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Metallic coatings — Review of methods of measurement of ductility

Revêtements métalliques — Vue d'ensemble sur les méthodes de mesurage de la ductilité

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Foreword

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Metallic coatings — Review of methods of measurement of ductility

1 Scope and field of application

1.1 This International Standard specifies general methods of measuring the ductility of metallic coatings of thickness below 200 µm prepared by electroplating, autocatalytic deposition or other processes (see the note).

The methods of measuring the ductility of metallic coatings can be divided into two main categories:

— tests on unsupported foils (separated from the substrate);

— tests of coatings on substrates.

NOTE — When specific methods of 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.

1.2 In the testing of unsupported foils separated from the substrate (see figure 1), the foils may consist of one or more metallic layers. Therefore it is possible to measure the ductility of composites and to determine the influence of individual layers on overall ductility. Methods of testing of unsupported foils are described in clause 3. Methods of producing foils for testing are discussed in annex A.

1.3 In the testing of coatings on substrates (see figure 2), it is especially important to determine the exact point of crack initiation of the top layer. Attention is drawn to different methods of discerning this point, by normal or corrected-to-normal vision or with a lens. See the guidance in the individual methods. These methods can also be used to detect embrittlement of the substrate that may have resulted from the coating process. Methods of testing of coatings on substrates are described in clause 4.

1.4 Although ductility is a property of the material and independent of the dimensions of the test piece, thickness of the coating may have an influence on the value of linear elongation ($\Delta l/l_0$).

1.4.1 Very thin layers have different properties as the build-up of the initial layers will be influenced by the properties of the substrate (epitaxy). High internal stresses may be incorporated into the initial layers and these may affect ductility.

1.4.2 It is essential that the test piece has uniform thickness, as thinner spots will give rise to premature cracking. Also, the current density is lower at thinner parts and higher at thicker parts of electroplated test pieces; in this way current density differences may result in different ductilities. The current density applied should be maintained as uniform as possible over the test piece, and its value reported.

2 Definitions

For the purpose of this International Standard, the following definitions apply.

2.1 ductility: The ability of a metallic or other coating to undergo plastic or elastic deformation, or both, without fracture or cracking.

2.2 linear elongation: The ratio of the elongation, Δl, to a definite initial length, l_0, of the test piece. This is taken as a measure of ductility.

Often this ratio is expressed as a percentage.

NOTE ON MEASURES OF DUCTILITY

Normally the test pieces are elongated. With some bending tests, the outer layer of the test piece, i.e. the plating, is elongated (see figure 3). In bulge tests, however, the surface of the foil is enlarged, requiring calculation of linear elongation from the reduction in the thickness. Using the component of deformation (stretching) in only one axis would give false information about the ductility of the material (see figure 4). In those cases the thinning of the foil, as calculated from the increase in the surface area, is a better measure of the ductility of the material (see annex B).

3 Tests on unsupported foils

These techniques involve measurement of a foil which has been separated from the substrate (see figure 1). In this case, the foil to be tested can also consist of several layers so as to allow measurement of the influence of undercoats on the ductility of the foil sandwich. Examples are gold flash on gold/copper alloys and chromium-plated nickel deposits. Methods of producing unsupported foils are given in annex A.

Five methods are described.

3.1 Tensile testing

3.1.1 Principle

Determination of the linear elongation of a foil, which is clamped into the jaws of a tensile testing machine. In this type of stressing the foil is lengthened, but both the width and the thickness of the foil diminish.

3.1.2 Apparatus

This method may utilize conventional mechanical testing equipment, available commercially and in many metallurgical laboratories.[1] For some applications, tensile testing equipment adapted to microscopic inspection during the test may be used.

3.1.3 Preparation of test pieces

Test pieces may be machined, chipped, punched or cut from the metallic foil or prepared by photoprinting with the help of light-sensitive lacquers or light-sensitive foils which are pressed onto a suitable substrate. After developing the pattern of the test piece it is plated into the final form. A similar method uses chemical or electrochemical milling of the desired shape from a foil on which has been applied a suitable resist by silk screen printing or by applying a photosensitive resist. These last methods are widely used in the printed circuit industry. The test pieces are usually rectangular in shape, but can be widened at both ends to avoid breaking in the clamping jaws (see figure 7).

Some methods of preparing the test pieces may cause microcracking at the edges that results in premature failure and erratic results. Test piece preparation involving photoprinting or electroforming is preferred to avoid edge defects.

Test pieces plated into the final form may have thicker edges unless shielding and other techniques are used to ensure uniform current distribution (see figure 10).

Make equidistant marks on the surface of the test piece as illustrated in figure 7. Determine the distance between the marks before testing.

3.1.4 Procedure

Clamp the test piece between the jaws of the tensile test equipment and apply strain using a selected cross-head speed. Determine the distance between the marks on the test pieces after testing (see figure 8).

3.1.5 Expression of results

3.1.5.1 Calculation

The ductility, D, expressed as a percentage, is given by the equation

$$D = \frac{l_1 + l_2 - l_0}{l_0} \times 100$$

where

l_0 is the distance between the marks before testing;

$l_1 + l_2$ is the distance between the marks after testing.

3.1.5.2 Coefficient of variation

Mechanically prepared test pieces can have coefficients of variation, $s/\bar{D}$ (where s is the standard deviation and $\bar{D}$ the mean ductility), as high as 20 %.

By plating into the final form using shields to ensure uniform current distribution, test pieces can be produced which have lower coefficients of variation.

3.1.6 Notes on procedure

3.1.6.1 Necking of the test piece (see figure 8) may require measurement of very small changes in length and the use of a microscope that has a vernier scale.

3.1.6.2 Mounting fragile thin test pieces into the jaws of a tensile testing machine may give rise to prestressed test pieces which thereby diminish the real value of elongation.

3.1.6.3 Care shall be taken to avoid twisting of the test piece (see figure 9).

3.1.6.4 When these sources of error (3.1.6.1 to 3.1.6.3) cannot be eliminated, other methods of measuring ductility should be used.

3.2 Bending (micrometer bend test)

3.2.1 General

This method is suitable only for the evaluation of metallic foils having low ductility.[2] The values obtained have no simple relation to values obtained by other methods. This method is useful for brittle metals such as bright nickel.

3.2.2 Apparatus

Micrometer.

3.2.3 Preparation of test pieces

Cut strips of 0,5 cm × 7,5 cm from the foil under test. The foils are usually 25 to 40 µm thick. The difficulties described in 3.1.3 and 3.1.6 apply likewise to this test. Measure the thickness of the test piece at the point of bending, using an instrument or method which enables the thickness to be determined to within 5 % of its nominal value.

3.2.4 Procedure

Bend the test piece into a U-shape and place it between the jaws of the micrometer so that as the jaws are closed the bend

remains between the jaws. Close the micrometer jaws slowly until the foil cracks.

Record the micrometer reading and the thickness of the foil (see figure 11).

Carry out the test at least in duplicate.

3.2.5 Expression of results

3.2.5.1 Calculation

Calculate the average of the micrometer readings (see 3.2.4).

The ductility, D, expressed as a percentage, is given by the equation (see figure 12)

$$D = \frac{\delta}{2\bar{r} - \delta} \times 100$$

where

δ is the thickness of the test pieces;

$2\bar{r}$ is the average of the micrometer readings.

3.2.5.2 Precision

As the value of D rises more rapidly than δ, it is essential that the value of δ be measured with high precision. If a foil of 20 µm is read as 25 µm, the following difference, supposing $2\bar{r}$ = 0,5 cm, will be found:

$$D_1 = \frac{20 \times 10^{-4}}{0{,}5 - 20 \times 10^{-4}} \times 100 = 0{,}4\ \%$$

$$D_2 = \frac{25 \times 10^{-4}}{0{,}5 - 25 \times 10^{-4}} \times 100 = 0{,}5\ \%$$

i.e. a difference of 0,5 − 0,4 = 0,1 %.

A thickness of 25 µm will give results that are 25 % higher than for a 20 µm thickness.

It is obvious that this method will give reproducible results only when δ is measured to within 1 µm and $2r$ to within 0,01 cm.

3.3 Folding (vice-bend test)

3.3.1 General

Although this test is simple and may have some utility, the nature of the test, the cold working that occurs as a result of bending, and other factors may lead to incorrect measures of ductility. The thickness of the test piece affects the results, but the influence of thickness cannot be calculated.

3.3.2 Apparatus

Machinist's vice, equipped with two small machined jaws to hold the test piece (see figure 13).

3.3.3 Preparation of test pieces

Cut rectangular strips, 1 cm wide by 5 cm long, from the metal foil.

3.3.4 Procedure

Grip the test piece between the jaws of the vice. Bend the test piece sharply through 90°, then bend it successively in opposite directions through 180° until fracture occurs.

3.3.5 Results

The number of bends is taken as a measure of ductility.

3.4 Hydraulic bulging

3.4.1 General

Hydraulic bulge-testing can be used to measure the ductility of thin sheet materials accurately. No machining of the test piece is required, there are no problems of achieving axial alignment as in tensile testing, and the test is especially useful for measuring the ductility of ductile materials. Until recently, the lack of commercially available equipment has prevented wider use of this method. [3]

3.4.2 Principle (see figure 14)

Clamping of a test piece between a bottom cylinder and an upper platen. The upper platen has a circular opening of the same diameter as the cylinder. Increasing the water pressure slowly and steadily to deform the test piece into a bulge or dome until the foil bursts.

3.4.3 Apparatus

See figure 15.

3.4.4 Procedure

With the equipment shown schematically in figure 15, fill the bottom cylinder with water to the rim. Place the test piece on the surface of the water. Use the upper platen, in the shape of a hollow cone, to clamp the test piece firmly in position.

Fill the hollow cone with water from the reservoir that is provided. The excess water will rise in the glass gauge. When the level of the water is above the light-sensing device, close the valve that controls the flow of water from the reservoir. Turn the motor on and slowly raise the light-sensing device. When the device is aligned with the meniscus, the beam of light within the device will be deflected; the drop in voltage that occurs as a result of this shuts off the motor.

The pressure under the test piece is increased by means of the plunger. When the meniscus in the glass gauge begins to rise, the motor will automatically begin to operate and the light-sensing device will track the rise in the level of water. By means of the potentiometer, record the increase in volume on an x-y recorder.

A pressure sensor in the cylinder simultaneously records the pressure beneath the test piece. In a commercial version of the equipment, a pressure-sensitive switch is used to shut the motor off at the moment of bursting so that the total volume of displaced water can be read directly from the digital display on the potentiometer.

3.4.5 Expression of results

3.4.5.1 Calculation

The ductility of the metal specimen may be calculated from the volume of the displaced water, which equals the volume inside the dome. The ductility, expressed as a percentage, is given by the equation derived and discussed in annex C. The tensile strength of the metal foil may also be determined from the value of the pressure at bursting (see annex C).

3.4.5.2 Coefficient of variation

Because only the centre of the foil (ϕ 3 cm) is tested, the current density and the thickness in this region are probably more constant than in the case of tensile tests. Values of $s/\bar{D}$ = 0,05, i.e. 5 %, are easily arrived at.

3.4.6 Notes on procedure

Pinholes in the test piece are one possible source of error. Pinholes can be detected prior to testing by "candling". A 100 W light bulb in a box with a hole slightly smaller in diameter than the opening in the top plate or cone is satisfactory.

When pinholes are present, it is possible to underlay the test piece with a very thin plastic foil which will stop the water from passing through the pinholes.

By visual observation it is possible to note the moment of cracking.

Stopping the motor of the light-sensing device at this moment will give fair indication of the ductility of the porous foil.

3.5 Mechanical bulging

3.5.1 General

Mechanical bulge tests are similar to hydraulic bulge tests. The dome, however, is formed mechanically as indicated schematically in figure 16.

3.5.2 Apparatus

Equipment for measuring the ductility of thin metal foils is not readily available, but can be easily assembled.

Two types of apparatus are used. The simplest one consists of a micrometer, a spindle extension with a steel ball and a pair of circular plates each with a round opening at the centre[4] (see figure 17).

3.5.3 Procedure

Place a foil test piece between the circular plates. Clamp the upper and lower plates together firmly by means of two screws. Then slowly push the test piece upward by turning the micrometer. Read from the micrometer the distance travelled by the steel ball from the initial contact with the metal film to the point of crack initiation.

The initial contact point between the steel ball and the test piece is detected electrically. A battery-operated lamp is fitted into the upper brass plate in such a way that the lamp lights at the instant the steel ball touches the test piece. The lamp stays lit throughout the test.

The visual detection of the initiation of rupture is accomplished with the aid of a magnifier (X 15) attached to the upper plate (but not shown in figure 17).

3.5.4 Expression of results

It is possible to calculate the ductility from the height of the cone by calculating the loss of thickness of the foil (see annex D).

3.5.5 Special cases

It may be preferable to use a slightly altered procedure. In the apparatus shown schematically in figures 18, 19 and 20, the steel ball remains stationary, but the two plates and the sample are moved downward with a motor until the test piece cracks.

The instrument is placed under a microscope which enables use of X 70 magnification when looking for the first cracks. At the start of the test, the motor stops when electrical contact between the steel ball and the test piece is made. At the moment of cracking the motor is stopped by hand. The height of the cone is measured by the displacement of a linear potentiometer with a resolution of 5 µm.

With the motor-driven apparatus, it is easier to obtain good results because

a) there is no twisting moment of the steel ball against the foil, which will be the case when turning the micrometer screw;

b) a microscope, preferably with interference lighting of the Nomarski-type, will indicate the moment of appearance of the first cracks with more reliability;

c) electrical measurement of the height of the cone is more precise than a micrometer;

d) better lighting and the fact that the distance between the microscope and the summit of the cone is constant give more reproducible data than in the case of the micrometer instrument. A value of $s/\bar{D}$ = 0,05, i.e. 5 %, is easily arrived at.

4 Tests on coatings on substrates

These techniques involve testing a foil on a substrate (see figure 2). In this case, the utmost care has to be taken to

find the exact point at which the top layer cracks. Attention is drawn to different methods of detecting this point, by normal or corrected-to-normal vision, or with a lens. See the guidance given in the descriptions of the individual methods.

Unless, therefore, very brittle electrodeposits are being assessed, the substrate has to be very ductile. Annealed copper or brass, or a suitable plastics material such as ABS, are preferred substrates. If the substrate is electroplated ABS, the moment of cracking can be found exactly by recording the electrical resistance of the test layer during the test (see figure 21). In certain cases, the purpose of the test is to measure the embrittlement of the substrate by the electroplating process, for example hydrogen embrittlement of zinc-coated steel.

Using these methods will avoid many of the drawbacks caused by handling very thin foils, but the problem of ascertaining the moment of cracking of the top layer arises.

Seven methods are described.

4.1 Tensile testing

4.1.1 Apparatus

See 3.1.2.

4.1.2 Preparation of test pieces

The coatings are deposited adherently on a substrate that shall be more ductile than the coatings.

Test pieces shall be machined and the sides can be polished, thus preventing edge-cracking. Necking is considerably reduced and it is easy to mount the test piece into the jaws of the tensile machine avoiding misalignment.

4.1.3 Procedure

See 3.1.4.

It is difficult to determine the exact moment of crack initiation, although in relatively brittle, high gloss electrodeposits like nickel it can be ascertained by visual observation. [5] In the case of electrodeposited coatings on plastics, the moment of cracking can be found exactly by recording the electrical resistivity of the metallic coatings during testing (see figure 21).

4.2 Three-point bending [6]

4.2.1 Principle

Application of a force transversely to the test piece in the portion that is being bent, usually at the centre of the test piece.

4.2.2 Apparatus

Bending forces may be applied by one of the three arrangements illustrated in figure 22. Various devices to hold the test piece are used for each of the loading arrangements. The devices may be mounted on a universal testing machine, or a special bending apparatus may be used.

4.2.3 Procedure

Inspect the surface periodically to determine the exact moment when the coating cracks. This is difficult to do unless a method of continuously observing the test piece during bending is incorporated in the test device.

A major source of error is the tendency of the specimen to kink or hinge in the test as illustrated in figure 23 a).

4.2.4 Expression of results

If kinking does not occur and the onset of cracking is accurately detected, the ductility, D, expressed as a percentage, is given by the equation

$$D = \frac{4\delta s}{l^2} \times 100$$

where

δ is the total thickness;

s is the vertical displacement;

l is the gauge length.

See figures 12 and 23 b).

4.3 Four-point bending [7]

4.3.1 General

This is similar to three-point bend testing (4.2) except that the test piece is subjected to two loads, symmetrical over the centre portion as illustrated in figure 24. The main advantage is that kinking of the test piece is avoided.

4.3.2 Expression of results

The ductility, D, expressed as a percentage, is given by the equation

$$D = \frac{\delta s}{l_2^2 + 2\, l_1 l_2} \times 100$$

where

δ is the total thickness;

s is the vertical displacement;

l_1 is one-half the distance between the two loads;

$l_1 + l_2$ is one-half the distance between the two supports.

See figure 24.

4.4 Cylindrical mandrel bending

4.4.1 Principle

Bending of the electroplated or coated test piece in the form of a narrow strip over mandrels of decreasing diameter. [4] Determination of ductility, expressed as a percentage, from the smallest diameter mandrel that does not cause the coating to fracture. [8]

4.4.2 Apparatus

Clamps and a series of mandrels having diameters of 5 to 50 mm in 3 mm steps (see figure 25).

4.4.3 Preparation of test pieces

The substrate thickness and temper shall permit bending around the smallest diameter mandrel without evidence of cracking; for example low-carbon steel or ductile copper, 1,0 to 2,5 mm thick, may be used. The substrate is electroplated or otherwise coated and used to prepare test pieces that are 10 mm wide and at least 150 mm long.

4.4.4 Procedure

Bend the test pieces round mandrels of decreasing diameter. Record the smallest diameter mandrel which does not cause the coating to fracture.

4.4.5 Expression of results

The ductility, D, expressed as a percentage, is given by the equation

$$D = \frac{\delta}{d + \delta} \times 100$$

where

δ is the total thickness;

d is the diameter of the smallest mandrel that does not cause cracking of the coating.

4.4.6 Notes on procedure

With this method it is possible to detect cracks by performing a porosity test on strips, which are bent over different cylinders (cracks will be enlarged in the same way as pores would be enlarged). An electrolytical porosity test with a jelly would develop any microcracks into distinct lines.

4.5 Spiral mandrel bending

4.5.1 Principle

Bending an electroplated or coated strip over a mandrel with a decreasing radius. Determination of the value at which cracking of the coating is observed. [9, 10]

4.5.2 Apparatus

Mandrel (see figure 26).

4.5.3 Procedure

The moment of cracking can be determined by electrical means (see figure 21) if the substrate is a non-conductive plastic material.

4.5.4 Expression of results

The angle of the bending lever (see figure 27) can be used as a measure of ductility to determine relative values. Otherwise, the calculation of ductility, expressed as a percentage, is as in 4.4.5.

4.6 Conical mandrel bending

4.6.1 Principle

Bending a square, electroplated or coated specimen over a cone-shaped mandrel (see figure 28).

4.6.2 Apparatus

Cone-shaped mandrel.

4.6.3 Procedure

Determine the position where cracking is initiated by examining the surface of the specimen with a magnifier (X 10). Also the device can be placed under a technical microscope, where a higher magnification can be used.

4.6.4 Expression of results

The ductility, expressed as a percentage, is calculated from the radius of curvature of the cone at the point of cracking, using the equation given in 4.4.5.

Because only a plate less than about 0,5 mm thick can be bent satisfactorily, this method is not suitable for determining the ductility of a coating with an elongation greater than 11 %.

$$\frac{\Delta l}{l} \leqslant \frac{0{,}5}{4{,}5} = 0{,}11 \qquad r = 2 \text{ mm} \qquad D = 11\ \%$$
$$\delta = 0{,}5 \text{ mm} \qquad d = 4 \text{ mm}$$

4.6.5 Special cases

A variation of this test involves winding electroplated or coated copper wire around a cone (see figure 29).

4.7 Mechanical bulging

4.7.1 Apparatus

See 3.5.2.

4.7.2 Preparation of test pieces

Using very ductile copper plates 0,1 mm thick as the substrate gives satisfactory results.

4.7.3 Procedure

See 3.5.3.

Ensure that the diameter of the hole is such that the field of view of the microscope (X 100) includes the area where cracks are expected. An 8 mm diameter hole and a 4 mm diameter ball give reproducible results.

4.7.4 Expression of results

4.7.4.1 Calculation

See annex D.

4.7.4.2 Precision

The precision of the method depends on the freedom from scratches of the substrate as these scratches initiate cracks at a lower height of bulge as would be consistent with the ductility of the coating.

5 Selection of test method

5.1 It is not possible to recommend one method of measuring the ductility of coatings that is applicable to all materials and applications. The guidelines given in the table may be helpful.

The results obtained with different methods are seldom comparable.

5.2 Coatings less than 10 µm thick should be tested on a suitable substrate. For brittle deposits, the tensile testing method is preferred but the bending tests (4.4 and 4.5) should be satisfactory. For ductile deposits, the bending test (4.3) is preferred.

5.3 Coatings thicker than 10 µm can be tested in the form of foils. Ductile foils can be tested by hydraulic bulging (3.4) or tensile testing (3.1). Brittle foils can be tested by the micrometer bend test (3.2) or by mechanical bulging (3.5).

5.4 Brittle and/or highly stressed coatings even when thicker than 10 µm may have to be tested when applied to a suitable, ductile substrate, in which case tensile testing (4.1) is preferred, although the cylindrical mandrel bending (4.4) or spiral mandrel bending (4.5) tests can be used.

6 Test report

The test report shall contain the following information:

a) a reference to the method used;

b) the results and the method of expression used;

c) details of the preparation of the test piece.

Annex A

Methods of producing foils

(This annex forms part of the standard.)

A.0 Introduction

Metal foils can be prepared by using a substrate from which the electrodeposit can be readily separated. Several methods can be used (see clauses A.1 and A.2).

A.1 Plating on to a soluble substrate

The substrate is dissolved after applying to it the coating to be tested. This is a method which can be used if the foil to be tested will not be affected by the dissolving solution. Even if the attack is only visible by a diminution in gloss, it would be possible that an embrittlement of the surface would enhance the beginning of cracking throughout the whole layer. For gold layers on copper substrates, dissolution of the copper with nitric acid solution is often used.

In the case of plated plastic it is possible to dissolve the plastic material into an organic solvent without affecting the quality of the test foil.

A.2 Plating on to a non-adherent substrate

Plating is carried out on a metal substrate to which the plating will not adhere; the foil is then peeled from the substrate.

A.2.1 Plating on stainless steel

Care shall be taken in this case that the surface of the stainless steel is free from scratches as these will be copied in the plated foil and will start the propagation of premature cracks. The stainless steel may require anodic cleaning for 15 s in a hot alkaline cleaner.

A.2.2 Plating on copper or bronze sheets

In the case of copper or bronze sheets it is easy to polish the surface before passivating. Also after the test, these sheets can be repolished and used again.

There exist several passivating methods (see A.2.2.1 to A.2.2.3).

A.2.2.1 Electroplating the copper or bronze substrate with 5 to 15 µm of bright nickel and subsequent passivation by immersion in a 1 to 5 % (m/m) solution of chromic acid for 30 to 60 s. Before immersion in the electroplating solution, the test surface should be connected to the negative electrical supply and the current switched on so as to prevent spoiling the passivation (live plating).

A.2.2.2 Plating the copper or bronze substrate with arsenic, using as electrolyte 59 g of arsenic trioxide (As_2O_3) and 21 g of sodium hydroxide (NaOH) in 1 litre of water. Plate for 5 min at 0,3 A/dm^2 and 18 to 30 °C, using as anodes carbon or graphite.

A.2.2.3 Immersion in a polysulfide solution — 50 g of sodium polysulfide (Na_2S_n) in 1 litre of water.

A.2.3 Plating on steel

A steel panel electroplated with nickel may be used. For example a piece of cold-rolled steel, of any convenient size, shall be properly cleaned, acid dipped and electroplated with about 7,5 µm of nickel. After rinsing, the test piece is either passivated (see A.2.2) or cleaned anodically for 15 s in a hot alkaline cleaner, acid dipped in 0,5 mol/l sulfuric acid, water rinsed, and placed in the electroplating solution of the metal to be tested. An electrodeposit of the desired thickness is electroplated on the prepared surface.