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Metallic coatings on metallic substrates — Review of methods available for testing adhesion — Part I : Electrodeposited and chemically deposited coatings

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FOREWORD

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Draft International Standards adopted by the Technical Committees are circulated to the Member Bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 2819/1 was drawn up by Technical Committee ISO/TC 107, *Metallic and other non-organic coatings*, and circulated to the Member Bodies in September 1972.

It has been approved by the Member Bodies of the following countries :

Egypt, Arab Rep. of	Italy	Sweden
France	Japan	Switzerland
Germany	New Zealand	Thailand
Hungary	Poland	Turkey
India	Portugal	United Kingdom
Ireland	Romania	U.S.S.R.
Israel	South Africa, Rep. of	

The Member Body of the following country expressed disapproval of the document on technical grounds :

Netherlands

Metallic coatings on metallic substrates — Review of methods available for testing adhesion — Part I : Electrodeposited and chemically deposited coatings

1 SCOPE AND FIELD OF APPLICATION

This International Standard describes methods of checking the adhesion of electrodeposited and chemically deposited coatings. It is limited to tests of a qualitative nature. Table 2 indicates the suitability of each test for some of the most usual types of metallic coatings.

It does not describe certain tests which have been developed at various times to give a quantitative measure of adhesion of metallic coating to a substrate since such tests require special apparatus and considerable skill in their performance which renders them unsuitable as quality control tests for production parts. Some of these quantitative tests may, however, be useful in research and development work.

When particular methods of adhesion testing are included in International Standards for individual coatings, they should be used in preference to the methods described in this International Standard and should be agreed upon beforehand by the supplier and the purchaser.

2 METHODS OF TEST

2.1 Burnishing test

If plated parts are subjected to burnishing in a localized area, the deposit will tend to work-harden and absorb frictional heat. If the coating is thin, separation of the coating from the basis metal as blisters will occur under these conditions in areas of poor adhesion.

When the shape and size of the part permit, an area of not more than 6 cm² of the plated surface should be rubbed with a smooth implement for about 15 s. A suitable implement is a steel rod 6 mm in diameter with a smooth hemispherical end.

The pressure shall be sufficient to burnish the coating at every stroke but not so great as to cut the coating. Poor adhesion is indicated by the appearance of a blister which grows as the rubbing is continued.

If the mechanical properties of the coating are poor, the blister may crack and the coating will peel from the basis metal. This test shall be limited to relatively thin deposits.

2.2 Ball burnishing test

Ball burnishing is frequently used for polishing, but it can be used also to test adhesion. Using a barrel or vibratory burnisher with steel balls about 3 mm in diameter and soap solution as lubricant, it is possible to produce blisters when the adhesion is very poor. The method is suitable for relatively thin deposits.

2.3 Shot peening test

There are some variations of the principle by which the hammering action of iron or steel balls, allowed to fall by gravity or forced by means of a pressure air stream onto the surface to be tested, produces deformation of the deposit.

If the coating is poorly bonded, it will become blistered. Usually, the intensity of peening necessary to cause non-adherent coatings to blister varies with the coating thickness, thin coatings requiring less than thick coatings.

One test can be performed using a tube 150 mm long, 19 mm internal diameter, as the reservoir for round iron or steel shot (0,75 mm diameter approximately) connected to a nozzle. Compressed air is brought to the apparatus with a pressure of 0,07 to 0,21 MN/m² and the distances between nozzle and specimen are 3 to 12 mm.

Another test, that appears to be the most suitable for checking the adhesion of electroplated coatings of silver during production of coatings from 100 to 600 µm in thickness, is described in the annex and employs a standard air-operated cabinet of the type used for shot-peening steel parts.

If the silver is poorly bonded, it will extend or flow and become blistered.

2.4 Peel test

This test is suitable for coatings less than 125 µm thick on substantially flat surfaces. A strip of tinned mild steel or brass, approximately 75 mm long X 10 mm wide X 0,5 mm thick, is bent at right angles 10 mm from one end and the shorter limb soldered flat to the coated surface. A load is applied to the free limb and normal to the soldered surface. The coating will be detached from the substrate if the adhesion is weaker than the soldered joint. If the adhesion of the coating is greater than this, however, failure will occur in the soldered joint or within the thickness of the coating.

This method is not widely used because the temperature reached during the soldering operation might alter the adhesion. Alternatively, the test can be performed using an adhesive of hard-setting synthetic resin of adequate tensile strength in place of solder.

Another test (the tape test) employs an adhesive cellulose tape, with an adhesion value of approximately 8 N per 25 mm width, whose adhesive side is applied to the coating under test, using a fixed-weight roller, care being taken to exclude all air bubbles. After an interval of 10 s, the tape is removed by applying a steady pulling force to the tape, perpendicular to the surface of the coating. The adhesion of the coating shall be such that there is no evidence of detachment of the coating. This test is particularly used for testing adhesion of coatings on the conductors and contacts of printed circuits. Coated conductors shall be tested over an area of at least 30 mm².

2.5 File test

A piece sawn off a coated article is held in a vice and a coarse mill file (one set of serrations only) is applied to the cut in such a manner as to attempt to raise the coating. The file is used in the direction from the basis metal to the coating at an angle of approximately 45° to the coated surface. No detachment of the coating shall occur. This test is not suitable for very thin coatings and for soft coatings such as zinc or cadmium.

2.6 Grinding and sawing tests

Grind an edge of the coated specimen with a grinding wheel with the direction of cutting from the basis metal to the deposit. If adhesion is poor, the deposit is torn from the base. A hacksaw can be substituted for the grinder. It is important to saw in such a direction that a force is applied that tends to separate the coating from the basis metal. Grinding and sawing tests are especially effective on harder coatings such as nickel and chromium.

2.7 Chisel test

The chisel test is normally used on coatings of considerable thickness (greater than 125 µm).

One variation of the test is to place a sharp chisel at the back of a coating overhang, and give it a sharp hammer blow. If the adhesion is good, the coating will break away or be cut through without the bond between basis metal and coating being affected.

Another type of "chisel test" is combined with the "saw test". The test is made by sawing the specimen perpendicular to the coating; if the adhesion is not very good, failure immediately becomes evident. In cases where there appears to be no separation at the fracture, a sharp chisel is used to try to raise the coating at the edge. If the coating can be peeled from the edge for an appreciable distance, poor or weak adhesion is indicated. The cutting edge of the chisel shall be sharpened prior to each test.

Thinner coatings can be tested by substituting a knife for the chisel and light tapping with a hammer may or may not be used. The chisel test is not suitable for soft coatings such as zinc or cadmium.

2.8 Scribe and grid tests

Using a hardened steel scribe which has been ground to a sharp 30° point, two parallel lines are scribed at a distance apart of about 2 mm. In scribing the two lines, enough pressure shall be applied to cut through the coating to the basis metal in a single stroke. If any part of the coating between the lines breaks away from the basis metal, the coating shall be deemed to have failed the test.

One variation of the test is to draw a square with a grid of 1 mm side and observe whether the coating peels from the basis metal within this area.

2.9 Bending test

The bend test consists in bending or flexing the coated products. The extent and nature of the distortion will vary with the basis metal, the shape, the nature of the coating and the relative thickness of the two layers.

The test is usually carried out by hand or with pliers, bending the specimen as sharply as possible first to one side then to the other, until the specimen breaks. The rate and the radius of bending can be controlled using suitable machines. The test produces a shearing stress between the basis metal and the deposit; if the deposit is ductile, the shearing force is much reduced because the coating flows and the basis metal can even break without the coating loosening.

A brittle deposit can crack but even so the test can give some information about the adhesion; the fracture must be inspected to determine whether the deposit peeled or can be removed with a knife or chisel.

Any sign of peeling, chipping or flaking is taken as indication of poor adhesion.

The coated specimen can be distorted with the coating on either the inside or the outside of the specimen. The behaviour of the coating is normally observed on the outside layer, although in some cases further information may be gained by examining the inside of the bend.

2.10 Twisting (winding) test

In this test the specimens (normally strips and wires) are twisted around a mandrel. Each part of the test can be standardized: the length and width of the strip, the rate of bending, the uniformity of bending movement and the diameter of the rod around which the piece is twisted.

Any sign of peeling, chipping or flaking is taken as indication of poor adhesion.

The coated specimen can be distorted with the coating on either the inside or the outside of the specimen. The behaviour of the coating is normally observed on the outside layer, although in some cases further information may be gained by examining the inside of the bend.

2.11 Tensile test

This is suitable only for certain types of coated article. The article is stressed in tension until it breaks. Some cracking of the coating will normally be evident near to the fracture, but no detachment of the coating from the basis metal shall be visible.

2.12 Thermal shock test

The adhesion of many deposits can be determined by heating the coated specimen and then suddenly cooling it. The principle involved in this test is the difference in coefficient of expansion between the coating and the basis metal.

It is therefore applicable when the coefficient of expansion of a coating is appreciably different from that of the basis metal. The test is performed by heating the specimen in an oven for a sufficient time to achieve the appropriate temperature shown in Table 1. The temperature shall be maintained within $\pm 10^\circ\text{C}$. Metals that are sensitive to oxidation shall be heated in an inert or reducing atmosphere or in suitable liquids.

TABLE 1 — Thermal shock test temperatures

Basis metal \ Coating metal	Chromium, nickel, nickel + chromium, copper and tin-nickel	Tin
Steel	300 °C	150 °C
Zinc alloys	150 °C	150 °C
Copper and copper alloys	250 °C	150 °C
Aluminium and aluminium alloys	220 °C	150 °C

The specimen shall then be quenched in water at room temperature. No separation, for example by blistering, flaking or exfoliation, of the coating from the basis metal shall occur.

It should be mentioned that heating generally improves the bond strength of electrodeposits¹⁾ so that any test method which requires heating of the test piece does not give a correct indication of the bond strength "as plated".

2.13 Drawing test

The drawing tests most commonly used on coated sheet metal are the "Erichsen cupping test" and the "Romanoff flanged cap test".

They produce a deformation of the deposit and basis metal into a cup or flanged depression by means of some sort of plunger.

In the Erichsen test, a ball-shaped plunger 20 mm in diameter is pushed into the specimen with a speed of 0,2 to 6 mm/s to a desired depth with a suitable hydraulic device. Poorly adherent deposits peel or flake from the basis metal after a few millimetres' distortion, while adherent deposits exhibit no peeling, even when the basis metal has been cracked by the penetrating mandrel.

The apparatus for the Romanoff test consists of an ordinary press with a set of adjustable dies for drawing a flanged cap. The flange is 63,5 mm in diameter and the cap 38 mm in diameter. The depth of the cap is adjustable from 0 to 12,7 mm. The specimens are usually tested to a point which will fracture the cap. The intact part of the draw shows how drawing affects the structure of the deposit. These methods are used specially for deposits of the harder metals, such as nickel or chromium.

In all cases, the results must be cautiously interpreted since the ductilities of both the deposit and basis metal are involved.

2.14 Cathodic test

The coated part is made cathodic in a solution from which only hydrogen is evolved. Blistering of the coating may take place due to pressure of gaseous hydrogen which diffuses through certain coatings and accumulates at the site of any discontinuities between the coating and the basis metal.

The test is performed using a 5 % solution of sodium hydroxide (ρ 1,054 g/ml) and treating the specimen for 2 min with a current density of 10 A/dm² at 90° C. Small blisters form at points where adhesion is poor. If the coating is still free from blisters 15 min afterwards, the adhesion may be regarded as good. Alternatively, a solution of sulphuric acid (5 % m/m) can be used at 60 °C with a current density of 10 A/dm². Weakly adherent coatings develop blisters in 5 to 15 min under these conditions.

The electrolytic test is limited in application to coatings which are permeable to cathodically discharged hydrogen. Nickel or nickel-chromium coatings react satisfactorily to the test if they are weakly adherent. Coatings of metals such as lead, zinc, tin, copper or cadmium are not suitable for testing by this method.

1) In other cases, the diffusion of the coating into the basis metal can create a brittle layer so that the peeling of the coating is caused by fracture rather than non-adhesion.

TABLE 2 — Adhesion tests appropriate for various coating metals

Coating metal Adhesion test	Cadmium	Chromium	Copper	Nickel	Nickel + chromium	Silver	Tin	Tin-nickel alloy	Zinc	Gold
Burnishing	*		*	*	*	*	*	*	*	*
Ball burnishing	*	*	*	*	*	*	*	*	*	*
Peeling (soldering method)			*	*		*		*		
Peeling (adhesive method)	•		*	*		*	*	*	*	*
File			*	*	*			*		
Chisel		*		*	*	*		*		
Scribe	*		*	*	*	*	*		*	*
Bending and twisting		*	*	*	*			*		
Grinding and sawing		*		*	*			*		
Tension	*		*	*	*	*		*	*	
Thermal shock		*	*	*	*		*	*		
Extrusion (Erichsen)		*	*	*	*			*		
Extrusion (Flanged cap)		*	*	*	*	*		*		
Shot-peening				*	*	*				
Cathodic treatment		*		*	*					