawing specification such as ARP5316 or ISO 27996. Specific packaging and labeling of parts shall be in compliance with AMS2817 unless specified otherwise by the customer documentation or purchase order.

6.1.1 Until final product is packaged, the manufacturer shall have a process for storing parts properly to prevent damage, contamination, or degradation of product.

6.2 Shipping

6.2.1 Prior to shipping, the shipment and paperwork shall be inspected to confirm that the parts are properly packaged and labeled and the correct certifications and test reports are included as required by the customer.

6.2.2 Each shipment shall include a Certificate of Analysis (CofA) signed by an authorized quality assurance representative of the manufacturer. Each certificate of analysis shall include the applicable information based on the specification, drawing, or purchase order.

6.2.3 The manufacturer shall retain a copy of the CofC and all appropriate job information in accordance with the manufacturer's retention policy.

7. LABORATORY ACCREDITATIONS/CERTIFICATIONS (CAPTIVE LABS ONLY)

7.1 Certification

7.1.1 Laboratories performing qualification or lot/batch acceptance test for Qualified Products Listing (QPL) shall have the laboratory certifications as required per AC7115 and PD2102 documents.

7.1.2 AC7115 captive laboratory certification requirements are found in AC7122-P, AC7122/1, AC7122/2, AC7122/3, and AC7122/4 as applicable to each laboratory.

7.2 ISO 17025 for Elastomers

7.2.1 ISO 17025 accreditation and applicable scope/AC7122 compliant is not a requirement for captive laboratories. If a laboratory is not ISO 17025 accredited with the applicable scope/AC7122 compliant then verification testing per PD2102 shall be performed at an ISO 17025 certified laboratory with the applicable scope for all new qualification submission for Qualified Product Listings (QPL). All acceptance and lot testing shall be performed in the manufacturer's laboratory or an ISO 17025 accredited laboratory.

7.2.2 An ISO 17025 accredited laboratory will not have to complete the additional NADCAP AC7122 audit as long as the scope of accreditation/compliance covers the required testing per the specification.

8. NOTES

8.1 Revision Indicator

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

PREPARED BY AMS COMMITTEE “CE”

APPENDIX A - OVEN TEMPERATURE UNIFORMITY SURVEY (TUS)

A.1 SCOPE

A.1.1 Purpose

This appendix details the technical and quality requirements for performing Temperature Uniformity Survey (TUS) on ovens used for post curing of elastomers. Manufacturers shall have a procedure for calibration of post cure ovens that require at a minimum, a nine point calibration that is NIST traceable, with an established calibration cycle maximum of 1 year. Other industry approved methods such as ASTM E145, D7969 with corresponding documentation are acceptable.

A.2 TECHNICAL REQUIREMENTS

A.2.1 TUS

Shall be performed to measure the temperature uniformity and establish the acceptable work zone and qualified operating temperature range. A TUS shall also be performed after any Oven modification or adjustment that could have altered the temperature uniformity characteristics of the Oven.

A.2.2 Any persons completing the calibrations in house must have been trained to do so and have up to date training records at the time of calibration. The calibrations must be completed using calibrated equipment which is traceable to NIST or other nationally recognized standard. Outside contractors must have appropriate accreditation for oven calibration.

A.2.3 Periodic TUS

Shall be performed annually and more frequently if failures in calibration occur.

A.2.4 Oven Parameters during TUS

During each survey all parameters shall reflect the normal operation of the equipment in production. The survey temperatures shall be the minimum and maximum temperatures of the qualified operating temperature range.

A.2.5 Shall be performed in an empty oven.

A.2.6 Location of TUS Sensors

A.2.6.1 For all production ovens a total of nine thermocouples shall be used with eight TUS sensors located at each of the corners and one located in the center.

A.2.7 TUS Data Collection

A.2.7.1 The Oven shall be held at the test temperature until all test sensors have stabilized. After stabilization, data collection shall continue for a minimum of 30 additional minutes. Once data collection begins, temperature data shall be recorded from all TUS sensors at a frequency of at least one set of all readings at least every 2 minutes for the duration of the survey.

A.2.7.2 All survey thermocouples shall be within the desired temperature range and shall not be changing such that they drift above or below the maximum or minimum temperature tolerance.

A.2.8 TUS Instrumentation

TUS shall be performed using calibrated independent test instrumentation traceable to the National Institute of Standards and Technology (NIST) or equivalent national standard.

A.2.9 Temperature Uniformity Pass/Fail Requirements

A.2.9.1 A survey shall be considered acceptable if the following requirements are met.

a. All readings of all TUS sensors and control or recording sensors are within the temperature tolerance requirement of ±15 °F.

b. TUS is run for the minimum required time.

A.2.10 TUS Failures

If the temperature uniformity is not within the tolerance specified, the cause of the deviation shall be determined and documented. A technical engineering review of parts processed in the oven since the last calibration shall be reviewed for quality impact. The equipment shall not be used for additional processing until the cause has been corrected and the TUS has been performed successfully.

A.3 QUALITY ASSURANCE PROVISIONS

A.3.1 Temperature Uniformity Survey Report

The following items shall be included in the temperature uniformity survey report.

a. Oven identification name or number

b. Survey temperatures

c. Testing company identification and address (if not performed in-house)

d. Name or identification of technician performing survey

e. Survey date

f. Calibration or survey method

g. Survey test instrument identification number

h. Indication of test pass or test fail

i. Time and temperature profile data showing TUS sensors

j. Summary of final plus and minus readings at each test temperature

k. Revision level of certification, if revised

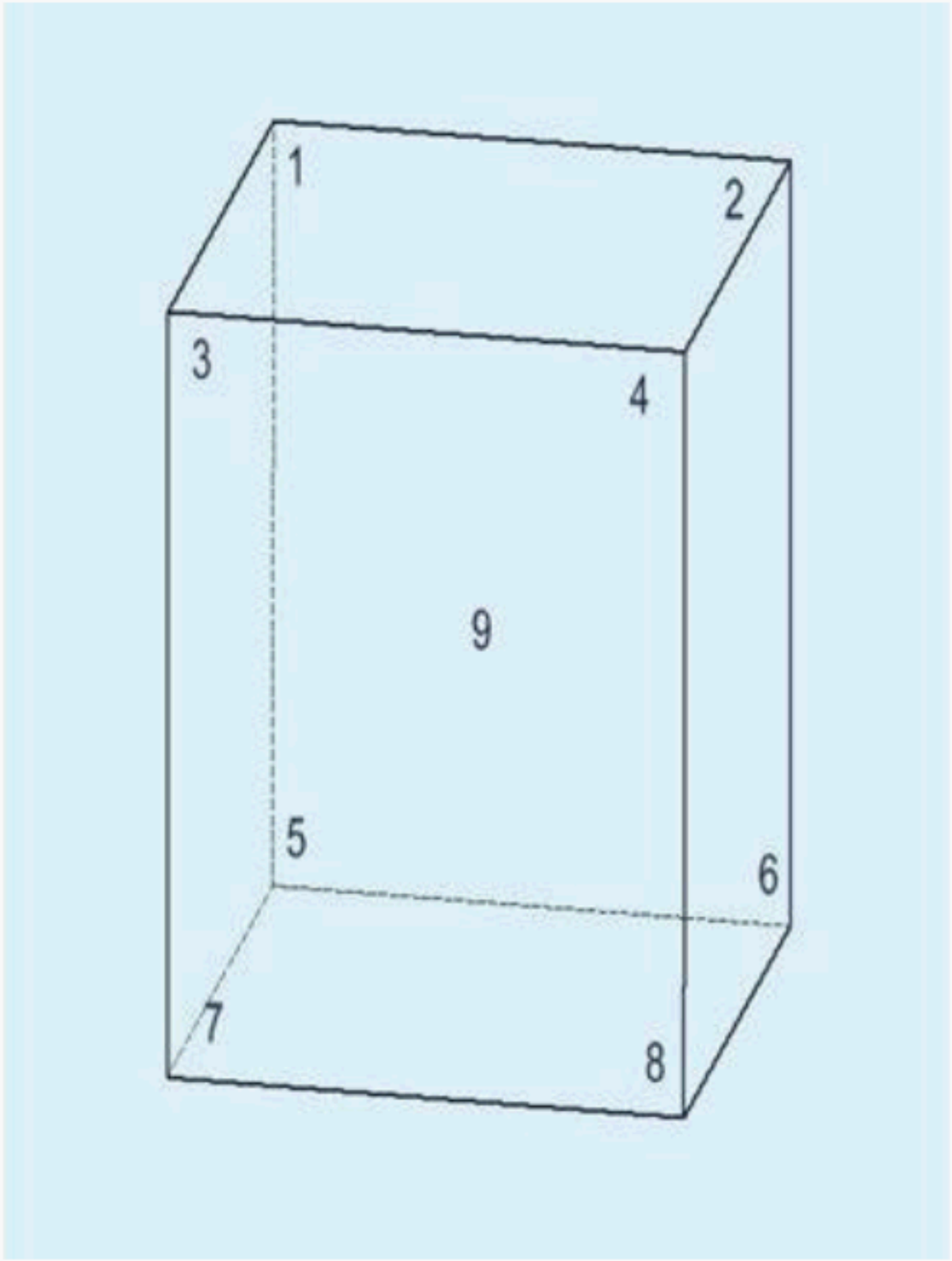

Figure A1 - Location example of thermocouple sensors – each corner and center

AEROSPACE MATERIAL SPECIFICATION	AMS2759™/6	REV. C
	Issued 1987-04 Revised 2018-06 Superseding AMS2759/6B	
(R) Gas Nitriding of Low-Alloy Steel Parts		

RATIONALE

AMS2759/6C results from a Five-Year Review and general revision of AMS2759 and its slash specifications.

NOTICE

- ORDERING INFORMATION: In addition to that listed in AMS2759, the purchaser shall supply the following information to the nitriding processor.
- AMS2769/6C
- Class of nitriding (see 1.3)
- Designated surfaces to be nitrided if selective areas only
- Current heat treat condition
- Last tempering temperature
- Part condition (see 3.4.1.1)
- Case Depth – designate total case or effective case depth (see 3.6.1)
- Case Depth – include minimum or maximum amount to be removed during subsequent machining/grinding
- Case surface hardness
- White layer depth
- White layer removal if it is to be performed
- Core hardness
- Purchaser to supply test part(s), test specimen(s), or specify otherwise

TO PLACE A DOCUMENT ORDER: **Tel: 877-606-7323 (inside USA and Canada)**
Tel: +1 724-776-4970 (outside USA)
Fax: 724-776-0790
Email: CustomerService@sae.org
SAE WEB ADDRESS: **http://www.sae.org**

1. SCOPE

1.1 Purpose

This document specifies the procedure and requirements for gas nitriding low-alloy and tool steels by the use of raw or dissociated ammonia. Additional alloys and steel families (e.g., stainless steels) can also be nitrided using parameters acceptable to the cognizant engineering organization.

1.2 Application

1.2.1 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 per AMS2753, ion nitriding per AMS2759/8, automated gas nitriding controlled by nitriding potential per AMS2759/10, automated gas nitrocarburizing by potential per AMS2759/12 and gas nitrocarburizing per AMS2757, are not included.

1.3 The provisions of this specification revision shall become effective 90 days after publication.

1.4 Classification

Processes covered by this specification are classified as follows:

Class 1: Two-stage nitriding with maximum applied white layer of 0.0005 inch (13 µm).

Class 2: One-stage nitriding with a maximum applied white layer of 0.001 inch (25 µm).

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

2. APPLICABLE DOCUMENTS

In addition to those listed in AMS2759, 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 +1 724-776-4970 (outside USA), www.sae.org.

AMS2418	Plating, Copper
AMS2429	Plating, Bronze, Nitriding Stop-off, 90Cu – 10Sn
AMS2750	Pyrometry
AMS2759	Heat Treatment of Steel Parts, General Requirements
AMS2759/1	Heat Treatment of Carbon and Low-Alloy Steel Parts, Minimum Tensile Strength Below 220 ksi (1517 MPa)
AMS2759/2	Heat Treatment of Low-Alloy Steel Parts, Minimum Tensile Strength 220 ksi (1517 MPa) and Higher
AMS2759/9	Hydrogen Embrittlement Relief (Baking) of Steel Parts
AMS2759/11	Stress Relief of Steel Parts
ARP1820	Chord Method of Evaluating Surface Microstructural 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, www.astm.org.

ASTM E18 Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials

ASTM E140 Standard Hardness Conversion Tables for Metals

ASTM E384 Knoop and Vickers Hardness of Materials

2.3 ANSI Accredited Publications

Copies of these documents are available online at http://webstore.ansi.org/.

ANSI B46.1 Surface Texture

3. TECHNICAL REQUIREMENTS

3.1 Pyrometry

Shall be in accordance with AMS2750.

3.2 Furnace Equipment

Shall be in accordance with AMS2759. Nitriding furnaces shall be Class 3 or better.

3.2.1 Nitriding Environment

Equipment shall be in place 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 in place to measure and maintain the dissociation of the process atmosphere going through the retort or furnace throughout the nitriding cycle.

3.2.2.1 Measurement

Equipment used for measuring the gas dissociation shall be at the exit of the retort or furnace and shall be checked periodically for accuracy. Water absorption techniques (burette) are acceptable, but infrared analysis or in-situ probes are preferred. The accuracy of burettes is defined as clean and readable.

3.3 Auxiliary Equipment

Shall be in accordance with AMS2759.

3.4 Procedure

3.4.1 Hardening

When heat treatment is specified for parts to be nitrided, it shall be performed as specified prior to nitriding. If a heat treatment document is not called out, then it shall be performed in accordance with AMS2759 and to applicable slash specifications.

3.4.1.1 Stress Relieving

Unless otherwise specified parts which have been ground, straightened, machined, or otherwise mechanically worked after heat treatment, shall be stress relieved prior to nitriding in accordance with AMS2759/11.

3.4.2 Cleaning

Parts shall be cleaned such that sufficient and uniform nitriding is able to take place. Care shall be exercised after cleaning to prevent recontamination.

3.4.3 Masking

Areas not required to be nitrided shall be masked. Alternatively, parts may be nitrided on all surfaces and the case ground off those surfaces not required to be nitrided.

3.4.3.1 Prohibition

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

3.4.3.2 Maskant

Shall be fine grained copper plate, not less than 0.001 inch (25 µm) in thickness, applied in accordance with AMS2418 or bronze plate, not less than 0.0005 inch (12.7 µm) in thickness, applied in accordance with AMS2429. Paints, pastes, or mechanical methods may be used if acceptable to the cognizant engineering organization. Additionally, maskants shall be checked visually for evidence of blistering after nitriding but before subsequent stripping and etching.

3.4.4 Visual Inspection

Masked or plated parts shall be visually inspected prior to and after nitriding. Parts exhibiting blistering, peeling, or porosity in the masking after nitriding shall be rejected (see 8.7).

3.4.5 Racking

Rack 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 approximately the same location or locations as the parts they represent.

3.4.6 Nitriding

3.4.6.1 Dissociation

For Class 2 and the first stage of Class 1 (approximately 20% of the total nitriding time) it is recommended that the ammonia gas dissociation set point be within the range of 12 to 35%. For the second stage of Class 1 it is recommended that the ammonia dissociation set point be within the range of 55 to 88%.

3.4.6.2 Nitriding Range

The recommended nitriding set point is 915 to 1050 °F (491 to 566 °C).

3.4.6.3 Nitriding Temperature

The nitriding temperature shall not be higher than 50 °F (28 °C) below the final tempering or aging temperature unless the core hardness can be verified on an actual or representative part (see 8.6). The nitriding temperature shall not exceed the tempering temperature.

3.4.6.4 Nitriding Procedure

The load shall be held at nitriding temperature(s) in an atmosphere in accordance with 3.4.6.1 for sufficient time to produce the specified depth of case.

3.4.7 Cooling

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 purged with inert gas before opening.

3.5 Post Nitriding Operations

3.5.1 Maskant Stripping

When required, stripping of maskant shall be accomplished by use of an appropriate stripping solution that shall not pit or etch the part. If an electrolytic stripping solution is used, parts shall be embrittlement relieved in accordance with 3.5.3.

3.5.2 Removal of White Layer

When white layer is partially or totally restricted, the white layer shall be removed to meet the requirements of paragraph 3.6.3 by lapping, honing, grinding, or etching with maximum stock removal of 0.002 inch (0.05 mm). If etching is used, parts shall be embrittlement relieved after etching in accordance with 3.5.3.

3.5.2.1 Limitation on Metal Removal

When any metal removal is performed, it shall not reduce the effective case depth or surface hardness to below specified requirements.

3.5.3 Embrittlement Relief

Pickled, plated, etched (to remove white layer), or electrolytically cleaned or stripped parts shall be baked to remove hydrogen as specified in AMS2759/9. Where pickling or electrolytic cleaning is performed as an integral part of a plating operation, baking is not required after each process but shall be performed following the final process provided not more than 4 hours elapse between such operations.

3.6 Properties

3.6.1 Case Depth

The depth shall meet the requirements of the engineering drawing. If the engineering drawing and/or purchase order specifies a case depth but does not specifically state that it is to be an effective case depth, total case depth, as defined below, shall apply. In cases where the engineering drawing or purchase order specifies post nitride surface removal (see ordering information), the reported case depth shall include the amount to be removed.

3.6.1.1 Total Case Depth

The total case depth shall be the depth of the continuous etching subsurface zone, determined metallographically from a section of the as-nitrided part or a control specimen prior to machining, or may be determined by a microindentation hardness traverse conducted in accordance with ASTM E384. On those alloys which do not respond by darker etching, the total case depth shall be the depth below the surface at which the hardness is 50 HK or 50 HV higher than the core below the nitride case, as determined by a Knoop or a Vickers hardness traverse respectively. The average of a minimum of three hardness readings taken at a minimum distance of 2X the case depth shall be used to determine the core hardness below the case.

3.6.1.2 Effective Case Depth

When required (see ordering information), the effective case depth shall be as specified. It shall be the as nitrided case depth at which the values specified in Table 1, converted from microindentation hardness, are obtained by performing a hardness traverse in accordance with ASTM E384. In cases where the engineering drawing or purchase order specifies post nitride surface removal (see ordering information), the reported effective case depth shall include the amount to be removed.

Table 1 - Hardness at effective case depth

Specification	Alloy	Hardness HRC or Equivalent (see 8.12)
AMS6470, AMS6471, AMS6472	Nitralloy 135 Mod	50
N/A	Nitralloy EZ	50
AMS6475	Nitralloy N	50
AMS6382, AMS6414, AMS6415	4140, 4340	40
AMS6431, AMS6438	D6AC	50
AMS6485, AMS6487, AMS6488, AMS6408	H11, H13	60
AMS 6496, AMS 6497, AMS 6498	GKP, GKPW, GKPYW	50
AMS 6481	GKHYW	50
Other alloys		As specified

3.6.2 Surface Hardness

Surface hardness shall be as specified in Table 2, determined in accordance with ASTM E18, or ASTM E384 after any material is to be removed. In case of dispute, the surface Rockwell superficial hardness shall govern provided the depth and hardness is appropriate to support the hardness value. If not, then the microindentation hardness at a depth of 0.002 inch (51 µm) from the surface shall take precedence. If material is to be removed as specified in the ordering information, the microindentation hardness values shall be taken at 0.002 inch (51 µm) from the predicted finished surface.

Table 2 - Minimum surface hardness requirements

Alloy	Hardness (HR15N) or Equivalent (see 8.12)
Nitralloy 135 modified	92.5
Nitralloy EZ	92.5
Nitralloy N	92.5
AISI 4140, AISI 4340	85.5
D6AC	85.5
H11, H13	92.1
GKP, GKPW, GKPYW	90.3
GKHYW	90.3
Other alloys	As Specified

3.6.3 White Layer

When white layer is permitted, its maximum applied thickness shall be 0.0005 inch (12.7 µm) for Class 1 and 0.001 inch (25 µm) for Class 2 as determined by metallographic examination in accordance with 4.3.3. White layer may be limited or totally restricted per the engineering drawing in some or all areas regardless of the nitriding class and shall be treated in accordance with 3.5.2.

3.6.4 Core Hardness

The core hardness shall be as specified, determined in accordance with ASTM E18 prior to nitriding. Alternatively, the core hardness may also be measured on a surface of the part or specimen after nitriding where no nitriding has occurred (such as on a surface that has been masked), or at a designated position on a cross section of a specimen or part.

3.6.5 Case Microstructure

The finished case in 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. Nitride networks are permissible at corners and sharp edges provided the following requirements are met: it does not exceed a depth of two grain boundaries from the outside surface and it does not exceed 20% of the case depth. Cracking at the surface or in the nitride layer is not permitted.

4. QUALITY ASSURANCE PROVISIONS

The responsibility for inspection, classification of tests, sampling and testing, approval, records, record retention, and report/certification shall be in accordance with AMS2759 and as follows.

4.1 Responsibility for Inspection

Where parts are required for destructive test, these shall be provided by the purchaser.

4.2 Classification of Tests

4.2.1 Acceptance Tests

Case depth (3.6.1), surface hardness (3.6.2), white layer (3.6.3), core hardness (3.6.4), and case microstructure (3.6.5) are acceptance tests and shall be performed on each lot of parts.

4.2.2 Periodic Tests

Shall be in accordance with AMS2759.

4.2.3 Preproduction Test

In addition to the tests specified in AMS2759, all tests are preproduction tests and shall be performed on specimens prior to initial production nitriding in each furnace. Nitrider shall perform at least one test run on a representative alloy and case depth range covered by this specification prior to production work to prove that the equipment is capable of meeting the requirements of 4.2.1.

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, 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 from 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.

4.3.2 Specimen Description

Specimens shall be of the same alloy as the actual parts having a thickness not less than 0.125 inch (3.2 mm) Alternatively, parts may be used in lieu of a specimen (see 4.3.1). 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. Visual examination for white layer or case microstructure shall be performed at a minimum of 400X magnification. In case of dispute, 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 Surface Hardness

Unless otherwise specified, a minimum of 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.

4.3.5 Core Hardness

When masking has not been used, a part or the process control specimen shall be tested to determine core hardness. When masking has been used, the masked control specimen(s) and 5% of each lot of nitrided parts shall be tested to determine core hardness. When the entire surface of parts has been nitrided, the control specimen shall be used for validation of the part hardness.

4.4 Reports

In addition to that required in AMS2759 the report shall show the results of tests for core hardness, microstructure, white layer, as-nitrided case depth and surface hardness, and state that nitriding conformed to the other specified requirements. When post-nitride stock removal is specified, the microindentation hardness traverse data (see 3.6.1.2) shall be reported along with a statement that the reported effective case depth includes the amount to be removed.

4.4.1 If finish machining is performed by the nitride processor the report shall show maximum and minimum stock removal, effective case depth, depth of white layer if present, and surface hardness after machining.

4.5 Resampling and Retesting

If any specimen used in the above tests fails to meet the specified requirements, disposition of the heat treated parts may be based on the results of testing a production part. Except as permitted in 4.5.1, failure of any retest specimen or part to meet the specified requirements shall be cause for rejection of the parts represented. Results of all tests shall be reported.

4.5.1 Parts that do not meet the minimum case depth or minimum hardness limits after processing as specified herein may be reprocessed by re-nitriding as necessary to meet specified requirements except that parts may be re-nitrided only once. Test specimens for such reprocessing shall be the remaining portions of specimens or parts used to determine the original nonconformance.

5. PREPARATION FOR DELIVERY

Shall be in accordance with AMS2759.

6. ACKNOWLEDGMENT

The heat treating processor shall mention this specification number, and its revision letter, in all quotations and when acknowledging purchase orders.

7. REJECTIONS

Parts not processed in accordance with this specification or to 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 AMS2759 and as follows:

8.1 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 for information only.

8.2 Other steels (e.g., 17-4PH, 17-7PH, 300 and 400 series corrosion-resistant steels) can also be nitrided using proprietary additives to the atmosphere, or other processes.

8.3 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.

8.4 Case depths up to 0.035 inch (0.88 mm) can be obtained by nitriding, depending on the base material, the nitriding process, and the time (12 to 100 hours) at the nitriding temperature.

8.5 The capability of a surface to accept a nitride case is influenced by its surface finish. Burnished or polished surfaces will not nitride satisfactorily. Abrasive blasting (200 to 1200-grit size) of all surfaces is recommended prior to nitriding, surface finish requirements in accordance with 4.3.2 still apply.

8.6 Nitriding may lower the core hardness if performed too close to the tempering temperature used during hardening of the material depending on the alloy. It is recommended that nitriding be performed at least 50 °F (28 °C) below the tempering temperature.

8.7 Blistering and peeling of the plating are typically caused by improper cleaning of parts prior to plating. Porosity is typically caused by improper plating practice.

8.8 The case depth is typically measured at the flank (pitch diameter) of the gear or spline (as measured at approximate midpoint between the root and tip of the tooth).

8.9 Sharp corners or edges on parts should be avoided, wherever possible. Growth of surfaces combines at the corners to form brittle projections which are likely to chip.

8.10 Internal cylinder surfaces with depth to diameter ratios greater than two can produce a case that is deeper at one end than the other. Similarly, gear and spline teeth normally exhibit a case depth that is greater at the tips of the teeth than at the tooth roots. For such surfaces, actual parts should be supplied and locations for determinations noted.

8.11 Hardness conversion tables for metals are presented in ASTM E140.

8.12 Terms not specifically defined herein are clarified in ARP1917.

8.13 Reference Photomicrographs

Case and core microstructures are illustrated in photographs 523, 524, 528, 531, 532, and 534 of the ASM Metals Handbook, Volume 7, 8th Edition, or Figures 81, 82, 86, 87, 88, 89 and 91 in the Case Hardening Steel section of Volume 9, 9th Edition.

8.14 Revision Indicator

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

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