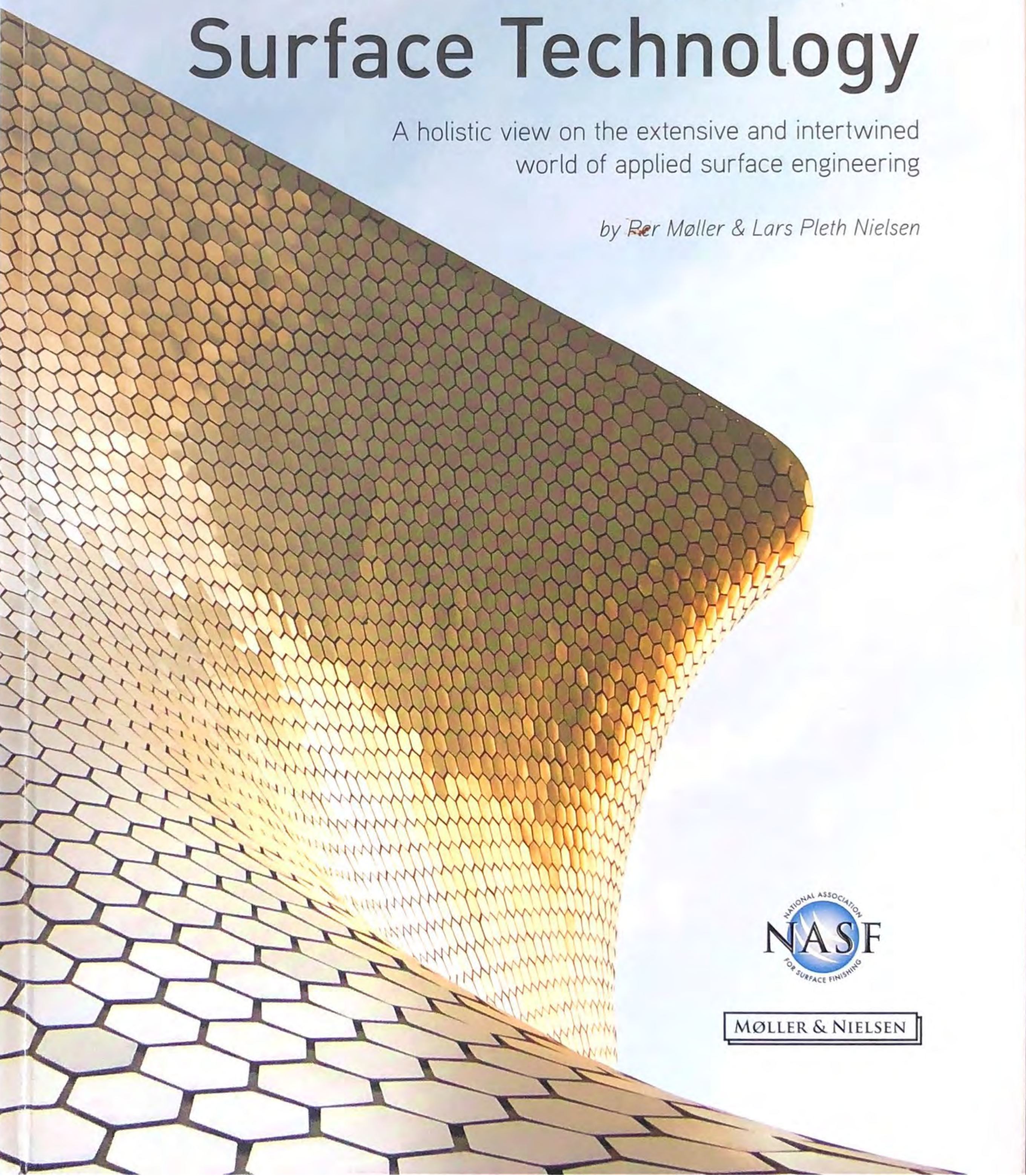
VOLUME 02

Advanced Surface Technology

A holistic view on the extensive and intertwined
world of applied surface engineering

by Per Møller & Lars Pleth Nielsen



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CHAPTER 33

QUALITY CONTROL; THICKNESS AND ADHESION OF COATINGS

33.1 INTRODUCTION

Destructive versus non-destructive testing

Coating thickness can be measured by a number of different techniques that are either destructive or non-destructive. Many end-users prefer to use non-destructive methods, since they can be used on the actual coated components and devices with the relevant geometries, without introducing defects in the surface coatings.

There are several non-destructive methods for determining coating thickness. Many of the methods are covered by relevant standards, which should be followed to achieve fast, accurate and reproducible results. The most commonly applied non-destructive methods are:

Non-destructive techniques

- X-ray fluorescence
- Eddy-current
- Magnetic induction
- Beta backscattering
- Micro-resistance
- Rutherford backscattering (RBS)
- Weight gain upon coating
- Ultrasonic
- Methods based on electrical resistance.

Furthermore, a number of destructive methods can also be applied, such as:

Destructive techniques

- Metallographic investigation (SEM/FIB-SEM)
- Cross-section analysis
- Grinding methods
- Dissolution of the coating followed by chemical analysis (e.g. titration)
- Weight loss upon removal of the coating.

In the following, we will discuss several non-destructive and destructive methods for determining coating thickness in further detail. When applying the various methods, it is recommended to consult the relevant standards covering the specific method.

Following the sections on determining coating thickness is a chapter discussing how to determine the reliability of coatings based on real weathering tests and accelerated application-oriented model tests.

33.2 MEASURING THE THICKNESS OF COATINGS

Measuring the thickness of coatings is of utmost importance for evaluating the expected specification of the coating, as well as validating and controlling the coating process in detail. Many coating processes do not provide a uniform material distribution across the entire component, and hence, it is important for the coating producer, as well as for the customer, to know that the coating thickness is within specifications. A coating thickness beyond maximum specification can become a major issue when coating high-precision components to tight tolerances. Similar critical demands can be put on the minimum thickness. This is especially the case when a component fails within its expected lifetime. It is clear that, when coating substrates with precious metals such as iridium, platinum, gold or silver, the reproducibility and control of the thickness attract special attention simply because the material cost of the coating represents a significant part of the overall coating cost. It is therefore optimal to stay within specification and not provide a thicker layer than necessary. As mentioned above, some of the methods applied to quantify coating thickness are destructive, and hence, it might be necessary to measure the coating thickness on dummy samples coated in the same batch or process line. Numerous international standards exist within the field of thickness quantification and they describe in detail how and where to measure representative coating thicknesses. Complicated components with several grooves and channels are not covered by standards since standards often only cover surface textures, which can be physically touched by a sphere of a given diameter.

Specifications

International standards

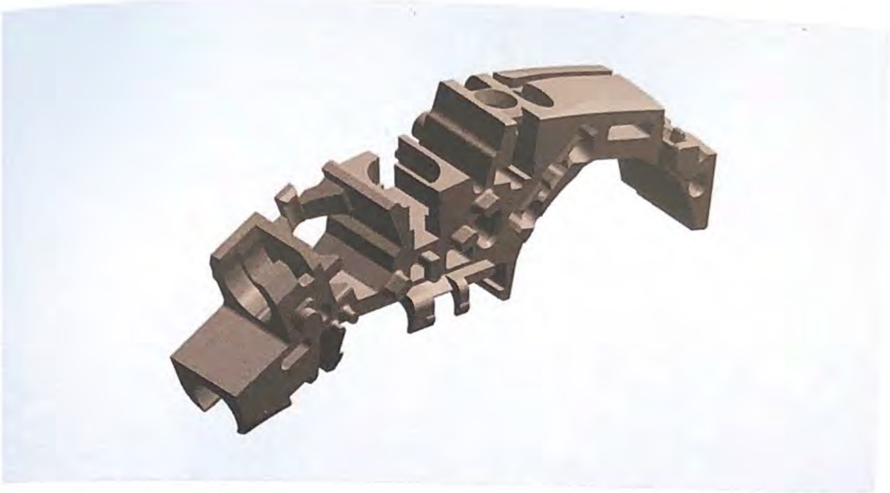

Figure 33.1: *Example of a difficult-to-coat component. Clearly, it will not be coated with equal coating thickness in all corners, recessions and edges when using electroplated coatings such as, e.g. hard chrome (see Chapter 11) or 'line-of-sight' PVD-based coatings (see Chapter 18). Image courtesy of Widex A/S, Denmark.*

There are a number of different methods for measuring the coating thickness. For a majority of these methods, there are commercial instruments available on the market. Most of the instruments and measuring procedures are, as mentioned earlier, covered by various international standards.

In the following, the most widely used instruments and methods for determining coating thickness will be discussed. Table 33.1 summarizes the different methods, some important standards are suggested in connection with the different types of measurements as well as potential coating types that the suggested technique can be used to quantify.

Coating	Substrate								
	Unalloyed magnetic steel	Austenitic unmagnetic steel	Al and alloys	Zn and alloys (die-cast)	Cu and alloys	Mg and alloys	Ti and alloys	Ceramic and glass	Polymer
Zn and alloys	A, B, C, CS, M, W, X	A, B, C, CS, W, X	A, B, C, CS, W, X	CS	B, C, CS, W, X	A, B, C, CS, W, X	A, B, C, CS, W, X	A, B, C, CS, E, W, X	A, B, C, CS, E, W, X
Cu	A, B, C, CS, M, W, X	A, B, C, CS, E, W, X	A, B, C, CS, W, X	A, B, C, CS, W, X	CS, W	A, B, C, CS, W, X	A, B, C, CS, W, X	A, B, C, CS, E, W, X	A, B, C, CS, E, W, X
Ni	A, B, C, CS, W, X	A, B, C, CS, W, X	A, B, C, CS, W, X	A, B, CS, W, X (†)	A, B, C, CS, W, X	A, B, C, CS, W, X (†)	A, B, C, CS, W, X	A, B, C, CS, W, X	A, B, C, CS, E, W, X
Electroless Ni[A]	A, B, C, CS, E, M, W, X	A, B, C, CS, W, X	A, B, C, CS, W, X	A, B, CS, W, X (†)	A, B, C, CS, W, X	A, B, C, CS, W, X (†)	A, B, C, CS, W, X	A, B, C, CS, E, W, X	A, B, C, CS, E, W, X
Cr	A, B, C, CS, E, M, W, X	A, B, C, CS, W, X	A, B, C, CS, W, X (†)	A, B, C, CS, W, X (†)	A, B, C, CS, W, X	A, B, C, CS, W, X (†)	A, B, C, CS, W, X	A, B, C, CS, E, W, X	A, B, C, CS, E, W, X
Au	A, B, C, CS, E, M, W, X	A, B, C, CS, E, W, X	A, B, C, CS, W, X (†)	A, B, C, CS, W, X (†)	A, B, C, CS, W, X	A, B, C, CS, W, X (†)	A, B, C, CS, W, X	A, B, C, CS, E, W, X	A, B, CS, W, X
Ag	A, B, C, CS, E, M, W, X	A, B, C, CS, E, W, X	A, B, C, CS, W, X (†)	A, B, C, CS, W, X (†)	A, B, C, CS, W, X	A, B, C, CS, W, X (†)	A, B, C, CS, W, X	A, B, C, CS, E, W, X	C
Sn	A, B, C, CS, E, M, W, X	A, B, C, CS, W, X	A, B, C, CS, W, X (†)	A, B, C, CS, W, X (†)	A, B, C, CS, W, X	A, B, C, CS, W, X (†)	A, C, CS, W	A, C, CS, W, X	A, B, C, CS, W, X
Vitreous enamel/glass	CS, M, W, X	CS, E, W, X	CS, E, W, X	–	CS, E, W, X	–	CS, E, W, X	CS, W, X	–
Paint	A, CS, M, U, W	A, CS, E, U, W	A, CS, E, U, W	A, CS, E, U, W	A, CS, E, U, W	A, CS, E, U, W	A, CS, E, U, W	A, CS, U, W	CS, U
Oxide coating[B]	CS, M, W, X	CS, E, W, X	A, CS, E	A, CS, E, W, X	CS, E, W, X	CS, E, W, X	CS, E, W, X	CS, W, X	CS, W, X
PVD/CVD[C]	A, CS, W, X	A, CS, W, X	CS, W, X	CS, W, X (†)	A, CS, W, X	CS, W, X (†)	A, CS, W, X	A, CS, W, X	A, CS, W, X

[A] Electroless nickel (above 8% P) unmagnetic

[B] Anodic coating upon aluminum or plasmasprayed oxide coatings

[C] For example: TiN, TiAlN, TiCN, CrN, DLC, TiO_2, Al_2O_3

† Strike or intermediate coating is a prerequisite before applying the specified top coat

Symbol	Method	Standards
X	X-ray fluorescence	ASTM B568
B	Beta backscatter	ASTM B567
E	Amplitude-sensitive eddy-current	ASTM B244, ISO 2360
M	Magnetic induction	ASTM B499, ASTM D7091, ISO 2178, ISO 2360
C	Coulometric	ASTM B504 - 90(2007), ISO 2177:2003
U	Ultrasonic	ASTM D6132-04/08
CS	Analysis of cross-section; Optical, SEM, calotte grinding, wedge cutting, etc.	ISO 2808, ASTM D4138, DIN 50986
W	Weight gain upon coating	–
A	Chemical measurement methods (after stripping of coating)	–

Table 33.1: *Overview of selected methods for measuring coating thickness on different materials. See also the discussion on TEM/SEM/FIB-SEM in Section 36.5, GDEOS in Section 36.9, SIMS in Section 36.10 and the different ion beam methods discussed in Section 36.12; especially Section 36.12.1 on RBS.*

33.2.1

WEIGHT GAIN UPON COATING

Non-destructive, weight-based measurements are best applied to rather thick coatings due to the uncertainty of the measurement technique. In the case of a simple geometry, the measured coating thickness can be compared with the weight difference before and after applying the coating. Assuming that the coating is uniform, the thickness of the coating can be determined from the following relationship:

Coating thicknes

$$d = \frac{\Delta W}{\rho_{coating} \cdot A} \tag{33.1}$$

Weight gain on test plates

Where d is the thickness of the coating, $\Delta W = W_{after} - W_{before}$ is the weight of the coating, $\rho_{coating}$ is the density of the coating, and A is the surface area of the coated component. The method is very attractive in the case of electroless plating as discussed in Chapter 14, simply because a plated surface can be treated together with the real component and be used as documentation, validating the correct average coating thickness. In this particular case it is possible to measure the thickness of the coating at submicron level, based on weight uptake quantified on test plates.

33.2.2 MECHANICAL MEASUREMENT

Measuring screw

Coordinate measuring machine

The simplest version of a mechanical measuring method is to apply a micrometer measuring screw on a simple geometry and measure before and after the coating has been deposited. Using a cylindrical geometry, the coating thickness will be measured twice, lowering the overall uncertainty. A more advanced geometric measurement can be performed with very high precision by a so-called *coordinate measuring machine* (CMM), which is a device capable of measuring the physical geometrical characteristics of an object in three dimensions (3D) with typical precisions in the order of 1 µm. However, new micro-CT (µCT) scanning methods can measure with precisions down to 5 nm. The principle of the CMM technology is to move a measuring head along three calibrated orthogonal axes, recording the x, y, and z coordinates for a number of pre-defined measuring points. An example of a CMM machine and a mechanical quartz probe, touching the samples to be measured mechanically, is shown in Figure 33.2.

Coordinate measuring machine

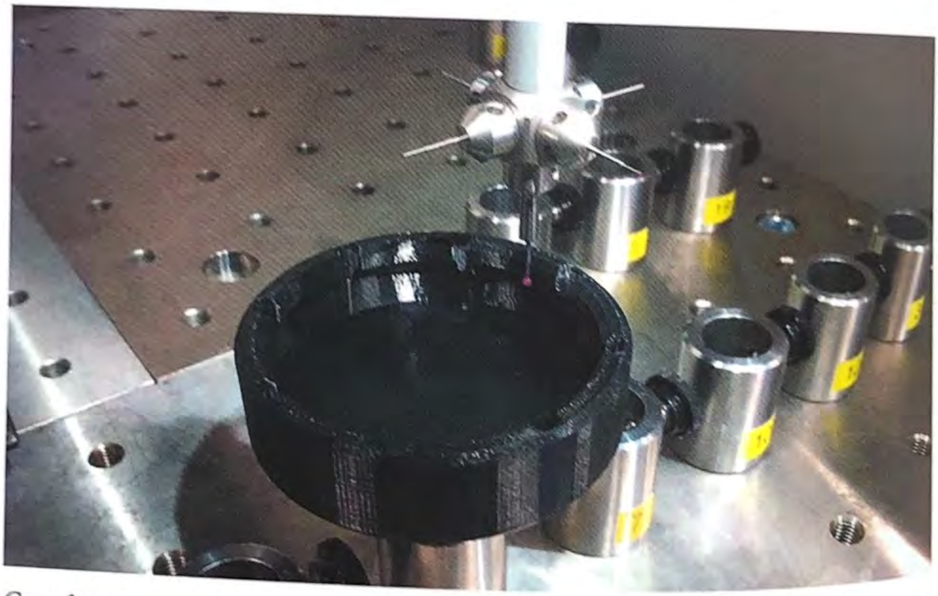

Figure 33.2: *Coordinate measuring machine that moves a probe around on a workpiece to be measured. The outcome of the measurement is a 3D-CAD file of the scanned object with micrometer precision. The workpiece is moved freely along the (x, y)-direction on an ultra-stable table with micrometer precision on an (x, y)-board. The third axis (z-axis) is provided by adding a vertical spindle or step motor, which moves up and down. The touch probe forms the sensing device on the end of the z-axis. Image courtesy of Danish Technological Institute.*

Different measuring principles (or probes) can be used, which may be based either on simple mechanical principles (i.e. probes physically touching the sample) or on more elaborated optical principles, based on interferometry using laser light or white light reflected from the object being scanned.

Mechanical/physical probes

Switches, strain sensors or cantilevers

A probe is moved to a number of pre-defined discrete measuring points and pressed against the workpiece. Upon contact between the probe and the surface, the point is defined as a surface point on the workpiece. The point of contact can be defined by applying switches, strain sensors or cantilevers. The mechanical probes are the least expensive and the most commonly applied type of probe.

Optical probes

The sensitivity is determined by the applied wavelength

This method scans the workpiece continuously, thereby generating a complete surface profile of the workpiece. Different light sources can be applied. In the case of lasers, the dimension of an object can be determined with accuracy down to one μm for most standard methods. The sensitivity can be increased even further by decreasing the applied wavelength into the X-ray region (see also the discussion in Section 36.14 on wavelengths).

33.2.3

CHEMICAL MEASUREMENT

Coating thickness measurement can also be based on chemical methods in cases where the coating can be stripped selectively from the workpiece in a suitable solution, which does not attack the base material.

Dissolving the coating

Stripping of coating followed by chemical analysis

The concentration of the dissolved coating material can be determined by *titration, atomic absorption* or *induced coupled plasma* (ICP). Alternatively, the component can be weighed before and after stripping the coating, and the coating thickness can be determined from the weight difference as in Equation (33.1). Chemical methods can be used to quantify the layer thickness of almost all metals. The method may also be used to determine the chemical composition of alloys and in some cases even the thickness of oxide coatings—if they can be removed selectively from the underlying substrate. These methods are relatively time-consuming and require a high degree of chemical knowledge and are therefore not suitable for standard process control. Such methods have earlier been used for quality control of zinc plated fasteners and nails (see Chapter 7). The method is clearly destructive and nowadays significantly more advanced methods have been introduced.

33.2.4

OPTICAL MEASUREMENT

Common for these methods is that they are based on a cutting/grinding of the subject perpendicularly to the coating to be measured, i.e. by making a metallographic cross-section, followed by an appropriate metallurgical preparation such as polishing. Optical methods are again destructive and incredibly time-consuming and require substantial training, if the uncertainty of the measurement is to be kept at an acceptable level.

Possible sources of error include:

- Surface roughness
- Non-perpendicular cross-section
- Deformation of coating
- Poor metallurgical preparation
- Uncertainty in the optical measuring instrument
- Non-representative area.

33.2.4.1 OPTICAL MICROSCOPY

In the case of optical measuring method, the thickness of the coating is determined by a calibrated optical microscope, where the thickness of the coating can be measured visually. Under optimal conditions, the thickness can be determined with an accuracy of about ±0.5 µm with these methods. For adequate layer thickness determination, there must be a clear contrast between the base material and the coating, so that the different layers can be distinguished unmistakably from each other. Polarization filters or etching can be applied to amplify the visual difference between different materials. The applied etching agent depends on the specific coating and substrate material. The method can be applied to surfaces with a thickness above one micrometer. If higher precision is needed or if the coating thickness is smaller, the thickness may also be determined by a *scanning electron microscope* (see the discussion in Section 36.5).

SEM methods

33.2.4.2 HARDNESS OR CHEMICAL COMPOSITION

Hardness and chemical composition perpendicular to the coating

If there is no color contrast between the surface and the base material, an alternative strategy can be applied to determine the coating thickness. In this case the thickness is defined as the point where either the chemical composition or the hardness of the coating changes. This change can be identified when scanning or moving from the surface region toward the base material in a sample cut, cleaved and polished perpendicular to the coating. Such a chemical analysis is typically carried out using EDAX/EDS as discussed in Section 36.6. In cases where the hardness of the coating is significantly different from that of the base material, it is possible to determine the layer thickness by quantifying a hardness profile by applying a nano-indenter or micro-Vickers hardness measurement (see Section 34.3). This is for example a standardized method for measuring the case hardening depth of thermo-chemical diffusion processes (see also Figure 22.6).

33.2.4.3

COATING EVALUATION ON PCB BOARDS

When evaluating the coating thickness and the quality of the production process in connection with through-hole plated printed circuit boards (PCB) with multilayers and blind holes, optical measurement methods are selected as the fastest and most reliable technique. Such types of measurements are often completely automatized based on small test structures integrated into the actual PCB product. Some of these test structures may even be positioned in such a way that they can be separated from the original PCB at a later stage for a comprehensive investigation based on a metallographic cross-section and SEM analysis.

33.2.4.4

THE WEDGE CUT METHOD

Projected optical images

The standardized wedge cut method is also an optical method. It is destructive since the specimen has to be cut in a well-defined angle by a carbide cutting tool. The resulting angular groove (see Figure 33.3) can be used to quantify the coating thickness. The thickness of the coating (d) is calculated from the projected dimensions (s) of the cut region, which is evaluated with an optical microscope. The coating thickness can be calculated from the known cutting angle α.

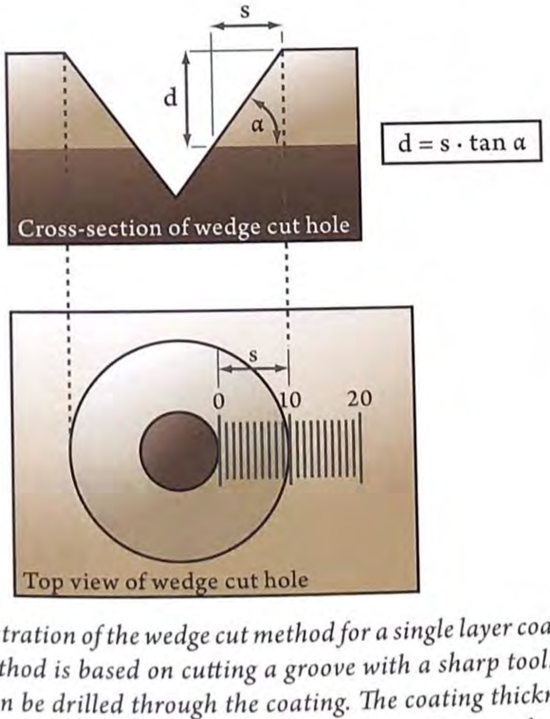

Figure 33.3:

Schematic illustration of the wedge cut method for a single layer coating system. This destructive method is based on cutting a groove with a sharp tool. Alternatively, a conical hole can be drilled through the coating. The coating thickness is calculated from the known angle a, and the projected distance s, or from the difference in the radii of the concentric circles (bottom view). The only thing needed is a calibrated optical microscope and a known angle a.

The method can also be used for measuring the individual layers of multi-layer coating systems (see Figure 33.4). It is suitable for measuring coatings such as micrometer ceramics on steel, paint systems on wood, concrete, plastic, metal, etc. The method is covered by several standards: ISO 2808, ASTM D4138 and DIN 50 986. The methods are reported for measuring thickness from a few microns up to several millimeters.

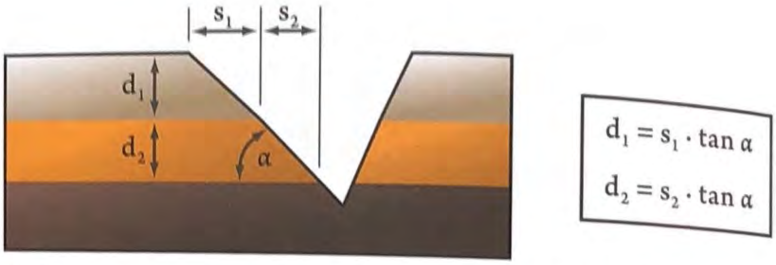

Figure 33.4: *A multi-layer coating system, where the thickness of the individual layers can be calculated using trigonometry and the measured surface projected distances s_1 and s_2.*

33.2.4.5 CALOTTE GRINDING

In the case of thin coatings (1–3 µm), the thickness can be quantified by a very simple method known as *calotte grinding*. The principle of the method is to rotate a hardened steel ball against the surface with an abrasive media (e.g. a diamond suspension), in order to grind through the coating. Subsequently, the projected surface area of the wear track can be evaluated.

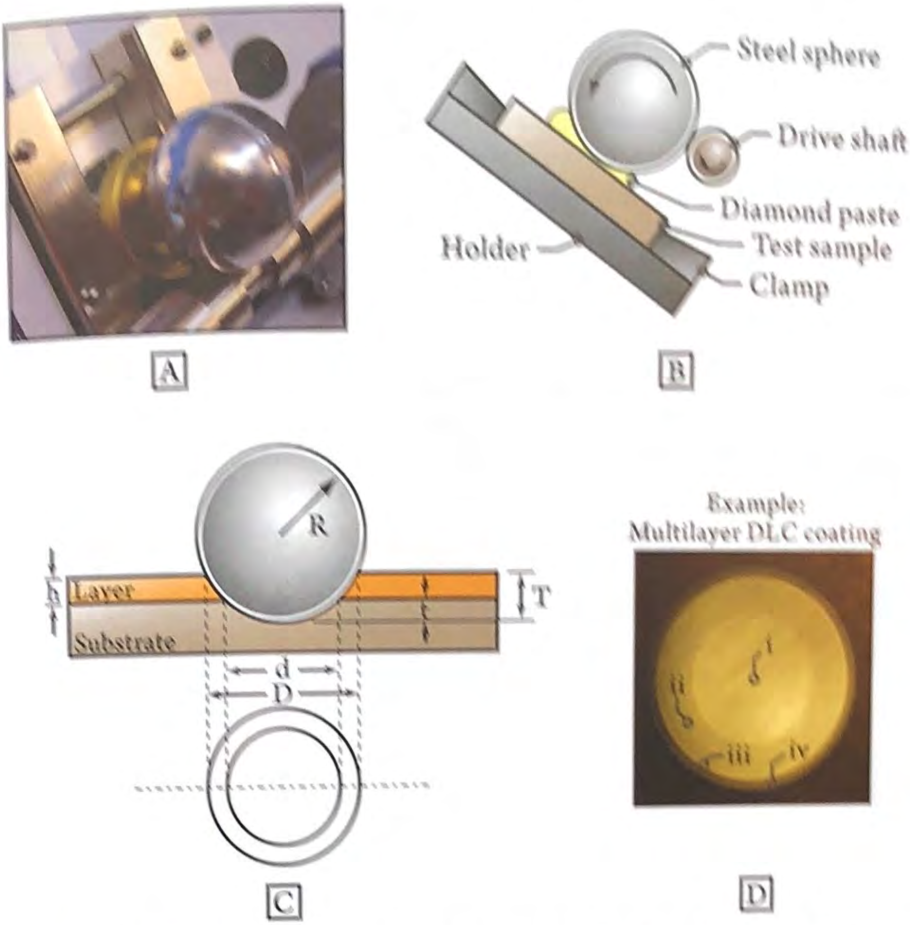

Figure 33.5: *Simple calotte grinding equipment using a rotating ball and an abrasive media (e.g. a diamond paste) for grinding through a single or multilayer surface coating. A and B: The ball is rotated by a spindle, mounted on a motor. The grinding time depends on the abrasive media, the coating thickness, as well as the hardness and wear resistance of the coating. Image of a piece of Calotest equipment courtesy of ST Instruments, the Netherlands, and CSM Instruments, Switzerland. C: Sketch of the wearing ball going through the coating. D: Optical microscope image of the grinding profile on a flat surface (multilayer DLC coating). Depending on the geometry, various grinding patterns will occur. Concentric circles appear on flat surfaces, whereas an ellipsoidal surface pattern of concentric ellipses will be obtained on curved surfaces. By measuring the dimension d and D, respectively, it is possible to calculate the coating thickness as:*

$$h = \left[R^2 - \frac{d^2}{4}\right]^{\frac{1}{2}} - \left[R^2 - \frac{D^2}{4}\right]^{\frac{1}{2}}$$

The DLC coating example comprises the following layers: (i) substrate, (ii) CrN adhesion layer, (iii) a CrN-CrN graded transition layer followed by (iv) the DLC top layer. The coating is deposited by PVD and is equivalent to the coating shown in Figure 20.16. The resolution of calotte grinding is approximately ±0.5 µm.

33.2.5 ELECTROCHEMICAL MEASUREMENT

The electrochemical method is also called the coulometric method

The electrochemical method is based on electrochemical removal of the coating material. The thickness of the coating is determined by measuring the accumulated charge needed when stripping the coating electrolytically from a known surface area. This is also called the *coulometric method* and is analogue to Faraday's law discussed in Section 4.3.

The method is shown schematically in Figure 33.6. During the dissolution process, the coating is the anode and the wall of the chemical cell acts as the cathode. The electrochemical dissolution process is carried out on a well-defined circular surface area. The consumed accumulated charge (Faraday's law) during the dissolution of the coating is measured and integrated over time. Combining this with the involved anodic dissolution reaction, it is possible to calculate the number of coating atoms dissolved from the coating, and hence, the coating thickness. When the coating is fully dissolved by the electrochemical process, the anodic potential will change and the measurement will be terminated. The method can also be used to determine the thickness of multilayer coating systems such as, e.g. a multilayer system comprised of a top layer of silver, followed by a copper layer and a thin nickel layer on top of a steel substrate. Each layer can be dissolved selectively by applying dedicated electrolytes specifically active in connection with each metal layer and/or specific anodic potentials suitable for dissolving each involved metal layer.

The measurement range of the coulometric method is in the range of 0.1 μm to about 50 μm. The clear advantage of the method is its flexibility as it can be used in connection with many different types of coatings and additionally the instrument is relatively cheap. One of the major drawbacks is that the measurements are rather time-consuming. Furthermore, the method requires some knowledge of relevant chemistry. Also, great care has to be taken to ensure that the surface area to be dissolved, is clean and not surface-passivated, preventing its complete dissolution. Clearly, the method is destructive since part of the coating is etched away.

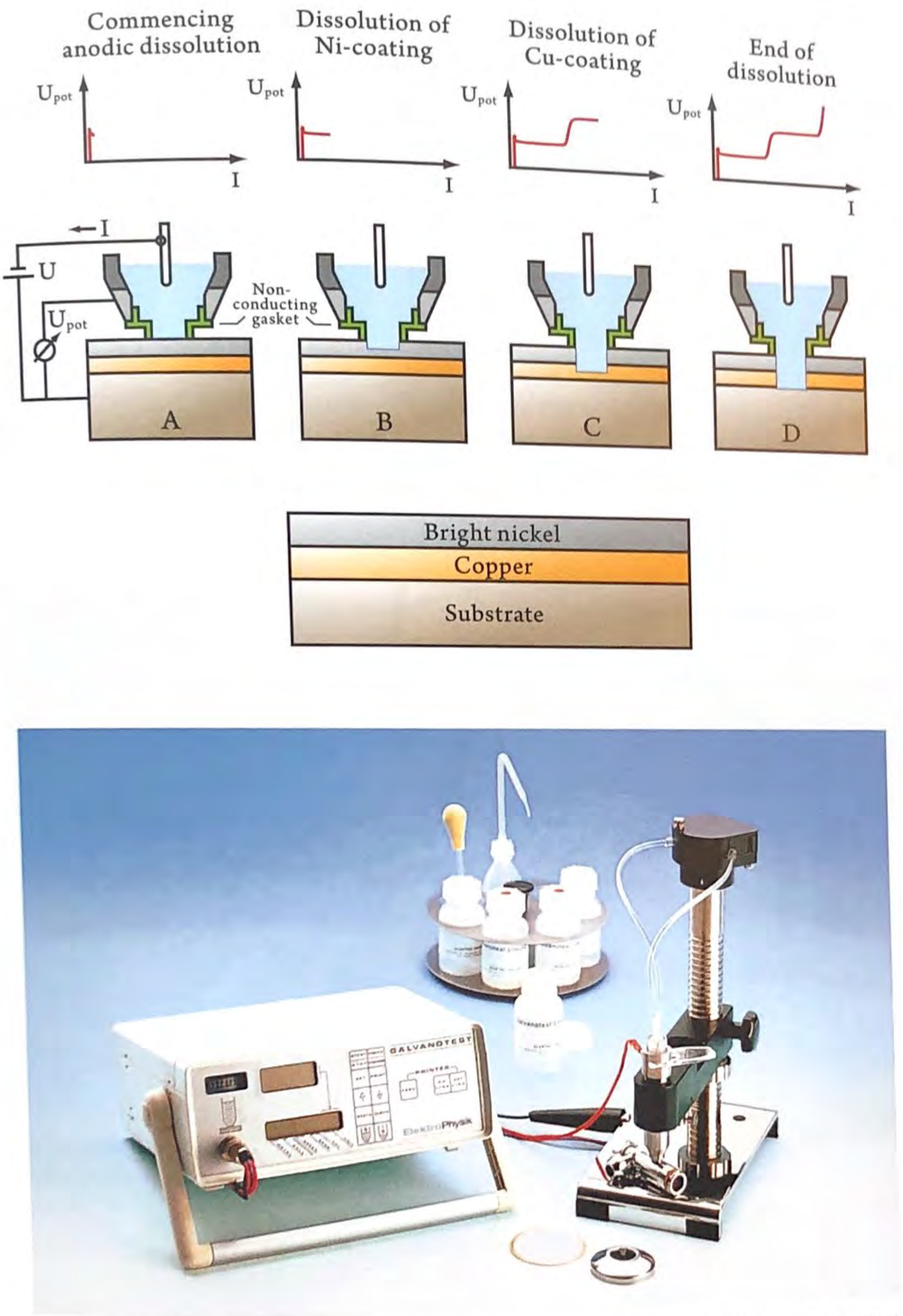

Figure 33.6: *Top: The principle of the coulometric method according to ASTM B504 - 90(2007) and ISO 2177:2003. The walls of the chemical cell act as cathodes and the surface coating to be dissolved is the anode. The packing material (i.e. a gasket) defines the surface area of the coating to be dissolved and seals the cell. For illustration purposes, the associated voltage change when dissolving, for example bright nickel and copper coatings, in the chemical cell, is presented above the process steps. Bottom: Representative equipment used for the coulometric method. Image courtesy of ElektroPhysik, Germany.*

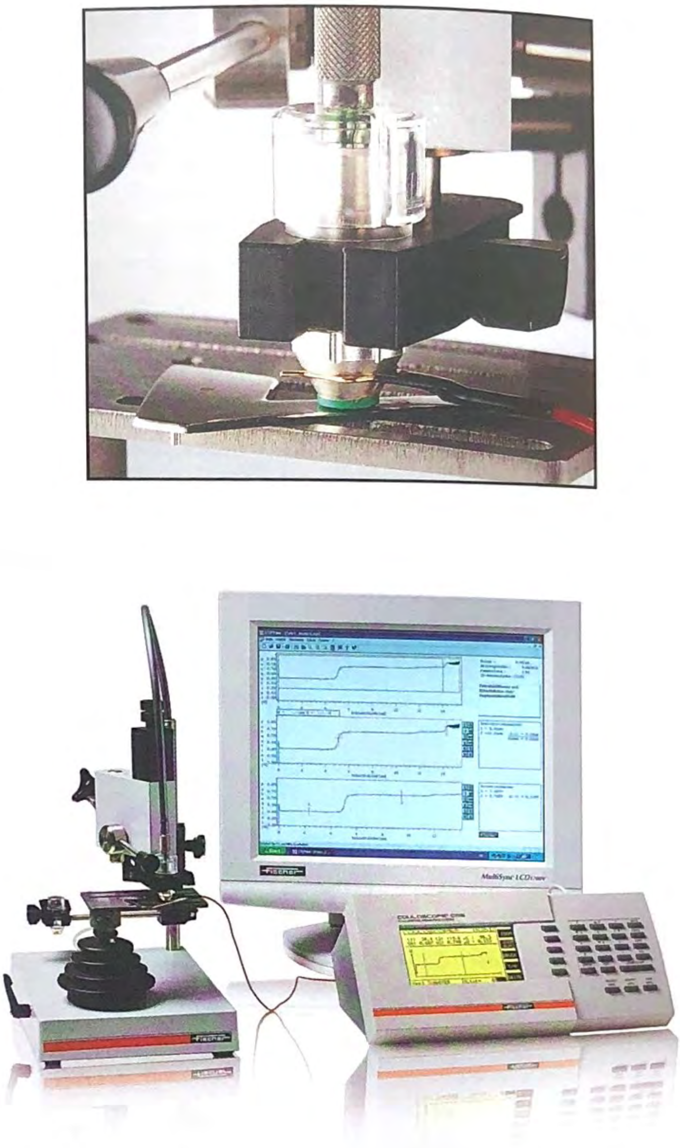

Figure 33.7: *The Helmut-Fischer COULOSCOPE® instrument for measuring coating thickness. Images provided by Helmut-Fischer, Germany and Chembo Overfladeteknik A/S, Denmark.*

MAGNETIC MEASUREMENT

33.2.6

Best for non-magnetic coatings on magnetic substrates

The magnetic method has for many years been the most prevalent method for determining the coating thickness of non-magnetic coatings on top of a magnetic substrate. The method is based on the fundamental fact that the magnetic attraction decreases as the thickness of a non-magnetic coating on top of an underlying magnetic substrate increases. The measuring principle is based on lifting a magnet away from the surface by means of a spring connected to a magnet arm. The force needed to lift the magnet is directly related to the thickness of the coating. If the coating is thick, the magnetic force is lowered. Often the magnetic force is recorded mechanically by a torsion spring or a linear spring. These gauges are designed to measure thickness of a non-magnetic coating on a magnetic substrate. However, in special cases it might also be possible to measure the thickness of a magnetic coating on a non-magnetic substrate. The measuring principle is also used for electroplated nickel (or other magnetic coatings) on top of a non-magnetic substrate such as, e.g. copper or aluminum.

The method is relatively uncertain (±10% and at least ±2.5 μm). Because of the large uncertainty, the method is only used in connection with relatively large coating thicknesses, where measurement accuracy and absolute thickness is not of major importance. The method is used to a certain extent for a rapid assessment of the thickness of zinc coatings on hot dip galvanized constructions.

Stable permanent magnet method

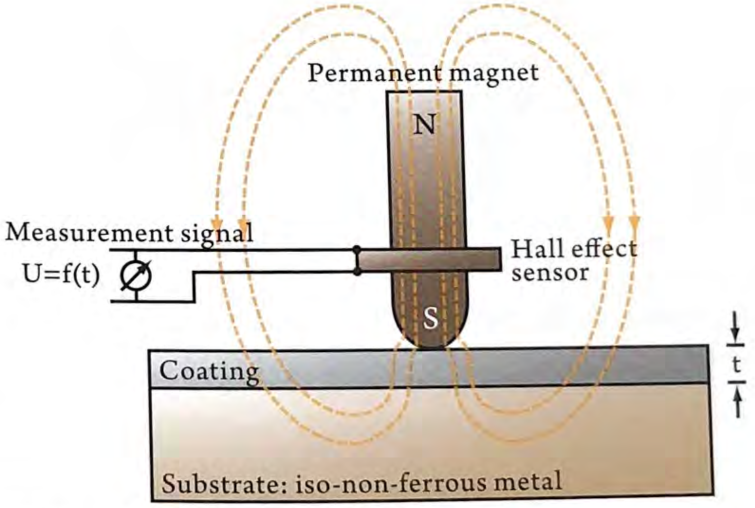

Figure 33.8:

A: Schematic illustration of the magnetic method. The surface to be characterized is approached with a permanent magnet. The magnetic field strength generated by the permanent magnet will be proportional to the distance between the probe tip and the substrate. The magnetic field strength is measured by a Hall sensor and the signal can be calibrated to the thickness of the coating (see also ISO 2808).

33.2.7 MAGNETIC-INDUCTIVE MEASUREMENT

Non-magnetic coating on a ferrous substrate

The magnetic-inductive method is an electromagnetic method, which can be used to determine the thickness of non-magnetic coatings on ferrous substrates and in some cases also the thickness of magnetic coatings on a non-magnetic substrate. When the probe is positioned on the sample, the induced magnetic field is measured as a function of the distance between the probe and the base material. It is one of the most commonly used methods for measuring coating thickness because the instrument is relative inexpensive, reliable and rather simple. The measurements are non-destructive, very fast, and can be carried out with a small measuring probe, e.g. the size of a pencil, which can easily be moved across the surface to be examined.

The method is mostly used for quantifying the coating thickness of non-ferromagnetic coatings on magnetic substrates such as, e.g. polymers, paint, lacquer, powder coatings, enamel as well as metal coatings such as chromium, copper, tin, zinc, cadmium or phosphate on top of, e.g. iron or steel.

Induced magnetic field can be corrolated to coating thickness

The measuring principle can be described as follows: a measuring probe is placed on the coated surface. A low frequency magnetic field is generated by applying a low frequency AC current through a coil. Placing a ferromagnetic material (i.e. the magnetic base material) in the magnetic field will change the magnetic flux. The change in the magnetic field is registered by a measuring coil, a so-called *pick-up coil*, and can be converted to a value, which indicates the distance from the probe to the magnet substrate, equal to the coating thickness.

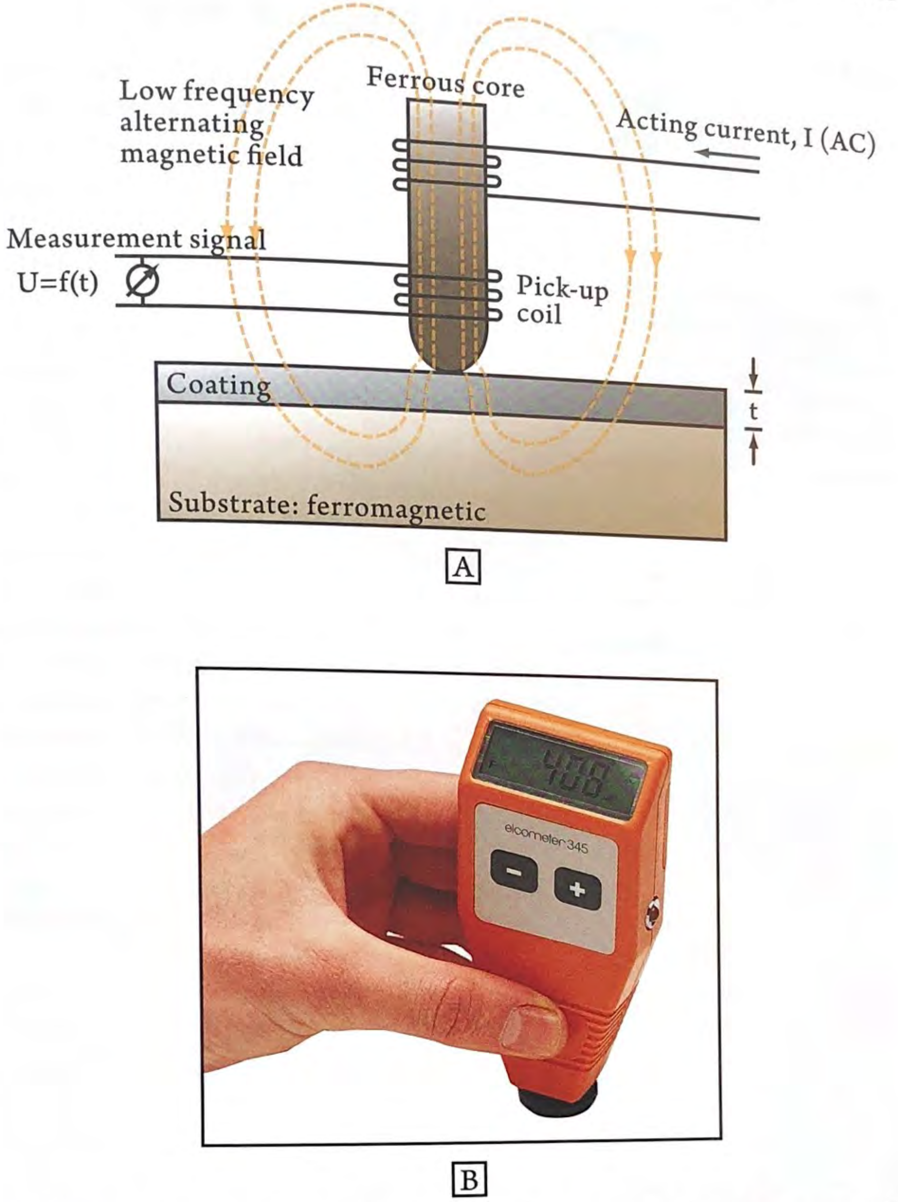

Figure 33.9: *A: Schematic illustration of the magnetic-inductive method used to measure the thickness of non-magnetic coatings (e.g. zinc, copper, organic coatings, etc.) on top of magnetic substrates. As mentioned above, the measuring principle is based on detecting the induced magnetic field in a secondary coil that, by means of a calibrating procedure, will provide the coating thickness (see ASTM B499 and ISO 2178). B: Example of handheld equipment for measuring the coating thickness of, e.g. paints on metal. Image courtesy of Elcometer Limited, England.*

33.2.8 EDDY-CURRENT MEASUREMENT

The eddy-current technique is used for measuring both non-magnetic, metallic coatings (zinc, cadmium, copper, etc.) on top of steel as well as non-conductive coatings on non-ferrous metals, such as anodized or painted aluminum. It might also be used for non-magnetic conductive coatings on non-conductive substrates (like aluminum deposited on a polymer substrate).

Induced eddy-current can be used to calculate the coating thickness

If a conductive material is placed in an alternating magnetic field (AC-field), there will, according to Maxwell's equation, be induced an eddy-current in a conductive material. The magnitude of these eddy-currents will be proportional to the applied frequency, the resistance of the conductive coating and the distance between the probing coil and the substrate. The induced eddy-current in the conductive layer generates an opposite directed magnetic field, which weakens the initial superimposed magnetic field. Thus, by measuring the combined magnetic field (the initial magnetic field and the generated magnetic field), it is possible to determine the layer thickness.

For non-conductive coatings, a gap between the probe and the non-magnetic basic material is introduced. This gap causes a loss in the eddy-current, which again can be used to determine the coating thickness.

For conductive coatings on steel, a magnetic field is generated in both the base material and in the coating. The eddy-current loss will then be a function of the thickness of both the surface coating and the basic material and will be somewhere between what can be measured directly on pure samples of each material. Clearly, the method is non-destructive.

Two types of eddy-current techniques

Two types of eddy-current techniques are used commercially: (i) *amplitude-sensitive eddy-current*, and (ii) *phase-sensitive eddy-current.*

The first method is a well-known method for measuring the thickness of a non-conductive coating on non-ferrous metal, such as an anodized layer on aluminum and magnesium or paint on light metals such as aluminum, manganese, and zinc. It might also be used for non-magnetic conductive coatings on non-conductive substrates (like aluminum deposited on a polymer or a ceramic substrate). The method is described in ISO 2360:2003 and ASTM B244-09.

The second method is able to measure many different coatings on various substrates when there is a significant difference in conductivity between substrate and coating. Phase-sensitive eddy-current can measure coatings such as zinc on steel with good precision. Another benefit of the phase-sensitive eddy-current method is that it can be used to accurately determine the thickness of a zinc coating sandwiched between a paint and a steel substrate.

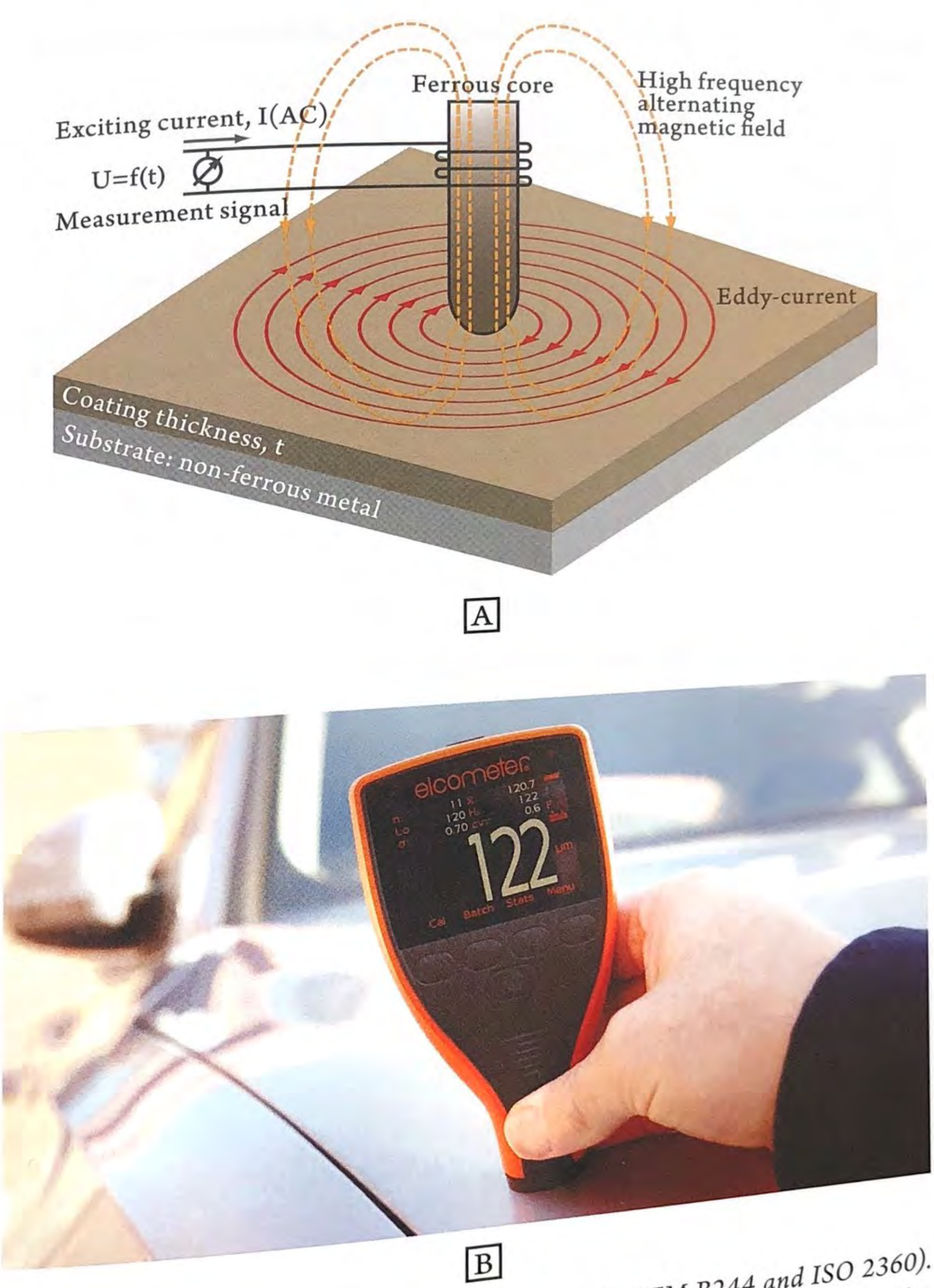

Figure 33.10: *A: Schematic drawing of the eddy-current method (ASTM B244 and ISO 2360). The principle can be applied to determine the layer thickness of a non-conductive coating on non-magnetic metals. B: Example of handheld equipment based on the eddy-current principle for measuring the coating thickness of, e.g. paints on aluminum. Image courtesy of Elcometer Limited, England.*

33.2.9 X-RAY FLUORESCENCE MEASUREMENT

As discussed extensively in Section 36.11.2, the X-ray fluorescence method relies on the formation of vacancies in surface atoms, when irradiating the surface with energetic X-rays, as illustrated in Figure 33.11 below.

Vacancy formation and re-exitation

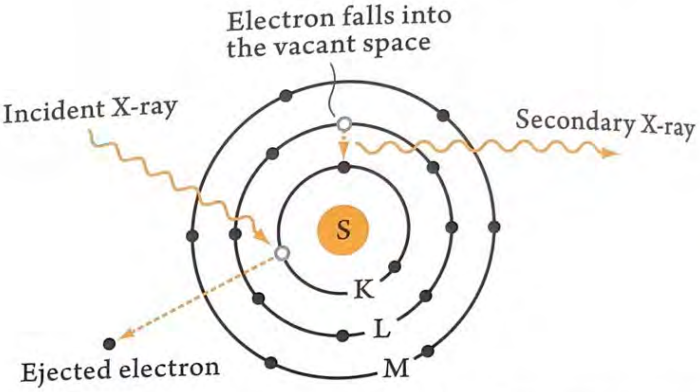

Figure 33.11: *Schematic illustration of the formation of a K-shell vacancy in a sulfur atom surrounded by 16 electrons. The K-shell electron is kicked out by an incoming X-ray. The vacancy is subsequently filled by the decay of a L-shell electron falling into the K-shell. The energy difference is converted to a secondary X-ray photon having energy unique for sulfur. Detection of photons with exactly this energy proves that the sample contains sulfur. The number of photons with exactly this energy can be calibrated to the concentration of sulfur.*

RoHS in PE Matrix

```
2
Cr: 100 ± 54 ppm
Cd: 2017 ± 85 ppm
Pb: 0 ± 755 ppm
Hg: 0 ± 877 ppm
Br: 0 ± 13 ppm
1
Cr: 89 ± 87 ppm
Cd: 2037 ± 62 ppm
Pb: 0 ± 751 ppm
```

Figure 33.12: *Example of an X-ray fluorescence setup capable of determining the composition and thickness of a coating. The insert shows a PCB being tested for cadmium. Images courtesy of Oxford Instruments plc, England.*

As indicated in Figure 33.11, an X-ray fluorescence photon is sent out through a process which fills the inner core vacancy formed by initially incoming primary X-rays. When the exited atoms decay to their ground state, they will release photons having specific X-ray energies known as X-ray fluorescence. The energy and intensity of the specific photons can be used to identify the type and the concentration of the specific type of atoms. Furthermore, since both the coating and the underlying substrate will produce X-ray fluorescence, it is possible to quantify the coating thickness since the substrate signal is reduced (attenuated) when passing through the coating. Since the degree of attenuation depends on the characteristics of the coating, in particular its density and thickness, it is possible to quantify the coating thickness when applying calibration samples. It is important to notice that a special application of the instrument (measuring an alloy coating thickness and its composition or the thickness of a multilayer system) will always be based on comprehensive calibration with several standards with different thickness and/or alloy compositions. See also the discussion in Section 36.11.2.

Attenuation of X-ray can be used to measure coating thickness

Thus, X-ray fluorescence provides the opportunity to determine alloy coating composition and thickness in the same measurement. The method can also be used to measure the thickness of two surfaces on a base material. Equipment of this type can measure thickness of both gold coatings as well as nickel coatings plated on top of each other, for example with copper as base material. The method is non-destructive.

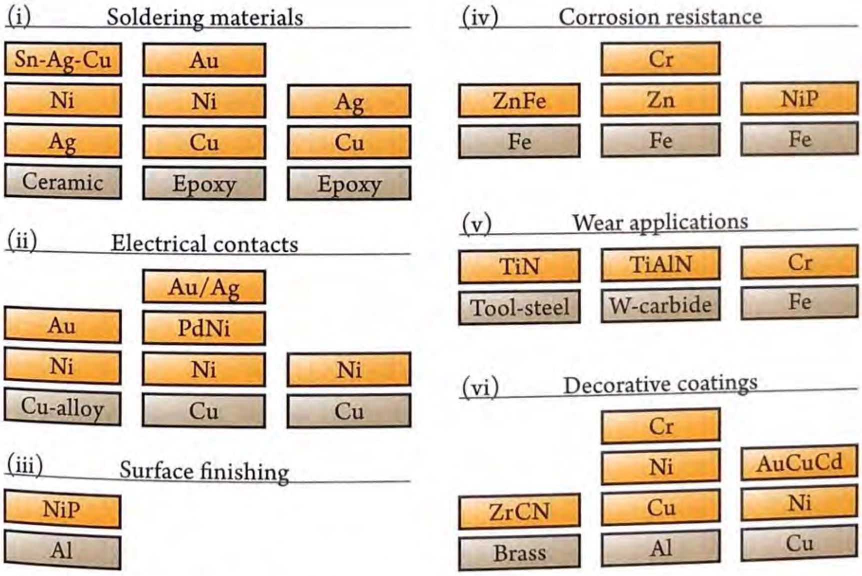

Figure 33.13: *Illustrative examples of where X-ray fluorescence is used as an extremely powerful technique enabling process control and characterization of combinations of many different materials in areas such as: (i) soldering materials, (ii) electrical contacts, (iii) surface finishing, (iv) corrosion resistant coatings, (v) coatings for wear applications, and for (vi) decorative coatings.*

X-ray fluorescence is the most precise measurement method, especially for small-diameter parts, or dual coatings such as gold and nickel on top of copper. Since the price of X-ray fluorescence equipment has been reduced drastically over the years and since new generations of user interfaces has made it significantly easier to operate the devices, it is increasingly becoming a standard technique for quantifying coating thickness. Hereto comes that the method is not only capable of determining the coating thickness, but can also determine the coating composition as well as identify impurities.

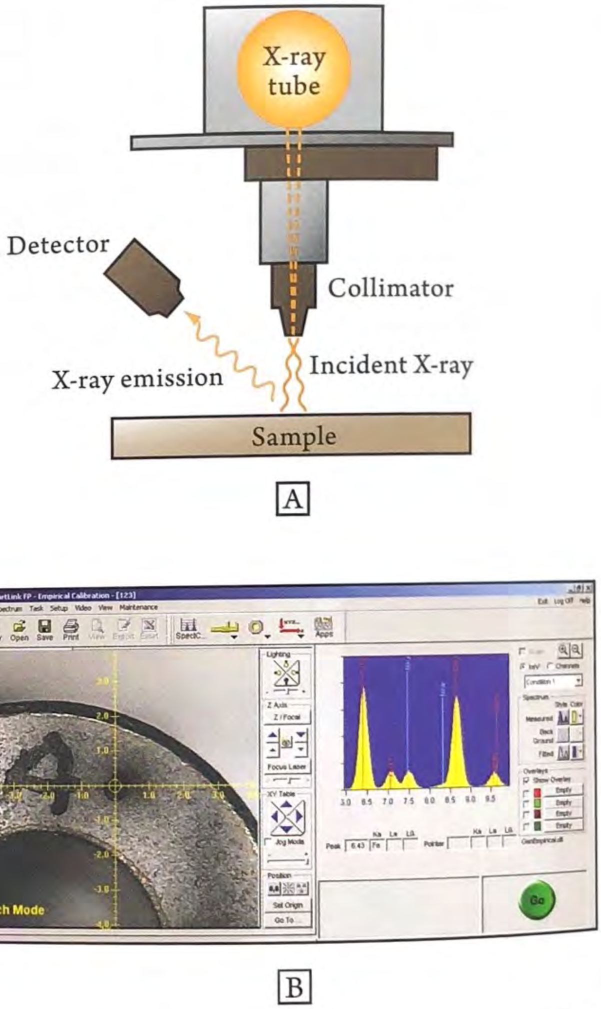

Figure 33.14: *A: Schematic illustration of the principle of X-ray fluorescence (ASTM B568). B: Example of an X-ray fluorescence spectrum. Image courtesy of Oxford Instruments plc, England.*

The function of the collimator is to restrict the measurement area to avoid it extending beyond the edges of the feature. X-ray fluorescence equipment makes it possible to measure areas as small as 150 μm in size.

33.2.10 BETA BACKSCATTER MEASUREMENT

The thickness of the coating is determined by the intensity of backscattered electrons

The principle of this method is based on the fact that beta rays (electrons) are generated from an unstable radioactive isotope (see also the discussion in Section 36.5.1, where the interaction of electrons with matter is discussed extensively). The beam of electrons is directed toward a surface (e.g. a printed circuit board plated with gold on nickel). Beta rays will penetrate the plating layer and be reflected back (back-scattered). Beta radiation can be collected and recorded using a detector such as, e.g. a standard Geiger-Müller tube (GM-tube). The intensity of the signal can be converted into a coating thickness. The method assumes that the atomic number of the coating must be sufficiently different from that of the base material (more than four atomic numbers) for measurements to be performed with reasonable accuracy. This method is suitable for measuring, e.g. gold coatings thicker than two μm, where the X-ray fluorescence method has some limitations. The method is non-destructive.

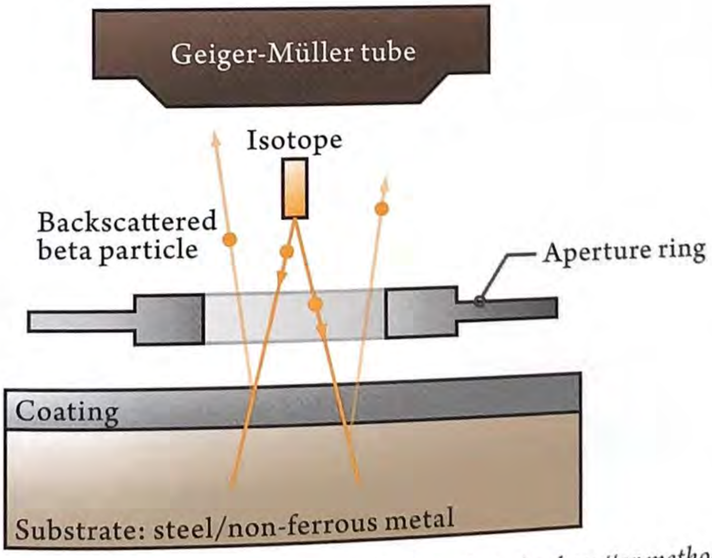

Figure 33.15: *Schematic illustration of the basic principle of the beta backscatter method (ASTM B567). The method can be used to measure the thickness of, e.g. paint, oil, lubricating films, plastic, enamel, ceramic and phosphate coatings on metals and some non-metals, as well as gold coatings thicker than two μm.*

33.2.11 ULTRASONIC MEASUREMENT

Reflection of sound pulses can be linked to coating thickness

If the coating and the substrate exhibit different acoustic properties, pulses of ultrasonic sound waves will be reflected at interphases and, since the propagation speed is material-dependent, it is possible to quantify coating thickness based on the timedelay of reflected sound pulses. Thus, using pulses of sound waves and collecting the reflected or transmitted waves as a function of time, it is possible to quantify individual coating thicknesses.

The ultrasonic method is mostly used for organic coatings. The ability to detect a distinct interface between a coating and the underlying substrate may be impeded, if the coating and the substrate are too similar, or, if the coating is non-homogeneous. If the materials have different acoustic properties like organic coatings on concrete, wood or wallboard substrates, an accurate measure of the film thickness can be carried out.

Multilayered coatings characterized by many interfaces are extremely difficult to characterize and most often only the interface, separating the two most acoustically different materials, will be identified. However, some of the more advanced instruments are able to detect and measure individual layer thicknesses in a multi-layer system.

The effective measuring range for the ultrasonic method is limited by the sensor design. Typically, thicknesses ranging from eight µm to 15 mm have been demonstrated according to the standard ASTM D6132 - 08.

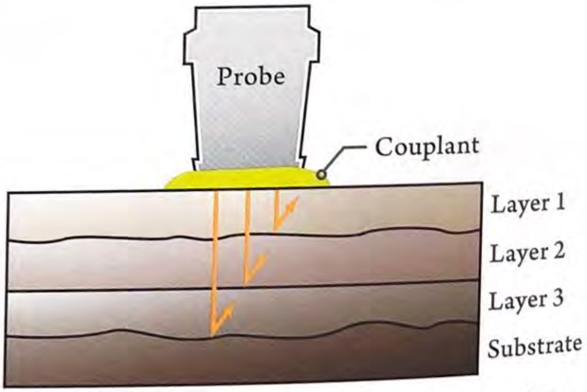

Figure 33.16: *Schematic illustration of the reflection of an ultrasound wave at different interfaces. An ultrasonic gauge can measure the thickness of coatings on non-metallic substrates. The probe contains an ultrasonic transducer that sends a pulse through the coating. The pulse reflects back from the interface, back into the transducer, where the sound wave is analyzed to determine the coating thickness. In some circumstances, individual layers in a multi-layer system can be measured. The typical tolerance of this device is ±3%. Standard methods for the application and performance of this test are available in ASTM D6132.*

ELLIPSOMETRY

33.2.12

Ellipsometry is a very versatile technique that can be used not only to determine the thickness of a coating, but also its optical properties, i.e. $n = n_1 + in_2$ corresponding to the real and imaginary part of the refractive index. Ellipsometry was also treated in Section 36.14.4.

Change in polarization upon reflection can be used to calculate the thickness and refractive index

In summary, ellipsometry is dealing with the interaction of a known polarization being reflected or transmitted from a surface and how the in-plane (s-component) and the out-of-plane (p-component) of an incoming light is changed upon interaction with a coated surface. Typically, the incident light is linear with both p and s components. The reflected light has normally undergone amplitude and phase changes for both p and s-polarized light. Ellipsometry measures this change and uses the information to calculate the coating thickness and the refractive characteristics of the involved films. The method can be used with one wavelength when all the optical properties are known, otherwise, several wavelengths have to be used to fit the multiparameter variable space.

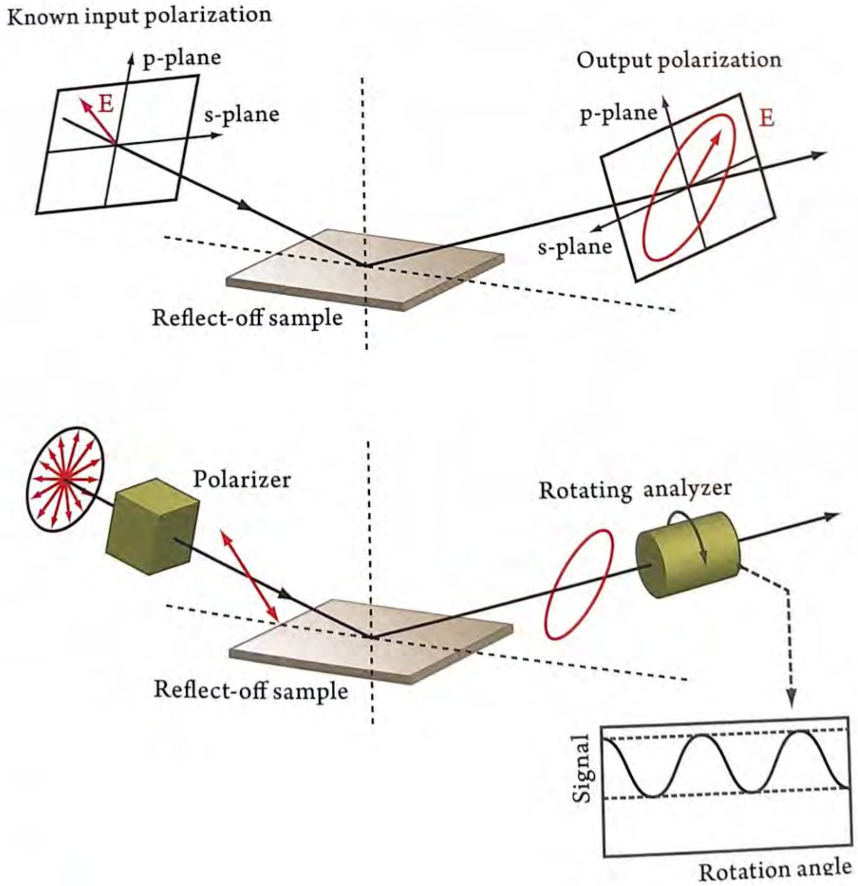

Figure 33.17:

Schematic illustration of the setup and measurement principle of an ellipsometer.

33.2.13 MEASUREMENT BEFORE CURING

This method is used for paint, varnish and lacquer before drying

Wet-film thickness (WFT) gauges can be used to determine how much material that has to be applied wet to achieve a specified dry-film thickness provided that the percent of solid per volume is known. The method can be used to measure all types of wet organic coatings, such as paint, varnish, and lacquer on flat or curved surfaces.

Measuring wet-film thickness during application will allow one to immediately correct and adjust the added paint thickness on site. A subsequent and later correction of the film thickness, after it has dried or chemically cured, requires costly extra labor time. Furthermore, it may lead to contamination of the film, and may even introduce problems of adhesion and integrity of the coating system.

The equations for determining the correct wet-film thickness (WFT), both with and without thinner can be done according to:

Without thinner:

$$\text{WFT} = \frac{\text{desired dry film thickness}}{\text{\% of solid per volume}} \tag{33.2}$$

With thinner:

$$\text{WFT} = \frac{\text{desired dry film thickness / \% of solid per volume}}{\text{100\% + \% of thinner added}}$$

Measuring the thickness of the wet paint

Wet-film is most often measured with a wet-film comb or wheel. The wet-film comb is a flat aluminum, plastic, or stainless steel plate with calibrated notches on the edge of each face. The gage is placed on the surface to be measured immediately after coating application and then removed. The wet-film thickness lies between the highest coated notch and the next uncoated notch. Notched gage measurements are neither accurate nor sensitive, but they are useful in determining approximate wet-film thickness of coatings on surfaces where size and shape prohibit the use of more precise methods. The gage should only be used on smooth surfaces, free from irregularities and should be used along the length of curved surfaces. Using a wet-film gage on quick-drying coatings will yield inaccurate measurements. A wet film wheel (eccentric roller) uses three disks. The gage is rolled in the wet film until the center disk touches the wet film. The point where it makes contact provides the wet-film thickness (see ASTM D1212 and D4414 for further detail).

Powder coatings can be measured prior to curing with a simple hand-held comb or, e.g. an ultrasonic gage. The uncured powder film comb works similar to the wet-film comb discussed above. The comb is dragged through the powder film and the thickness lies between the highest numbered tooth, which made a mark and has powder clinging to it, and the next highest tooth, which left no mark and has no powder clinging to it. These gages are relatively inexpensive with accuracy of ±5 μm. They are only suitable as guidelines since the cured film may be different after flow. Marks left by the gage may affect the characteristics of the cured film.

An ultrasonic device can be used non-destructively on uncured powder coatings on smooth metallic surfaces to predict the thickness of the cured film. Measurement uncertainty is of the order of ±5 μm.

33.2.13.1

Standards are available from, e.g. NIST

CALIBRATION THICKNESS STANDARDS

Coating thickness gages are calibrated to known thickness standards. There are many sources of thickness standards, but it is best to ensure they are traceable to a national measurement institute such as, e.g. National Institute of Standards & Technology (NIST). It is also important to verify that the standards are at least four times as accurate as the gage they will be used to calibrate. A regular check against these standards verifies the gage is operating properly. When readings do not meet the accuracy specification of the gage, the gage must be adjusted or repaired and then recalibrated again.

33.3 QUANTIFYING COATING ADHESION

It is recommended to consult standards

There are many different test methods for quantifying adhesion between a coating and the underlying substrate. Each coating laboratory has often developed 'inhouse' application-oriented standards, inspired by official ASTM or ISO standards, modified to their relevant production lines and products. Nevertheless, it is recommended to consult relevant standards covering the individual test methods. It is important to always apply a test method, which simulates the real application for a given coating and use the given application to guide the choice of best suitable laboratory test method. All test methods will have limitations linked, not only to the physical properties of the underlying substrate, but also to the properties of the added coating. It is therefore important to consider the limitation of the underlying substrates, when choosing a suitable test method. The most important properties of the coating are:

- Thickness
- Wear resistance
- Elasticity
- Hardness
- Ductility.

The different test methods are summarized in Table 33.2 below, with indication of where the different test methods can be applied.