THICKNESS TESTING OF ELECTROPLATED AND RELATED COATINGS

VOLUME 2

Edited by S.W. Baier

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INSTITUTE OF METAL FINISHING Exeter House, 48, Holloway Head, Birmingham B1 1NQ INDUSTRIAL AND TECHNICAL COMMITTEE (Chairman: Robert Pinner)



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Foreword to Volume 2

As stated in the Foreword to Volume 1 the purpose of this book is to describe the methods which can be used to measure the thickness of metallic coatings (on both metallic or non-metallic substrates) and of inorganic coatings (such as anodic oxide and phosphate coatings) on metallic substrates, but excluding the measurement of paint or other organic coatings although some of the methods may also be satisfactory for these coatings. The book attempts to give sufficiently detailed treatment of the test methods so that any of those described can be carried out without referring to any other literature, apart from instructions by instrument suppliers on their particular instruments.

For convenience the book has been divided into two parts—Volume 1, dealing with "Destructive Testing Methods" and Volume 2, with methods which in general are "Non-Destructive".

In contrast, most of the non-destructive methods, such as the magnetic, eddy current, beta back-scatter and X-ray spectrographic methods, can only be considered as "secondary" methods as the test instruments must be calibrated, often at frequent intervals, with calibration standards having known thicknesses (measured by primary methods) of the specific coatings on appropriate substrates. However, there are also a few non-destructive (or almost non-destructive) optical and profilometric methods which do not require calibration standards and can, therefore, be considered as primary methods, although except for a few optical methods used for measuring oxide films on aluminium, they are seldom used except for measuring the coatings on calibration standards required for other methods.

Introduction to Volume 2

As stated in the Introduction to Volume 1 the Institute of Metal Finishing Industrial and Technical Committee, under the chairmanship of Robert Pinner, realised that a publication giving comprehensive details of the methods which can be used for measuring the thickness of metallic and related coatings would be of great value both to metal finishers and those employing their products. A working party was therefore given the task of producing these 2 volumes.

The types of coatings for which satisfactory thickness testing methods are available include:

- (1) Electroplated coatings on both metallic and non-metallic basis materials—or 'substrates', as they are usually called nowadays when they are considered as the materials underlying the coatings.
- (2) Electroless coatings, such as autocatalytic nickel-phosphorus alloy coatings, on both metallic and non-metallic substrates.
- (3) Chemical replacement coatings on metallic substrates, such as tin coatings on aluminium.
- (4) Hot dipped coatings on metallic substrates, such as zinc (hot-dip galvanized) coatings on steel and hot-dip tin coatings on steel and copper.
- (5) Anodic coatings on aluminium and other metals.
- (6) Conversion coatings on metallic substrates, such as phosphate coatings on steel and chromate (passivation) coatings on zinc, cadmium or aluminium.
- (7) Vitreous enamel coatings on metals.

A number of the methods, especially some of the non-destructive methods are equally applicable to measurement of paint and other organic coatings on metals; but only brief references are made to these applications as they were not considered to come within the scope of this particular I.M.F. working group.

Before giving guidance on the most suitable methods to use it might be useful to consider the basic reasons for carrying out thickness tests. In a few cases, such as building-up worn parts with electrodeposited nickel or chromium, the main reason for thickness testing is to ensure that the dimensions of the coated parts will fit properly when assembled. However, in most cases some definite minimum thickness of coating is required to ensure some specific surface property, such as resistance to wear, corrosion

protection, solderability or electrical conductivity (or perhaps insulation in the case of anodic coatings on aluminium).

TESTING FOR MINIMUM THICKNESS

Even with coatings considered as purely decorative, such as decorative chromium or gold plating, it is necessary to have some minimum thickness in order to resist normal wear in handling (except when the coatings are protected by lacquering as is normal with decorative brass coatings over bright-nickel or vacuum evaporated aluminium coatings). On the other hand where considerable resistance to wear is needed—e.g., with hard chromium or hard anodising for engineering purposes—greater coating thicknesses will obviously be needed although for these applications it is also important that the coatings should have a specified wear-resistance, as shown by a direct wear-test and/or by a definite hardness range, as well as an appropriate minimum thickness.

Probably the greatest use of thickness testing is to ensure compliance with specifications for protective coatings. The service life of most decorative and protective coatings is greatly affected by the thickness of the coating and with sacrificial coatings such as zinc and cadmium, which are anodic to the substrate, the life of the coating is directly proportional to its thickness. Even with coatings which are strongly cathodic to the basis metal, the deposit thickness is critical in respect of the porosity of the coating and its ability to withstand pitting corrosion.

A minimum thickness of deposit is, therefore, an essential requirement of most coating standards. The choice of the most appropriate method for thickness testing, as well as being dependent on the particular coating, is also obviously dependent on the purpose for which the test is required. For routine inspection, or quality control, to ensure that production is up to specification thickness, the test method should be as rapid and economic as possible and wherever practical non-destructive methods should be chosen. It is generally considered, especially in American Standards, that accuracies of $\pm 10\%$ are quite satisfactory for this purpose; although for economic reasons, particularly with gold and other precious metal coatings it is often better to get greater accuracy in order to avoid making the coatings of these expensive metals even a few per cent thicker than they need be.

NON-DESTRUCTIVE TESTING FOR MINIMUM THICKNESS

Non-destructive testing is obviously the only way if testing should be required on 100% of the articles being coated and is obviously also the most economic way when large numbers of similar articles have to be tested. However, it should always be realised that non-destructive testing instruments have to be calibrated at fairly frequent intervals with standards having

accurately known thicknesses* of the appropriate coating on the appropriate substrate; and these can sometimes be rather costly to produce or purchase. Therefore, non-destructive testing may not prove really economic when only small samples, or a variety of different coatings, on various substrates have to be tested.

In practice, the two most frequently used non-destructive methods are:

- (1) Magnetic methods, which are widely used for measuring non-magnetic metallic (and organic) coatings on iron and steel;
- (2) The Eddy Current method which is mainly used for anodic oxide coatings on aluminium.

Many commercial instruments are available for both these methods, from quite cheap, but rather inaccurate, pull-off magnetic gauges to rather more expensive, but more accurate, electromagnetic and eddy current instruments.

A great advantage with both these methods is that plastics foils of known thicknesses can be used quite satisfactorily for calibrating these instruments for the normal ranges of coating thicknesses. For magnetic instruments, simple calibration with foils, placed on samples of mild steel will serve for calibration for testing any non-magnetic metallic coatings, such a zinc, cadmium, tin or copper, or paint or other organic coatings, on almost every type of mild steel; but with eddy current instruments calibration must be done with foils placed on a bare sample of the particular aluminium alloy which has been anodized. (The most recent development in magnetic testing is the production of special proprietary commercial instruments designed for testing nickel coatings on steel-i.e., for testing weakly magnetic coatings, with somewhat variable magnetic properties in the case of electroplated nickel, on strongly magnetic substrates. Such instruments, which are claimed to give very satisfactory results, could be expected to be very useful for routine testing of nickel plated steel articles, although they normally have to be calibrated with accurately known thickness of nickel coatings, and not with foils.)

The third very important non-destructive method is the beta back-scatter method, which is now used to a very considerable extent for measuring the thickness of gold or other precious metal coatings, particularly for the

$$E_{\text{(abs)}}$$
(Absolute measurement error) = $\sqrt{a^2 + b^2 + c^2}$

where a = Instrument error

b = Calibration standard error

c = Operational error.

Most instrument manufacturers provide error data for their instruments and calibration standards which they supply, but what cannot be pre-defined is error due to operation.)

^{(*}It should also be realised that the accuracy of any non-destructive method can never be better than the accuracy of the calibration standards and is normally considerably less accurate since the absolute measurement error depends on instrument and operation errors as well, as given by the formula:

electronics industries. Although beta back-scatter instruments are much more expensive than even the best electro-magnetic or eddy current instruments, they are usually well worth using since, on the one hand, it is most important to the electronics industries to be assured that the gold or other coatings they require are up to the thickness specified while, on the other hand, the electroplater wants to be sure that he is not wasting expensive metals by plating any thicker than he has to in order to meet the specifications—a matter which can make the difference between profit and loss. However, calibration of beta back-scatter instruments is nothing like as simple as calibration of magnetic or eddy-current instruments, calibration standards coated with very accurately known thicknesses being needed to enable the instruments to measure the various types of gold coatings or other coatings with the required accuracy. Also it is important that these calibration standards should have practically identical coatings, e.g., bright, dull or alloyed gold coatings, to those on the articles being tested and that the substrate of the standards should correspond to those of the articles. However, since there are no simple alternative methods (either destructive or non-destructive) for measuring the thickness of gold or other precious metal coatings, the beta back-scatter method has to be used wherever there is a call for regular routine testing of these coatings, i.e., except when the number of samples is so limited that microsectioning or chemical stripping might be used instead.

These three non-destructive methods each measure "local"* thickness as is needed to ascertain "minimum"* specified thicknesses on coated articles. In particular the beta back-scatter method is practically the only method, apart from microsectioning, that can be used to measure the local thickness on very small areas, or very small components, as is often required with electrical or electronic components, where it is often only necessary to meet a specified minimum thickness over quite small "significant surfaces".*

MEASUREMENT OF AVERAGE THICKNESS

In cases where coatings tend to be fairly uniform in thickness, as on barrel plated work and with hot-dip and sherardized zinc coatings and anodic coatings on aluminium, it would seem perfectly satisfactory to measure the average thickness over reasonably large areas (say, around $10 \, \mathrm{cm}^2$) instead of measuring the local thickness over an area of about $1 \, \mathrm{cm}^2$ or less. For this purpose strip and weigh methods are obviously satisfactory, although non-destructive tests may be applicable in some cases.

^{*}Definitions of these terms, "local thickness", "minimum thickness", "significant surfaces" (and "average thickness") and how they should be measured are given in the recent British Standard, 5411 (Part 1)—"Definitions and Conventions Concerning the Measurement of Thickness".

CHAPTER 5

Magnetic Methods for Measurement of Coating Thickness

INTRODUCTION

The thickness of any non-magnetic material applied as a coating on to a magnetic substrate can be measured by techniques loosely called "magnetic". There are, in fact, several methods available which in some way use the magnetic properties or a change in magnetic properties of the coating/substrate combination as a measure of coating thickness. For the purposes of this survey the methods have been divided into two main parts:

- (a) Permanent Magnet Methods
- (b) Electromagnetic Methods

The Permanent Magnet Method may be defined as a method using self generated magnetism derived from special magnetic retentive materials, such as alloys of iron, cobalt and nickel, and the Electromagnetic Method is one in which magnetism is generated by an applied electric current. The Permanent Magnet Method is frequently used commercially for small, cheap pocket sized devices giving accuracies at least within $\pm 10\%$ although under carefully controlled conditions there are some instruments with which it is possible to improve on this figure in individual cases. Electromagnetic methods are used increasingly in commercial instruments as these methods are capable of continuous further development and give accuracies within ± 2 to 5%, but at higher cost.

As well as measuring the thickness of non-magnetic coatings on magnetic substrates, magnetic methods, especially some of the electromagnetic methods, can be used for measuring weakly magnetic coatings on both non-magnetic and strongly magnetic substrates, such as electroplated nickel coatings both on non-magnetic materials such as brass and copper, and on strongly magnetic materials such as steel.

In the case of measuring non-magnetic coatings on magnetic substrates, the instruments (or gauges) either measure the reduced attraction of a permanent magnet to the substrate caused by the intervening coating or they measure a change in the magnetic properties of the sensor (probe) of the instrument, caused by the intervening coating. Therefore, they cannot give direct measurements of coating thickness unless they have first been calibrated with calibration standards. For measurement of these non-magnetic coatings, calibration can often be made very simply by using plastics (or non-magnetic metallic) foils or shims of known thickness placed over specimens of the appropriate substrate* as calibration standards, and the thickness of these foils can often be conveniently checked by the operator with conventional micrometers.

Alternatively, calibration standards can be produced from specimens of the appropriate substrates which have been coated with known thicknesses of coatings. However, these coatings have to be measured by suitable means† which are normally much more difficult than the measurement of the thickness of foils; but such coated calibration standards have to be used when the instruments are used for measuring weakly magnetic coatings, such as electroplated nickel coatings on both non-magnetic and strongly magnetic substrates.

Some of the simpler instruments, such as magnetic pull-off gauges, often display their results directly in thickness units—i.e., they have to be calibrated by the makers to suit the strengths of the permanent magnets and releasing springs employed in them. These "manufacturers calibrations" are usually quite satisfactory for testing non-magnetic coatings on mild steel substrates, which all have very similar magnetic properties, although rather more accurate results can frequently be obtained by actual calibration of the gauges by the operators employing them, especially on curved surfaces or where the gauge has been exposed to vibration, elevated temperature or strong external magnetic fields.

PERMANENT MAGNET METHODS

TYPES OF INSTRUMENTS

Instruments designed to measure the thickness of non-magnetic coatings (and in some cases para magnetic coatings, such as nickel) on magnetic substrates using permanent magnet techniques fall into three main categories:-

- (1) Direct Pull-Off
- (2) Controlled Pull-Off
- (3) Magnetic Flux Measurement

^{*}Calibration foils or shims are often particularly useful for calibrating gauges or instruments that are to be used for measuring the thickness of coatings on curved surfaces, since calibration with flat standards is often not applicable to measurements on curved surfaces.

[†]ASTM B499 suggests that either microsectioning or the coulometric test can be used for this purpose.