AEROSPACE MATERIAL SPECIFICATION

AMS03-28	PART 3
Issued 2015-06	

Physical Vapor Deposition of Metals:
Physical Vapor Deposition of Titanium Nitride for Surface Protection

RATIONALE

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FOREWORD

REVISION NOTE

This Defence Standard was raised to Issue 4 to update its content and incorporate the latest MoD/DStan policy in place at the time.

HISTORICAL RECORD

This standard supersedes the following:

Def Stan 03-28 (Part 3) Issue 1 dated 8 February 1991

Def Stan 03-28 (Part 3) Issue 2 dated 19 December 1997

Def Stan 03-28 (Part 3) Issue 3 dated 4 February 2005

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INTRODUCTION

Titanium nitride deposited by physical vapor deposition techniques is applied to improve surface properties, in particular wear resistance, surface hardness and to reduce the tendency for galling. The coating is resistant to corrosion under ambient conditions and to oxidation below 450 °C. It is particularly suitable for the protection of items which need to be handled and is compatible with many energetic materials. Surfaces containing oxide, rust, black oxide scale, grinding burns or other machining marks, cracks or burrs are not suitable for coating. Processing conditions used for the coating of tempered steels must be carefully controlled so that their metallurgical properties and structure are not impaired by substrate heating. The presence of a thin titanium metal underlayer improves the adhesion and corrosion resistance of titanium nitride coatings. However, this underlayer may be omitted if its inclusion compromises the performance of the substrate/titanium nitride system as a whole.

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1. SCOPE

This SAE Standard specifies the requirements for coatings of titanium nitride on non-corrosion resisting and corrosion resisting steels, cast irons and other suitable materials. Standard engineering items such as coated tools, drills and dies would not normally be included.

2. WARNING

This section which appeared in DEF STAN 03-28 Part 3 Issue 4 has been deliberately deleted.

3. NORMATIVE REFERENCES

3.1 The publications shown below are referred to in the text of this standard. Publications are grouped and listed in alpha-numeric order.

BS ISO 6344-1	Coated Abrasives – Grain Size Analysis. Grain Size Distribution Test
Def Stan 03-2	Cleaning and Preparation of Metal Surfaces
AMS03-2	Cleaning and Preparation of Metal Surfaces
Def Stan 03-4	The Pretreatment and Protection of Steel of Specified Maximum Tensile Strength Exceeding 1450 N/mm^2
AMS03-4	The Pretreatment and Protection of Steel of Specified Maximum Tensile Strength Exceeding 1450 N/mm^2
Def Stan 03-21	Mechanical Methods for the Inducement of Compressive Surface Residual Stresses

3.2 This section which appeared in DEF STAN 03-28 Part 3 Issue 4 has been deliberately deleted.

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4. DEFINITIONS

4.1 For the purposes of this Part of the Standard the following definitions apply:

4.2 DESIGN AUTHORITY

The approved firm, establishment or branch responsible for the detailed design of material to approved specifications and authorized to sign a certificate of design or to certify sealed drawings.

4.3 PROCESS CONTROL SCHEDULE

The document which specifies/defines:

a. The sequence of manufacturing operations and processes.

b. The control parameters and their tolerances for each individual process within the total sequence.

4.4 SIGNIFICANT SURFACE

That area of the item, covered or to be covered by the coating, and for which the coating is essential for serviceability and/or appearance.

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5. INFORMATION TO BE SUPPLIED TO THE PROCESSOR

The following information shall be given on the drawing, contract or order:

a. The number of this AMS Material Specification or its superseded Defence Standard.

b. The specification and metallurgical condition of the basis metal;

c. The significant surface (see 4.4), including the coating of holes and recesses (see Section 12 (a)).

d. The minimum thickness of titanium nitride coating on the finished item (see Section 10); the maximum thickness may also be specified.

e. The requirements for coating adhesion (see Section 16).

6. PROCESS CONTROL

6.1 A Process Control Schedule suitable of achieving the requirements of this Standard shall be prepared by the processing contractor(s) prior to the commencement of production.

6.2 Details of the coating process, including all preparatory and post coating treatments, processing, chemical composition of the source material and coating (where appropriate), thickness of the coating required, processing temperature range and gas pressure during coating, significant surfaces, tests and all other processes and treatments shall be included in the Process Control Schedule.

6.3 In accordance with the general requirements of AMS03-2/Def Stan 03-2 and AMS03-4/Def Stan 03-4 as appropriate, all stages in the complete Schedule shall follow each other without delay.

7. PREPARATION FOR COATING

All items to be coated shall be treated in accordance with the General Requirements Section of AMS03-2/Def Stan 03-2 or AMS03-4/Def Stan 03-4, as appropriate.

8. PHYSICAL PREPARATION

8.1 Cold Working

All cold working processes (e.g., forming, straightening, grinding and machining) where required, shall be completed before any preparation, stress relieving and coating unless otherwise specified in the drawing, contract or order.

8.2 Heat Treatment

8.2.1 Steel items shall be stress relieved prior to the deposition of titanium nitride in accordance with AMS03-2/Def Stan 03-2 or AMS03-4/Def Stan 03-4, as appropriate.

8.2.2 All heat treatment operations, including those designed to reduce the susceptibility to hydrogen embrittlement as a consequence of cleaning operations, shall be completed prior to the deposition of titanium nitride in accordance with the requirements of this Part of the Standard.

8.3 Surface Condition

8.3.1 Surfaces to be coated shall be smooth and free from oxides, tool marks, pitting, intergranular attack or other defects (see 8.4.1).

8.3.2 Due allowance shall be made for surface preparation (especially abrasive cleaning) and the application of a uniform coating thickness (see Section 10 and Table 1), in order to obtain the specified surface finish and dimensional tolerance quoted in the Process Control Schedule (see Section 6).

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8.3.3 Any shot peening of the basis metal shall be carried out before the application of the coating and shall be performed in accordance with Def Stan 03-21.

8.3.4 Immediately prior to deposition, surfaces to be coated shall be conditioned, for example by sputter cleaning (see 9.2).

8.4 Cleaning

8.4.1 All surfaces shall be free from soils, such as soap, drawing compounds, oil, grease, machining fluid, polishing compounds, etc., discoloration, oxide, scale or other foreign matter. All items shall be degreased (see AMS03-2/Def Stan 03-2) prior to the commencement of a cleaning sequence which shall be in accordance with AMS03-2/Def Stan 03-2 or AMS03-4/Def Stan 03-4, as appropriate, to produce a chemically clean surface. Drying by means of chlorinated solvents is not permitted.

8.4.2 Abrasives used on any metal or alloy shall not have been used on any dissimilar metals or alloys. Separate abrasive cleaning media shall be used for different alloy families, e.g., iron and its alloys, aluminum and its alloys, etc. After abrasive cleaning, residues shall be removed from the surfaces of items prior to subsequent processing.

8.4.3 Areas Required to be Kept Free of Coating

Areas on which physical vapor deposition of titanium nitride is not required shall be masked prior to the application of the coating. Corrosion-resisting steel masking, foil or plugs shall be used to cover areas where the coating is not required. Organic masking materials shall not be used.

8.4.4 Contamination of cleaned items (e.g., handling with bare skin) shall be avoided (e.g., by the use of clean and dry lint free cotton gloves or any other suitable gloves).

9. PROCESS REQUIREMENTS

9.1 Equipment

9.1.1 The equipment used shall consist of a coating chamber with associated pumps, controls and power supplies containing:

a. Means of producing energetic bombardment of the substrate prior to coating and at a reduced intensity, throughout deposition of the growing film (see 9.2.3).

b. Means of producing a coating flux of titanium, such as an evaporative or sputtering source, or an arc cathode. According to the technique used it may be necessary to include a shutter, which may be interposed between the titanium source and the items to be coated except during coating (see 9.3.6).

NOTE: A shutter may be necessary to reduce radiant substrate heating from the titanium source.

c. Means of holding and manipulating the items to ensure the specified coating uniformity.

d. Means of monitoring the substrate temperature and/or controlling it within specified limits.

9.1.2 The equipment shall be capable of producing a vacuum of 7×10^{-3} Pa (5×10^{-5} torr), or better, in the coating chamber.

NOTE: Where water-cooled surfaces are present in the chamber, including chamber walls, then a means of heating the circulating water shall be used to ensure that the chamber surfaces are warm enough to prevent condensation when open to the atmosphere.

9.1.3 Ultra high purity argon and nitrogen (at least 99.998% pure) shall be used during substrate conditioning and coating as required. The titanium metal used shall contain not less than 99.50% titanium by weight.

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9.2 Substrate Conditioning

9.2.1 After cleaning (and drying as necessary (see 8.4)) items shall be transferred as soon as practicable into the coating chamber. Where this is not possible, items may be kept in a controlled atmosphere provided that there is not deterioration in the substrate condition. Items shall only be handled by operators wearing suitable gloves (see 8.4.4).

9.2.2 The coating chamber shall then be pumped to the vacuum specified in the Process Control Schedule (see Section 6).

9.2.3 Items will then be subjected to substrate conditioning, to remove adsorbed and chemically bound contaminants and thus ensure adequate coating adhesion. Substrate conditioning may be achieved by intimate contact with a glow discharge plasma (sputter cleaning), bombardment with energetic ions, radicals or atoms or by reactive plasma cleaning or by other suitable methods. Certain conditioning treatments may require the coating chamber to be back filled with argon or other gas of sufficiently high purity (see 9.1.3) and/or manipulation of the items to ensure complete exposure.

9.2.4 When the specified substrate conditioning has been completed, there shall be a smooth, uninterrupted transition to the coating deposition stage of the process (see 9.3.5).

NOTE: The chamber shall not be vented to the atmosphere after substrate conditioning prior to coating. Should this occur, the substrate conditioning stage shall be repeated. In some circumstances, it may be necessary to unload and re-clean the items before restarting the process.

9.3 Titanium Nitride Coating

9.3.1 The equipment, process and materials used to produce physical vapor deposited titanium nitride shall be capable of producing a uniform coating in accordance with the detailed requirements of this Part of the Standard.

9.3.2 The equipment and process shall be such that any temperature rise in the items will have no adverse effects on the properties of the basis metal or the coating, or on the adhesion characteristics between the basis metal and the coating.

9.3.3 The equipment and process shall be capable of covering completely all significant surfaces (see 4.4), including roots of threads, recesses, sharp corners and other areas specified in the drawing, contract or order. Suitable rotating and revolving jigs shall be used to ensure that all significant surfaces are cleaned and coated evenly.

9.3.4 Only items of a similar basis metal, e.g., corrosion resisting steels, shall be processed together.

9.3.5 The coating process shall be carried out in the evacuated coating chamber in which the items had just been surface conditioned (see 9.2.4). At the conclusion of the substrate conditioning, a smooth transition shall be effected by introducing the titanium coating flux (for example by opening the shutter, see 9.1.1(b)) and adjusting the process conditions.

9.3.5.1 It is essential that a sufficiently energetic ion/radical/atom bombardment of the growing film occurs throughout the deposition process according to the technique used, for example by maintaining the glow discharge. Where necessary the items shall be rotated and/or revolved to ensure uniform coating deposition and if specified in the Process Control Schedule (see Section 6), an initial thin layer of titanium metal shall be deposited.

9.3.5.2 Subsequent deposition of titanium nitride shall then be effected usually by the introduction of a suitable partial pressure of nitrogen in the coating chamber (see 9.1.3), until the specified coating thickness has been achieved (see Section 6).

9.3.6 The titanium source shutter (if fitted) should then be closed, the titanium sources switched off and the coated items and sources allowed to cool before air is admitted to the coating chamber. The items shall then be removed from the coating chamber (see 10.3). Masking material (see 8.4.3) shall then be carefully removed from the items prior to examination (see Sections 10 to 16).

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9.3.7 The substrate conditioning techniques and the coating conditions will vary with the nature, size and shape of the items to be coated. These variable factors shall be determined experimentally for each type of item, to ensure that the requirements for thickness and adhesion of the coating are met. Once established, the variable factors for each type of item shall be defined in the Process Control Schedule.

10. COATING REQUIREMENTS

10.1 The coating shall be smooth, continuous, uniform, adherent, fine-grained and free from stains, burns, blisters, pits, nodules, cracks, uncoated areas or other defects, with minimal porosity and edge build up. The titanium nitride coating shall show no indication of contamination or improper operation of equipment, such as darkened areas or other defects. The coating shall completely cover all significant surfaces (see 4.4), which may include roots of threads, recesses, holes and sharp corners, and shall conform to the thickness requirements detailed in Table 1. Masked areas shall be clean, free from titanium nitride and free from surface or other defects. The formation of nitrided substrate (white layer) underneath the coating shall be avoided.

NOTE: It may be possible to avoid white layer formation on certain substrates by the deposition of a titanium metal underlayer.

Table 1 - Coating thickness requirements

Application	Thickness Range (µm)
Coatings for general surface protection	2 - 6
Coatings for protection under more severe conditions	>6

NOTE: There may be an increased risk of spalling of coatings if the thickness exceeds approximately 10 µm.

10.2 Allowances shall be made in the manufacture of threaded items for dimensional tolerance to permit the necessary coating thickness to be applied.

10.3 Coated items should be removed from the equipment and handled in such a way as to avoid contamination (e.g., skin contact) by wearing clean and dry lint-free cotton gloves, or any other suitable gloves and examined in accordance with Sections 10 to 16 of this Part of the Standard.

11. PROCESS CONTROL

Inspection shall verify that the mandatory requirements of the sections included in this Part of the Standard have been carried out in accordance with the agreed Process Control Schedule (see Section 6).

12. INSPECTION

All coated items shall be visually inspected by appropriate methods for appearance. Inspection shall be carried out to ensure that:

a. All coated items conform to the mandatory requirements of Section 10. The coating shall completely cover all significant surfaces and shall extend into holes and recesses to a distance specified in the drawing, contract or order.

b. All masking material, if applied prior to the application of the coating, is removed. Masked areas shall be clean and free from any defect and titanium nitride coating. The line of demarcation between the coated and uncoated area shall be clear cut. Lifting of the coating at the edges shall not be acceptable.

c. The adhesion of the coating is satisfactory and meets the requirements of the drawing, contract or order (see Sections 5, 6, 16, and 13.2).

d. The thickness of the coating conforms to the requirements of Table 1 (see Sections 6, 10, and 15) and the finished items are in accordance with the drawing, contractor order (see Section 5).

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