Process Specification for Ion Nitriding

Engineering Directorate

Structural Engineering Division

April 2020

National Aeronautics and
Space Administration

Lyndon B. Johnson Space Center
Houston, Texas

Process Specification for Ion Nitriding

Prepared by:	Signature on File John Figert Materials and Processes Branch/ES4	4/29/2020 Date
Reviewed by:	Signature on File Daila Gonzalez Materials and Processes Branch/ES4	4/29/2020 Date
Approved by:	Signature on File Brian Mayeaux Materials and Processes Branch/ES4	4/29/2020 Date

REVISIONS		
VERSION	CHANGES	DATE
Baseline	Original version	9/07/05
Revision A	Allowance for other methods for passivation removal in section 2.0. A trace of white layer is acceptable in section 3.0.	6/28/06
Revision B	Changed application temperature in section 2.0. Changed “coating” to “case” in section 4.1. Added statement about avoiding sharp corners in the design in section 7.0	09/15/11
Revision C	Recommended that a radius of 0.060 inch or greater be used for corners and edges in section 3.0.	8/13/14
Revision D	Updated Signature Sheet. Applicable to wrought steel alloys. Added some information about surface requirements prior to nitriding. Updated references. Added bullets in process verification. Added definitions.	4/29/20

1.0 SCOPE

This process specification establishes the engineering requirements for producing a hard, wear-resistant ion nitrided surface on wrought steel parts.

2.0 APPLICABILITY

Ion nitriding (also known as plasma ion nitriding) is a surface hardening heat treatment that uses diffused nitrogen to form various nitrogen compounds. The nitrogen ions are diffused into a part (or sample) in a vacuum through the use of high voltage electrical energy. The application or set temperature is usually 750- 1110 F which produces a typical case depth of 3-30 mils. The set temperature selected is a minimum of 50 °F (28 °C) below the final tempering or aging heat treatment temperature.

Ion nitriding is generally performed on alloy steels, nitriding steels, and/or stainless steels for extended wear life and a reduced fatigue failure rate. This PRC applies to wrought steel alloys, it does not apply to non-ferrous alloys, cast items, nor additively manufactured items. Typical components processed include: gears, tools, shafts, die, molds, and bearing rollers. Components are typically ion nitrided in the final machined dimensions; there is excellent dimensional stability and minimal distortion with ion nitriding. One of the major benefits of ion nitriding over gas nitriding is that the brittle, white layer that forms with gas nitriding can readily be avoided or minimized with ion nitriding. No more than 0.0005 inches (class 1 per AMS 2759/8) of white layer is acceptable in as- deposited condition in this PRC.

All stainless steel parts shall be passivated before ion nitriding whenever possible to remove embedded tooling carbides and free iron from the part’s surfaces. However, since the passive chromium oxide layer (from passivation) prevents the nitride layer from forming, the passive chromium oxide layer must be removed just prior to ion nitriding.

Sharp corners are a common design problem with nitriding, these sharp corners often chip and crack. For most applications, it is recommended that edges and corners have a radius of 0.060 inch or greater.

This specification shall be applicable whenever the ion nitride process is invoked per section 4.0, “Usage”.

Unless otherwise specified on the engineering drawing, all parts shall be heat treated to the required temper and final machined prior to ion nitriding.

Parts which have been ground, straightened, or otherwise mechanically-worked after hardening, shall be stress relieved before ion nitriding. Abusively machined surfaces shall not be considered acceptable for ion nitriding.

For all parts which have been electrical discharge machined (EDM), the oxidized zone* must be removed prior to ion nitriding. For martensitic steels and martensitic stainless steels, removal of both the recast layer and untemper martensite layer (heat affected zone) is also required.

* The oxidized zone contains gaps, voids and laps between highly oxidized particles that are partially fused to the remelt zone layer. This porous, oxidized zone can trap liquids during subsequent processing if it is not removed. In addition, this oxidized layer contains brass particles from the EDM wire.

3.0 USAGE

Coupons that are dimensionally representative of the part(s) shall be batch processed with the parts being ion nitrided. These coupons shall be from the same material lot and heat treat as the parts. These coupons shall be sectioned and metallographically examined to determine case depth in the critical locations.

An example of a drawing note for either a 15-5 PH or 17-4 PH gear would be:

ION NITRIDING SHALL BE PERFORMED IN ACCORDANCE WITH PRC 2004. A CASE DEPTH OF 0.004 – 0.008 INCHES AT THE GEAR'S PITCH DIAMETER IS REQUIRED.

An example of a drawing note for either a 15-5 PH or 17-4 PH on a shaft diameter would be:

ION NITRIDING SHALL BE PERFORMED IN ACCORDANCE WITH PRC 2004. AN OVERALL CASE DEPTH OF 0.004 – 0.008 INCHES IS REQUIRED.

3.1 SPECIAL NOTATIONS RELATED TO CASE DEPTH DETERMINATION

Verification of ion nitriding is generally performed by determining the case depth and measuring the surface superficial hardness. The case depth is the distance from the surface of the representative test coupon including oxide scale to the depth where the hardness is 50 HRC (by conversion from a microhardness number). For some materials like 15-5 PH, the case depth can be measured visually without microhardness testing due to the sharp transition between base material hardness and the ion nitrided case hardness (almost no diffusion zone). As-polished metallographical samples utilizing colloidal silica as the last process sequence reveals a line where the transition occurs between base metal and case.

Sample coupons shall be used to verify the case depth of the ion nitriding heat treat rather than the actual part. Samples must be representative of the parts being ion nitrided. Sometimes it may be easiest to use a section out of an extra or scrap part because geometry is a critical component in this process. Sample parts shall be made from the same raw material lot and same heat treatment lot; the sample must be processed in an identical manner as the production parts. There must be at least two samples for each production run. Critical or high wear locations (or surface) can be identified and checked on the samples. An example drawing note would be:

LOCATIONS A AND B ARE CRITICAL LOCATIONS WHERE CASE DEPTH NEEDS TO BE VERIFIED ON THE METALLOGRAPHICAL SAMPLES.

4.0 REFERENCES

All documents listed are assumed to be the current revision unless a specific revision is listed.

SAE AMS 2759/8	Society of Automotive Engineers, Inc., Aerospace Material Specification: Ion Nitriding
SAE AMS 2759	Society of Automotive Engineers, Inc., Aerospace Material Specification: Heat Treatment of Steel Parts: General Requirements
ASTM E18	American Society for Testing and Materials Specification, Standard Test Methods for Rockwell Hardness of Metallic Materials
ASTM E384	American Society for Testing and Materials Specification, Standard Test Method for Microindentation Hardness of Materials
ASTM A370	American Society for Testing and Materials Specification, *Standard Test*

Methods and Definitions for
Mechanical Testing of Steel Products

The following references were used in developing this process specification:

SOP-007.1	Preparation and Revision of Process Specifications
JSC 8500.4	Engineering Drawing System Requirements

5.0 MATERIALS REQUIREMENTS

This process is specific for alloy steels, nitriding steels, and/or stainless steels only. This PRC applies to wrought steels, it does not apply to non-ferrous alloys, cast items, nor additively manufactured items.

6.0 PROCESS REQUIREMENTS

All heat treatable steel shall be hardened or tempered prior to ion nitriding. All parts shall be final machined, unless otherwise specified on the engineering drawing. Sharp corners or edges should be avoided on parts to be nitride due to the susceptibility to chipping. When sharp corners or edges are unavoidable, brittleness may be reduced by nitriding one side only especially if the surface is not a wearing surface.

Tools, equipment, and technical requirements for the equipment shall be as-specified in SAE AMS 2759/8 and SAE AMS 2759. Safety precautions and warning notes shall be as-specified in SAE AMS 2759/8 and SAE AMS 2759.

Microhardness shall be done in accordance with ASTM E384. Conversions from one hardness scale to another shall be done in accordance with ASTM A370. Please note that 15N hardness is not acceptable method to determine case depth in this PRC.

7.0 PROCESS QUALIFICATION

All new ion nitride vendors shall on an initial qualification return the ion nitrided samples to the Materials and Processes Branch (ES4) for case depth verification at the critical or drawing designated area. Two samples per batch are required. Surface hardness testing in accordance with SAE AMS 2759/8 and ASTM E18 shall also be performed by Materials and Process Branch (ES4) for qualification.

Two type of qualifications will be provided for ion nitride vendors:

Group I: Complex shapes—including all gear teeth—that are very difficult to ion nitride with a uniform case depth.

Group II: Normal shapes—flat surface and diameters—that are easy to ion nitride with a uniform case depth.

8.0 PROCESS VERIFICATION

Verification of furnace temperatures shall be accomplished by recording the furnace temperatures on strip charts or other suitable hard copy recordings. Furnace charts for heat treatment shall be maintained with the hardware's work order router package (or equivalent documentation).

Other information that shall be included:

- 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
- Furnace 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.

For a qualified/certified vendor, the ion nitrided samples for subsequent production runs can be returned to ES4 for metallographic analysis or a metallurgical report on the ion nitrided samples must be supplied that verifies the case depth at the critical locations indicated on the drawing. Please note that the metallographic laboratory performing the evaluation must be approved by NASA- JSC/ES4 (Materials and Processes Branch) prior to the job being contracted.
The ion nitride vendor shall perform the required surface hardness testing in accordance with SAE AMS 2759/8 and ASTM E18 and report the data with the other required verification paperwork.

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

9.0 TRAINING AND CERTIFICATION OF PERSONNEL

All ion nitrided parts used on flight hardware shall be ion nitrided by company qualified or certified operators.

10.0 DEFINITIONS

Low Alloy Steels: Low-alloy steels constitute a category of ferrous materials that exhibit mechanical properties superior to plain carbon steels as the result of additions of alloying elements such as nickel, chromium, and molybdenum. Total alloy content can range from 2.07% up to levels just below that of stainless steels, which contain a minimum of 10.5% **Cr**. For many low-alloy steels, the primary function of the alloying elements is to increase hardenability in order to optimize mechanical properties and toughness after heat treatment.

Material Lot: A single batch (bar, forging, extrusion, etc.) of material that is produced by the vendor and is documented by a certificate of compliance.

Passivation: A non-electrolytic finishing process that makes stainless steel more rust-resistant. The passivation process typically uses nitric or citric acid to remove free iron from the surface. This results an inert, thin protective oxide layer that is less likely to chemically react with air and cause corrosion.