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Method and requirements for plasma nitriding and follow-up PVD hard coatings on cold-work mould steels

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 107, *Metallic and other inorganic coatings*, Subcommittee SC 9, *Physical vapor deposition coatings*.

Any feedback or questions on this document should be directed to the user’s national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Cold-work moulds in automotive application are subjected to heavy wear and severe pressures, while meeting key criteria including high surface hardness and good core strength. Due to higher hardness and better wear resistance compared with the physical vapour deposition (PVD) coating, the duplex treatment based on plasma nitriding and follow-up PVD hard coating has been widely used to improve the lifetime of cold-work moulds in the automotive industry. Plasma nitriding is contributed to improve the loading capacity of the substrate prior to PVD coating deposition. It prevents the plastic deformation of the substrate and delamination of thin and brittle coating, and provides proper stress and hardness gradients between the coating and the substrate, which contributes considerably to the increase in performance of the PVD coating. However, there is no standard to qualify the process, specification and quality of the duplex treatment, which hinders the further development of duplex PVD coatings in the cold-work moulds.

Method and requirements for plasma nitriding and follow-up PVD hard coatings on cold-work mould steels

1 Scope

This document specifies a method and requirements for plasma nitriding and follow-up PVD hard coatings intended for use in cold-work moulds. This document provides the necessary information, such as the original structure of nitriding steels, process requirements, surface quality and adhesion of duplex PVD coatings, to create an optimal combination of high performance.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 6508-1, *Metallic materials — Rockwell hardness test — Part 1: Test method*

ISO 27831-1, *Metallic and other inorganic coatings — Cleaning and preparation of metal surfaces — Part 1: Ferrous metals and alloys*

ISO 18203, *Steel — Determination of the thickness of surface-hardened layers*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
adhesion
amount of energy required to separate the coating from the substrate, that ensures the coating remains adhered to the surface for long especially under aggressive conditions

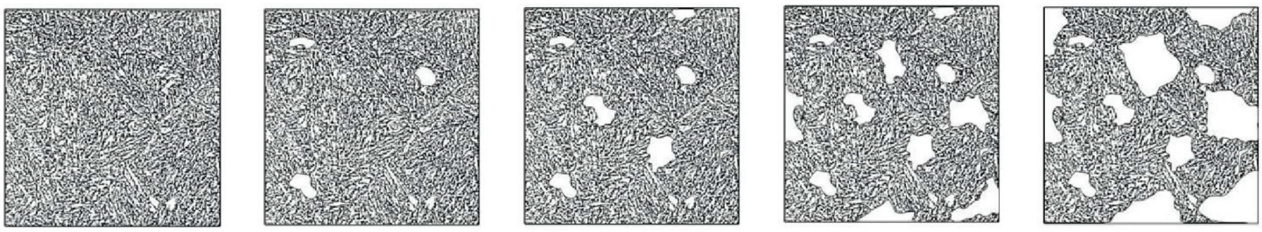
4 Requirements of cold-work mould steels

The alloying elements in cold-work mould steels, such as chromium, vanadium, aluminium, tungsten and molybdenum, are beneficial in plasma nitriding because they can form stable nitrides at nitriding temperatures.

For different service conditions, cold-work mould steels are subjected to conventional heat treatment such as thermal refining by quenching and tempering prior to plasma nitriding. The specimen should be made of cold-work mould steels with a hardness of HRC45 to HRC60. Rockwell hardness test shall conform to the requirements of ISO 6508-1. The test piece for the structure and properties evaluation shall be treated in the same batch as the moulds. The original structure is observed and evaluated by the optical microscope when magnified 500 times. Classes 1 to 5 are specified in Table 1. The schematic drawings of original structures are shown in Figure 1. Classes 1 and 2 reveal acceptable structures.

Table 1 — Description of original structures of cold-work mould steels prior to nitriding

Class	Microstructure
1	Homogeneous fine acicular sorbite with minimal free ferrite content
2	Homogeneous fine acicular sorbite with free ferrite accounting for less than 5 %
3	Fine acicular sorbite with free ferrite accounting for 5 % to 15 %
4	Fine acicular sorbite with free ferrite accounting for 15 % to 25 %
5	Sorbite with free ferrite accounting for more than 25 %



a) Class 1 b) Class 2 c) Class 3 d) Class 4 e) Class 5

Figure 1 — Schematic drawings of original structures of cold-work mould

5 Process requirements

5.1 Requirements for plasma nitriding

5.1.1 Surface polishing

The surface roughness, R_z, of the mould steels shall be less than 1 µm.

5.1.2 Surface cleaning

The surface shall be metallic blank and free of oxidation and foreign residues.

5.1.3 Protection against plasma nitriding

A solution shall be applied for local protection when the areas are not required to be nitrided. In addition, for complex-shaped objects with deep holes, the hollow-cathode-effect can lead to over-ionization inside the holes and uncontrolled heating. These holes shall be blinded using the suitable tools.

5.1.4 Plasma nitriding process

The control of plasma nitriding process is necessary to ensure formation of an acceptable metallurgical case. There are several parameters influencing the plasma nitriding process such as reactive gas, temperature, hold time and pressure. An example of plasma nitriding process is shown in Table 2. For thin-walled substrate, it its necessary to use a plasma nitriding process shall be with low temperature and long holding time. After plasma nitriding, a compound layer and an underlying diffusion zone is formed at the surface of the mould steels. In some cases, nitriding treatments without the formation of a compound layer can be achieved by adjusting the process parameters.

Table 2 — Example of plasma nitriding process

Concentration of alloying element mass fraction %	Ratio of nitrogen to hydrogen	Temperature °C	Hold time h
≤10	≤1	480 to 550	8 to 12
>10	≤0,5	430 to 520	10 to 20
For thin-walled substrate, the plasma nitriding process with low temperature and long holding time is recommended.			

5.2 Requirements for PVD coatings

5.2.1 Mechanical grinding and polishing

In the case of nitriding treatments with the formation of a compound layer, it is necessary to do a mechanical treatment of the surface in order to be coated. The nitriding process produces a fairly rough porous surface not suitable for PVD coatings. In addition, the compound layer inhibits the diffusion of the coating elements in the substrate, which is harmful for adhesion strength. Therefore, the compound layer shall be removed by mechanical grinding and polishing before deposition. The surface roughness, R_z, of the mould steels after plasma nitriding shall be less than 1µm.

5.2.2 Surface cleaning

The nitrided substrates shall be as clean as possible before coating. Debris shall be properly removed in accordance with ISO 27831-1.

5.2.3 PVD coating deposition

For cold-work applications such as forming, cold extrusion and powder compacting, etc., the typical failures on the mould surface are abrasive wear, adhesive wear, galling and friction. The PVD hard coatings with low-friction properties and outstanding high wear resistance dramatically increase the system efficiency and mould lifetime. The design of PVD coatings is mainly depended on roughness, friction coefficient, toughness and anti-sticking.

5.3 Nitriding compound layer and case depth

The compound layer is mainly composed of ε-nitride ($Fe_{2-3}N$) and γ'-nitride (Fe_4N). The ε phase is close to the surface and the γ' phase is near to the diffusion zone. After mechanical treatment, the compound layer thickness cannot be measured by microscopic methods, and the ε-nitride phase in nitriding layer can hardly be detected from X-ray diffraction test.

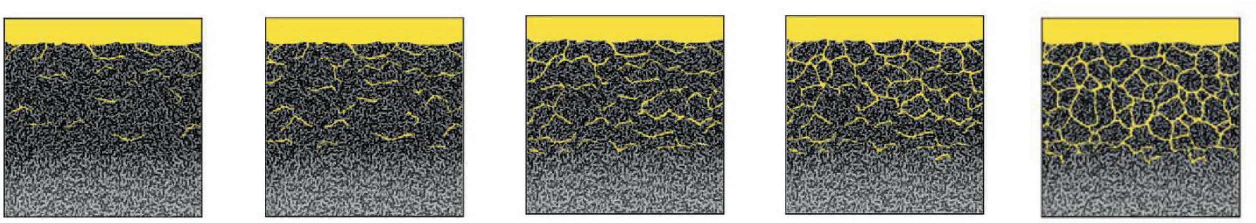
The nitriding case depth shall be calculated from at least five measurements in accordance with ISO 18203. Nitriding case depth shall depend on the type of application of the tools and the cold work tool steel used.

5.4 Nitriding diffusion layer

The nervation and wave like nitrides dispersed around the grain boundary can liable to result in the formation of stress concentration belt. The dispersed nervation and wave like nitrides also enhance the brittle of tool surface and easy to cause cracks initiate due to uneven brittleness. The nitrides in diffusion layer shall be examined by an optical microscope with a magnification of ×500. Classes of 1 to 5 are specified in Table 3. Figure 2 shows pictorial representations of the classes defined in Table 3. Class 1 shall be acceptable.

Table 3 — Description of nervation and wave like nitrides in diffusion layer

Class	Distribution of nervation and wave like nitrides
1	Very little nervation and wave like nitrides
2	A spot of nervation and wave like nitrides
3	More nervation and wave like nitrides
4	Serious nervation and a spot of discontinuous net nitrides
5	Continuous net nitrides



a) Class 1 b) Class 2 c) Class 3 d) Class 4 e) Class 5

Figure 2 — Pictorial representations of the classes defined in Table 3

5.5 Brittleness of nitrided layer

The brittleness of the nitrided layer determined from the appearance of the diamond pyramid impression depends not only on the inherent brittle strength of the layer but also on the compressive stresses. Acting as a four-sided wedge, the diamond pyramid produces compressive stresses in the layer in cross-sections parallel to its sides. The brittleness of nitride layer is tested by using Vickers hardness tester with the load of 98,1 N, and evaluated by impression optical microscope images. Classes of 1 to 5 are specified in Table 4. Figure 3 shows schematic drawings of the brittleness classes in Table 4. Classes 1 and 2 reveal acceptable brittleness.

Table 4 — Brittleness classification of nitrided layer

Class	Distribution of spalls at diamond imprints
1	No side or corner spalls
2	One side or corner
3	Two sides or corners
4	Three sides or corners
5	All sides or corners

