Treatments & Tests of Electroplated Coatings on Steel Fasteners

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Introduction

Fasteners play the role of hardware joint part(s). The hardware joins two or more objects together, and this affixing is crucial to the fabrication of numerous products across the expanse of industries. Unlike welds, fasteners are not permanent generally. It's no surprise that hardware joints and objects shall exist in harmony that the detrimental changes of physical and chemical properties between joints, such as welds and fasteners, and objects need to be considered. The effect of its performance should extend far beyond shapes and sizes of hardware joints and objects.

In the use of fasteners, the fasteners should be exposed to air or their working environments. In the atmosphere of air or working environments there shall be certain corrosion conditions to cause the chemical reaction of materials. In the phenomena of detrimental corrosion there shall be negative status to physical performance. After the surface treatment, the appearance, optical, or electrical performance of fasteners should be improved. It is important that the capability of anti-rust and corrosion resistance should be increased and strengthened. The service life of product is also improved by the surface treatment.

Most fasteners are made of steel material in the form of iron alloy phase. If there is no protection of appropriate metals, materials or bonding other chemicals, the iron alloy shall be attacked easily, reacted, deteriorated to be corroded. The main techniques of surface treatments for fasteners include Conversion coating such as phosphate coating or chromate coating, electroless plating, electoplating, galvanizing, anodizing, painting, etc. Electroplating is the process of plating one metal onto another through hydrolysis, and it's often necessary for fasteners because they, whether in the form of bolts, screws, nuts, or studs, are composed of steel. The interaction and application between electoplating coatings and other coatings become the electroplated coating systems. the quality of electoplating coatings and electroplated coating systems result in the performance of surface treatments. Therefore, electoplating coating and electroplated coating system may be the key to dominating the value of fasteners.

Characteristics of Electroplated Coating Systems

Coating of Metals or Alloys on Fasteners

Electroplated coating systems for steel fasteners are primarily applied for corrosion protection and functional properties, such as torque/clamp force relationship. In addition, other functional properties or decorative properties can be specified such as chemical resistance, electrical conductivity, galvanic corrosion, and cleanliness. Table 1 shows commonly used electroplated coatings in relation with their main purposes and references to related ISO standards.

The schematic representation for build-up of basic electroplated coating systems is shown in Figure 1. A conversion coating increases corrosion protection on zinc, zinc alloys and cadmium coatings. It may be a passivation (chromium VI free) or a chromatation (chromium VI containing). The conversion coating can also provide better adhesion for additional layer(s) and/or additional color/paint.

An additional sealant/top coat (with or without integral lubricant) may be chosen to increase corrosion resistance and to achieve other specific properties (e.g., torque/clamp force properties, resistance to chemicals, mechanical resistance, aspect, color, thermal stability, increased electrical resistance, UV radiation resistance). The selection of the nature of a sealant or top coat should be based on desired additional properties. An additional lubricant may be applied to adjust or amend the torque/clamp force relationship.

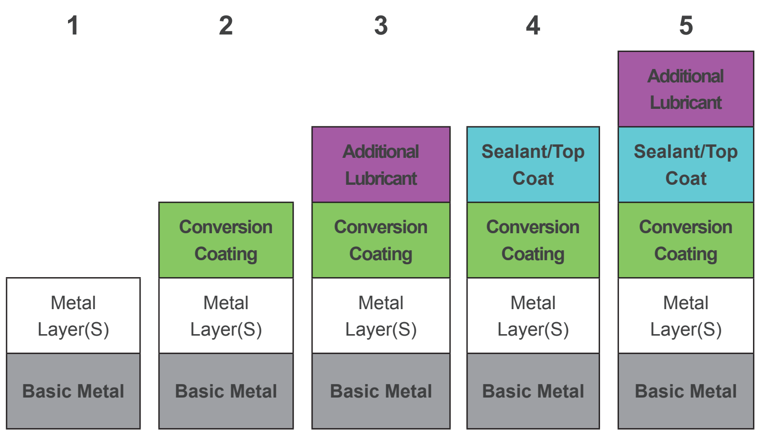

Figure 1. Schematic representation for build-up of basic electroplated coating systems

Table 1. Coatings combination in accordance with their main purposes and related ISO standards

Electroplated Coating + Inorganic Coatings		
Metallic Coating	**Inorganic Coating**	**ISO Standard**
Electroplated coatings of nickel, nickel + chromium, copper + nickel and of copper + nickel + chromium	Inorganic	ISO 1456
Zinc on iron or steel	None or plus inorganic	ISO 2081
Zinc and zinc alloys on iron or steel with Cr(VI)-free treatment	None or plus inorganic	ISO 19598
Cadmium on iron or steel	None or inorganic	ISO 2082
Electroplated coatings of tin	None	ISO 2093
Electrodeposited silver and silver alloy	None or plus inorganic	ISO 4521
Electrodeposited gold and gold alloy coatings	None	ISO 4524-3, ISO 4524-6
Electroplated coatings of nickel plus chromium on plastics materials	None	ISO 4525
Electroplated coatings of nickel for engineering purposes	None	ISO 4526
Electrodeposited coatings of chromium for engineering purposes	None or plus inorganic	ISO 6158
Electroplated coatings of tin-lead	None	ISO 7587
Electrodeposited zinc alloys with nickel, cobalt or iron	None or plus inorganic	ISO 15726
Electrodeposited nickel-ceramics composite coatings	None or plus inorganic	ISO 19487
Electrodeposited gold and gold alloy coatings for electrical, electronic and engineering purposes	None	ISO 27874
Other Coating + Inorganic Coatings		
Metallic Coating	**Inorganic Coating**	**ISO Standard**
Autocatalytic (electroless) nickel-phosphorus alloy coatings	None or plus inorganic	ISO 4527
Mechanically deposited coatings of zinc	None or plus inorganic	ISO 12683
Autocatalytic nickel over autocatalytic copper for electromagnetic shielding	None or plus inorganic	ISO 17334

Electroplated Coating Processes

The type and geometry of the fasteners should be considered when selecting a coating system and the related coating process as well as hydrogen embrittlement considerations.

The issues for type and geometry of the fasteners should be the following.

- Threads of fasteners;
- Fasteners with captive washer;
- Washers and similar fasteners;
- Fasteners with adhesive or patch;
- Prevailing torque nuts;
- Fasteners with recess, internal drive, cavity or hole;
- Screws which form their own mating threads;
- Clips and retaining rings.

Before selecting a coating system, all functions and conditions of the assembly should be considered and not just the fastener.

The functional properties of assembly should be taken into consideration such as assemblability, mountability, chemical resistance, electrical conductivity, galvanic corrosion, cleanliness, etc.

Electroplated coating systems can be applied in bulk process using barrels or in rack process. Top coats are usually applied using a dip-spin process. Electroplating for fasteners is generally a mass process. When lots of small quantities are to be coated, a suitable coating line and/or process can be necessary in order to achieve the required properties and performances for the coated fasteners. For fasteners of large size or mass, or when the risk of thread damage is to be reduced or avoided, rack processing instead of bulk/barrel processing may be considered.

When top coats are applied, the curing process (especially with higher temperature and/or longer duration) can have a detrimental effect on the properties/performances of fasteners, e.g. for cold worked fasteners, for fasteners with threads rolled after heat treatment where intentionally introduced residual stresses may be reduced, for prevailing torque nuts with non-metallic inserts.

Sealants and/or top coats are generally intended to improve the corrosion resistance of zinc based coating systems. If chemical resistance is required, organic top coats applied on electroplated coating systems are typically more resistant against acids and alkaline chemical than inorganic top coats or sealants. The electrical conductivity of electroplated coating systems with a sealant is generally sufficient for application of electrophoretic coatings and antistatic purposes. Electroplated coating systems in combination with sealants and top coats are usually not suitable for electrical grounding. In order to reduce the risk of galvanic corrosion, each part of the assembly should be considered (coated fasteners and clamped parts). A direct metal contact of coated fasteners with non-coated clamped parts should be avoided, especially for stainless steel, aluminium, magnesium, copper or copper alloys, carbon fiber materials, and carbon filled rubber. Due to their higher insulating effect, organic top coats can improve the resistance against galvanic corrosion. The most suitable measure to reduce the risk of galvanic corrosion is to select a coating or coating system for the fastener with the same or a similar electrical potential compared to the clamped parts.

Internal Hydrogen Embrittlement (IHE)

Hydrogen embrittlement is the coating process of electroplated coating systems by which hydride-forming metals become brittle and fracture due to the introduction and subsequent diffusion of hydrogen into the metal. Susceptibility to hydrogen-induced cracking ('embrittlement') is often a result of the introduction of hydrogen during forming, coating, plating, cleaning, and finishing operations, often referred to as 'internal embrittlement'. Hydrogen also may be introduced over time (so-called 'external embrittlement' through environmental exposure (soils and chemicals, including water), corrosion processes (especially galvanic corrosion), cathodic protection, and/or from hydrogen generated by corrosion of a coating.

There is a risk of Internal Hydrogen Embrittlement (IHE) if the three following conditions are concurrently present for fasteners:

• with high tensile strength or hardness or which have been case hardened and tempered or cold worked to high hardness,

• which are under tensile stress, and

• which have absorbed hydrogen.

The susceptibility to IHE increases with increasing hardness of the fastener material.

Kinds of Clean, Pretreatment or Post-coating treatments of iron or steel to reduce the risk of hydrogen embrittlement of ISO standard should be recommended in Table 2.

Table 2. Recommended treatments to reduce the risk of hydrogen embrittlement of ISO standard

Treatment	ISO Standard
Pretreatment	ISO 9587
Post-coating treatments	ISO 9588
Cleaning and preparation of metal surfaces for ferrous metals and alloys	ISO 27831-1
Cleaning and preparation of metal surfaces for non-ferrous metals and alloys	ISO 27831-2

Appropriate measures for prevention of IHE for quenched and tempered fasteners depending on hardness are specified in ISO 4042:2018. It is shown in Table 3. Measures related to IHE in accordance with ISO 898-1, ISO 898-2, ISO 898-3 are listed in Table 4 for the fastener hardness between 360 HV and 390 HV.

When electroplating fasteners with specified maximum hardness above 390 HV, baking is required.

ISO 898-1, ISO 898-2 and ISO 898-3 and ISO 4042:2018 apply.

Table 3. Measures related to IHE for quenched and tempered fasteners with regard to hardness

360HV 390HV

A	B	C
No supplemental process verification or product testing with regard to IHE AND No baking necessary	Supplemental process verification and/or product testing with regard to IHE OR Baking (at the choice of the fastener manufacturer)	Supplemental process verification and/or product tests with regard to IHE AND Baking (baking temperature and duration shall be specified)

Table 4. Measures related to IHE in accordance with ISO 898-1, ISO 898-2, ISO 898-3

Bolts. screws, studs in accordance with ISO 898-1	Property class		
	≦ 9.8	10.9	12.9 and 12.9.
Measures related to IHE	A	B	C
Nuts in accordance with ISO 898-2	Property class		
	≦ 12		
	Nuts with specified maximum hardness ≦ 360 HV	Nuts with specified maximum hardness ≧ 360 HV	
Measures related to IHE	A	B	
Flat washers in accordance with ISO 898-3	Property class		
	≦ 200HV	300 HV	380HV
Measures related to IHE	A	B	C

Supplementary Treatments

When baking is performed, baking conditions including temperature and duration shall be based on fastener material properties, electroplating process, and coating material.

Baking is usually performed before application of a conversion coating and/or before application of an additional sealant/top coat. In case of passivations (with or without sealant) and depending on baking temperature, baking in the passivated and/or sealed condition may be suitable, provided that corrosion resistance is not impaired.

The minimum baking temperature and duration factors are temperature, duration, permeability of the coating and coating thickness. For susceptible fasteners (e.g. with hardness/core hardness above 390 HV) that are zinc electroplated, 8h to 10h at 190 °C to 220 °C is a minimum recommended baking duration. The common practice of baking zinc electroplated fasteners for 4h at approximately 190 °C is inadequate for extracting hydrogen because zinc is an effective barrier to hydrogen diffusion. It has been shown that a baking duration of 4h can even be detrimental and can lead to occasional failures.

Property class 10.9 fasteners that are correctly manufactured to the intended material and metallurgical properties, as specified in ISO 898-1, are not susceptible to fail due to IHE, and do not need to be baked. Property class 10.9 fasteners are sometimes baked as a precaution against manufacturing errors or out-of-control process that could render the material susceptible.

The maximum temperature and duration used in a baking process are limited by the following considerations. It should not exceed the temperature at which the fasteners were originally tempered, and should not impair the performance of the coating. Excess of temperature and/or duration can impair the beneficial effect of thread rolling after heat treatment.

Zinc electroplated parts are usually baked at a temperature not higher than 220 °C.

Cadmium electroplated parts are usually baked to a temperature not higher than 200 °C.

Baking criteria as specified in ISO 2081, ISO 9588 and ISO 19598 may be the references and recommendation to reduce the risk of IHE.

Case-hardened and tempered fasteners include self-tapping screws (see ISO 2702), thread-forming screws for metallic materials, self-drilling screws (see ISO 10666) and screws for soft materials (e.g. plastic, wood). The surface of these screws is usually intentionally hardened to fulfil their specific functions. Requirements for case-hardened and tempered fasteners (except for self-tapping screws and screws for soft materials) are specified in Table 5 (except self-tapping screws and screws for soft materials). Requirements for self-tapping screws and screws for soft materials are specified in Table 6.

Table 5. Measures related to IHE for case-hardened and tempered fasteners

Case-hardened and tempered fasteners	Core Hardness	
	≦370 HV	> 370 HV
Measures related to IHE	B	C
	Supplemental process verification with regard to IHE AND Product testing and/or baking	Supplemental process verification with regard to IHE AND Product testing and/or baking AND Product testing for each manufacturing lot

Table 6. Measures related to self-tapping screws and screws for soft materials

Self-tapping screws and screws for soft materials	Core Hardness	
	≦390 HV	> 390 HV
Measures related to IHE	B	C
	Supplemental process verification with regard to IHE AND Product testing and/or baking	Supplemental process verification with regard to IHE AND Product testing and/or baking AND Product testing for each manufacturing lot

Corrosion Protection and Tests

Corrosion resistance is considered to be a product characteristic that can be altered as a consequence of the following factors such as physical damage to the coating from handling and transportation, and oxidation of the coating or reaction with the environment during transportation and storage. The frequency and duration of wetting and service temperatures, contact with corrosive chemicals and contact with other metals and materials (galvanic corrosion/contact corrosion) can influence the protective performance of coatings. Coatings of zinc, zinc alloys and cadmium provide cathodic protection to the steel basis metal. In contrast, It can intensify corrosion of the fastener if the coating is damaged or pitted and metals & coatings cannot provide cathodic protection (e.g. nickel, copper, silver). Accelerated tests are commonly used to evaluate the corrosion resistance of the coating system. The neutral salt spray test (NSS) in accordance with ASTM B117, ISO 9227, DIN 50021, or JIS Z2371 is used to evaluate the corrosion resistance of the coating system. Sulfur dioxide test (Kesternich test) is intended for outdoor building fasteners with zinc based coating systems. DIN 50018, ISO 6988 ,ISO 3231, ASTM G87 or ASTM G85 Annex 4 should be applied if specified. The other test methods may be applied such as AASS (Acetic Acid Salt Spray), CASS(Copper Accelerated Acetic Acid Salt Spray) to evaluate the corrosion resistance of the coating system.

The corrosion protection of an electroplated coating system depends to a considerable extent on the thickness of the metal layer(s). Conversion coatings and/or sealants/top coats on zinc, zinc-iron, zinc-nickel and cadmium coatings provide protection against coating metal corrosion (formation of white corrosion).,thus providing additional protection against basis metal corrosion.

Coating thickness has a significant influence on gaugeability and assemblability; therefore thread tolerance and clearance in the thread shall be taken into account. The coating shall not cause the zero line to be exceeded in the case of external threads, nor shall it fall below in the case of internal threads. One of the test methods shall be used to determine the local thickness of the metal layer(s) in Table 7.

The coated fastener shall be free from blisters. Delamination and uncoated areas which can adversely affect the corrosion protection. Local excess of coating (e.g. in the case of top coats) shall not impair functional properties (gaugeability, torque/clamp force relationship, etc.).

It is possible to add dyes/pigments to the passivation or chromatation solution in order to give a colored aspect to the conversion layer often used for distinction purposes. Dyes/pigments may also be added to sealants/top coats to obtain colored surfaces.

If the environment temperature of fastener use is taken into consideration, elevated temperature can affect the corrosion protection of coated fasteners. Corrosion resistance related to temperature should be carried out. The corrosion resistance may be tested after a specified heating cycle. Temperature and duration shall be agreed upon, e.g.1 cycle & 1h at 120 °C, 1 cycle per 2h to 24h at 120 °C, 1 cycle 1h at 150°C. After heating the fasteners at part temperature for a specified cycle, the corrosion resistance shall still be the specified requirements.

Table 7. Test methods for determining the local thickness of the metal layer(s)

Test methods	Standard	Remark
X-ray techniques	ISO 3497	
Coulometric method	ISO 2177	This method should not be used when additional non-conductive layers are present
Microscopic method	ISO 1463	On any area(s) of the fastener
Magnetic inductive techniques	ISO 2178	Before addition of any sealant and/or lubricant this method can also be used to determine the total local thickness
Eddy current (phase sensitive) testing	ISO 21968	

Torque/clamp force relationship may be determined with electroplated coating systems including sealants and/or top coats with integral lubricant and/or subsequently added lubricant.

The test method shall be agreed in accordance with other relevant technical specifications, e. g. fasteners with ISO metric thread is in accordance with ISO 16047. When required, the presence or absence of Cr(VI) shall be determined in accordance with ISO 3613:2010.

The following mandatory tests shall be carried out for each lot of fasteners:

- coating thickness;
- gaugeability/assemblability of fasteners;
- appearance.

The following tests are not intended to be applied for each fastener lot but shall be used for in-process control:

- corrosion resistance: NSS test, sulfur dioxide test or alternatively;
- supplemental/additional process verification with regard to IHE.

In-process control may be used to be final test results the same as the final process control.

Corrosion resistance related to temperature, Torque/clamp force relationship, determination of presence or absence of hexavalent chromium are all the integral performance of fasteners with Electroplated Coating System.

Reference:

ISO 4042:2018 Fasteners -- Electroplated coating systems

ISO 9587:2007 Metallic and other inorganic coatings -- Pretreatment of iron or steel to reduce the risk of hydrogen embrittlement

ISO 9588:2007 Metallic and other inorganic coatings -- Post-coating treatments of iron or steel to reduce the risk of hydrogen embrittlement

ISO 27831-1:2008 Metallic and other inorganic coatings -- Cleaning and preparation of metal surfaces -- Part 1: Ferrous metals and alloys

ISO 27831-2:2008 Metallic and other inorganic coatings -- Cleaning and preparation of metal surfaces -- Part 2: Non-ferrous metals and alloys.