

# **METAL PLATING PROCESSES AND METHODS OF MEASURING SURFACE HARDNESS AND THICKNESS OF COATINGS**

Technical Report  
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## 1.0 OVERVIEW

Plating is the application of a surface finish to another material; it provides the surfaces of manufactured parts with a number of desirable physical, chemical, and appearance qualities. Nearly all manufactured or fabricated products made of metal or having metal components have some type of surface finishing. Plating increases sturdiness, prevents corrosion, provides hardness, or gives an attractive finish to an object.

## 2.0 SUMMARY

Electroplating is inexpensive, reliable, efficient, and applicable to a wide variety of shapes and sizes. However, traditional electroplating technologies often result in uneven surface finishes, and have inherent pollution problems.

Electroless plating has increased in popularity due to improvements in solution stability, pretreatment cycles, reducing agents, and equipment. In addition, electroless plating provides uniform thickness and deposits, especially in deep recesses, bores, and blind holes.

Alternative plating methods such as PVD, CVD, and thermal spraying reduce the amount of contaminated wastewater produced by plating, but have high unit-plating costs.

## 3.0 COATING TYPES

There are 46 different processes regulated under metal finishing standards featuring different technologies, operational steps, inputs, and outputs. Some of the more common plating technologies are the following:

### 3.1 Electrodeposited / electroplating

Electroplating is used to change the surface properties of a metal part by adding a metal coating by the action of electric current; it is also called "electrodeposition,". The object to be coated receives a negative charge and is immersed into a solution that contains a salt of the metal to be deposited. The metallic ions of the salt carry a positive charge and are attracted to the object.

Surface pretreatment by chemical or mechanical means is important for electroplating, as the successful adhesion of the surface coating depends on removing contaminants and films from the substrate.

In addition, the geometric shape and contour of the object affects the thickness of the deposited layer. Objects with sharp corners and features will have thicker deposits on the outside corners and thinner ones in the recessed areas, because the current flows more densely to prominent points than to less accessible areas. This characteristic of electroplating limits applications with uneven surfaces or that have depressions or hidden holes.

Some metals used in electroplating are aluminum, brass, bronze, cadmium, copper, chromium, iron, lead, nickel, tin, and zinc, as well as precious metals such as gold, platinum, and silver. Different types

of coatings can be achieved through control of parameters such as voltage, amperage, temperature, residence times, and purity of the bath solutions.

Applications of electroplating are numerous because it is an inexpensive and simple method; it is used in all aspects of electronics, optics, and the automobile industry where, for example, chrome plating is used to enhance the corrosion resistance of metal parts

Below is a chart that shows the diversity of electroplating metals and the associated applications:

Chart #1– Electroplating Characteristics and Applications

<b>Metal Coating</b>	<b>Characteristics</b>	<b>Typical Applications</b>
Zinc	Excellent corrosion protection for steel; economic & safe	Connecting elements, auto and construction industry, plant engineering
Alloys – zinc, copper, and precious metal	Excellent corrosion protection, decorative finish, wear resistant	Extreme corrosion resistance combined with high thermal stress, e.g., car exhausts, engine elements, and electronics parts
Copper, Nickel, Chrome	Good corrosion protection, decorative finish	Motor vehicles, plumbing fittings, steel furniture, shop fittings
Hard Chrome	Extreme hardness, resistance to wear, corrosion protection, improved lubricating properties	Hydraulics, mold making, vehicles, shafts, and bearings
Silver, silver alloys	Good conductivity for heat and electricity, antibacterial, decorative finish with enhanced value.	Jewelry, electrical and electronic parts, household goods
Gold	Flexible, non- reactive with other elements, good electrical conductivity, resistance to corrosion, enhanced value	Jewelry, electrical and electronic parts, household goods
Bronze	Resistance to wear and corrosion, flexible	Bearing shells
Tin	Softness, flexible, solderable	Household applicanes, electronics, printed circuits, food industry

## Electroless Nickel

Electroless nickel plating operates without electricity; the process action is purely chemical. Coating is achieved through metal ion exchange using chemical reduction in a hot aqueous solution

An electroless nickel coating is uniform; it will not build up on corners or projections. The deposited metal layer has an even thickness over all surfaces of the component regardless of its shape; this cannot be achieved with electrodeposited coatings.

Typical thickness of electroless plating can be as thin as .0005" and up to .010". Electroless coatings, used on a broad range of base materials, provide exceptional hardness and resistance to wear and corrosion.

Parts to be plated have to be pre-treated, and each type of material requires a specific pre treatment. The deposit properties of an electroless nickel layer and the performance of the plated component depend upon the phosphorous content, the purity, the substrate, the pre treatment, and the thickness of the coating.

Electroless nickel solutions operate in a pH 4 - 9 medium at a temperature of between 77 to 198°F (25 - 92°C.) The plating speed of the solution is between 2 to 25 micrometers per hour (um/hr) and the deposit thickness generated on the parts depends upon the operating conditions and time.

Electroless nickel deposits may be heat treated to improve abrasion resistance, increase hardness, and enhance adhesion. The heat treatment temperature must be above 464°F (240°C), and to obtain the maximum hardness, it is necessary to treat parts at a temperature of 752° F (400°C) for 1 hour. Heat treatment achieves hardness of about 1000 V P N (Vickers) or HK 0.05/HV 0.1 (Knoop.)

Coating thicknesses for engineering uses vary from 0.0005 inch to 0.0015 inch (0,012 mm to 0,038 mm) with hardness from approximately 48 to 52 Rockwell C. When the plating is heat treated approximately 750°F (400°C) for one hour, the obtainable hardness varies from 58 to 64 Rockwell C.

The microelectronics industry uses electroless nickel plating to create a barrier between base metal and the final overplate. Industries such as mining, petroleum, aerospace, molds and dies, medical, dental, and pharmaceutical, use electroless nickel plating over steel for its excellent wear and corrosion resistance and for its ability to provide a coatings with a uniform deposit. Electroless nickel layers of about 50 um on steel have very good resistance to marine environments. In many cases, it is possible to use electroless nickel plated steel as a substitute for stainless steel.

Electroless nickel coatings are resistant to a wide range of chemicals, which makes them suitable for many applications in the chemical industry, for example, stirrers, valves, reaction tanks, and covers.

Some benefits of electroless nickel plating are the following:

- Superior corrosion resistance.
- Natural flexibility, providing excellent mold release properties.
- Freedom from porosity..
- A solderable, hard coating for light metals.
- Overall uniformity of deposit with no edge buildup.

See Bal Seal report *TR-16 Rev.C, Electroless Nickel Plating*

## 2.3 Anodizing

The anodizing process creates a coating both on the surface and into the metal; it is usually performed on aluminum for protection and cosmetic purposes. Thin coatings, 2 to 25 µm (100 to 1000 µin) can be coated on most aluminum.

Anodizing refers to the electrochemical oxidation of a metal in a suitable electrolyte such as sulfuric acid in various concentrations. When the temperature and current density are controlled, the aluminum oxide develops both on the surface and into the aluminum. The coating penetrates into the base

material as much as it builds up on the outside. The term "thickness" includes both the buildup and penetration

**Soft** anodizing refers to coatings produced in electrolytes that operate at room temperature. Soft anodize coatings tend to be more porous and can be dyed a wide variety of colors and then sealed. Since the coating is an integral part of the base metal, anodic coatings are very durable and not subject to chipping under harsh service conditions.

In **hard** anodizing, the electrolytes operate at lower temperatures. The chemical action on the aluminum oxide is slower, but eventually produces a harder and thicker coating than soft anodizing.

See Bal Seal report *TR-17, Rev.C, Hard Anodizing of Aluminum Alloys and Its Effects on Bal Seal Performance*

## 3.4 Carburizing / carbonizing

Carburizing is the introduction of carbon into the surface layer of steel with low carbon content. The process includes heating the components in a liquid or gaseous carbon-containing medium. The time and temperature parameters of the treatment control the depth of penetration of carbon into the surface.

The most typical carburizing temperature is 1750°F, although lower temperatures may be used to reduce distortion or improve control of the case depth tolerance. Uniform temperatures must be present to produce uniform carburizing and hardening, and to control distortion. It is important to maintain the temperature of the steel, rather than just furnace temperature

After carburizing, the work is either slow-cooled for later quench hardening, or quenched directly into various liquids.

The combination of hard surface and soft interior withstands very high stress and fatigue, and offers low cost and superior flexibility in manufacturing. Gears, ball and roller bearings, and piston pins are often carburized.

## 3.5 PVD/CVD

The physical vapor deposition (PVD) method deposits the coating over the entire object simultaneously, rather than in localized areas.

PVD is a surface modification process that takes place in a vacuum chamber heated at approximately 750°F (400°C). The PVD process directs a concentrated, high-energy plasma onto a metal surface. The

thickness of the coating is approximately 2 to 4 microns (0.000079 to 0.00016 inch or 0.002 mm to 0.004 mm), resulting in improved corrosion resistance.

All PVD hard coating processes combine the following:

- A method for depositing the metal
- Combination with an active gas, such as nitrogen, oxygen, or methane
- Plasma bombardment of the substrate to ensure a dense, hard coating

PVD methods differ in the means for producing the metal vapor and the details of plasma creation. The primary PVD methods are ion plating, ion implantation, sputtering, and laser surface alloying.

Chemical vapor disposition (CVD) is a subset of PVD; the variations are in the way that the gases are converted. CVD occurs in a vacuum chamber filled with a reacting chemical vapor heated at a temperature of approximately 1650°F (900°C).

Companies use CVD to improve corrosion and wear resistance, however, the facilities required to create the extreme temperatures make CVD more expensive than other processes.

Specialty processes such as PVD/CVD and plasma spray coating are relatively more expensive plating methods.

See Bal Seal report *TR-24, Rev. B, ION implantation, PVD, and CVD, and their effects on Bal Seal Performance*.

### **3.6 Plasma spray coating**

Plasma spray is a high-speed flame spray process that produces a dense, high-quality, machinable coating. In plasma spray coating, powders are injected into the plasma stream to melt the material. The molten material is propelled and bonded onto the substrate.

The coatings thickness is dictated by the size of the feedstock for powders, the size of the droplets for arc spraying, and the size of the atomized droplets created by the liquid spray process. Coatings are typically applied to a thickness of 0.003 to 0.010 inches (0.076 to 0.25mm), but can be up to 0.020inches (0.51mm) without detrimental impact to the coating properties.

Plasma spray coating enhances, protects, and alters surface properties for many purposes, including dimensional restoration, thermal barriers, conductivity, wear resistance, and chemical resistance.

Plasma spray coating is used for fiber and textile-like applications where a long-lasting wear resistant coating is needed. Plasma spray processes are used to coat electronic components, automotive sub assemblies, medical instruments, jet engines, and other structural parts, as well as orthopedic implants, and consumer products.

See Bal Seal report *TR-3 Rev.D Plasma Spray Coated Shafts for Rotary and Reciprocating Service in Contact with Bal Seals*

### 3.7 Cost

Cost comparison for various plating methods depends on design, market, quantity, and other factors. Plating processes not only perform different functions, but also apply to different substrates. For example, anodizing applies to aluminum and not steel, whereas carburizing applies to steel and not aluminum.

In addition, the processes often go together. A part to be carburized, for example, a gear, may be selectively copper electroplated to prevent hardening in the bore area. Some gold finishes employ a PVD topcoat on top of electro-deposited plating.

Therefore, without considering such variables, the relative costs of plating methods are as follows:

Chart #2 Relative Cost of Plating Methods

Plating Method	Cost
Electro-deposited / electroplating	\$\$
Hard anodizing	\$\$
Carburizing / carbonizing	\$\$\$
Electroless	\$\$\$
Plasma spray coating	\$\$\$\$
PVD/CVD	\$\$\$\$

## 4.0 COATING MEASUREMENT

Hardness and thickness are important dimensions of plating materials because the effectiveness, longevity, and cost of the final product depend, not only on the appropriate selection of coating material, but also on the amount applied, and the final surface texture.

### 4.1 Hardness

Hardness is the property of a material that enables it to resist deformation, however, is not an intrinsic material property dictated by precise definitions in terms of fundamental units of mass, length, and time. A hardness property value is the result of a defined measurement procedure.

Hardness tests characterize the materials and determine if they are suitable for their intended use. All of the hardness tests described in this section involve the use of a specifically shaped indenter, significantly harder than the test sample. The indenter is pressed into the surface of the sample using a specific force. Either the depth or size of the indent is measured to determine a hardness value.

Establishing a correlation between the hardness result and the desired material property makes hardness tests useful in industrial and R&D applications.



Four major hardness scales described are the following:

- Rockwell
- Brinell
- Vickers
- Microhardness (Knoop)

Each of these scales uses a specifically shaped diamond indenter, made from carbide or hardened steel. The indenter is pressed into the material with a specific force using a defined test procedure. The hardness values are determined by measuring either the depth of indenter penetration or the size of the resultant indent. All of the scales are arranged so that the hardness values increase, as the material gets harder.

Current methods such as Rockwell or Brinell, use a minimum load of approximately 5 grams force (gf.) However, the resulting indentation may be too large for thin film coating, and the underlying layer interferes with accurate hardness measurement. Therefore, an ultra-low load hardness test, a microhardness test, was developed for thin coatings.

#### **4.1.1 Rockwell**

The Rockwell test method is the most commonly used hardness test method, since it is easier to perform and more accurate than other types. Rockwell is used on all metals except where the test metal structure or surface conditions introduce too much variation, where the indentations is too large for the application, or where the sample size or shape prohibits its use.

The Rockwell hardness test measures the depth of indentation produced by the preliminary and total test forces. First, a preliminary test force is applied; this is the zero or reference position. Next, an additional test force is applied to reach the total required test force. This additional force is held for a predetermined amount of time and released, but the preliminary test force still applied. The indenter reaches the final position at the preliminary force and the distance traveled from the major load position is measured and converted to a hardness number.

#### **4.1.2 Brinell**

The Brinell hardness test method consists of indenting the test material with a hardened steel or carbide ball. The diameter of the indentation left in the test material is measured with a low powered microscope. The Brinell harness number is calculated by dividing the load applied by the surface area of the indentation.

Compared to the other hardness test methods, the Brinell ball makes the deepest and widest indentation, so the test averages the hardness over a wider amount of material, which will more accurately account for multiple grain structures and any irregularities in the uniformity of the material.

The Brinell method is the best for achieving the bulk or macro-hardness of materials with heterogeneous structures.

Some applications where the Brinell hardness test is used are the following:

- Forgings/castings
- Heavy truck/bulldozer parts
- Engine blocks and heads
- Non-homogeneous materials
- Rear-end housings
- Springs
- Variety of large, coarse-surface parts

#### 4.1.3 Vickers

In the Vickers hardness test method, the indenter is a right pyramid with a square base and an angle of 136 degrees between opposite faces. The two diagonals of the indentation left in the surface of the material after removal of the load are measured using a microscope and their average calculated. The area of the sloping surface of the indentation is calculated. The Vickers hardness is the quotient obtained by dividing the kgf<sup>1</sup> load by the square millimeter area of indentation.

The Vickers test has two force ranges micro (10 to 1000gram) and macro (1 to 100kilogram.) The indenter is the same for both ranges, and the Vickers hardness values are continuous over the total range of hardness for metals (typically HV100 to HV1000).

The advantages of the Vickers hardness test are that extremely accurate readings can be taken, and only one type of indenter used for all types of metals and surface treatments. Because of the wide test force range, the Vickers test can be used on almost any metallic material. The part size is only limited by the testing instrument's capacity.

#### 4.1.4 Microhardness (Knoop)

The Microhardness test method, according to ASTM E-384, specifies a range of loads using a diamond indenter to make an indentation that is measured and converted to a hardness value.

There are two types of microhardness indenters, a square base pyramid shaped diamond used in a Vickers tester and a narrow rhombus shaped indenter for a Knoop tester. Typically, loads are very light, ranging from a few grams to one or several kilograms. The term microhardness refers to static indentations made with loads not exceeding 1 kgf.

The procedure for testing is very similar to that of the standard Vickers hardness test, except that it is done on a microscopic scale with higher precision instruments. Precision microscopes used to measure the indentations, have a magnification of approximately X500, and measure an accuracy of  $\pm 0.5$  micrometers.

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<sup>1</sup> kgf A force equal to a kilogram weight or a one-kilogram mass times the acceleration of gravity

Like the Vickers test, the Knoop microhardness test has a wide test force range, and it can be used on almost any metallic material. The primary application of the microhardness test is measuring the hardness of a thin film coating.

The following table shows hardness test methods by material application:

Chart #3      Material Application Table

Materials	Test Method			
	Rockwell	Vickers	Microhardness (Knoop)	Brinell
Copper and aluminum alloys	✓	✓	✓	✓
Steel and titanium	✓	✓	✓	✓
Magnesium alloys	✓	✓	✓	✓
Annealed copper alloys, thin soft sheet metals	✓	✓	✓	
Beryllium copper, zinc, lead	✓	✓	✓	✓
Tin	✓	✓	✓	
Aluminum	✓	✓	✓	✓
Zinc	✓	✓	✓	✓
Paint and organic coatings			✓	
Hard rubber	✓			
Plastics	✓	✓	✓	

#### 4.1.6 Conversion Charts

Many conversion charts are available for the different hardness tests. Some conversion charts cover specific materials and hardness scales. Conversion charts are covered under specification ASTM E-140-02, which advises that, due to the error involved, conversion charts between different hardness scales should only be used when it is impossible to test under the conditions specified.

#### 4.1.7 Cost

Hardness testers today are designed for portable operation in field and shop applications. They can operate on battery or AC power, and have direct digital hardness readout in Rockwell, Vickers or Brinell scales. Since most testers now can test using different hardness scales, one machine can supply a range of requirements, and the cost comparison would be the choice between a less expensive portable tester and a high end table or floor model.

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## 4.3 Thickness

Coatings applied to base materials provide properties not inherent in the base, including corrosion and wear resistance, conductivity, color, and solderability. The amount of coating applied to a material, that is, the coating thickness, is crucial to the product's final use and cost.

This section describes four non-destructive<sup>2</sup> methods of measuring coating thickness. Each method was devised to achieve cost-effective, accurate, and repeatable results, and is particularly suited to a specific coating(s)/substrate combination.

The four methods described are:

- X-Ray fluorescence
- Eddy-current
- Magnetic induction
- Beta backscatter

### 4.3.1 X-ray Fluorescence

When a material is subjected to x-ray bombardment, some of its electrons will gain energy and leave the atom, releasing a photon of x-ray energy known as x-ray fluorescence. The energy level or wavelength of fluorescent x-rays is proportional to the atomic number of the atom and is characteristic for a particular material. The quantity of energy released will be dependent upon the thickness of the material being measured.

The X-ray fluorescence unit consists of an x-ray tube and a proportional counter. Emitted photons ionize the gas in the counter tube proportional to their energy, permitting spectrum analysis for determination of the material and thickness.

X-ray fluorescence is the most precise measurement method; it is used to measure the thickness of small-diameter parts, or dual coatings such as gold and nickel over copper.

### 4.3.2 Eddy Current

The Eddy current technique is used to nondestructively measure the thickness of nonconductive coatings on nonferrous metal substrates, for example, paint on aluminum and acrylic on copper.

Eddy current inspection is based on the principles of electromagnetic induction and therefore has many similarities to the electromagnetic induction test method. Magnetically induced eddy currents generate an opposing magnetic field, which alters the circuit reactance and the output voltage. The change in output voltage is used to calculate the coating thickness.

Eddy current gauges use a constant pressure probe and display results on an LCD; they can store measurement results or perform instant analysis of readings, and output the data to a printer or computer for further examination. The typical tolerance is  $\pm 1\%$ . Testing is sensitive to surface roughness, curvature, substrate thickness, type of metal substrate, and distance from an edge.

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<sup>2</sup> Nondestructive testing (NDT) is test methods used to examine an object, material, or system without impairing its future usefulness.

Some applications of the Eddy current method are the following:

- Crack detection
- Material thickness measurements
- Coating thickness measurements
- Conductivity measurements

#### 4.3.3 Magnetic Induction

Magnetic induction is used to measure the thickness of a non-magnetic coating (zinc, cadmium, paint, powder coating, etc.) over a steel substrate. Most coatings on steel and iron are measured this way.

A probe system is part of a transformer circuit that reacts to the presence of a magnetic material. The circuit efficiency and output voltage increase when the probe is brought near a magnetic surface, providing parameters, which may be used to measure the distance (coating thickness) from the magnetic surface.

#### 4.3.4 Beta Backscatter

The Beta backscatter method is similar to the X-ray fluorescent in that the area tested is the target of radiation, and the energy emitted from the surface is measured. Beta rays are electrons emitted from unstable radioisotopes. The electrons penetrate the plating material and reflect back (back scatter) toward the source. They are collected and counted with a Geiger-Mueller tube for conversion to coating thickness.

Beta-backscatter measuring technique, in compliance with ASTM B567 specification, measures many typical thickness applications, including gold on nickel, copper on epoxy, silver on copper, titanium nitride on steel, and tin-lead alloys. Probe systems are designed for precise measurement on a variety of surfaces, from small parts (connectors and pins) to large parts (circuit boards).

The following table indicates the measurement method best suited to a particular coating:

Chart #4

Coating	Thickness Testing Method			
	X-ray Fluorescence	Eddy Current	Beta Backscatter	Magnetic Induction
Gold	✓		✓	
Electroplated nickel	✓	✓		✓
Copper	✓	✓	✓	✓
Powder coatings		✓		✓
Electroless nickel	✓			✓
Chrome	✓	✓	✓	✓
Anodize		✓	✓	

## 4.4 Cost

X-ray fluorescence is the most versatile system for measuring coating thickness, and it is quickly becoming more affordable. However, there are applications where the other methods are more cost-effective, for example, if the parts to be measured are too big to fit into an X-ray chamber, or if the plated surface may be inaccessible to an X-ray beam.

Chart #5

Thickness Measurement Method	Cost
Magnetic Induction	\$\$
Eddy Current	\$\$
Beta Backscatter	\$\$\$
X-ray Fluorescence	\$\$\$\$

The chosen method is determined by the coating material, the substrate type, the coating thickness, and the part size and shape.

All the above non-destructive measurement methods are used in commercial applications; destructive thickness measurement methods are rarely used outside the laboratory.

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## 5.0 References

- 5.1 ASM International Handbook Vol. 5 Surface Engineering
- 5.2 ASTM-E 140-02 Standard Hardness Conversion Tables for Metals. Relationship Among Brinell Hardness, Vickers Hardness, Rockwell Hardness, Superficial Hardness, Knoop Hardness, and Scleroscope Hardness
- 5.3 ASTM E-384 Standard Test Method For Microindentation Hardness of Materials
- 5.4 TR-1, Rev.B Treatment of Metal Surfaces to improve Bal Seal Performance in Dynamic Applications
- 5.5 TR-3 Rev.D Plasma Spray Coated Shafts for Rotary and Reciprocating Service in Contact with Bal Seals
- 5.6 TR-16 Rev.C, Electroless Nickel Plating
- 5.7 TR-17, Rev.C, Hard Anodizing of Aluminum Alloys and Its Effects on Bal Seal Performance
- 5.8 TR-24, Rev.B, ION Implantation, PVD, and CVD, and Their Effects on Bal Seal Performance.
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