AIPS
Airbus Process Specification

Physical Vapor Deposition of titanium nitride for coating of nickel alloys, stainless steels and titanium alloys

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Contents

1 Scope

This Airbus Process Specification defines the requirements for the titanium nitride protection of nickel alloys, stainless steels and titanium alloys.

The purpose of this specification is to give Design and Quality requirements to manufacturers.
Although the essential requirements of a process will be described in detail, the specification does not give complete in-house operation instructions; these shall be given in the manufacturers supporting work instructions.

This specification shall not be used as an inspection document unless parts or assemblies have been manufactured according to this specification.

It shall be applied when mentioned in the relevant standard material specification or drawing.

1.1 Scope

By adding titanium nitride to the surface of nickel alloys, titanium alloys or stainless steels, the process provides resistance to sliding wear and to abrasion without affecting the mechanical properties (static and fatigue) of the substrate.

1.2 Classifications

Not applicable.

2 Normative references

This Airbus specification incorporates by dated or undated reference provisions from other publications. All normative references cited at the appropriate places in the text are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this Airbus specification only when incorporated in it by amendment of revision. For undated references, the latest issue of the publication referred to shall be applied.

AIMS 03-10-008	High strength titanium alloy
AITM 2-0059	Test method for thickness measurement by rotative abrasion
ASNA 3360	NC19FeNb - (Inconel 718 aged condition)
ASNA 3311	E-ZI CNDAT 12-10 - (Marval X12 H)
ASNA 3360	NC19FeNb - (Inconel 718 solution heat treatment condition)
ASNA 3307	TA6V α,β (annealed condition) bar
ASNA 3106	Z6 CNT18/10
ASNA 3304	TA6V α,β (annealed condition) plate
EN9103	Quality management systems - Variation management of key characteristics
ISO 6507	Metallic materials – hardness test – Vickers test
ISO 6508	Metallic materials – hardness test – Rockwell test

3 Applicability, limitations and definitions

3.1 General description of process

Titanium nitride is a thin layer applied by physical vapour deposition on nickel alloys, titanium alloys or stainless steels for its sliding wear properties.

3.2 Definitions

Titanium nitride is also written as TiN.

3.3 Applicability

This Airbus specification is applicable when invoked by the drawing directly or through another document for the purpose given in the scope. When processing to AIPS 02-03-003 is required, it shall be invoked on the drawing by the words “Titanium nitride protection of nickel alloys, stainless steels and titanium alloys” according to AIPS 02-03-003.

This specification defines the requirements for the titanium nitride protection of nickel alloys, stainless steels and titanium alloys.

3.4 Limitations

Titanium nitride is resistant to sliding wear under a light load. Therefore, as soon as the maximum acceptable pressure for the material / titanium nitride coating pair is exceeded, the layer may begin to flake and this produces a third abrasive body.

This coating does not provide corrosion protection.

A number of different processes to deposit the coating are commercially available, this AIPS only allows to use the Physical Vapour Deposition process, in order to ensure properties of the substrate. Depending on the alloy, maximum temperature for deposition shall not change the static properties of the base alloy (see relevant Technical Design Directive).

4 Requirements

4.1 Technical requirements

The required surface textures (roughness and ripple) must be obtained by machining and/or grinding before applying the deposit. Indeed the thinness of the coating does not allow a grinding operation to be performed after applying the deposit. However, the theoretical thickness of the titanium nitride on the dimensions of the parts to be coated must be taken into account (cylindricity, dimensional tolerances on diameter, etc.).

The colour of the deposit varies according to its composition (see table 1).

Table 1 – Colour criteria as a function of the stoechiometry

Colour	Silver	Gold	Reddish
Stoechiometry	too rich in titanium	Ti/N stoechiometry	too rich in nitrogen
Criteria	**Unacceptable**	**Acceptable**	**Unacceptable**

4.2 Engineering requirements

The final Titanium nitride coating shall meet the following requirements:

- An homogeneous visual aspect
- A coating thickness
- An adhesion of the coating to the base material

4.2.1 Visual inspection

The titanium nitride coating shall completely cover the surface to be plated. The coating must have a golden and bright appearance and be free of all visible defects (burns due to arc damage, powdery appearance, etc.) with a naked eye observation.

4.2.2 Coat thickness

The thickness can be measured by:

– micro-section,

– β-ray backscatter or X-ray fluorescent method,

– by rotative abrasion in accordance with AITM 2-0059.

The thickness of the titanium nitride shall be in the allowed range of:
- $1.5 < \varepsilon < 5$ µm for nickel alloys and stainless steels
- 4 µm ± 1 µm for titanium alloys

The coat must be continuous, smooth and of uniform thickness.

4.2.3 Coat adhesion

When tested with a Vickers hardness test according to ISO 6507 or to a Rockwell HRC hardness test according to ISO 6508 with a load of 150 kg, the adhesion must be grade 0. Grades 1 and 2 are accepted provided that a reinforced inspection is conducted in series-production follow-up (defined in appendix D).

4.3 Quality requirements

4.3.1 General requirements

When processing according to this AIPS all installations shall be continuously operated within the parameters defined in the Process Instruction.

All installations shall comply with the general and cleanliness requirements as defined in the Process Instruction.

This shall be ensured by regular control of:
- the facilities (installation and equipment) and the products used.
- conformance of the application process conditions to the requirements of this document and the Process Instruction.

All records of test / inspection results shall be stored adequately under the control of the quality assurance of the shop.

All operators of the process shall be adequately trained.

The following minimum properties and corresponding quality requirements are considered necessary to verify the above specified design requirements.

4.3.2 Test frequency

The details and frequency for permanent and regular quality control checks shall be defined by the quality assurance of the shop.

4.3.3 Visual appearance

A visual inspection of the surface after application of deposits is conducted on all treated test specimens and parts. The coat must be uniformly golden and bright, and free of all visible defects (burns due to arc damage, powdery appearance, darkening, etc.).

4.3.4 Coat thickness

The thickness can be measured by :

– micro-section,

– β-ray backscatter or X-ray fluorescent method,

– by rotative abrasion in accordance with AITM 2-0059.

The thickness shall be:
- $1.5 < \varepsilon < 5$ µm for nickel alloys and stainless steels
- 4 µm ± 1 µm for titanium alloys.

The coat must be continuous, smooth and of uniform thickness.

4.3.5 Coat adhesion

The adhesion must be checked on a type 2 follow-up test specimen according to the test method described paragraph 5.2.1.3. Following the acceptance criteria given in appendix C, the adhesion must be grade 0. Grades 1 and 2 are accepted provided that this is due to a worn penetrator.

4.3.6 Quality check of the process - summary

All series production checks are summarised in table 2.

Table 2- Serials checks and minimum checking frequencies

Property	Requirement	Frequency	Material	Number of specimens per alloy
Visual appearance	Para 4.3.3	All parts and follow-up specimen	NA	NA
Thickness of plating	Para 4.3.4	Regular (- one test specimen per **batch** accompanying class 1 and 2F parts - one test specimen per set of 2S and 3 parts and at least one for each week of treatment - when classification applicable)	Nickel alloys and stainless steel: on part or on test specimen Titanium alloys: on test specimen	Parts or 3 specimens for each
Adhesion of plating	Para 4.3.5	Regular (- one test specimen per **batch** accompanying class 1 and 2F parts - one test specimen per set of 2S and 3 parts and at least one for each week of treatment - when classification applicable)	All alloys	5 specimens for each
(1) A batch shall consist of the set of parts treated at same time in the chamber				

4.4 Key Characteristic

Key Characteristics acc. to EN9103 are defined by responsible engineering based on a risk analysis for parts manufactured by this process. Key characteristics shall be defined on product level and if necessary also on process level.

They shall be subject to variation control by production organization according to EN9103.

Key Characteristics do not relieve the production organization from meeting all engineering requirements defined in this document.

Table 3: Key Characteristic

Product Key Characteristic			Process Key Characteristic		
No.	**Designation**	**Requirement/ Limit**	**Sub.-No.**	**Designation**	**Requirement/ Limit**
1	Visual appearance	Correct stoichiometry is defined by the colour, See §4.1 and §4.2.1	1.1	PVD process parameters	Refer to P.I.
		Bright appreance free from defects. See §4.2.1	1.2	PVD process parameters	
2	Thickness	1,5<e<5µm for Ni alloys and stainless steels 4µm ±1µm for Ti alloys	2.1	PVD process parameters	
3	Adhesion	According to §4.2.3	3.1	Surface preparation	
			3.2	PVD process parameters	

5 Process qualification

For technical qualification, refer to AIRBUS qualification procedure for Manufacturing Process.

5.1 Basic qualification requirement

The general process qualification, the qualification of industrial facilities and Airbus quality assurance are described in prevailing procedure documents.

The titanium nitride coating process must be stable and reproducible before launching the technical qualification, so that the selected set of process steps and parameters is taken for the qualification.

The production shop must use its facilities under serial production conditions for the performance of the qualification tests.

Process parameters, environmental conditions etc. applied for qualification shall ensure process performance within the entire process window applied at the relevant shop.

Qualification test program (QTP)

The means for showing compliance with the requirements for achieving technical qualification of the titanium nitride coating process must be defined by the qualification test program.

This includes the standard qualification test defined by this AIPS hereinafter (chapter 5.2) as well as any amendment or deviation.

5.2 Standard qualification test program

This chapter provides the details of the standard test program, which shall be taken into account for the definition of the specific qualification test program (QTP) for the technical qualification of a shop.

In addition to the acceptance criteria, specific test requirements are defined as amendment and/or deviation from the specified test method.

5.2.1 Demonstration of the required performance and quality level

5.2.1.1 Visual inspection

A visual inspection of the surface of all the test specimens and parts treated is conducted after applying the deposit. The coating must have a golden and bright appearance and be free of all visible defects (burns due to arc damage, powdery appearance, etc.).

5.2.1.2 Coat thickness

The coat must be continuous, smooth and of uniform thickness. The thickness is measured by micro-section, using β-ray backscatter or X-ray fluorescent method or by rotative abrasion.

The measurement can be performed by a micrographic section on 3 test specimens (1 section per test specimen). Measurements obtained must be 1.5 µm minimum (Type 2 test specimen).

The measurement can be made using a β-ray backscatter or X-ray fluorescent method. The mean coat thickness from 10 measurements made on test specimen must be 1.5 µm minimum (Type 2 test specimen defined appendix A). The thickness measurement will be confirmed by a micrographic section on 3 test specimens (1 section per test specimen). Measurements obtained must be 1.5 µm minimum (Type 2 test specimen).

The measurement using a calotest will be conducted on type 1 test specimens defined in appendix 2. The method used is described in AITM 2-0059. The thickness must be checked on three test specimens. On each test specimen, three measurements must be made in top, centre and bottom positions of test specimen respectively. In case of doubt, a fractographic section may be requested (observation with scanning electron microscope).

The thickness shall be :

- $1.5 < \varepsilon < 5$ µm for nickel alloys and stainless steels, measured with β-ray backscatter or X-ray fluorescent method (see note 1)
- 4 µm ± 1 µm for titanium alloys, measured by rotative abrasion.

5.2.1.3 Coat adhesion

The adhesion must be checked on a type 2 test specimen.

The adhesion is checked with a Vickers hardness test according to ISO 6507 or to a Rockwell HRC hardness test according to ISO 6508 on five type 2 test specimens.
A Vickers penetrator or Rockwell C penetrator with a load of 150 kg is used. The indentation created produces radial microcracks which may cause separations of the deposit at the edge of the indentation. This separation is inversely proportional to the degrees of adhesion. A complete map of the edge of the indentation must be produced for each indentation. On each test specimen, three measurements must be made in the top, centre and bottom positions of the test specimen respectively. Cracking around the edge of the indentation is observed with a magnification factor of X200. Adhesion must be grade 0. Grades 1 and 2 are accepted provided that this is due to a worn penetrator. The acceptance criteria are given in appendix D.

5.2.2 Standard qualification test program

Test specimen definition is given in table 3.

5.2.3 Qualification test report (QTR)

The qualification test report shall document the qualification tests performed and its results.
It has to be written in English language.

The test report shall make reference to this AIPS and include at least all the following information:
- Name and address of the production shop,
- Process instruction,
- All the details regarding surface preparation,
- Detailed process parameters (bath compositions, temperatures, times, voltage, current density, etc.),
- Equipment, test method, test parameters and test conditions used, etc.,
- Detailed report of the test results. This report shall present all the test values. For the visual inspection, the workshop to be qualified shall supply pictures of treated samples.
- Any incident that may have affected the results and any deviation from this AIPS

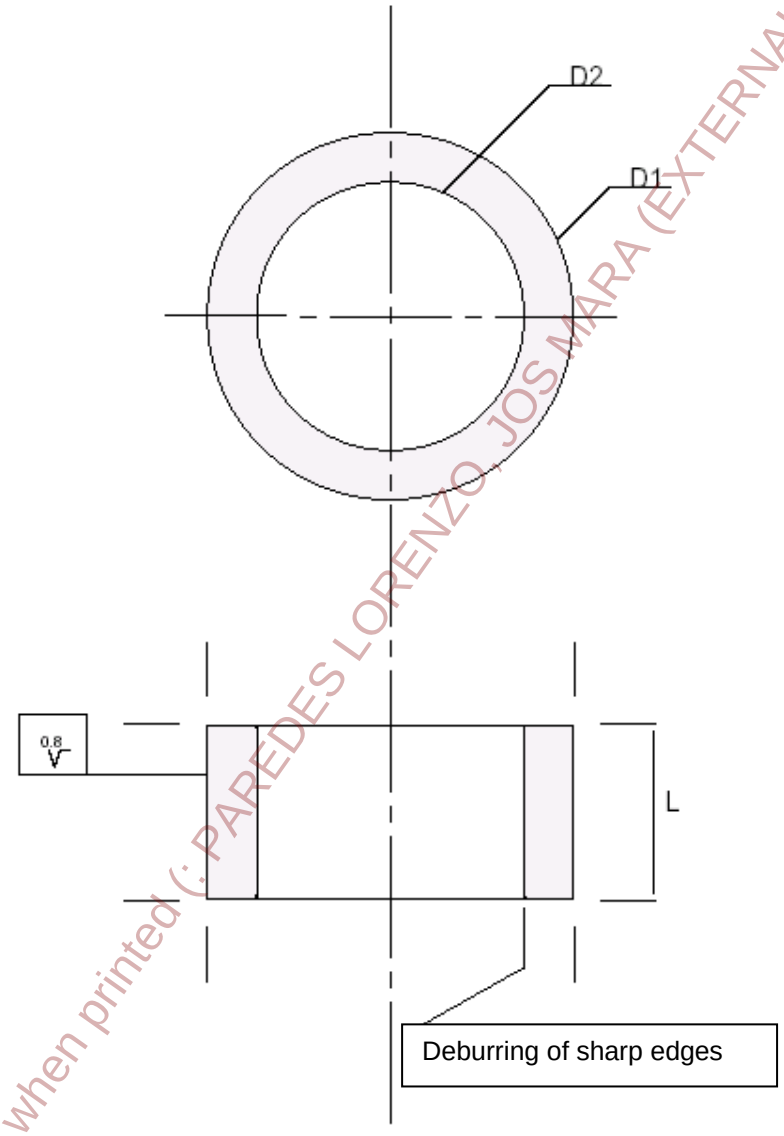
Table 4- Test specimen definition

Test	Test specimen		No. of spec. To be tested.
	Material	Minimum dimensions (mm)	
Visual appearance	All	NA	NA
Thickness of Plating	All	Specimen type 1 or 2	3 for each
Adhesion of Plating	All	Specimen type 1	5 for each

APPENDIX A: TYPE 2 TEST SPECIMEN

Hollow cylindrical test specimen for adhesion and β-ray backscatter or X-ray fluorescent thickness inspection

(The series-production follow-up test specimen must be supplied to the qualified TiN treatment workshop at the time as the parts by the company doing the machining operation)



<u>TiN workshop qualification test specimen</u>: D1 = 50, D2 = 30, L = 20.

Figure A.1:

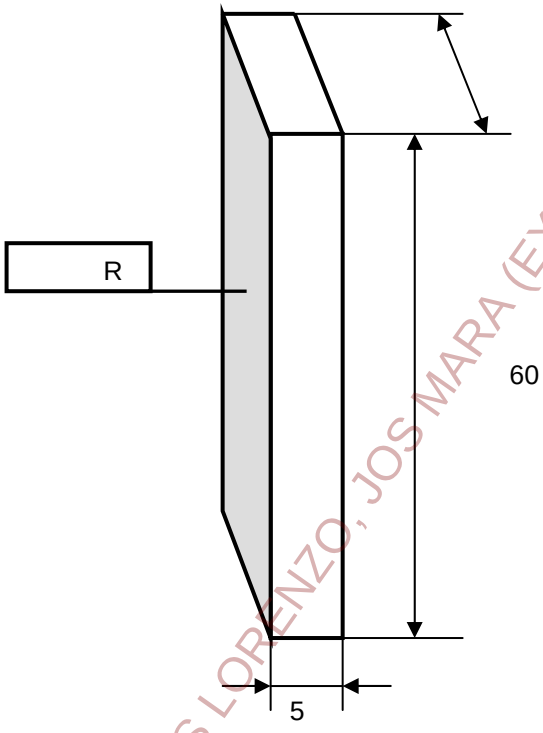
<u>Series-production follow-up test specimen</u>:

- The **material of the test specimen** depends on the material of the parts treated (given in the table above).
- **Test specimen dimensions D1, D2 and L must be defined by the qualified workshop in agreement with the Airbus Quality Department** according to the geometry of the parts treated with D1 > 25 mm. The follow-up test specimen can be used as mask on a part of the batch (especially on the axes). If these dimensions are not defined, default values D1, D2 and L are those of the qualification test specimen (value above).

APPENDIX B: TYPE 1 TEST SPECIMEN

Flat test specimen for thickness inspection using AITM 2-0059

This follow-up test specimen is imposed for inspection using rotative abrasion and, more especially, for the thickness inspection on titanium alloys.



The qualified workshop must use (and provide proof of) a fixture which rotates the test specimen in such a way as to reproduce the kinematics of the parts in the chamber during treatment. A “mannequin” (sample holder) must simulate as best as possible, on the follow-up test specimen, the kinematics of the application of the coating on the surfaces of the part to be treated.

Qualification test specimen (thickness by rotative abrasion):
ASNA 3304 mandatory for titanium alloys.

Figure B.1:

Thickness follow-up test specimen:

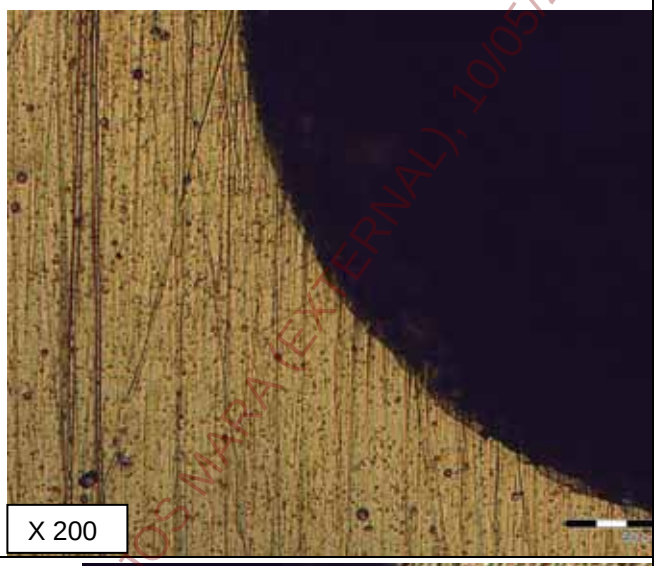
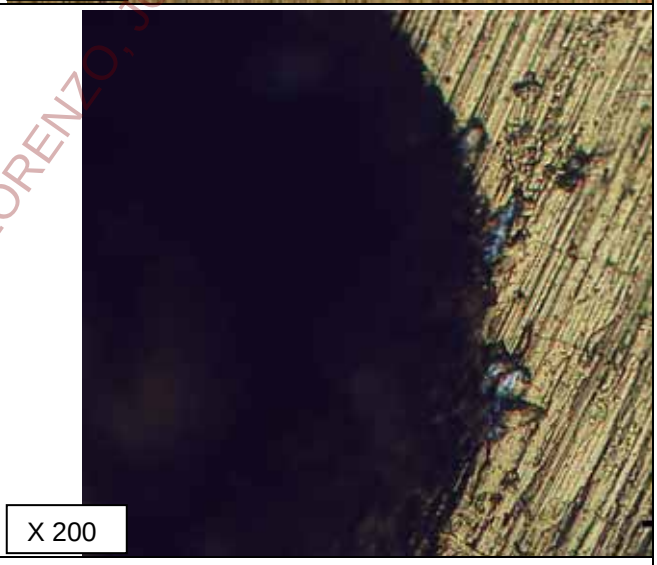
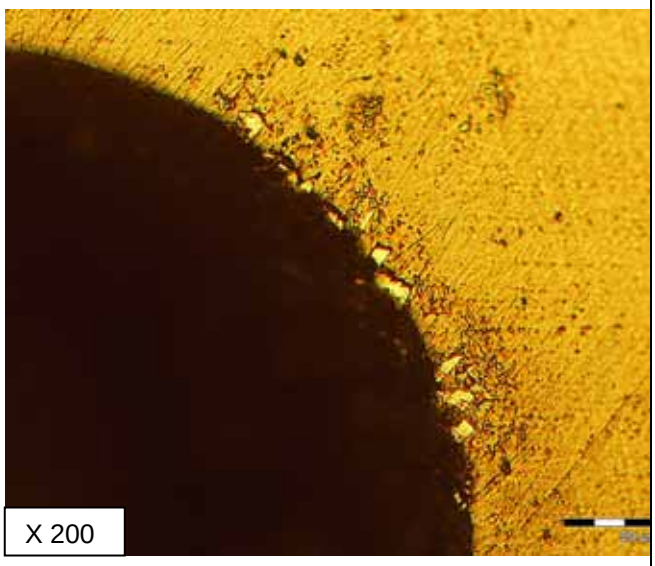
The materials used for the thickness inspection test specimen depend on the material treated:

Table B.1:

Material treated (series-production part)	TYPE of test specimen for thickness inspection	Thickness follow-up test specimen material
Titanium alloy	TYPE 1 imposed	ASNA 3304
Nickel alloy	TYPE 2 recommended	-
Stainless steel	TYPE 2 recommended	-

APPENDIX C: Classification of adhesion test results on TiN coating

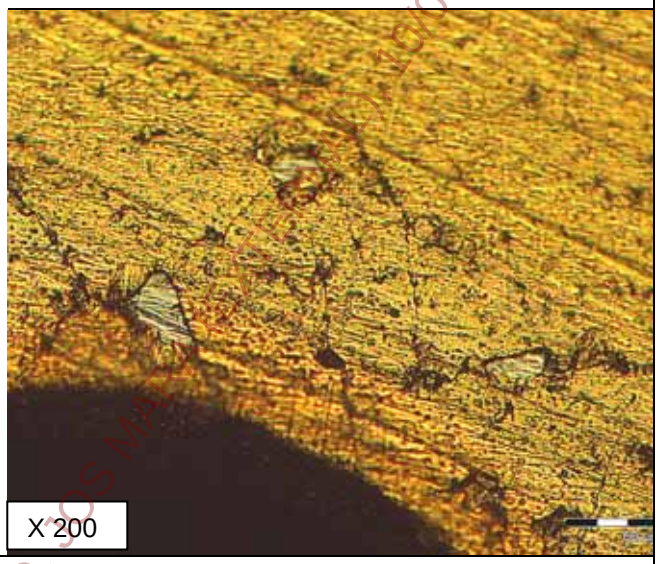
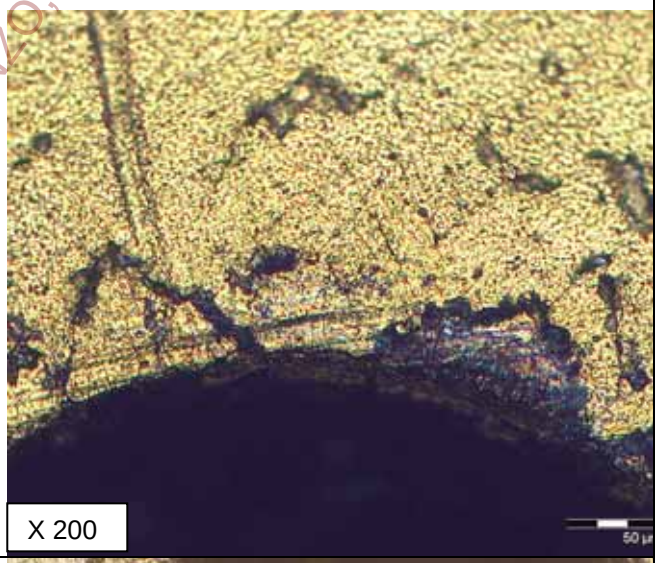
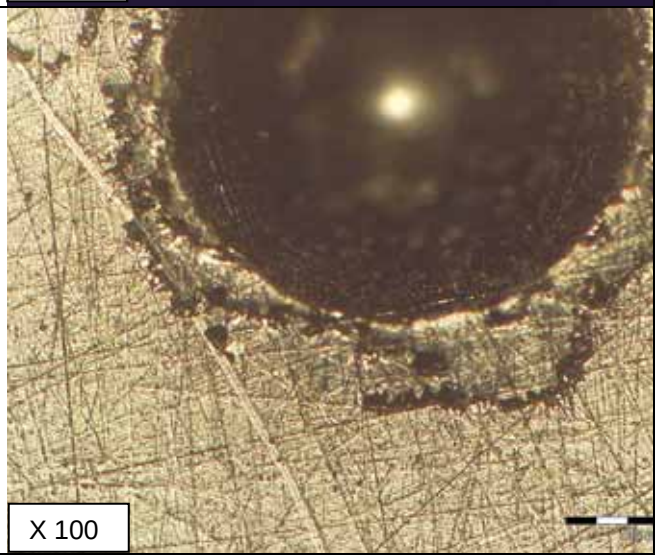
Table C.1:

Classification	Description	Observation of the surface of a portion of the indentation (magnification factor X 200)
Grade 0	The surface of the deposit is cracked but shows no flaking. On the figure opposite, the cracks are radial to the indentation.	 X 200
Grade 1	Some flaking can be seen (between the microcracks) on the immediate edge of the indentation.	 X 200
Grade 2	Flaking is still limited to the edge of the indentation but is more substantial. There are more flakes but they remain limited to the immediate edge of the indentation.	 X 200

(Continued)

APPENDIX C: Classification of adhesion test results on TiN coating

Table C.1:

Classification	Description	Observation of the surface of a portion of the indentation (magnification factor X 200)
Grade 3	Flaking is not limited only to the edge of the indentation. The cracks in the coating are no longer rectilinear and form closed contours where the flaking is observed. Flaking at the edge of the indentation can still be seen.	
Grade 4	Change in relation to grade 3: the flaking occurs in blocks and reaches the edge of the indentation.	
Grade 5	Change in relation to grade 4: generalised flaking.	

Test conducted by HRC 150 kg indentation.

RECORD OF REVISIONS

Issue	Clause modified	Description of modification
1 02/07	_	New Standard
2 07/10	4.4	KC-update