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Anodizing of aluminium and its alloys — Measurement of abrasion resistance of anodic oxidation coatings

Anodisation de l'aluminium et de ses alliages — Détermination de la résistance à l'abrasion des couches d'oxyde anodiques



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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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 8251 was prepared by Technical Committee ISO/TC 79, Light metals and their alloys, Subcommittee SC 2, Organic and anodic oxidation coatings on aluminium.

This second edition cancels and replaces the first edition (ISO 8251:1987) as well as ISO 8252:1987, which have been technically revised.

The main changes compared to the first edition are as follows:

- a) the inclusion of the test previously described in ISO 8252:1987;
- b) the inclusion of the falling sand test;
- c) the use of the methods for coatings produced by hard anodizing has been moved to ISO 10074:2010.

Introduction

The resistance of anodic oxidation coatings to abrasion is an important property. As it is dependent upon the composition of the metal, the thickness of the coating and the conditions of anodizing and sealing, it can give information about the quality of the coating, its potential resistance to erosion or wear and its performance in service. For example, the effect of an abnormally high anodizing temperature, which could cause potential deterioration in service by chalking of the surface layers, may be readily detected by means of an abrasive wear resistance test.

Anodizing of aluminium and its alloys — Measurement of abrasion resistance of anodic oxidation coatings

1 Scope

This International Standard specifies the following three test methods:

- a) **abrasive-wheel-wear test method**, determining the wear resistance and the wear index of anodic oxidation coatings on flat specimens of aluminium and its alloys;
- b) **abrasive jet test method**, comparing the resistance to abrasion of anodic oxidation coatings on aluminium and its alloys with that of a standard specimen or, alternatively, a reference specimen, by use of a jet of abrasive particles;
- c) **falling sand abrasion method**, determining the abrasion resistance with falling sand applied to thin anodic oxidation coatings.

The use of these methods for coatings produced by hard anodizing is described in ISO 10074.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 565:1990, Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings

ISO 2360:2003, Non-conductive coatings on non-magnetic electrically conductive basis materials — Measurement of coating thickness — Amplitude-sensitive eddy-current method

ISO 6344-1, Coated abrasives — Grain size analysis — Part 1: Grain size distribution test

ISO 8486-1:1996, Bonded abrasives — Determination and designation of grain size distribution — Part 1: Macrogrits F4 to F220

3 Terms and definitions

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

3.1

test specimen

specimen on which the test is to be carried out

3.2

standard specimen

test specimen produced in accordance with the conditions specified in Annex A

3.3

reference specimen

test specimen produced under conditions agreed between the anodizer and the customer

3.4

double stroke

ds

one complete reciprocal movement made by the abrasive wheel

Characteristics of abrasion tests 4

There are three kinds of abrasion tests: abrasive-wheel-wear test, abrasive jet test and falling sand abrasion test.

4.1 Abrasive-wheel-wear test

Determination of the resistance to abrasion by movement of a test specimen relative to an abrasive paper under a specified pressure. The wear resistance or the wear index of the layers of oxide near the surface, or of the whole oxidation coating thickness, or of any selected intermediate zone may be determined by the method described. For most purposes the wear index (see 5.4.3) or the mass wear index (see 5.4.4) will be the most appropriate characteristic to determine.

The method is applicable to all anodic oxidation coatings of thickness more than 5 µm on flat aluminium or its alloy specimens.

This method is not applicable to concave or convex specimens; these may be examined using the abrasive jet test method which will give an average value for the abrasive resistance of the coating (see 4.2 and Clause 6).

NOTE Minimum test specimen dimensions of 50 mm × 50 mm are normally required.

4.2 Abrasive jet test

Determination of the resistance to abrasion by the impact of abrasive particles projected onto a test specimen. The mean specific abrasion resistance of anodic oxidation coatings may be determined.

Different batches of the same abrasive are liable to give different results and for this reason the test is a NOTE 1 comparative one.

With a suitably designed abrasive jet and film-thickness-measuring devices with a small probe, it is possible to conduct a depth survey which indicates how abrasion resistance varies through the coating thickness (see Annex B). However, this property is preferably measured using the abrasive-wheel-wear test.

The method described is applicable to all anodic oxidation coatings of thickness more than 5 µm on aluminium or its alloys. It is primarily intended for surfaces which are not flat. If suitable flat test surfaces are available, the abrasive-wheel-wear test is the preferred method. Production components may be tested without cutting if the apparatus chamber can accommodate these.

NOTE 3 This method is particularly suitable for small test specimens because the individual test area required is only about 2 mm in diameter.

Falling sand abrasion test

Determination of the resistance to abrasion by the impact of freely falling abrasive particles onto anodic oxidation coatings.

The method described is applicable to the thin anodic oxidation coatings.

5 Abrasive-wheel-wear test

5.1 Principle

The anodic oxidation coatings on a test specimen are abraded, under defined conditions, by reciprocal motion against a strip of silicon carbide paper attached to the outer circumference of a wheel. After each double stroke, the wheel turns through a small angle to bring an unused portion of the abrasive strip into contact with the test area. The decrease in coating thickness or mass obtained is used to calculate the wear resistance or wear index. This result is compared with that obtained using a standard specimen (see Annex A) or reference specimen (see 3.3).

The method normally requires an eddy-current meter with a probe of less than 12 mm diameter. If this is not available, the method of loss in mass should be used.

NOTE A complete presentation of the wear characteristics of the anodic oxidation coatings can be obtained by progressively abrading the test area, until the substrate metal is revealed, and then constructing a graph to show the relation between the coating thickness removed and the number of double strokes used. This is referred to as a depth survey of the anodic oxidation coatings (see Annex B).

The testing environment should be at room temperature and the relative humidity should be under 65 %.

5.2 Apparatus

5.2.1 Abrasive-wheel-wear test apparatus

The apparatus consists of a clamping device or pressure plate for holding the test specimen (see 5.3.2) level and rigid, and a 50 mm diameter wheel to the outer circumference of which is attached a 12 mm wide strip of silicon carbide paper (see 5.2.2). The force between the wheel and the test surface shall be capable of being varied from zero to at least 4.9 N with an accuracy of ± 0.05 N. The abrasive action is produced either by the fixed wheel sliding to and fro in a horizontal plane in parallel contact with the test surface over a 30 mm length or, alternatively, by the test specimen sliding in a similar way over the stationary wheel. Typical apparatus is illustrated in Figure C.1.

After each double stroke, the wheel is advanced through a small angle to bring a fresh area of the silicon carbide paper into contact with the surface before making the next double stroke. The angle of rotation is such that, after 400 ds, the wheel will have made one complete revolution. At this stage, the strip of silicon carbide paper shall be renewed. The relative speed of movement shall be (40 ± 2) ds per minute. The number of double strokes can be registered by means of a counter, and provision is normally made for the apparatus to switch off automatically after a preset number of double strokes has been reached (400 ds maximum). The test surface shall be kept free from loose powder or abrasion detritus during the test.

5.2.2 Abrasive strip

The abrasive strip consists of P320 silicon carbide paper (in accordance with ISO 6344-1) 12 mm wide. Its length shall be such that it covers the abrasive wheel without overlapping, and it shall be either bonded or mechanically clamped into position.

NOTE P320 paper is 45 µm grade (320 mesh).

5.2.3 Eddy-current meter

An eddy-current meter with a suitable diameter probe is described in ISO 2360.

5.3 Procedure

5.3.1 Standard specimen

Prepare the standard specimen using the method specified in Annex A.

5.3.2 Test specimen

Cut a suitably sized test specimen from the item to be tested without damaging the area to be tested.

Test dimensions of 50 mm \times 50 mm are usually required.

5.3.3 Calibration of apparatus

- **5.3.3.1** Select and mark the area of the standard specimen (see 5.3.1) to be abraded. Accurately measure the anodic oxidation coating thicknesses in each of at least three positions along the test area by means of the eddy-current meter (see 5.2.3) in accordance with the method specified in ISO 2360 and calculate an average thickness value (d_1) .
- **5.3.3.2** Clamp the standard specimen into position on the apparatus (see 5.2.1).
- **5.3.3.3** Attach a new strip of silicon carbide paper (see 5.2.2) to the circumference of the abrasive wheel. Adjust the abrasive wheel, in accordance with the manufacturer's instructions, so that it gives uniform abrasion across the width of the test area. Adjust the force between the wheel and the test surface to $3.9 \ N \pm 0.1 \ N$.
- **5.3.3.4** Allow the apparatus to run for 400 ds or an adequate number of double strokes corresponding to the coating thickness and the kind of aluminium alloys. Keep the abrasive action uniform by adjusting and maintaining the alignment of the abrasive wheel in accordance with the manufacturer's instructions. Continuously remove any abrasion detritus by suction, blowing or frequent wiping with a fine brush.
- **5.3.3.5** Remove the standard specimen from the apparatus, wipe carefully to remove any loose oxide and determine the average thickness of the coating at the test area (d_2) using the eddy-current meter in accordance with 5.3.3.1.
- A 3 mm length at one extremity of the test area may be subject to extra wear because of the continual wheel rotation which takes place at this point; this area should be ignored when taking the thickness measurements.
- **5.3.3.6** Carry out at least two further determinations on the standard specimen in test areas that do not overlap, using the procedure specified in 5.3.3.1 to 5.3.3.5.
- **5.3.3.7** Calculate the wear rate for the standard specimen (see 5.4.3) from the average of the determinations.

5.3.4 Determination

Take the test specimen (see 5.3.2) and carry out the procedure specified in 5.3.3.1 to 5.3.3.6 using abrasive strips from the same batch as that used for the calibration. If the test specimen is not rigid, bond it firmly with an adhesive to a rigid metal sheet with a flat surface before carrying out the determination.

Calculate the wear rate for the test specimen and, from the wear rates for the standard specimen and for the test specimen, calculate the wear index in accordance with 5.4.3.

5.3.5 Use of a reference specimen

5.3.5.1 **General**

Because of the relatively high abrasion resistance of integral colour anodized specimens, testing of these finishes normally requires the use of a reference specimen produced by the same process (see 3.3) in a comparative-wear-testing method (see 5.3.6).

5.3.5.2 Initial determination

Carry out an initial determination in accordance with 5.3.4. If the thickness loss in the test area is less than $3 \mu m$, adjust the abrasion conditions either by increasing the force between the wheel and the test specimen surface, or by employing a coarser grade of silicon carbide paper. Alternatively, an increased number of double strokes may be used.

Unless a depth survey is being carried out (see Annex B), the abrasion conditions should be adjusted to give a coating thickness loss of (5 ± 3) µm after 400 ds. If loss of mass is to be determined, the mass equivalent of (5 ± 3) µm coating thickness is required to be known. This necessitates an assumption to be made about the coating density or, alternatively, this should be estimated by means of ISO 2106.

5.3.5.3 Determination

Determine the loss in thickness or loss in mass of the test specimen and the reference specimen under the conditions established in 5.3.5.2, following the procedure specified in 5.3.6.

Calculate the comparative wear rate in accordance with 5.4.5, or the comparative mass wear rate in accordance with 5.4.6, as appropriate.

5.3.6 Comparative wear testing

5.3.6.1 **General**

Comparison of the abrasion of the test specimen (see 5.3.2) can be made with that of a reference specimen (see 3.3) or with the standard specimen (see 3.2). In these cases, either comparative loss in thickness or comparative loss in mass can be determined. The comparative wear rate is expressed as a percentage of that of the reference specimen.

5.3.6.2 Comparative loss of thickness

Determine the loss in thickness of the test specimen and of the reference specimen using the procedure specified in 5.3.4.

Calculate the comparative wear rate in accordance with 5.4.5.

5.3.6.3 Comparative loss of mass

- **5.3.6.3.1** Select and mark the area of the test specimen to be abraded. Weigh the test specimen to the nearest 0,1 mg (m_1) . Carry out the procedure specified in 5.3.3.2 to 5.3.3.4.
- **5.3.6.3.2** Remove the test specimen from the apparatus, wipe to remove any loose oxide and weigh to the nearest 0,1 mg (m_2) .

Carry out at least two further determinations on the test specimen in test areas that do not overlap.

NOTE Freshly exposed anodic oxidation coatings can gain in mass by absorbing water vapour. Multiple tests on a single panel can therefore be subject to errors dependent upon variations in atmospheric humidity.

5.3.6.3.3 Repeat the procedure specified in 5.3.6.3.1 and 5.3.6.3.2 on the reference specimen. Calculate the comparative mass wear rate in accordance with 5.4.6.

5.4 Calculation of results

The calculation of results shall be chosen from the following.

5.4.1 Wear resistance

Calculate the wear resistance, WR, in double strokes per micrometre, using Equation (1):

$$WR = \frac{400}{d_1 - d_2} \tag{1}$$

where

is the average thickness, in micrometres, before abrasion (see 5.3.3.1);

is the average thickness, in micrometres, after 400 ds abrasion (see 5.3.3.5).

5.4.2 Wear resistance coefficient

Calculate the wear resistance coefficient, WRC, using Equation (2):

$$WRC = \frac{WR_{t}}{WR_{s}} = \frac{d_{1s} - d_{2s}}{d_{1t} - d_{2t}}$$
 (2)

where

WR_t is the wear resistance, in double strokes per micrometre, of the test specimen;

WR_s is the wear resistance, in double strokes per micrometre, of the standard specimen;

is the average thickness, in micrometres, of the standard specimen before abrasion (see 5.3.3.1); d_{1s}

is the average thickness, in micrometres, of the standard specimen after 400 ds abrasion (see d_{2s} 5.3.3.5):

 d_{1t} is the average thickness, in micrometres, of the test specimen before abrasion (see 5.3.3.1);

is the average thickness, in micrometres, of the test specimen after 400 ds abrasion (see 5.3.3.5). d_{2t}

The wear resistance coefficient is the reciprocal of wear index and is a measure of resistance to abrasive wear. The wear resistance coefficient of a standard specimen is 1. Values greater than 1 indicate a lower degree of wear than that on the standard specimen. Values less than 1 indicate a greater degree of wear than that on the standard specimen.

5.4.3 Wear index

Calculate the wear index, WI, using Equation (3):

$$WI = \frac{W_{t}}{W_{s}} = \frac{d_{1t} - d_{2t}}{d_{1s} - d_{2s}}$$
 (3)

where

is the wear rate of the test specimen, in micrometres per 100 ds;

$$W_{\mathsf{t}} = \frac{d_{1\mathsf{t}} - d_{2\mathsf{t}}}{\Delta}$$

 d_{1t} and d_{2t} are as defined in 5.4.2.

 $W_{\rm s}$ is the wear rate of the standard specimen, in micrometres per 100 ds;

$$W_{S} = \frac{d_{1S} - d_{2S}}{4}$$

 d_{1s} and d_{2s} are as defined in 5.4.2.

NOTE The wear index is a ratio and is dimensionless; it is an indication of the relative rate of wear and is the reciprocal of the wear resistance coefficient. The wear index of a standard specimen is 1. Values greater than 1 indicate a greater degree of wear than that on the standard specimen. Values less than 1 indicate a lower degree of wear than that on the standard specimen.

5.4.4 Mass wear index

Calculate the mass wear index, MWI, using Equation (4):

$$MWI = \frac{MW_{t}}{MW_{s}} = \frac{m_{1t} - m_{2t}}{m_{1s} - m_{2s}}$$
 (4)

where

MW_t is the mass wear rate of the test specimen;

MW_s is the mass wear rate of the standard specimen;

 m_{1t} is the average mass, in milligrams, of the test specimen before abrasion (see 5.3.6.3.1);

 m_{2t} is the average mass, in milligrams, of the test specimen after 400 ds abrasion (see 5.3.6.3.2);

 m_{1s} is the average mass, in milligrams, of the standard specimen before abrasion (see 5.3.6.3.1);

 m_{2e} is the average mass, in milligrams, of the standard specimen after 400 ds abrasion (see 5.3.6.3.2).

NOTE The mass wear index is a ratio and is dimensionless; it is an indication of the relative rate of wear. The mass wear index of a standard specimen is 1. Values greater than 1 indicate a greater degree of wear than that on the standard specimen. Values less than 1 indicate a lower degree of wear than that on the standard specimen.

5.4.5 Comparative wear rate

Calculate the comparative wear rate, CWR, expressed as a percentage, using Equation (5):

$$CWR = \frac{W_{r}}{W_{t}} \times 100 = \frac{d_{1r} - d_{2r}}{d_{1t} - d_{2t}} \times 100$$
 (5)

where

 $W_{\rm r}$ is the wear rate of the reference specimen, in micrometres per 100 ds;

$$W_{\mathsf{r}} = \frac{d_{\mathsf{1r}} - d_{\mathsf{2r}}}{\mathsf{4}}$$

 d_{1r} is the average thickness, in micrometres, of the reference specimen before abrasion;

 d_{2r} is the average thickness, in micrometres, of the reference specimen after 400 ds abrasion;

 W_{t} is the wear rate of the test specimen, in micrometres per 100 ds;

$$W_{\mathsf{t}} = \frac{d_{1\mathsf{t}} - d_{2\mathsf{t}}}{4}$$

 d_{1t} and d_{2t} are as defined in 5.4.2.

5.4.6 Comparative mass wear rate

Calculate the comparative mass wear rate, CWR_m, expressed as a percentage, using Equation (6):

$$CWR_{m} = \frac{MW_{r}}{MW_{t}} \times 100 = \frac{m_{1r} - m_{2r}}{m_{1t} - m_{2t}} \times 100$$
 (6)

where

is the comparative mass wear rate of the reference specimen, in milligrams per 100 ds;

$$\mathsf{MW}_\mathsf{r} = \frac{m_{\mathsf{1r}} - m_{\mathsf{2r}}}{4}$$

is the average mass, in milligrams, of the reference specimen before abrasion (see 5.3.6.3.1); m_{1r}

is the average mass, in milligrams, of the reference specimen after 400 ds abrasion (see 5.3.6.3.2); m_{2r}

is the comparative mass wear rate of the test specimen, in milligrams per 100 ds;

$$\mathsf{MW}_{\mathsf{t}} = \frac{m_{\mathsf{1}\mathsf{t}} - m_{\mathsf{2}\mathsf{t}}}{4}$$

 m_{1t} and m_{2t} are as defined in 5.4.4.

Abrasive jet test

Principle 6.1

Dry silicon carbide particles are projected in a stream of dry air or inert gas under carefully controlled conditions onto a small area of the surface to be tested. The test is continued until the substrate metal is exposed, after which the abrasion resistance of the coating is calculated from either the time taken or the mass of silicon carbide used. The result is compared with that obtained using a standard specimen (see Annex A) or reference specimen (see 3.3).

Apparatus 6.2

Abrasive jet test apparatus

The abrasive jet test apparatus is shown in Figures D.1 to D.3.

Abrasive jet assembly, consisting essentially of two glass or metal tubes supported rigidly and 6.2.1.1 coaxially. The outer tube is connected to a supply of clean, dry, compressed air or inert gas, which can be delivered at a carefully regulated flow rate. Dry abrasive particles are supplied to the inner tube, at the exit end of which they mix with the air stream to form an abrasive jet which is directed onto the test specimen.

No restriction is placed upon the design of the abrasive jet assembly, except that it shall give reproducible results in successive tests, and that it shall allow consistent measurements to be made.

NOTE A number of satisfactory designs of the jet assembly have been constructed but it has proved difficult in practice to manufacture a series of jets which give identical results, or to make any that are not subject to drift and variations. Designs that have proved satisfactory are shown in Annex D.

- **6.2.1.2 Test specimen support**, comprising an inclined platform on which the test specimen is firmly and rigidly supported such that the angle between the plane of the test area and that of the jet axis is in the range 45° to 55°. The jet is usually vertical.
- NOTE D.2 describes an apparatus where the angle is 55°; D.3 describes a different form of apparatus where the angle is approximately 45°. The larger angle produces a less elliptical test area, more rapid abrasion, and a sharper end point.
- **6.2.1.3** Air or inert gas supply, fed to the outer tube from a compressor or gas cylinder and controlled accurately by means of a regulating valve and a flowmeter or manometer situated near the apparatus. The air or inert gas shall be dry, or have constant low humidity.
- NOTE 1 The gas can be conveniently dried by passing it through tubes containing silica gel. Compressed air passed through a holding reservoir where condensed water vapour is collected will have a satisfactory and fairly constant humidity.
- NOTE 2 Flow rates depend on the apparatus. Gas pressures can be from 7,5 kPa to 15 kPa, where the latter can produce flow rates typically from 40 l/min to 70 l/min. When the flow rate has been selected for any particular jet assembly, it should, as far as possible, be maintained throughout the life of the jet nozzle.
- **6.2.1.4 Hopper**, for storage of the abrading medium and capable of supplying this at a steady rate of 20 g/min \pm 1 g/min to 30 g/min \pm 1 g/min.

6.2.2 Abrading medium

Silicon carbide particles of a grade recommended by the manufacturer of the apparatus used. A suitable grade of abrasive is 125 µm mesh size: F100 in accordance with ISO 565:1990 and ISO 8486-1:1996.

The abrading medium shall be free from moisture and shall be dried before use and passed through a coarse sieve (for example, of $180 \mu m$ or $300 \mu m$ nominal aperture size) to ensure freedom from large particles or fibres which might interfere with the rate of abrasive flow.

The dried medium may be re-used up to 50 times; after each use, the medium should be dried, passed through a coarse sieve and stored in a clean, tightly closed container.

NOTE Ambient humidity has little effect on the test result, but can have a very considerable effect if the medium is re-used without drying.

The testing environment should be at room temperature and the relative humidity should be under 65 %.

6.3 Procedure

6.3.1 Standard specimen

Prepare the standard specimen using the method specified in Annex A.

6.3.2 Test specimen

Cut a suitably sized test specimen from the item to be tested without damaging the area to be tested.

6.3.3 Calibration of apparatus

6.3.3.1 Select and mark the areas of the standard specimen (see 6.3.1) to be abraded. Accurately measure the anodic oxidation coating thickness (d) in each test area by means of an eddy-current meter in accordance with the method specified in ISO 2360.

- **6.3.3.2** Fix the standard specimen in position in the test apparatus (see 6.2.1) with the selected test area beneath the jet orifice and at the correct angle to the jet axis.
- **6.3.3.3** Fill the hopper (see 6.2.1.4) with sufficient silicon carbide (see 6.2.2) for the test. If the abrasion resistance is being determined in terms of the mass of abrasive used, weigh the hopper and contents to the nearest 1 g.
- **6.3.3.4** Set the air flow rate, or the pressure, to the specified (or selected) value (see 6.2.1.3), which shall be accurately maintained throughout each test and any series of tests.

The air or gas flow rate should be adjusted to give a rate of abrasion that is convenient for both the standard specimen being tested and for the test specimen. The preferred rate, or pressure, is normally indicated by the instrument manufacturer but it may be necessary to vary this for very soft, hard or thin coatings.

- **6.3.3.5** Start the flow of abrading medium (see 6.2.2) and simultaneously start a timer. Throughout the test, ensure that the abrasive flows freely.
- **6.3.3.6** Keep the standard specimen under observation; when a small black spot appears in the centre of the abraded area and rapidly enlarges to approximately 2 mm in diameter terminate the test by stopping the abrasive flow and the timer simultaneously. It is recommended that the end point be determined by using a circuit tester to measure the electrical resistance at the abraded area on the standard specimen.
- **6.3.3.7** Record the time, in seconds, taken for the test and, if required, weigh the hopper and residual contents to the nearest 1 g.

From the two weighings (see 6.3.3.3 and 6.3.3.7), calculate the mass, in grams, of silicon carbide used to penetrate the coating.

The results give the abrasion, S_s , of the standard specimen in either seconds or grams.

6.3.3.8 Carry out at least two further tests (see 6.3.3.1 to 6.3.3.7 inclusive) on other parts of the standard specimen.

6.3.4 Calibration of jet

6.3.4.1 General

Since individual jets may vary with use and one with another, it is necessary to correct each set of measurements by means of calibration determinations using a standard specimen as in 6.3.3. This enables the abrasive jet factor (see 6.4.1) for the set of measurements to be calculated.

6.3.4.2 Change of jet or abrasive characteristics with time

For any series of test measurements, repeat the procedure specified in 6.3.3 once or twice daily in order to allow a correction to be made for changes of jet or abrasive characteristics with time.

6.3.4.3 Jet replacement

After jet replacement, repeat the procedure specified in 6.3.3 to allow correction for changes in jet characteristics.

6.3.5 Determination

Carry out the procedure specified in 6.3.3 using the test specimen instead of the standard specimen.

6.3.6 Use of a reference specimen

Under some circumstances, for example for control purposes, it is common practice to use a reference specimen for comparison and, if this is required, the procedure specified in 6.3.3 shall be followed using the reference specimen in place of the standard specimen.

6.4 Calculation of results

The calculation of results should be chosen from the following.

6.4.1 Abrasive jet factor

Calculate the abrasive jet factor, K, in micrometres per second or micrometres per gram, using Equation (7):

$$K = \frac{d_s}{S_s} \times 10 \tag{7}$$

where

- $d_{\rm s}$ is the original coating thickness (see 6.3.3.1), in micrometres, of the standard specimen in the area tested;
- $S_{\rm s}$ is the abrasion, in seconds or grams, of the standard specimen.

When the abrasive jet factor has been determined for a jet used under any specified set of conditions, it is essential that measurements made with that jet should be multiplied by this factor.

6.4.2 Mean specific abrasion resistance

Calculate the mean specific abrasion resistance, R, of the coating at any test point with reference to the value obtained on a standard specimen, using Equation (8):

$$R = \frac{KS}{d} \tag{8}$$

where

- K is the abrasive jet factor (see 6.4.1);
- S is the abrasion (see 6.3.3.7), in seconds or grams, of the test specimen;
- d is the original coating thickness, in micrometres, of the test specimen in the area tested (see 6.3.3.1).

The values quoted shall be the mean of not less than three determinations.

NOTE 1 The mean specific abrasion resistance is a ratio with no dimensions. The standard specimen has an arbitrary value of 10 (see 6.4.1).

NOTE 2 Anodic oxidation coatings can be variable through their thickness and the measured value is an average property for the whole coating thickness.

Comparison with an agreed reference specimen

If the abrasive jet apparatus is being used for comparison with an agreed reference specimen, calculate the relative mean specific abrasion resistance, R_{rel} , as a percentage, using Equation (9):

$$R_{\text{rel}} = \frac{S}{d} \times \frac{d_{\text{r}}}{S_{\text{r}}} \times 100 \tag{9}$$

where

is the abrasion, in seconds or grams, of the reference specimen; S_{r}

 d_{r} is the original coating thickness, in micrometres, of the reference specimen in the area tested;

S and d are as defined in 6.4.2.

The value quoted shall be the mean of not less than three determinations for both the test specimen and the reference specimen.

Falling sand abrasion test

Principle 7.1

Dry silicon carbide particles fall onto a small area of the surface to be tested. The test is continued until the substrate metal is exposed, after which the abrasion resistance of the coating is calculated from the time taken.

7.2 **Apparatus**

7.2.1 Falling sand abrasion test apparatus

The test apparatus for this abrasion test comprises a hopper, a funnel, a shutter plate and a guide tube. The components of the apparatus shall comply with the requirements in Table 1; an example of a test apparatus is shown in Figure E.1.

Table 1 — Requirements for the falling sand abrasion test apparatus

Apparatus	Requirements				
Funnel	The funnel shall be made of glass, have an angular aperture of 60° , an inside diameter at the hopper end of 70 mm, a leg length of 50 mm, an inside diameter of the leg of 5,0 mm \pm 0,4 mm, with a smooth finish on the inside lower part of the funnel and inside the leg, and be capable of delivering abrasive particles at the rate of 320 g/min \pm 10 g/min. The rate of delivery of abrasive particles shall be controlled by moving up and down a control bar suspended at the centre of the funnel.				
Guiding tube	The guiding tube shall measure 970 mm in length and 20 mm in inside diameter.				
Specimen-supporting stage	The specimen-supporting stage shall be capable of fixing a test specimen at $45^{\circ} \pm 1^{\circ}$ to the vertical, and of adjusting the distance between the lower end of the guiding tube and the test specimen to 30 mm \pm 2 mm.				

7.2.2 Ohmmeter

The ohmmeter which is used for checking the exposed substrate metal surface of the test specimen shall meet the following conditions:

- a) scale of 5 000 Ω shall be accurately indicated;
- b) tip of contact probe shall have a smooth, spherical surface.

7.2.3 Abrading medium

The grit designation of abrasives shall be silicon carbide of F80 (see ISO 8486-1:1996). The abrasive shall not be reused more than 50 times.

7.3 Test specimen

The test specimens shall be extracted from the significant surface of the products. However, if they cannot be taken from real products, substitute test specimens which represent real products may be used for testing.

The standard size of a test specimen is preferably 100 mm \times 100 mm.

The test specimen shall be cleaned before testing.

7.4 Test environment

Testing should be at room temperature at relative humidity under 65 %.

7.5 Test conditions

The angle of the test specimen to the falling sand shall be $45^{\circ} \pm 1^{\circ}$.

The distance through which the abrasive particles fall shall be 1 000 mm.

The rate of delivery of the abrasive particles shall be 320 g/min \pm 10 g/min.

7.6 Test procedure

The test shall be carried out by one of the following procedures.

7.6.1 Electrical conductivity method

In this method, the end point shall be determined by measurement using an ohmmeter.

- a) Put abrasives into the hopper and open the shutter to let them fall for about 1 min. Then confirm the quantity of falling abrasives to be in the specified range (320 g/min \pm 10 g/min). If they are not in this range, adjust the control bar up or down to correct the position.
- b) Fix the test specimen on the specimen-supporting stage, keeping the surface of the specimen to be tested at an angle 45° to the vertical.
- c) Fix the specimen-supporting stage in the position where the surface of the test specimen is 30 mm from the lower edge of the guide tube.
- d) Open the shutter to let the abrasives fall until the substrate metal surface is exposed (visible by a colour change) and determine the time (t), in seconds, during which the abrasives were falling.

- e) Repeat the operation described in d) at several places on other areas of the test specimen for times $t \pm 10$ %. Select the subsequent test positions at 15 mm horizontally and 25 mm orthogonally to the horizontal on the specimen surface. It is not permitted to stop the abrasive falling during a test.
- f) After stopping the sand falling, clean the surface of the test specimen with a soft, dry cloth.
- g) Measure the electrical resistance between the substrate metal and the tested surface with the ohmmeter. Measurements shall be carried out three times at each test area using the tip of the contact probe. It shall be deemed that the substrate metal has been exposed if at least one test value is below 5 000 Ω .

7.6.2 Spot diameter method

In this method, the end point shall be determined by visual observation.

- a) Put abrasives into the hopper and open the shutter to let them fall for about 1 min. Then confirm the quantity of falling abrasives to be in the specified range (320 g/min \pm 10 g/min). If they are not in this range, adjust the control bar up or down to the correct position.
- b) Fix the test specimen on the holder, keeping the surface of the specimen to be tested at an angle of 45° to the vertical.
- c) Fix the holder in the position where the surface of the test specimen is 30 mm from the lower edge of the guide tube.
- d) Open the shutter to let the abrasives fall until the surface of substrate metal is exposed as an area about 2 mm in diameter (visible by a colour change) and record the time (t), in seconds, during which the abrasives fell.
- e) Repeat the operation as described in d) two or more times at other areas of the test specimen for times $t \pm 10$ %. Select the subsequent test positions at 15 mm horizontally and 25 mm orthogonally to the horizontal on the specimen surface. It is not allowed to stop the abrasive falling during a test.

7.7 Expression of results

7.7.1 Electrical conductivity method

The test results shall be expressed either as the shortest time (t_{min}), in seconds, needed for the abrasion of the anodic oxidation coatings to expose the substrate metal or as the wear resistance, WR_F, in units of s/µm, using Equation (10):

$$WR_{F} = \frac{t_{\min}}{d} \tag{10}$$

where

 t_{min} is the shortest time, in seconds, for the substrate metal to be exposed;

d is the coating thickness before the wear resistance test.

7.7.2 Spot diameter method

The test results shall be expressed as the shortest time (t_{min}), in seconds, needed for the abrasion to expose the substrate metal as a small black spot about 2 mm in diameter.

8 Test report

The test report shall include at least the following information:

- a) a reference to this International Standard, i.e. ISO 8251:2011;
- b) identification of the test specimen and, if appropriate, the reference specimen;
- c) the apparatus used;
- d) the number of test points and their location on the test surface;
- e) any other observations concerning the conduct of the test or the nature of the test specimen or test area;
- f) the date of the test;

and for the abrasive-wheel-wear test:

- g) the force between the abrasive wheel and test surface;
- h) the abrasive medium used;
- i) the calculated values, as required, of wear resistance, wear resistance coefficient, wear index, mass wear index, comparative wear rate or comparative mass wear rate (see 5.4);

or for the abrasive jet test:

- j) the angle between the plane of the test area and the jet axis;
- k) the abrasive medium and its particle size;
- I) the gas flow rate and pressure;
- m) the value of the mean specific abrasion resistance, R, or the relative mean specific abrasion resistance, R_{rel} (see 6.4);

or for the falling sand abrasion test:

- n) the abrasive medium used and its particle size;
- o) the calculated values of wear resistance in the case of an electrical conductivity method or the shortest abrasive time in the case of a spot diameter method.

Annex A

(normative)

Preparation of standard specimen

A.1 Aluminium specification

The standard specimen for abrasion test purposes shall be prepared from polished or bright-rolled aluminium sheet as follows:

Aluminium specification: AI 99,5 Al min. 99,5 %

H14 Temper:

Normal test specimen size: 140 mm \times 70 mm

Thickness: 1,0 mm to 2,0 mm

A.2 Pretreatment

Degreasing only (light caustic etching, electropolishing or chemical polishing is permissible).

A.3 Anodizing

Bath composition:

Free sulfuric acid concentration: $180 \text{ g/l} \pm 2 \text{ g/l}$

Aluminium concentration: 5 g/l to 10 g/l

Rest: deionized water (ion-exchanged water)

Conditions of anodizing:

Temperature: 20 °C ± 0,5 °C

Current density: $1.5 \text{ A/dm}^2 \pm 0.1 \text{ A/dm}^2$

Agitation: with compressed air or solution circulation

Anodizing time: 45 min

Anodic coating thickness: $20 \mu m \pm 2 \mu m$

The standard specimen shall be anodized vertically with the longer axis horizontal in the bath, maintaining vigorous agitation over the anodic surface and smooth direct current with not more than 5 % ripple. Not more than 20 standard specimens shall be anodized at one time and the volume of the electrolyte shall be not less than 10 I per standard specimen.

NOTE 1 The standard specimens are most accurate and reproducible if anodized singly with careful control of all the conditions.

NOTE 2 The standard specimens have inherent variations of \pm 10 %.

A.4 Sealing

Sealing shall be carried out for 60 min in boiling deionized water (ion-exchanged water) containing 1 g of ammonium acetate per litre, at pH 5,5 to 6,5.

Annex B

(informative)

Depth survey of abrasion resistance

B.1 General

It may be required to establish the way in which abrasion or wear resistance changes through the thickness of anodic oxidation coatings and it will be necessary to carry out a depth survey to determine this. This annex describes methods for determining the resistance to abrasion of anodic oxidation coatings on aluminium, progressively throughout the coating.

B.2 Abrasive-wheel-wear method

B.2.1 Principle

A test specimen is abraded on a single test area with a succession of abrasions until the substrate metal is revealed. The coating thickness is measured at the beginning and after each abrasion.

B.2.2 Apparatus

Use the apparatus specified in 5.2.

B.2.3 Procedure

B.2.3.1 Determination

- Measure the average coating thickness of the test specimen as specified in 5.3.3.1. Locate the test specimen precisely in the apparatus by means of positioning pins or stops attached to the specimen-supporting stage, so that successive abrasions can be carried out on a single test area.
- Carry out the first abrasion as specified in 5.3.3.3 and 5.3.3.4, using only 20 to 50 ds according to the anticipated hardness. Remove the test specimen and determine the average coating thickness in the area as described in 5.3.3.5.
- Relocate the test specimen accurately in the apparatus and repeat the procedure as specified in B.2.3.1.2 for a further appropriate number of double strokes (see Note 1). Repeat the operations of abrasion and thickness determination using an appropriate number of double strokes until the substrate metal is just revealed.
- NOTE 1 A typical sequence of abrasion intervals is: 50 - 100 - 200 - 400 - 800 and 1 200 ds.
- Where accurate relocation of the test specimen is difficult or doubtful, the same result can be achieved by successive tests of increasing duration on a series of adjacent test areas.

B.2.4 Expression of results

Calculate the change in resistance to wear through the coating thickness, and the appropriate wear indices or wear resistance coefficients for any part of the coating using, if required, a graph of coating thickness plotted against the number of double strokes used.

NOTE The result of a depth survey can be related to that carried out on a standard specimen, if required.

B.3 Abrasive jet method and falling sand method

B.3.1 Principle

A test specimen is abraded at a series of positions for increasing periods of time, the maximum time being that taken to penetrate or reveal the coating completely (see 6.3.3.6 and 7.6). The mean specific abrasion resistance or wear resistance is calculated at required depths through the coating.

B.3.2 Apparatus

Use the apparatus specified in 6.2 and 7.2.

B.3.3 Procedure

B.3.3.1 Test specimen

Use a test specimen not less than 70 mm × 70 mm, prepared as follows.

- a) If using the falling sand method, clean the specimen.
- b) Mark the positions of between 6 and 12 test sites on the surface of the test specimen so that they are separated by intervals of 10 mm and 20 mm along and across the test specimen respectively.
- c) Using an eddy-current meter fitted with a small probe of tip diameter less than 1 mm, measure the initial coating thickness accurately at each test site, using the method specified in ISO 2360.

B.3.3.2 Determination using the abrasive jet method

- **B.3.3.2.1** Prepare the apparatus in accordance with 6.3.3 and 6.3.4.
- **B.3.3.2.2** Position the first test site of the test specimen beneath the jet orifice at the correct angle and abrade the test specimen until the coating is just penetrated completely.

Note the time (t) taken, in minutes, and divide this by the number of remaining test sites (n-1) to derive a unit value for abrasion time, t^*

where

$$t^* = t/(n-1)$$

- **B.3.3.2.3** Using the same conditions, abrade the second test site for t^* min, the third test site for $2t^*$ min and so on, until all of the test sites have been abraded. Record the mass of silicon carbide used for each test site.
- **B.3.3.2.4** After completing abrasion at a given test site, remove the test specimen, clean the test surface with a soft cloth and, using the method specified in ISO 2360, carefully measure the minimum coating thickness remaining at that site. The eddy-current meter used for the measurement should be provided with a probe of diameter less than 1 mm. Confirm the zero thickness using, for example, a low-voltage continuity probe.
- **B.3.3.2.5** Carry out a separate test on a standard specimen (see Annex A) to determine the abrasive jet factor, K (see 6.4.1).

B.3.3.3 Determination using the falling sand method

B.3.3.3.1 Prepare the apparatus in accordance with 7.4, 7.5 and 7.6.1 a).

Position the first test site of the test specimen where the sand will fall on it in accordance with 7.6.1 b) and 7.6.1 c), and abrade the test specimen until the coating is just penetrated completely. This may be verified by using an ohmmeter when the contact resistance is below 5 000 Ω , or by visual observation when the exposed area of the substrate metal surface is about 2 mm in diameter (visible by a colour change). Note the time (t) taken, in minutes, and divide this by the number of remaining test sites (n-1) to derive a unit value for abrasion time. t^*

where

$$t^* = t/(n-1)$$

- **B.3.3.3.3** Using the same conditions, abrade the second test site for t^* min, the third test site for $2t^*$ min and so on, until all of the test sites have been abraded. Record the mass of silicon carbide used for each test site.
- After completing abrasion at a given test site, remove the test specimen, clean the test surface B.3.3.3.4 with a soft cloth and, using the method specified in ISO 2360, carefully measure the minimum coating thickness remaining at that site. The eddy-current meter used for the measurement should be provided with a probe of diameter less than 1 mm.

B.3.4 Expression of results

- B.3.4.1 For each test site calculate
- the thickness, in micrometres, of the coating removed,
- the corresponding abrasion value using the following formulae:

for the abrasive jet method

$$t_s K$$
 or mK ;

for the falling sand method

$$t_s$$
 or m ;

where

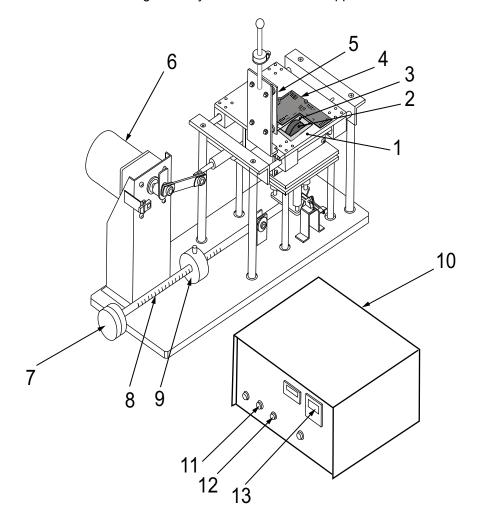
- is the abrasive jet factor; K
- is the abrasion time, in seconds;
- is the mass, in grams, of silicon carbide powder delivered. m
- Plot a graph with the number of micrometres of coating removed as the abscissa, and the B.3.4.2 corresponding abrasion values (see B.3.4.1) as the ordinate. The slope at any point on the graph is the specific abrasion resistance of the coating over the depth of the coating.

The abrasive jet method is not applicable to coating of less than 5 µm, in thickness. The portion of the graph immediately below this value should be extrapolated from the point just above it, although results obtained from this part of the graph can be subject to experimental error.

Annex C (informative)

Design of abrasive-wheel-wear test apparatus

- **C.1** No restriction is placed upon the design of the abrasive-wheel-wear test apparatus, provided that it complies with the general principles given in 5.2.
- **C.2** Figure C.1 shows the basic design and layout of a suitable test apparatus.



Key

- 1 specimen stage
- 2 abrasive wheel
- 3 specimen
- 4 specimen guide
- 5 specimen press
- 6 specimen reciprocating motor
- 7 load

- 8 load scale
- 9 load adjust
- 10 specimen reciprocating control unit
- 11 start button
- 12 stop button
- 13 double-stroke indication

Figure C.1 — Example of abrasive-wheel-wear test apparatus

Annex D (informative)

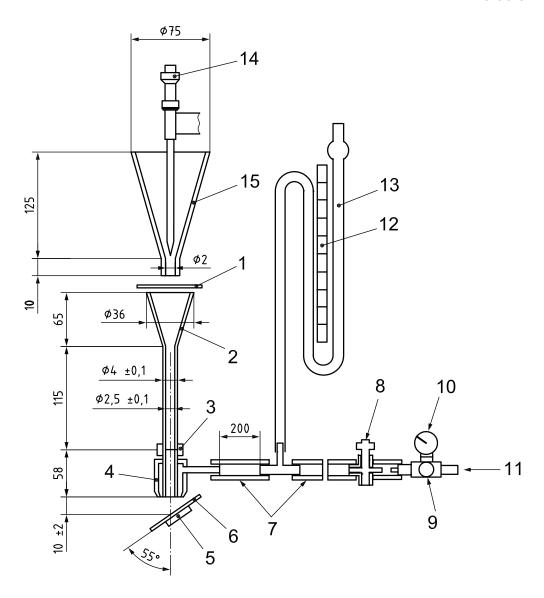
Design of abrasive jet test apparatus

D.1 No restriction is placed upon the design of the jet test apparatus, provided that it complies with the general principles given in 6.2.

Many jet assembly designs have been used for abrasion testing of anodic oxidation coatings and these have varied with regard to the sharpness of the end point, sensitivity to small changes in operating conditions, and tendency to drift due to wear. Two designs (see D.2 and D.3) have been found satisfactory and are recommended.

- **D.2** Figure D.1 shows the basic design and layout of a suitable test apparatus but does not include the cabinet which houses the jet assembly and test specimen and retains the used abrasive powder. The jet nozzle, shown in detail in Figure D.2, is constructed from brass and stainless steel, and the design ensures minimum wear of the nozzle; in conjunction with a test specimen angle of 55°, it produces rapid abrasion and a sharp end point.
- **D.3** Figure D.3 shows an alternative design of the jet assembly. The materials of construction are optional but the outer (8,5 mm diameter) tube should be made of glass and should be removable from the metal collar, as the outer tube is subject to wear (with consequent drifting of results and loss of sharpness of the end point). A specific glass tube should be used for each rate of air flow. The test specimen angle is generally 45° but may be from 45° to 55°.

Dimensions in millimetres



Key

1	shutter	9	control valve
2	funnel	10	pressure gauge
3	joint	11	air or inert gas supply
4	nozzle	12	scale
5	specimen-supporting stage	13	water manometer
6	test specimen	14	abrasive flow-rate adjuster
7	high-pressure-resisting rubber tube	15	hopper
8	stop cock		

NOTE Dimensions are not critical where no tolerances are given.

Figure D.1 — General layout of abrasive jet test apparatus

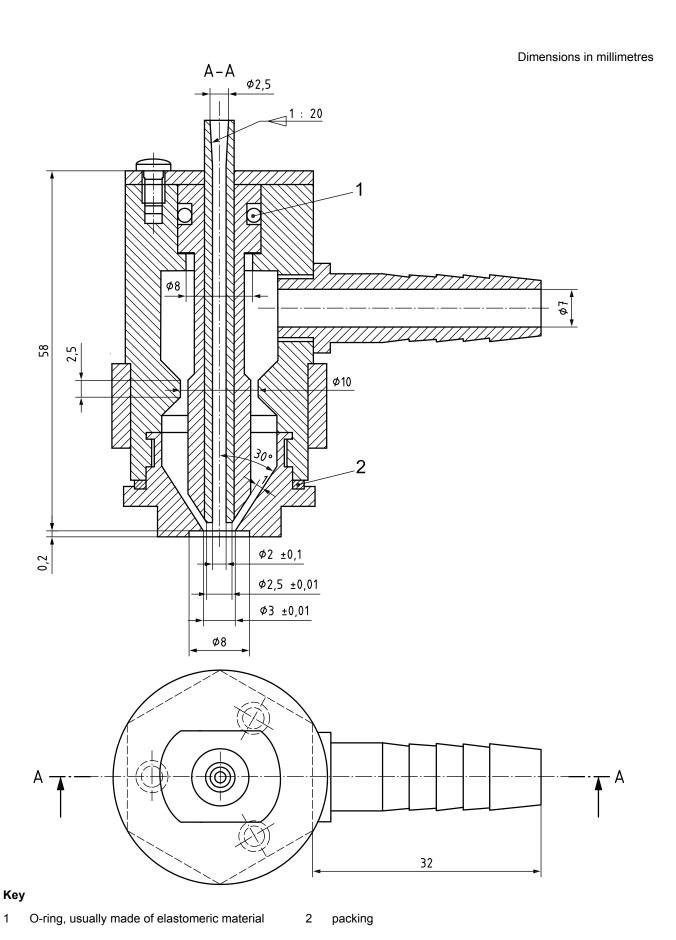
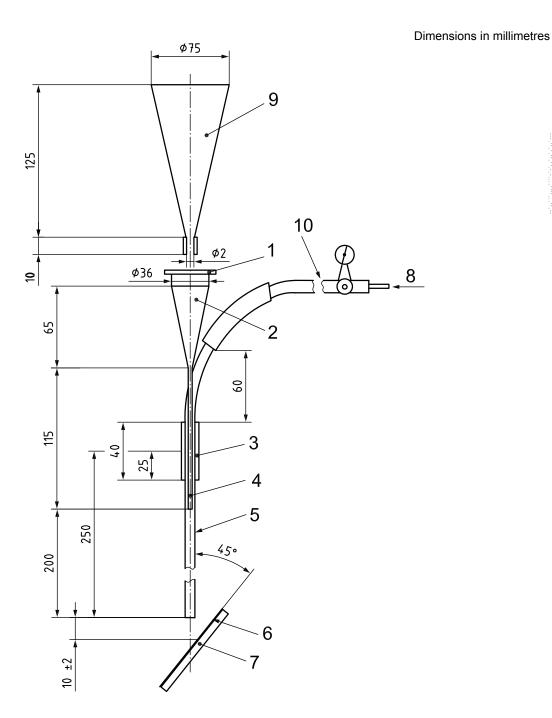


Figure D.2 — Details of suitable jet nozzle



Key

- 1 shutter
- 2 funnel
- 3 metal collar
- outer tube (push fit into metal collar) internal diameter (6,5 \pm 0,1) mm external diameter 8,5 mm minimum
- 6 test specimen
- 7 specimen-supporting stage
- 8 air or inert gas supply
- 9 hopper for abrasive powder
- 10 manometer or gas flowmeter (not shown)

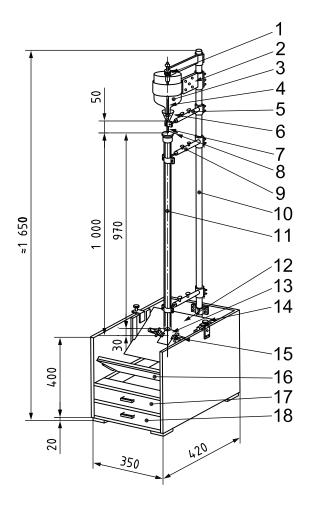
NOTE Dimensions are not critical where no tolerances are given.

Figure D.3 — General layout of alternative abrasive jet test apparatus

Design of falling sand abrasion test apparatus

- No restriction is placed upon the design of the falling sand abrasion test apparatus, provided that it E.1 complies with the general principles given in 7.2.
- Figure E.1 shows the basic design and layout of a suitable test apparatus.

Dimension in millimetres



Key

1	adjusting screw for falling quantity	7	funnel (leg)	13	test specimen
2	hopper supporter	8	shutter	14	screw for height control
3	hopper	9	supporter of guiding tube	15	stopper for test specimen
4	control-bar falling quantity	10	pipe prop	16	receiving plate for abrasives
5	funnel supporter	11	guiding tube	17	receiving box for abrasives
6	funnel	12	specimen-supporting stage	18	receiving box of auxiliary abrasives

Figure E.1 — Example of falling sand abrasion test apparatus

Bibliography

- [1] ISO 2106, Anodizing of aluminium and its alloys Determination of mass per unit area (surface density) of anodic oxidation coatings Gravimetric method
- [2] ISO 10074:2010, Anodizing of aluminium and its alloys Specification for hard anodic oxidation coatings on aluminium and its alloys

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