INTERNATIONAL STANDARD

ISO 4527

First edition
1987-07-15

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION
ORGANISATION INTERNATIONALE DE NORMALISATION
МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ

Autocatalytic nickel-phosphorus coatings — Specification and test methods

Dépôts autocatalytiques de nickel-phosphore — Spécifications et méthodes d'essai

Reference number
ISO 4527 : 1987 (E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 4527 was prepared by Technical Committee ISO/TC 107, *Metallic and other non-organic coatings.*

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

Printed in Switzerland

Contents

Figures

Autocatalytic nickel-phosphorus coatings — Specification and test methods

0 Introduction

Autocatalytic[1] nickel-phosphorus coatings can be obtained from baths formulated to produce bright, semi-bright or dull deposits. Their appearance depends upon the brightness and smoothness of the substrate. The deposits are normally used without machining or polishing.

These coatings are produced by the catalytic reduction of nickel metal ions in hot, usually mildly acidic aqueous solutions at atmospheric pressure using sodium hypophosphite as the reducing agent. Suitable solutions, proprietary or otherwise, are those that produce a deposit which will meet the requirements of this specification.

The coatings produced are uniform in thickness on irregularly shaped parts, provided that the processing solution circulates freely over all surfaces.

The coating is a metastable alloy of nickel and phosphorus containing up to 15 % (m/m) of phosphorus. The physical and chemical properties and the structure of autocatalytic nickel are dependent upon the coating composition, the chemical make-up of the plating bath, the pretreatment of the substrate, and heat treatment after plating.

It should be noted that heat treatments above 220 °C may reduce the corrosion resistance of the coatings. Heat treatments up to 200 °C used to improve adhesion or to give relief from hydrogen embrittlement do not impair the corrosion resistance, or substantially modify the hardness, or the wear properties of the coatings.

It should be recognized that autocatalytic nickel-phosphorus coatings tend to be less ductile than those of electrodeposited nickel. Certain coatings of less than 10 µm thickness will generally permit limited deformation without cracking or spalling.

1 Scope and field of application

This International Standard specifies requirements and test methods for autocatalytic nickel-phosphorus coatings.

This International Standard is not applicable to nickel-boron alloys.

2 References

ISO 468, *Surface roughness — Parameters, their values and general rules for specifying requirements.*

ISO 1462, *Metallic coatings — Coatings other than those anodic to the basis metal — Accelerated corrosion tests — Method for the evaluation of the results.*

ISO 1463, *Metallic and oxide coatings — Measurement of coating thickness — Microscopical method.*

ISO 2064, *Metallic and other non-organic coatings — Definitions and conventions concerning the measurement of thickness.*

ISO 2177, *Metallic coatings — Measurement of coating thickness — Coulometric method by anodic dissolution.*

ISO 2178, *Non-magnetic coatings on magnetic substrates — Measurement of coating thickness — Magnetic method.*

ISO 2819, *Metallic coatings on metallic substrates — Electrodeposited and chemically deposited coatings — Review of methods available for testing adhesion.*

ISO 2859, *Sampling procedures and tables for inspection by attributes.*[2]

ISO 3497, *Metallic coatings — Measurement of coating thickness — X-ray spectrometric methods.*

ISO 3543, *Metallic and non-metallic coatings — Measurement of thickness — Beta backscatter method.*

ISO 3768, *Metallic coatings — Neutral salt spray test (NSS test).*

ISO 3769, *Metallic coatings — Acetic acid salt spray test (ASS test).*

1) Autocatalytic nickel-phosphorus coatings are also known as "electroless" or "chemical" nickel.

2) At present at the stage of draft. (Revision of ISO 2859-1974.)

ISO 3770, *Metallic coatings — Copper-accelerated acetic acid salt spray test (CASS test).*

ISO 4516, *Metallic and related coatings — Vickers and Knoop microhardness tests.*

ISO 4519, *Electrodeposited metallic coatings and related finishes — Sampling procedures for inspection by attributes.*

ISO 4538, *Metallic coatings — Thioacetamide corrosion test (TAA test).*

ISO 4540, *Metallic coatings — Coatings cathodic to the substrate — Rating of electroplated test specimens subjected to corrosion tests.*

ISO 4541, *Metallic and other non-organic coatings — Corrodkote corrosion test (CORR test).*

ISO 4793, *Laboratory sintered (fritted) filters — Porosity grading, classification and designation.*

IEC Publication 68-2-20, *Basic environmental testing procedures — Part 2 — Test T: Soldering.*

3 Definition

significant surface: The part of the article covered or to be covered by the coating and for which the coating is essential for serviceability and/or appearance.

(Definition taken from ISO 2064.)

4 Information to be supplied by the purchaser to the plater

NOTE — Close liaison between designers, manufacturers and platers is desirable in order to obtain satisfactory autocatalytic nickel-phosphorus coatings and to avoid adverse effects on the mechanical properties of the article.

4.1 Essential information

The following information shall be supplied by the purchaser to the plater:

a) the number of this International Standard;

b) the thickness, in micrometres, of the coating and of any undercoats;

c) details of significant surfaces, to be indicated on drawings or by the provision of suitably marked samples — similarly, any areas on which autocatalytic nickel coatings may not be present shall be indicated;

d) the sampling procedure to be adopted (see clause 8);

e) the methods of adhesion testing to be employed (see 6.5);

f) the nominal composition or specification and metallurgical condition of the basis metal.

4.2 Additional information

The following additional information may be required and, if so, shall be specified by the purchaser:

a) any requirements for stress-relieving treatment before autocatalytic plating and/or hydrogen embrittlement reduction after plating (see 5.2 and 7.2);

b) any requirements for treatment to induce compressive stress (for example peening before plating) (see 5.3);

c) any special requirements for, or restrictions on, pretreatment;

d) any special requirements for, or restrictions on, heat treatment (see 5.2 and clause 7);

e) any requirements for maximum coating thickness, especially for the build-up of worn or over-machined parts. It shall also be specified whether these thicknesses shall be as-plated, or as obtained after any machining of the coating;

f) the necessity for degaussing (demagnetizing) steel parts before plating to minimize the inclusion of magnetic particles or swarf into the coating;

g) the final surface roughness of the coating (see 6.1 and 6.2);

h) the hardness of the coating and the method of test to be used in verification (see 6.4, clause 7 and annex J);

i) the type, size, extent and location of permissible surface defects in the coating (see 6.1);

j) any special requirements for the chemical composition of the coating (see 6.11);

k) any requirements for corrosion resistance;

l) any requirement in respect of porosity and, where relevant, the method of test (see 6.6 and annex C);

m) any requirements for wear resistance (see 6.9 and annex L);

n) any requirements for solderability (see 6.10 and annex F);

o) any other special requirements.

5 Treatment of basis metal before plating

5.1 Surface condition

The significant surfaces shall be examined by the plater using the unaided eye or corrected vision for visible surface defects which may be detrimental to the final finish. Any defects shall be brought to the attention of the purchaser before processing.

5.2 Stress relief before plating

5.2.1 Before being plated, steel parts shall be stress relieved if specified by the purchaser. The appropriate conditions given in table 1 shall be used, unless otherwise specified.

Table 1 — Heat treatment conditions for stress relief before plating (excluding surface-hardened parts)

Maximum specified tensile strength of steel, $R_{m\,max}$	Temperature	Time (minimum)
MPa	°C	h
$R_{m\,max} \leqslant 1\,050$	None required	—
$1\,050 < R_{m\,max} \leqslant 1\,450$	190 to 220	1
$1\,450 < R_{m\,max} \leqslant 1\,800$	190 to 220*	18*
$1\,800 < R_{m\,max}$	190 to 220	24

* Or for a shorter period at a higher temperature.

5.2.2 If stress relief is given after shot peening, the temperature shall not exceed 220 °C.

5.2.3 Surface-hardened parts shall be heat treated at 130 to 150 °C for not less than 5 h, or for shorter periods at higher temperatures if the resulting loss of surface hardness of the substrate is acceptable.

5.2.4 Stress-relieving heat treatment is not normally required for non-ferrous metals. However, brass and copper alloys that undergo stress or surface cracking in the presence of ammonia or solutions of ammonium salts shall be stress relieved.

5.3 Shot peening

5.3.1 General requirement

If peening is specified to induce compressive stress, an appropriate method shall be applied as specified by the purchaser (see 4.2.6). In order to prevent different stress conditions at the surface, there shall be 100 % coverage during peening.

NOTE — Since autocatalytic nickel-phosphorus coatings can cause a serious loss of fatigue strength, the introduction of compressive stresses into the surface of parts by shot peening is generally beneficial for both sustained load and fatigue properties. The loss of fatigue strength is due to delayed crack propagation from the coating to the base metal during cyclic loading. The introduction of compressive stresses into the surface of the part by shot peening will help to minimize the loss of fatigue strength of parts subsequently requiring autocatalytic nickel-phosphorus plating.

Peening using steel balls may produce inclusions in the surface of the substrate which may adversely affect the corrosion resistance of the coating.

5.3.2 Requirements for steels

If shot peening is necessary to improve the fatigue strength, the peening intensity, unless otherwise specified (see the note), shall be such that, when measured by the method described in annex H, the arc height is at least

— 0,3 mm for steels of tensile strength less than 1 100 MPa

— 0,4 mm for steels of tensile strength 1 100 MPa or greater.

Unless otherwise specified the peening shall be performed so that the area concerned is completely covered, that is, the ball marks completely overlap each other.

NOTE — Lower intensities may be necessary on thin sections to avoid distortion, but may not be fully effective in avoiding loss in fatigue strength.

5.3.3 Requirements for non-ferrous metals

For non-ferrous metals, the peening intensity shall be specified by the purchaser.

5.4 Undercoats

Undercoats may be necessary on certain basis metals for any of the following reasons:

a) to promote adhesion;

b) to prevent diffusion;

c) to prevent plating bath contamination.

To prevent diffusion and process contamination, a continuous undercoating of thickness 2 to 5 µm of either electroplated copper or electroplated nickel shall be used. Such undercoats shall be applied on basis metals containing more than trace quantities of antimony, arsenic, bismuth, cadmium, lead, magnesium, tin, and zinc, except bronzes and brasses.

To promote adhesion of certain metals, a continuous undercoat of thickness up to 2 µm of either electroplated copper or electroplated nickel shall be used. Such undercoats shall be applied on basis metals containing more than trace quantities of chromium, lead, molybdenum, nickel, tin, titanium or tungsten.

6 Requirements for coatings

6.1 Appearance

If specified by the purchaser, the appearance of the significant surface in the as-plated condition shall be bright, semi-bright, or dull. When examined by the unaided eye or corrected vision, the surface shall be uniform and free from defects such as pits, cracks, blisters, exfoliation, or growths except when permitted [see 4.2 g) and i)].

NOTE — Defects which are present on the basis metal before plating, including hidden defects, may be reproduced by the coating (see 5.1). In addition, stains and discolorations may result from post-plating heat treatment. It is advisable for the interested parties to agree on the acceptability of such defects.

6.2 Roughness

If the roughness is specified by the purchaser, the method of measurement shall be that given in ISO 468.

NOTE — It should be recognized that the surface finish of the coating is not usually superior to that of the substrate before plating.

6.3 Thickness

The finished minimum thickness of the autocatalytic nickel-phosphorus alloy plated on the significant surface and of any undercoat(s), and the method of measurement, shall be specified by the purchaser [see 4.1 b) and clause A.1]. Guidance on thicknesses to be used is given in annex K.

If the purchaser specifies the use of coupons plated simultaneously, as a measure of the autocatalytic nickel coating thickness, this shall be permitted.

6.4 Hardness

Hardness values, if required, shall be measured after heat treatment, and shall be carried out by the method specified in ISO 4516. The deposit hardness measured shall be within ± 10 % of that specified by the purchaser (see annex J and 7.3).

6.5 Adhesion

The autocatalytic nickel-phosphorus coating shall be adherent to the basis material. Coatings shall be capable of passing one or more of the adhesion tests given in annex B, as specified by the purchaser [see 4.1 e) and 7.4] in accordace with the following criteria.

a) When tested using the bend test described in B.1, no detachment of the coating shall occur. Cracks in the coating on the tension side of the bend shall not be taken as an indication of inadequate adhesion;

b) When tested using the thermal shock test described in B.2 no blistering or peeling of the coating shall occur;

c) When tested using the punch test described in B.3, no blistering or flaking of the coating shall occur;

d) When tested using the file test described in B.4, no lifting of the coating shall be observed.

6.6 Porosity

When tested by the appropriate method described in annex C and evaluated in accordance with ISO 1462 and ISO 4540, the rating shall normally be not lower than 8, unless otherwise specified. However, in some critical applications requiring a total absence of porosity, the rating shall be not lower than 10.

6.7 Corrosion protection of the substrates

When specified, autocatalytic nickel-phosphorus coated parts may be subjected to one of the tests specified in ISO 3768, ISO 3769, ISO 3770, ISO 4538, or ISO 4541. The results of the test employed shall be evaluated in accordance with ISO 1462 and ISO 4540 and the acceptance level stated by the purchaser.

6.8 Corrosion resistance of the coating

If required, the corrosion resistance of the coating shall be specified by the purchaser, together with the method of testing and evaluation [see 4.2 k) and annex G].

6.9 Wear resistance

If required, the wear resistance of the coating shall be specified by the purchaser, together with the method of testing and evaluation [see 4.2 m)]. Annex L gives guidance on the different test methods that are available.

6.10 Solderability

If required, the solderability of the coating shall be specified by the purchaser, together with the method of testing and evaluation [see 4.2 n) and annex F].

6.11 Chemical composition

If required, the chemical composition of the nickel-phosphorus alloy shall be specified by the purchaser [see 4.2 j) and annex D]. If the chemical composition is not specified, coatings shall be supplied according to table 2 and shall conform to the range specified as typical. In this case, verification of chemical composition will not normally be supplied.

Table 2 — Chemical composition of the deposit

Values as percentages by mass

Element	Composition		
	Minimum	Maximum	Typical
Nickel	85	98	88 to 95
Phosphorus	2	15	5 to 12
Other elements (Al, As, B, Bi, C, Cd, Co, Cr, Cu, Fe, H, Mn, Mo, N, Nb, Pb, S, Sb, Se, Si, Sn, V, Zn)*	0	2	0,05

* For certain specific applications, control of these elements within the deposit may be required. Close communications with engineering, quality control and purchasing departments will help with problems not covered by the essential information when controlling these elements.

7 Heat treatment after plating

NOTE — Work is at present being undertaken that may further refine the contents of this clause.

7.1 General

When required by the purchaser, heat treatment after coating shall be carried out

a) to give hydrogen embrittlement relief (see 7.2);

b) to increase the hardness of the coatings (see 7.3);

c) to increase the adhesion of the coatings on certain substrates (see 7.4).

If heat treatment for hardening of the coating is carried out, separate hydrogen embrittlement reduction heat treatments may not be needed (see 7.2). Heat treatment shall be carried out before any mechanical finishing. Delay in applying heat treatment is particularly undesirable with respect to steels of tensile strength of 1 400 MPa and greater (see table 3).

7.2 Heat treatment for hydrogen embrittlement relief after plating

7.2.1 The heat treatment of plated steel articles shall be in accordance with the requirements given in table 3.

The timing of heat treatment shall start from when the article reaches the required temperature and shall not include cooling time.

Table 3 — Heat treatment conditions for hydrogen embrittlement relief after plating (excluding surface-hardened parts)

Maximum specified tensile strength of steel, $R_{m\,max}$	Temperature	Time	Maximum permissible delay after plating
MPa	°C	h	h
$R_{m\,max} \leqslant 1\,050$	None required	—	—
$1\,050 < R_{m\,max} \leqslant 1\,450$	190 to 220	8	8
$1\,450 < R_{m\,max} \leqslant 1\,800$	190 to 220	18	4
$1\,800 < R_{m\,max}$	190 to 220	24	0

7.2.2 Unpeened parts may be heated for a shorter period at a higher temperature if such conditions have been shown to be effective in reducing hydrogen embrittlement. However, hardening of the deposit will also occur as a consequence (see annex J).

Parts shall not be heat treated to a temperature higher than 50 °C below their tempering temperature.

7.2.3 Surface-hardened parts shall be heat treated at 190 to 220 °C for not less than 1 h, or at a higher temperature if the resulting loss of surface hardness of the substrate is acceptable.

7.2.4 The specified parts shall be tested after heat treatment by one of the methods described in annex E.

7.3 Heat treatment to increase hardness

The application of heat treatment to increase the hardness of autocatalytic nickel-phosphorus coatings is summarized in table 4.

Table 4 — Application of heat treatment to increase hardness of autocatalytic nickel coatings (see annex J)

Hardness range (HV)	Nickel-phosphorus
500 minimum	As deposited
600 to 800	Heat treatment as in annex J
800 to 1 100*	Heat treatment as in annex J

* Coatings in this hardness range may be micro-cracked.

7.4 Heat treatment to improve adhesion

Heat treatment to improve the adhesion of autocatalytic nickel-phosphorus coatings on certain basis metals shall be carried out as specified by the purchaser or by following the recommendations given in table 5 for coatings of thickness 50 µm or less on alloys not affected by the temperature given. (Thicker coatings will require proportionately longer times.)

Table 5 — Recommendations for heat treatment to improve adhesion

Material	Time	Temperature
	h	°C
Beryllium and beryllium alloys	1 to 1,5	155 ± 5
	4	140 ± 5
Age-hardened aluminium and aluminium alloys	1 to 1,5	130 ± 10
Non age-hardened aluminium and aluminium alloys	1 to 1,5	160 ± 10
Magnesium and magnesium alloys	2 to 2,5	190 ± 10
Copper and copper alloys	1 to 1,5	190 ± 10
Nickel and nickel alloys	1 to 1,5	230 ± 10
Titanium and titanium alloys	10	280 ± 10
Carbon and alloy steels	1 to 1,5	210 ± 10
Molybdenum and molybdenum alloys	2 to 2,5	200 ± 10

8 Sampling

8.1 A sampling programme as specified in ISO 2859 or ISO 4519 shall be selected from the inspection lot unless the purchaser and plater agree to another representative sampling plan.

8.2 When the sampling plan requires the plating of separate test specimens, these shall be plated simultaneously with the articles.

NOTE — The autocatalytic nickel process is subject to rapid changes in solution concentration and daily sampling of the coating deposited is recommended. For coatings specified to have a certain phosphorus content and those requiring corrosion testing, more frequent sampling should be considered as an option [see 4.2 j) and 4.2 k)].

All such specimens used in the sampling plan shall be made of the same basis material as articles being plated to this specification.

8.3 All specimens shall be provided by the purchaser unless otherwise agreed by the plater.

Annex A

Determination of coating thickness

(This annex forms an integral part of the standard.)

A.1 General

The thickness shall be measured at any place on the significant surface designated by the purchaser and the measurement shall be made with a measurement uncertainty of less than 10 % by a method selected by the purchaser. If there is a dispute regarding the thickness, the referee method shall be that specified in clause A.3.

A.2 "Weigh, plate, weigh" method

A.2.1 Principle

Plating of a test specimen of similar substrate material to the article being plated. Determination of the increase in mass due to plating. Calculation of the thickness of the plating.

A.2.2 Procedure

A.2.2.1 Test specimen

Determine the area of the test specimen and ensure that it consists of a substrate material similar to that of the article being plated.

A.2.2.2 Determination

Weigh, to at least the nearest 0,001 g, the clean, dry test specimen. Plate it under process conditions. Clean and dry the plated test specimen and weigh it to a similar accuracy, at the same temperature used for weighing the uncoated article.

A.2.3 Expression of results

The coating thickness, in millimetres, is given by the formula

$$\frac{10\,(m_2 - m_1)}{\varrho\, A}$$

where:

m_1 is the mass, in grams, of the test specimen before plating;

m_2 is the mass, in grams, of the plated test specimen;

ϱ is the density, in grams per cubic centimetre, of the coating (see table 6);

A is the area, in square centimetres, of the plated surface of the test specimen.

NOTE — The density of the coating varies with its phosphorus content. Typical values are given in table 6.

Table 6 — Density of typical nickel-phosphorus coatings

Phosphorus content	Density
% (m/m)	g/cm^3
6,0	8,0
9,0	7,85
12,5	7,68

A.3 Metallographic sectioning

Use the method specified in ISO 1463.

A.4 Direct dimensional measurements before and after plating

A.4.1 Principle

Measurement of the thickness at a specific position on a part or test specimen before and after plating. Calculation of the thickness of the coating.

A.4.2 Procedure

A.4.2.1 Test specimen

The test specimen shall be either a part or a test coupon of similar substrate material to the part.

A.4.2.2 Determination

Using a suitable measuring device, for example a micrometer, measure, to the nearest 0,002 mm, the thickness of the test specimen at a specific position. Plate it under process conditions. Clean and dry the plated test specimen and measure its thickness, at the same position and to a similar accuracy, at the same temperature used for measurement of the uncoated article.

A.4.2.3 Expression of results

The coating thickness, in millimetres, is given by the formula

$$\delta_2 - \delta_1$$

where

δ_1 is the thickness, in millimetres, of the test specimen before plating;

δ_2 is the thickness, in millimetres, of the plated test specimen.

In the case of test specimens plated on both sides, divide the figure obtained from the above formula by a factor of 2.

A.5 Coulometric method

A.5.1 Use the method specified in ISO 2177. Use the solution recommended by the instrument manufacturer.

NOTE — A 600 g/l solution of sodium nitrate in distilled or deionized water has been found to be suitable for use with some instruments. It is nevertheless essential to check its suitability before using it with a particular instrument.

A.5.2 The instrument shall be calibrated against the coating to be used as the result is significantly influenced by a change in phosphorus content or heat treatment.

NOTE — This method is only recommended for use on steel substrates having a maximum coating thickness of 10 µm in the "as-deposited" condition.

A.6 Beta backscatter method

Use the method specified in ISO 3543.

The coating thickness can be measured by the use of a beta backscatter device. The use of the beta backscatter method is restricted to basis metals which have an atomic number less than 18 or greater than 40. The actual phosphorus content of the coating shall be taken into consideration; consequently, the measuring device shall be calibrated using standard specimens of the same substrate having the same phosphorus content in the coating as the articles to be tested.

A.7 Magnetic method

The method specified in ISO 2178 is applicable to magnetic substrates plated with autocatalytic nickel deposits which contain more than 9 % (m/m) phosphorus (non-magnetic) and which have not been heat treated. The instrument shall be calibrated with deposits plated in the same bath, on steel, and whose thickness has been determined by the microscopic method described in ISO 1463.

A.8 Scanning electron microscope method

A.8.1 Applicability

This clause specifies a method for the determination of metallic coating thicknesses by examination of cross-sections with a scanning electron microscope (SEM). It is destructive and has an uncertainty of less than 10 % or 0,1 µm, whichever is the greater.

A.8.2 Definitions

For the purpose of this clause, the definitions of ISO 2064 apply.

A.8.3 Principle

Cutting, grinding and polishing of a test specimen for metallographic examination by a SEM of a cross-section of the coating.

The measurement is made on a conventional micrograph or a photograph of the video waveform signal for a single scan across the coating.

A.8.4 Apparatus

A.8.4.1 Scanning electron microscope (SEM), having a resolution capability of 50 nm. Suitable instruments are available commercially.

A.8.4.2 SEM stage micrometer, for calibration of the magnification of the SEM. The stage micrometer shall have a measurement uncertainty of 5 % or better for magnifications of X 1 000 to X 20 000. Suitable stage micrometers are available commercially.

A.8.5 Procedure

A.8.5.1 Factors influencing the measurement results

The following factors may affect the accuracy of a measurement of coating thickness.

A.8.5.1.1 Surface roughness

If the coating or its substrate is rough relative to the coating thickness, one or both of the interfaces bounding the coating cross-section may be too irregular to permit accurate measurement of the average thickness in the field view.

A.8.5.1.2 Taper of cross-section

If the plane of the cross-section is not perpendicular to the plane of the coating, the measured thickness will be greater than the true thickness. For example, an inclination of 10° to the perpendicular will contribute a 1,5 % error.

A.8.5.1.3 Specimen tilt

Any tilt of the specimen (plane of cross-section) with respect to the SEM beam may result in an inaccurate measurement. The instrument should always be set for zero tilt.

A.8.5.1.4 Deformation of coating

Detrimental deformation of the coating can be caused by excessive temperature or pressure during the mounting and preparation of cross-sections of soft coatings or coatings melting at low temperatures, and by excessive abrasion of brittle materials during the preparation of cross-sections.

A.8.5.1.5 Rounding of edges of coating

If the edge of the coating cross-section is rounded, i.e. if the coating cross-section is not completely flat up to its edges, the observed thickness may differ from the true thickness. Edge rounding can be caused by faulty mounting, grinding, polishing, or etching. (See A.9.1 and A.9.2.)

A.8.5.1.6 Overplating

Overplating of the test specimen serves to protect the coating edges during preparation of cross-sections and thus to prevent an inaccurate measurement. Removal of the coating material during surface preparation for overplating can cause a low thickness measurement.

A.8.5.1.7 Etching

Optimum etching will produce a clearly defined and narrow dark line at the interface of the two metals. A wide or poorly defined line can result in an inaccurate measurement.

A.8.5.1.8 Smearing

Polishing may leave smeared metal that obscures the true boundary between two metals and results in an inaccurate measurement. This may occur with soft metals like lead, indium, and gold. To help identify whether or not there is smearing, repeat the polishing, etching, and measurement several times. Any significant variations in readings indicates possible smearing.

A.8.5.1.9 Poor contrast

The visual contrast between metals in a SEM is poor when their atomic numbers are close together. For example, bright and semi-bright nickel layers may not be distinguishable unless their common boundary can be brought out sufficiently by appropriate etching and SEM techniques. For some metal combinations, energy dispersive X-ray techniques may be helpful. (See A.10.)

A.8.5.1.10 Magnification

For a given coating thickness, measurement errors tend to increase with decreasing magnification. If practical, the magnification should be chosen so that the field of view is between 1,5 and 3 times the coating thickness.

The magnification readout of a SEM is often poorer than the 5 % accuracy often quoted and the magnification has been found for some instruments to vary by 25 % across the field.

Magnification errors are minimized by appropriate use of a SEM stage micrometer.

A.8.5.1.11 Uniformity of magnification

Because the magnification may not be uniform over the entire field, errors can occur if both the calibration and the measurement are not made over the same portion of the field. This can be very important.

A.8.5.1.12 Stability of magnification

A.8.5.1.12.1 The magnification of a SEM often changes or drifts with time. This effect is minimized by mounting the stage micrometer and test specimen side by side on the SEM stage so as to keep the transfer time short.

A.8.5.1.12.2 A change in magnification can occur when adjustments are made with the focusing and other SEM electronic controls. Such a change is prevented by not using the focus controls or other SEM electronic controls after photographing the stage micrometer scale except to focus with the x, y and z controls of the stage. Appropriate manipulation of x, y and z controls will bring the specimen surface to the focal point of the SEM beam.

A.8.5.1.13 Stability of micrographs

Dimensional changes of micrographs can take place with time, and with temperature and humidity changes. If the calibration micrograph of the stage micrometer scale and the micrograph of the test specimen are kept together and time is allowed for stabilization of the photographic paper, errors from this source will be minimized.

A.8.5.2 Preparation of cross-sections

Prepare, mount, grind, polish and etch the test specimen so that:

a) the cross-section is perpendicular to the plane of the coating;

b) the surface is flat and the entire width of the coating image is simultaneously in focus at the magnification to be used for the measurement;

c) all material deformed by cutting or cross-sectioning is removed;

d) the boundaries of the coating cross-section are sharply defined by no more than contrasting appearance, or by a narrow, well defined line;

e) if the video waveform signal is to be measured, the signal trace is flat except across the two boundaries of the coating.

NOTE — Further guidance on the preparation of test specimens is given in clause A.9.

A.8.5.3 Calibration of instruments

A.8.5.3.1 General

Before use, the SEM shall be calibrated with the SEM stage micrometer (A.8.4.2). Appropriate attention shall be given to