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Metallic and other inorganic coatings - Measurement of mass per unit area - Review of gravimetric and chemical analysis methods

金属和其他无机覆盖层 单位面积质量的测定

重量法和化学分析法评述

(ISO 10111:2000, IDT)

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WARNING - The methods referred to in this Standard can involve hazardous materials, operations and equipment. This Standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this Standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to its use.

1 Scope

1.1 General

This Standard outlines general methods for determining the average surface density over a measured area of anodic oxide or of a coating deposited autocatalytically, mechanically, by chemical conversion, by electrodeposition, by hot dip galvanizing and by vacuum using gravimetric and other chemical analysis procedures that have attained some degree of standardization.

A variety of procedures are described and include:

- gravimetric procedures for chemical or electrochemical dissolution of the coating or the substrate to determine the coating surface density;

- analytical procedures that utilize dissolution of the coating for photometric or volumetric determination of the coating surface density;

- non-destructive instrumental physical analysis of the coating to determine the surface density.

With the exception of the gravimetric method described in GB/T 9792, this Standard does not give the measurement uncertainties of the methods cited.

1.2 Sources

The stripping methods cited in annex A are described in specifications in the open literature or have been used routinely by at least one laboratory.

1.3 Restrictions

The procedures described can be used for many coating-substrate combinations. They cannot be used where neither the coating nor substrate material can be completely removed, one from the other by chemical or physical means and there is a constituent common to both that is not readily separated (e.g. nickel phosphorus alloy on nickel).

Note: The measurement of very thin coatings on very small pieces can result in a reduction in accuracy and a lack of repeatability. Several measurements using a combination of different procedures on similar samples might overcome this situation.

1.4 Limitations

In principle, the gravimetric procedures can be used to measure very thin coatings or to measure coatings over small areas, but not thin coatings over small areas. The limits depend on the required accuracy; e.g., 2.5 mg/cm^2 of coating might require 1 cm^2, but 0.1 mg/cm^2 of coating would require 25 cm^2 to obtain 2.5 mg of coating. These limitations do not apply to the chemical analysis methods.

The gravimetric method does not indicate the presence of bare spots or sites with thicknesses lower than the specified minimum in the measuring areas. In addition, the single value obtained from each measuring area is the mean thickness of that area. There can be no further mathematical analysis of this single value, for example, for statistical process control purposes.

2 Normative references

The following documents contain provisions which, through reference in this Standard, constitute provisions of this Standard. For the dated references, their subsequent amendments (excluding corrections) or revisions do not apply to this Standard. However, the parties who enter into agreement based on this Standard are encouraged to investigate whether the latest editions of these documents are applicable. For undated reference documents, the latest editions apply to this Standard.

GB/T 3138-1995 Terminology for metallic coating, chemical treatment and related process (neq ISO 2079:1981)

GB/T 8015.1-1987 Test methods for thickness of anodic oxidation coatings on aluminium and aluminium alloys - Gravimetric method (idt ISO 2106:1982)

GB/T 9792-2003 Conversion coatings on metallic materials - Determination of coating mass per unit area - Gravimetric methods (ISO 3892:2000, MOD)

GB/T 9799-1997 Metallic coatings - Electroplated coatings of zinc on iron or

6 Preparation of test specimen

6.1 Size

The specimen shall be large enough to permit area and mass measurement of adequate accuracy. (See clauses 8 and 9.)

6.2 Shape

The shape of the test specimen should be such that the surface area can be readily measured without difficulty. A rectangular or circular test specimen is usually suitable.

6.3 Edge condition

If the area to be measured is small and has to be known accurately, the edges may need to be dressed to remove smeared coating, to remove loose burrs and to provide well-defined and (for rectangles) straight edges. This should be considered for areas less than 100 mm^2.

One method of dressing the edges of a rectangular specimen involves clamping the specimen between two plastic or metal blocks with the edge of the specimen flush with the edges of the blocks and then grinding and polishing the edges metallographically.

6.4 Heat treatment

If the substrate has to be dissolved in such a way as to leave the coating intact, it may be desirable to first heat-treat the test specimen so that the coating will not curl up tightly or fall apart. Some gold deposits of 1.5 mg/cm^2 (< 0.9 μm) will fall apart when their substrates are dissolved, but will support themselves after heat treatment at 120 °C for 3 h. If the thickness of a coating (instead of its surface density) is being determined, a heat treatment that could change the density of the coating material shall not be used.

7 Measurement of coated area

7.1 Measurement method

Since the accuracy of the area measurement shall be greater than the desired accuracy of the surface density measurement, the method of measuring the area depends on the desired accuracy and the specimen size. The measurement uncertainty of the gravimetric method is normally less than 5 % over a wide range of thicknesses (see GB/T 9792).

there may be several analytical methods.

9 Gravimetric determination of mass of coating

9.1 Specimen size

Since the measurement uncertainty of the mass measurement shall be less than the desired measurement uncertainty of the surface density measurement, the test specimen shall be large enough for the coating to be weighed with the desired accuracy.

9.2 Gravimetric analysis equipment

A balance is required for gravimetric analysis, but the required sensitivity of the balance depends on the size of the test specimen, the coating thickness (coating mass) and the required accuracy of the measurement. The analytical balance shall be capable of weighing to an accuracy of 0.1 mg for weighing the test pieces under examination before and after dissolution of the coatings. For anodic and cathodic dissolution, a constant d.c. source is necessary.

9.3 Procedure

9.3.1 General

The mass of coating may be determined by:

a) weighing the test specimen before and after dissolving the coating (see annex A) and taking the difference (see 9.3.2) or

b) dissolving the substrate (see annex A) and weighing the coating directly (see 9.3.3).

Note: The first time a gravimetric method is used, it should be evaluated in accordance with 9.3.2.2 and 9.3.3.2.

9.3.2 Difference method

9.3.2.1 First, clean the test specimen of any foreign material, then rinse it with alcohol (methanol, ethanol, isopropanol) or other suitable solvent, blow it dry with clean air and weigh it. Immerse the specimen in the appropriate reagent (see annex A) to dissolve the coating either chemically or electrochemically, rinse it with water, then with alcohol, blow it dry with clean air and weigh it again. The loss of mass is the mass of the coating.

9.3.2.2 To determine if any dissolution of the substrate has occurred, repeat the process with the stripped substrate making sure that the substrate is immersed

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