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# Guidelines for Chromium(III) Coating Test Parameters

Metropolia University of Applied Sciences

Bachelor of Engineering

Biotechnology and Chemical Engineering

Bachelor's Thesis

19.4.2024

# Tiivistelmä

Tekijä: Juho Vuorela  
Otsikko: Ohjeistus kromi(III)pinnoitteen testiparametreille  
Sivumäärä: 29 sivua + 6 liitettä  
Aika: 19.4.2024

Tutkinto: Insinööri (AMK)  
Tutkinto-ohjelma: Bio- ja kemiantekniikka  
Ammatillinen pääaine: Materiaali- ja pinnoitetekniikka  
Ohjaajat: Lehtori Arto Yli-Pentti  
Teknologiajohtaja Jussi Räisä

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Tämän opinnäytetyön tarkoitus oli laatia ohjeistus kolmenarvoisen kromipinnoitteen testausta varten. Opinnäytetyö tehtiin Savroc Oy:lle.

Työn teoriaosuus käsittelee kromipinnoitteita sekä mahdollisia korvaavia pinnoitteita, kuten suurnopeusliekkiruiskutettuja pinnoitteita, kemiallista nikkelöintiä ja kaasufaasipinnoitusta.

Työn kokeellisessa osiossa testattiin kromipinnoitteen pinnankarheutta, paksuutta, kovuutta, adheesiota, huokoisuutta ja korroosionkestoa. Tarkoituksena oli perehtyä kromipinnoitteen testausmenetelmiin ja -parametreihin. Kokeellinen osuus tehtiin Metropolian pintalaboratoriossa.

Tehtyjen testien ja niiden tulosten perusteella kirjoitettiin ohjeistukset kromipinnoitteen pinnankarheuden mittaukseen pinnankarheusmittarilla, pinnoitepaksuuden mittaukseen kalvonpaksuusmittarilla sekä mikroskooppisella menetelmällä, kovuuden mittaukseen Vickers-mikrokovuusmittarilla, adheesioon mittaukseen naarmutustestillä, huokoisuuden mittaukseen ferroksyyli-testillä ja korroosionkeston testaukseen suolavesikokeella.

Avainsanat: kromaus, kolmenarvoinen kromi

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Tämän opinnäytetyön alkuperä on tarkastettu Turnitin Originality Check -ohjelmalla.

## Abstract

Author: Juho Vuorela  
Title: Guidelines for Chromium(III) Coating Test Parameters  
Number of Pages: 29 pages + 6 appendices  
Date: 19 April 2024

Degree: Bachelor of Engineering  
Degree Programme: Biotechnology and Chemical Engineering  
Professional Major: Materials and Surface Engineering  
Supervisors: Arto Yli-Pentti, Senior Lecturer  
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The purpose of this thesis was to develop guidelines for testing trivalent chrome plating. The thesis was conducted for Savroc Ltd.

The theoretical part of the thesis discusses chrome coatings and possible alternatives for chrome coatings such as high velocity oxygen fuel coatings, electroless nickel plating and vapor deposition methods.

In the experimental part of the thesis the surface roughness, thickness, hardness, adhesion, porosity and corrosion resistance of chrome coatings were tested. The goal was to become familiar with the testing methods and parameters for chromium coatings. The experimental part was conducted in the surface treatment laboratory of Metropolia.

Guidelines for measuring hard chrome plating's surface roughness with a surface profilometer, coating thickness with a coating thickness gauge and microscopic method, hardness using a Vickers microhardness tester, adhesion using a scratch test, porosity using a ferroxyl test and corrosion resistance using a salt spray test were written on the basis of conducted tests and their results.

Keywords: chrome plating, trivalent chromium

The originality of this thesis has been checked using the Turnitin Originality Check software

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## **List of Abbreviations**

REACH: European Union regulation regarding Registration, Evaluation, Authorisation and Restriction of Chemicals.

ECHA: European Chemicals Agency.

HVOF: High Velocity Oxygen Fuel.

## 1 Introduction

This thesis was conducted for Savroc Ltd. Savroc is the developer of TripleHard trivalent hard chrome coating. The company was founded in 2012 and is located in Kuopio. Figure 1 presents different types of TripleHard coatings [1].



Figure 1. TripleHard coatings.

The goal of this thesis was to create guidelines for the testing of trivalent hard chrome coatings, and the thesis consists of three parts: theory section, testing part and testing guidelines. The theory section addresses chrome plating and methods that can be used to test the properties of chrome coatings. It also briefly studies possible alternatives for hard chrome plating. Testing part covers the testing of chrome coating samples and its purpose was to get familiar with testing parameters.

## 2 Chrome plating

Chrome plating is a surface treatment process which involves the application of a thin layer of chromium onto a substrate. It is used to improve the plated object's surface hardness, corrosion resistance and appearance. The concept of

chrome plating goes as far back as 1854 when a German chemist Bunsen published a scientific report about it. [2, p. 58.]

Chrome plating can be roughly separated into decorative chrome plating and hard chrome plating. Decorative chrome plating is typically 0.2 – 0.5  $\mu\text{m}$  thick. It is mainly used for aesthetic purposes and it provides a shiny and reflective finish to the coated object. It can also help to protect the object against scratching, tarnishing and corrosion. Decorative chrome plating is typically used in items such as wheels, motorcycle parts and household fixtures. [3, p. 192.]

Hard chrome plating refers to chrome plating that has a thickness of over 10  $\mu\text{m}$ . Hard chrome plating provides the plated object with increased surface hardness, corrosion resistance and wear resistance. It also decreases friction on slip surfaces which makes it a good surface treatment option for rotating or sliding parts. Due to the benefits hard chrome plating offers, it is often used in products like hydraulic rods, piston rings and aircraft landing gears. In some applications like piston rings chrome plating can increase service life the coated product to roughly five times compared to an uncoated piston ring that is made of the same material. [3, p. 177.]

Hard chrome plating can also be used to restore and salvage worn or mismachined parts and tools to their original dimensions and is especially useful in restoring parts which require good wear resistance and low friction coefficient. Some factors like chrome deposits potentially reducing the fatigue strength of a salvaged part can have a restriction on which applications the use of chrome plating is viable. [3, p. 178.]

## 2.1 Hexavalent chromium

The most common source of chromium in chrome plating is chromium trioxide  $\text{CrO}_3$  which is a chemical compound where chromium ions have +6 oxidation state. The use and production of Chromium trioxide in European Union is restricted by REACH regulation and it is categorized as a substance of very high



concern by European Chemical Agency. The toxicity of hexavalent chromium is based on its high oxidization potential and it can enter into cells through sulfate channels due to its structural similarity with sulfates. Figure 2 shows the molecular structure of chromium trioxide [4.]

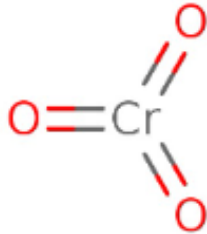


Figure 2. Molecular structure of chromium trioxide [4.]

Hexavalent chromium is also carcinogenic and workers that are exposed to it are at increased risk of developing lung cancer [4.]

## 2.2 Trivalent chromium

Chromium plating using trivalent chromium has been a studied topic for many years due its advantages compared to hexavalent chromium when it comes to environmental effects and safety. Some estimates point to trivalent chromium ions being hundred times less toxic than hexavalent chromium ions. These advantages and various restrictions placed hexavalent chromium have incentivized the development of plating processes which use trivalent chromium. First commercially viable decorative chromium plating processes using trivalent chromium began in the United Kingdom in 1975 and the United States in 1976. [3, p. 198.]

In addition to environmental and safety advantages, processes using trivalent chromium can also have some productivity advantages. Processes using trivalent chromium produce lower amounts of toxic chromic acid mist and have lower waste treatment costs. Chromium in the rinse water is in trivalent state,

thus, it does not require reduction step which hexavalent chrome ions require. Some processes using trivalent chromium ions are also faster than processes using hexavalent chromium ions and have higher tolerance to current interruptions which increases productivity and decreases the amount of rejected parts. [3, p. 198.]

### **3 Possible alternatives for hexavalent chrome**

#### **3.1 High velocity oxygen fuel spraying (HVOF)**

HVOF is a thermal spraying coating process which uses a mix of oxygen and fuel to create a high pressure and temperature gas which is used to deposit a melted or partially melted coating material onto a substrate with high velocity. Common coating materials used in HVOF include chromium carbide, tungsten carbide and tungsten carbide cobalt powders. [5, p. 23.]

The high speed with which the coating material hits the substrate results in a dense and cohesive coating. Coatings made with HVOF can have superior wear resistance and corrosion protection when compared to chrome plating achieved with hexavalent chromium. Hardness of the coatings are typically 1100 – 1400 HV so their hardness is within the same range as hardness of typical hard chrome plating or somewhat higher. Some of the coating materials are more hazardous to health than others, but generally they are less hazardous than hexavalent chromium. [5, p. 23.]

HVOF works best for visible and easily accessible surfaces but using it for hard-to-reach surfaces can be difficult or impossible. Thermal spraying can also cause residual stresses when the coating contracts as it cools down. These residual stresses can cause the substrate to distort if the substrate is too thin. The materials that can be coated using HVOF are also limited to materials that can tolerate the temperatures of the process. [5, p. 24.]

### 3.2 Electroless plating

Electroless nickel plating is a process in which nickel is deposited on the surface of a substrate with the use of autocatalytic chemical reduction by aminoborane, borohydride or hypophosphite instead of the use of electric current. Electroless nickel plating offers excellent wear resistance and corrosion resistance and results in uniform thickness on all areas where the object to be coated is in contact with electroless nickel solution. Due to these properties, electroless nickel plating is used in applications such as turbine blades, hydraulic components and piston rods. [3, p. 290.]

Electroless nickel plating can be used in some of the same applications as hard chrome plating and one of its main advantages is the uniformity of the resulting coating. Electroplated coatings can have large variation in the coating thickness depending on the shape of the coated part which could require additional finishing which increases labor costs. On the other hand, higher chemical cost for electroless nickel plating baths and slower plating rate can make it less productive and less economically viable than hard chrome plating in cases where thicker coating is required. [3, p. 294.]

Electroless nickel coatings can reach similar hardness as hard chrome plating through heat treatment but the treatment can have negative effects on the coating's corrosion resistance as changes in the microstructure of the coating can cause cracking. It also can also limit the available base materials the coating can be used on. Additionally, like hexavalent chromium, nickel is also a toxic substance and changing from the use of one toxic substance to another might be pointless in some cases. [6, p. 56.]

### 3.3 Vapor deposition methods

#### 3.3.1 Physical vapor deposition

Physical vapor deposition is a process where solid state coating material is evaporated and deposited on the surface of a substrate where it forms a thin layer. The process is done in a vacuum or low-pressure plasma system. [6, p. 109.]

Properties of the coating depend on the used material but generally physical vapor deposition coatings are very hard, dense and have excellent wear resistance. Possible coating materials that can be applied with physical vapor deposition include chromium nitride, titanium nitride, tungsten carbide and diamond-like carbon. [6, p. 110.]

The main property limiting the use of physical vapor deposition coatings as replacement hard chrome plating is coating thickness. Maximum coating thickness of most of the coatings is around 5  $\mu\text{m}$ . Thicker coatings can have high internal stress and a negative effect on fatigue strength. Due to these limitations, physical vapor deposition cannot be used in applications where a thicker coating is required. Even though the coatings generally have excellent wear resistance, the lifespan of a thin coating can be low if it used in high wear environment. Additionally, the method cannot be used in restoring damaged and worn-out components and tools in the same way as chrome plating can. While some of the physical vapor deposition coatings are reported to be inert, their thickness also limits their corrosion protection properties. In cases where the coating gets damaged, their corrosion protection can weaken significantly more than that of similarly damaged chrome plating. [6, p. 111.]

The process itself also has some limitations when compared to hard chrome plating as the vacuum chamber limits what size and what type of components can be coated. Most physical vapor deposition methods are line of sight processes which also limits their applications. The process temperatures can also have an effect on what type of substrates can be coated. [5, p. 26.]

### 3.3.2 Chemical vapor deposition

Chemical vapor deposition is a process where gaseous or evaporated compounds containing the coating material are brought to a reaction chamber and passed over a substrate. The compounds then react with the substrate or disintegrate on its surface forming the coating. The process can be used to deposit layers of carbon, silicon, nitrides, oxides and almost any metal. Metals typically used in the process include tungsten, nickel and molybdenum. [3, p. 510 – 513.]

Coatings produced with chemical vapor deposition processes generally have similar properties as coatings produced with physical vapor deposition processes, but the method can also be used to produce thicker coatings that are typically around 50  $\mu\text{m}$  thick. These thicker coatings can be used in similar applications as chrome coatings. [6, p. 81.]

Chemical vapor deposition processes also have similar restrictions as physical vapor deposition methods as the process is done in a vacuum chamber and the process temperatures can reach as high as 1000 °C which limits the usable substrates. On the other hand, the processes are not limited to line of sight can be used to coat inner surfaces. [5, p. 26.] [6, p. 82.]

## 4 Testing methods

Testing part of thesis was executed in Metropolia's surface treatment laboratory. The tests were conducted on test pieces prepared from objects that were coated with Cr(III) coating and Cr(VI) coating or on the coated objects themselves. Figure 3 shows the coated objects. The two larger objects were coated with Cr(III) coating and the smaller object on the right was coated with Cr(VI) coating.



Figure 3. Chrome plated objects

Sample preparation that involved cutting was performed with the use of Struers Discotom-50 automatic tabletop cut-off machine which is shown in Figure 4. Cut-off wheels used in sample preparation were suitable for hard ferrous metals and rotational speed was set to 1750 rpm.



Figure 4. Struers Discotom-50 automatic tabletop cut-off machine

The main goal of the testing part was to become familiar with testing parameters and creating guidelines for testing the properties of chrome plating based on those parameters.

#### 4.1 Surface roughness

Surface roughness of a coating can be measured using a profilometer. Contact profilometers use a stylus that traces the profile of the surface as it is dragged across it. Vertical movements of the stylus are recorded and used to calculate desired roughness parameters.

The most common parameter that is used to measure surface roughness is arithmetic average roughness  $R_a$  which measures the surface profile's average deviation on a specified sampling length. Another useful parameter the surface profile's maximum height  $R_z$  which measures the deviation between lowest valley and the highest peak on a specified sampling length. [7, p. 3.]

Parameters for surface roughness measurements for different metallic products are presented in the standard SFS-EN 10049. Cut-off  $\lambda_c$  is an especially important parameter because if it is set too low, the measured surface roughness values could be lower than the actual surface roughness. If it is set too high, the waviness of the surface could be included in calculation of the surface roughness which means the calculated surface roughness values could be higher than the actual values. Sample length is also the same as  $\lambda_c$ . [7, p. 2.] [8, p. 11.]

Measuring surface roughness of a coating is important because surface roughness has an effect on the coating's friction coefficient and wear resistance. Knowing the surface roughness of a coating is also important when testing its other properties such as hardness and adhesion.

Test pieces for surface roughness testing should be flat. Before measuring they should be degreased and cleaned from any contaminants that could have an effect on the test results. [8, p. 10.]

## Testing

Surface roughness of the coatings were measured with Mitutoyo SurfTest SJ-210 surface profilometer with cut-off  $\lambda_c$  set to 0,8 mm. The surface profilometer is shown in Figure 5.





Figure 5. Mitutoyo surface profilometer

The test results are presented in Table 1.

Table 1. Surface roughness measurements.

Measurement	Cr(III) Ra ( $\mu\text{m}$ )	Cr(III) Rz ( $\mu\text{m}$ )	Cr(VI) Ra ( $\mu\text{m}$ )	Cr(VI) Rz ( $\mu\text{m}$ )
1	0.796	4.250	0.041	0.416
2	0.652	4.228	0.040	0.323
3	0.517	3.610	0.041	0.392
4	0.591	4.087	0.034	0.294
5	0.581	4.030	0.038	0.300
6	0.593	3.917	0.067	0.480
Mean	0.622	4.020	0.0435	0.368

Measured surface roughness of Cr(VI) was lower than the measured surface roughness of Cr(III) coating due to surface of the Cr(VI) coating being polished. Surface roughness of Cr(III) is higher which could have an effect on the results of microhardness tests.

## 4.2 Thickness

Thickness of a coating can be determined by examining cross-section of the coating using a microscope. This method can be used as a reference method to which results from other tests can be compared to.

Cross-section for microscopic examination is prepared by cutting a small piece off the coated object. This piece is then mounted so that the cross-section is perpendicular to the coating. Mounted piece is then ground and polished using abrasive paper so that the surface is flat and all deformed material is removed. The cross-section can then be etched so that its layers become more clearly defined. [9, p. 8.]

There are also non-destructive testing methods to measure coating thickness. Figure 6 shows coating thickness gauge that uses a magnetic method to measure the thickness of a coating.



Figure 6. Elcometer coating thickness gauge

Magnetic methods can be used when the substrate is magnetic and the coating material is non-magnetic, which makes it suitable for determining the thickness of chrome plating on a steel substrate.

### Testing

Coating thickness of the samples were tested with Elcometer coating thickness gauge and microscopical method. The measurements taken with Elcometer are presented in Table 2.

Table 2. Coating thickness measurements taken with Elcometer coating thickness gauge.

Measurement	Cr(III) coating thickness ( $\mu\text{m}$ )	Cr(VI) coating thickness ( $\mu\text{m}$ )
1	45.6	27.0
2	37.1	28.9
3	42.2	26.4

4	42.3	30.3
5	39.5	28.3
6	37.6	27.5
Mean	40.7	28.1

Samples for microscopical method were prepared by cutting small pieces from the coated objects. The pieces were mounted in resin using Struers Pronto-Press-20. Mounted pieces were then ground and polished following the instructions of the standard SFS-EN ISO 1463:2021 by first using 180 grit grinding paper. Then 240, 400, 800 and 1200 grit grinding papers were used for 40 seconds each while changing the angle of scratches by 90° every time the paper was changed. Samples were then polished with the use of polishing cloth and diamond suspension for 3 minutes.

Additional samples were prepared with the help of a hard coating sample preparation method made for Struers Tegramin automatic polishing system. This preparation method used grinding and polishing discs made by Struers. The measurements taken from specimens prepared in these two different ways were similar to each other. When making of guidelines is taken into account, sample preparation method provided by the standard SFS-EN ISO 1463:2021 is probably more useful since it does not rely on equipment or accessories provided by a specific manufacturer.

Samples were then etched for 30 seconds with 5 % nital solution to make the visual difference between the coating and the base material more clearly defined. Coating thicknesses were then measured using Leica DM 2500M microscope and Leica LAS X software's measurement tool. Figure 7 shows cross-section of Cr(III) coating.

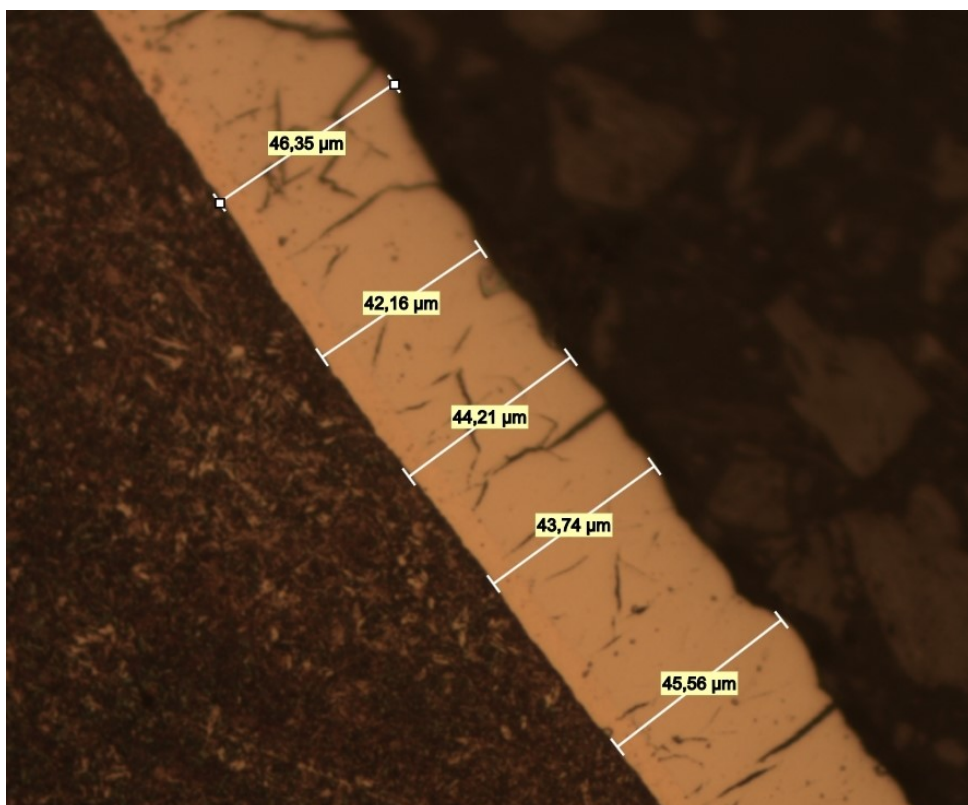


Figure 7. Cross-section of Cr(III) coating.

Measurements are presented in Table 3.

Table 3. Coating thicknesses measured from cross-sections of plated objects.

Measurement	Cr(III) coating thickness (μm)	Cr(VI) coating thickness (μm)
1	46.4	20.8
2	42.2	20.8
3	44.2	21.1
4	43.7	21.4
5	45.6	21.3
6	46.6	20.3
7	45.3	20.2
8	46.7	20.4
9	46.5	20.5
10	45.1	20.1
Mean	45.2	20.7

Both measurement methods gave similar results for Cr(III) coating. Coating thickness measurements obtained with the Elcometer were slightly lower than those obtained with the microscopical method which could be due to the coating having a thin layer of nickel. Nickel is magnetic but not as magnetic as iron. The thickness of the nickel layer in the coating was approximately 5  $\mu\text{m}$  and the measurements made with Elcometer are relatively close to the thickness of the coating without the nickel layer. Measurements for Cr(VI) coating, on the other hand, varied significantly between measurements methods. This might be due to magnetic method not being as reliable when measuring round surfaces as when measuring flat surfaces. Both coated objects had a round surface but the one coated with Cr(VI) coating had a smaller diameter which might have affected the results.

### 4.3 Microhardness

Hardness of chrome plating can be tested using Vickers or Knoop microhardness test. In both of these tests an indenter is pressed on the surface of a test sample and is held there for a specified time under a specified load. After removing the indenter, the resulting indentation is measured with the help of a microscope and the diagonals of the indentation can be used to calculate the microhardness of the coating. [10, p. 8.]

Microhardness of a coating can be measured directly from its surface or from a cross-section. When measured directly from the surface the thickness of the coating needs to be measured and the applied force should be such that the indentation's depth is less than one tenth of the coating's thickness. If the indentation reaches close to the substrate the results of the test can be affected. Minimum thickness of the coating when measuring directly from surface for both Vickers and Knoop tests is 15  $\mu\text{m}$  but should also be at least 1.4 times the average length of the diagonals for the Vickers test and at least 0.35 times the length of longer diagonal for the Knoop test according to the standard SFS-EN ISO 4516. [10, p. 25.]

According to the standard SFS-EN ISO 4516 when measuring on cross-section of a hard coating using Vickers indenter the thickness of the coating should be at least 80  $\mu\text{m}$ . When Knoop indenter is used the thickness of the coating should be at least 25  $\mu\text{m}$ . One of the reasons why microhardness testing on a cross-section is more common is that it removes the influence of base material and surface roughness on the testing. The arithmetic average roughness of the test surface should not exceed 0.1  $\mu\text{m}$  and a compatible surface roughness can be reached during preparation of the cross-section. The test piece should be prepared the same way as the test piece for microscopical method for coating thickness measurement. [10, p. 25.]

Applied test force has a greater effect on the results of microhardness tests than on the results of microhardness tests. The maximum force that is compatible with the thickness of the coating has to be used to determine the most accurate microhardness value. Standard SFS-EN ISO 4516 presents graphs that can be used to determine the maximum compatible force. The velocity with which the indenter is pressed onto the surface of the test sample also has an effect on the results of the test. The correct velocity for the test can be determined by gradually reducing the velocity until the results of the test no longer vary. [10, p. 17, 21.]

## Testing

Microhardness of both coatings were tested from cross-section and directly from the surface of the coating. The tests were performed with Matsuzawa microhardness tester with a Vickers indenter. Figure 8 presents a micro hardness tester.

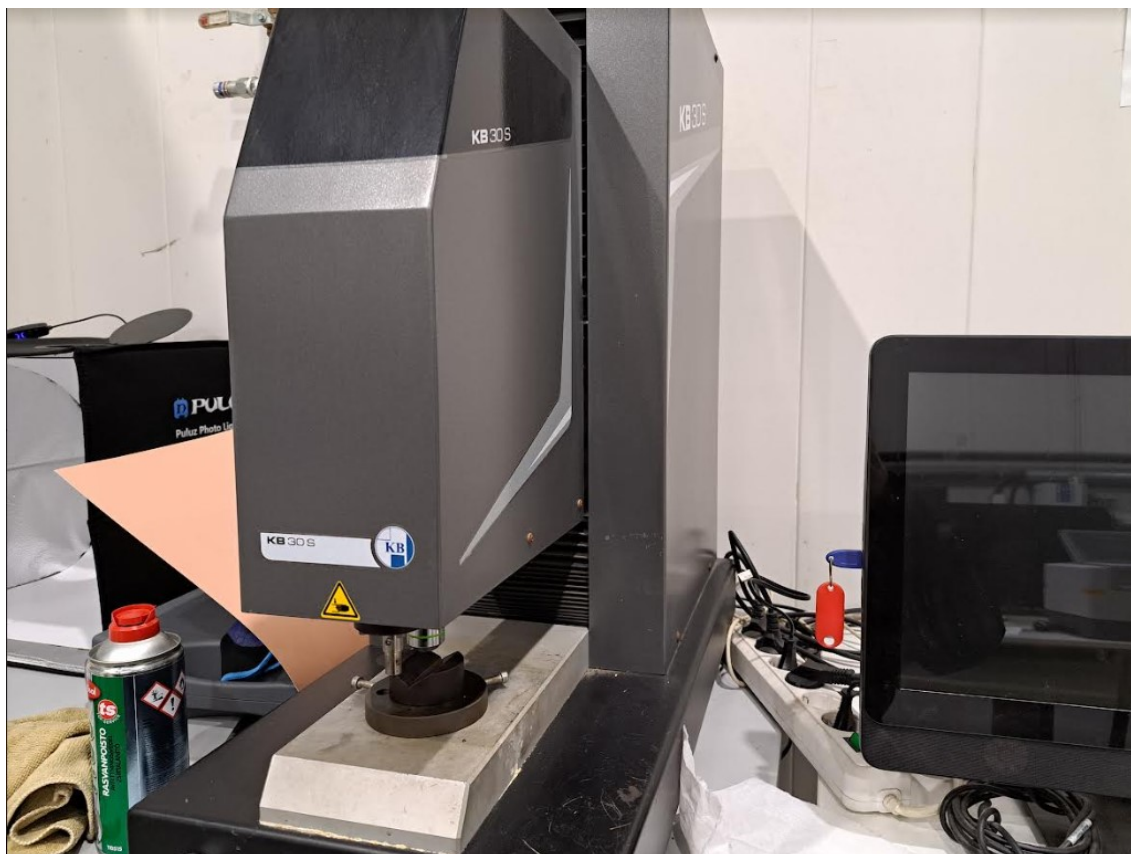


Figure 8. Vickers microhardness tester

Cross-sections were prepared the same way as the samples for microscopical coating thickness measurement. Microhardness from the surface was measured with the force of 100 g and pressing time of 15 seconds. The test results are presented in Table 4. Microhardness from the cross-section was measured with a force of 25 g and pressing time of 15 seconds. The resulting indentations were not properly formed so the microhardness could not be accurately measured.

Table 4. Microhardness values measured from the surfaces of the coated objects.

Measurement	Cr(III) microhardness (HV 0.1)	Cr(VI) microhardness (HV 0.1)
1	1123	808
2	1168	835



3	1140	787
4	1084	825
5	1118	833
6	1104	792
Mean	1123	813

The results were within the range of hardness that can be achieved with hard chrome plating. However, the coefficient of variation of the test results for both coatings were over 5 %.

#### 4.4 Adhesion

Practical adhesion of chrome plating can be tested with a quantitative single point scratch test. In this test the coating of the test piece is scratched by drawing a diamond indenter across its surface at constant speed and constant or continuously increasing force causing progressive mechanical damage. [10, p. 1.]

The scratch damage can be assessed using optical examination by microscope or scanning electron microscope, monitoring changes in drag force during the test and monitoring acoustic emission during the test. Optical examination is the primary method used to determine critical scratch load. Because different coatings can fail in different ways, it is important to specify which coating damage features are used to define critical scratch load or loads for coating that is being tested. Figure 9 displays coating damage features in a test where the load is progressive. [11, p. 3.]

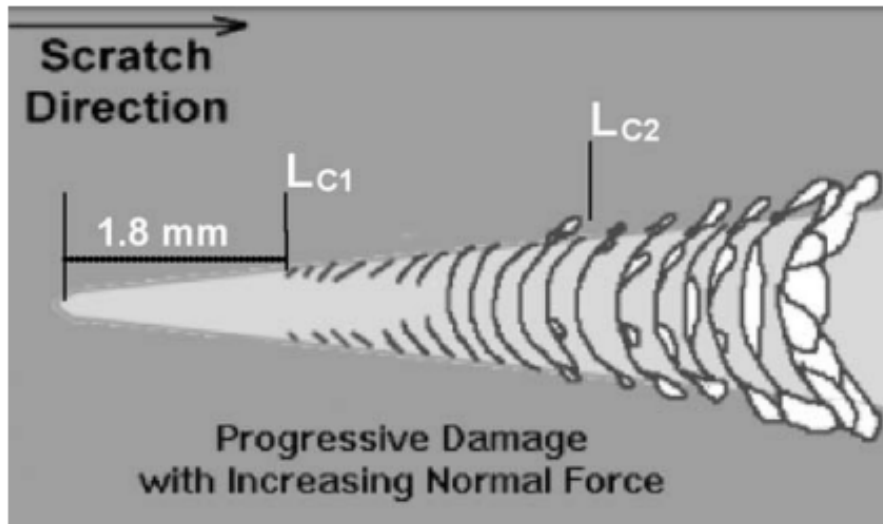


Figure 9. Coating damage features in a progressive load single point scratch test [10, p. 3.]

$L_{c1}$  indicates cohesive failure within the coating and is characterized by chevron cracking.  $L_{c2}$  indicates adhesive failure between the base material and the coating and is characterized by start of chipping damage. [11, p. 15.]

In tests where the load is constant, the critical scratch load is the force used in the test. In tests where the load is increasing the critical scratch load is calculated using equation 1. [10, p. 16.]

$$L_{CN} = \left[ L_{rate} \cdot \left( \frac{l_n}{X_{rate}} \right) \right] + L_{start}, \quad (1)$$

Where  $L_{CN}$  = critical scratch load for a defined type of damage,  $L_{rate}$  = rate of force application (N/min),  $l_n$  = Distance between the start of the scratch track and start point of defined type of damage (mm),  $X_{rate}$  = rate of horizontal displacement (mm/min) and  $L_{start}$  = Preload (N).

Table 5 presents common parameter values for both progressive load and constant load scratch tests where the maximum load is over 20 N.

Table 5. Parameter values for scratch tests.

<b>Parameter</b>	<b>Values</b>
Displacement Rate	10 mm/min
Loading Increment in constant load tests	1/5 of maximum load
Loading Rate in progressive load tests	100 N/min
Scratch length	≤ 10 mm
Minimum spacing between scratches	1 mm

Maximum load should be selected so that it causes selected coating damage features however it should not go over the critical load too much since higher loads can worsen the condition of the diamond stylus. [11, p. 13.]

Pre load for progressive load tests should be selected so that it produces recognizable opening indentation but no visible local damage in the coating. In constant load tests the load of the first test should be selected so that it is 20 % of maximum load. Subsequent test can then increase the load in 20 % increments until maximum load is reached. Other increments can also be used. [11, p. 13.]

The coating of the test pieces should be uniform across the whole surface area and its thickness must be known. It is also important to know the surface roughness of the coating because it has an effect on how the stresses within the coating develop. Before testing the surface of the test piece should be clean from grease, dust and other contaminants which could have an effect on the test results. [11, p. 11.]

## Testing

Samples for adhesion test were prepared by cutting 8 cm long pieces from the coated objects. The tests were performed with Ducom UniTest testing platform equipped with scratch testing device which is shown in Figure 10. The tests were executed using progressive load method and were setup according to parameters shown in standard ASTM C1624-05.  $L_{c2}$  was selected as the damage feature to define critical load and maximum load was set to 200 N.

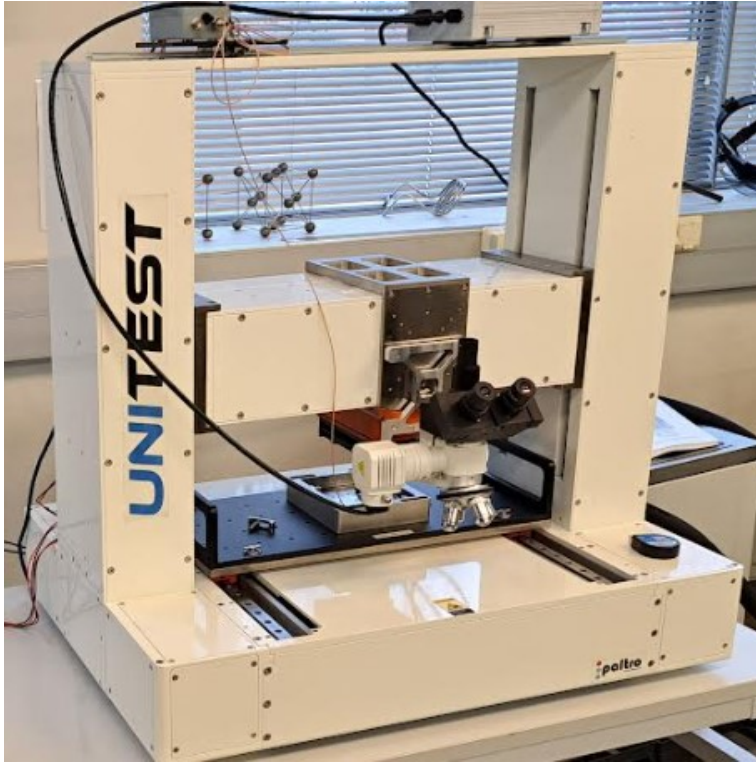


Figure 10. Ducom UniTest testing platform.

Test results are presented in Table 6. Measured critical load for Cr(VI) coating was higher than the critical load measured for Cr(III) coating. This could be due to Cr(VI) coating's smoother surface.

Table 6. Scratch test results.

Measurement	Cr(III) critical load (N)	Cr(VI) critical load (N)
1	93	152
2	99	150
Mean	96	151

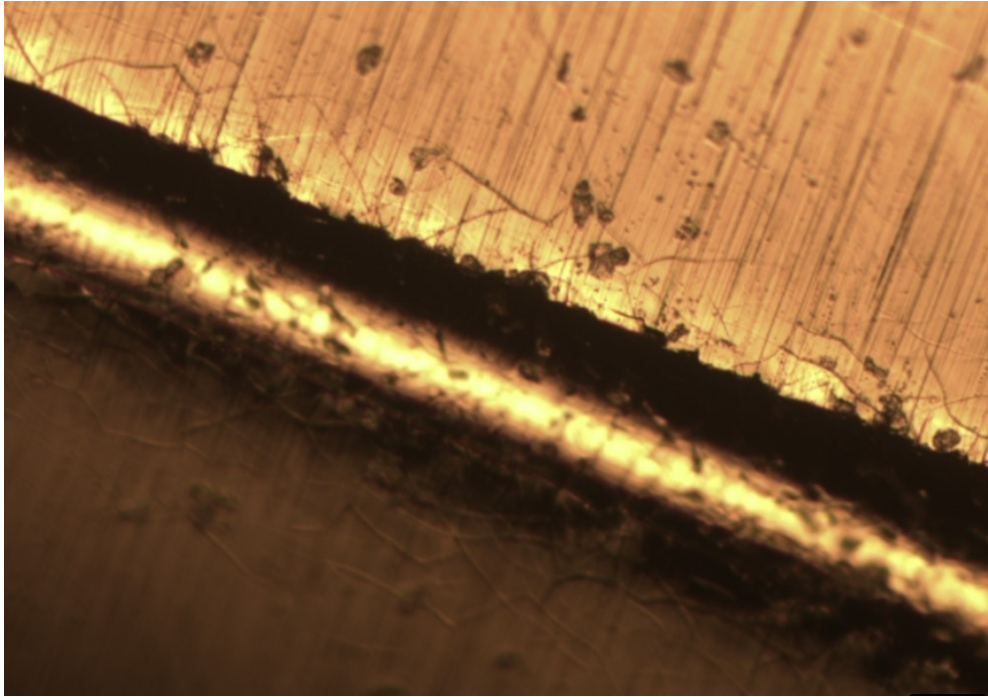


Figure 11. Scratch track and coating damage on a test piece.

#### 4.5 Porosity, microcracking and macrocracking

The amount of cracks that reach the base material in a coating on a ferrous base materials can be assessed with a ferroxyl test. During the test, pieces of test paper immersed with sodium chloride solution or gel are placed on a cleaned surface of a coating. The sodium chloride solution then reacts with the exposed base material and its iron ions are transferred to the test paper. After 10 minutes the paper is removed from the coating's surface and is placed in potassium hexacyanoferrate(III) solution where the reaction between the solution and iron from the base material forms blue markings on the paper in the spots where iron is present. The blue markings are then counted without the aid of magnification and the results can then be expressed as an average amount of pores per  $\text{cm}^2$  or as the maximum number of pores per  $\text{cm}^2$ . Figure 12 shows a blue markings on a test paper after testing. [12, p. 6-7.]

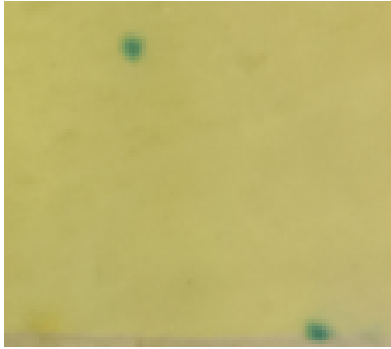


Figure 12. Blue markings on a test paper after testing.

Concentration of the sodium chloride solution should be 50 g/l and the same amount of agar or gelatin can be dissolved into the solution to provide it with gelling properties. Concentration of the potassium hexacyanoferrate(III) solution should be 10 g/l and its pH  $6.0 \pm 0.2$ . Chemicals used in the solutions should be analytical reagent grade and the water either distilled or deionized. Before testing the test paper should be tested for iron contamination by placing it in the sodium chloride solution, blotting and then placing it in the potassium hexacyanoferrate(III) solution where possible contamination will be revealed by blue markings or change in the color of the dried paper. [12, p. 5.]

Pores and cracks in chrome coatings are not desirable when it comes to corrosion resistance properties of the coating but microcracks can have a positive effect on wear properties in applications where lubricants are used. Pores improve wetting of the coating and helps lubricant retention after the lubricant is applied. A macrocrack is a crack that goes through the entire thickness of the coating. [3. s, 187]

### Testing

Before testing 200 ml of potassium hexacyanoferrate(III) solution was prepared by dissolving 2 g of potassium hexacyanoferrate(III) into deionized water. The pH of the solution was measured with pH paper to make sure it was within acceptable range. Same amount of sodium chloride solution was prepared by dissolving 10 g of sodium chloride in deionized water.

After the solutions were prepared the surfaces of the specimens were cleaned with ethanol and the test paper was checked for iron contamination by first placing it in sodium chloride solution and then in potassium hexacyanoferrate(III) solution. There was not any indication of iron contamination in the test paper.

Pieces of test paper were then immersed in sodium chloride solution and were placed on a glass plate to drain for 1 minute. After draining the pieces of test paper were placed on the coated objects for 10 minutes. Larger pieces of test paper did not keep in contact with the round surface of the coating properly so they were discarded. Smaller 1 cm<sup>2</sup> pieces of paper placed next to each other remained in contact with the surface much better and were placed the potassium hexacyanoferrate(III) solution. Blue marks developed in spots where cracks had reached the base material and the marks were counted. The test results are presented in Table 7.

Table 7. Ferroxy test results.

	<b>Cr(III)</b>	<b>Cr(VI)</b>
<b>Tested Surface Area</b>	6 cm <sup>2</sup>	6 cm <sup>2</sup>
<b>Pores / cm<sup>2</sup></b>	3.7	1.8

Cr(III) had higher amount of pores that reached the substrate than Cr(VI) which was to be expected since Cr(III) typically have a higher tendency to form macrocracks.

#### 4.6 Corrosion resistance

Corrosion resistance of chrome plating can be assessed using salt spray tests which can be performed in three standardized ways. In this method of testing a test piece is continuously sprayed a salt spray solution with specific pH and composition in specified temperature for a specified time inside a salt spray cabinet. Before the test the samples need to be carefully cleaned and arranged so that they are exposed to unimpeded circulation of the spray. Recommended

test periods are 2 h, 6 h, 24 h, 48 h, 96 h, 168 h, 240 h, 480 h, 720 h, and 1008 h. [13, p. 7, 13-14.]

After removing the test pieces from the cabinet, they should be allowed to dry for 0,5 – 1 hour and then cleaned so that the residues of the spray solution are removed. After that the test pieces can be examined for whether or not the coating meets certain requirements like appearance, number and distribution of corrosion defects, change in mass or change in mechanical properties. The purpose of the test and basis for evaluation of the coating to be tested should be defined before testing. [13, p. 15.]

#### 4.6.1 Neutral salt spray test (NSS)

Salt solution for the NSS test is prepared by dissolving sodium chloride in deionized or distilled water so that the concentration of the solution is  $50 \pm 5$  g/l. pH of the solution should be between 6.5 and 7.2 and required adjustments should be made by adding analytical grade hydrochloric acid, sodium bicarbonate or sodium hydroxide. [13, p. 8.]

#### 4.6.2 Acetic acid salt spray test (AASS) and copper accelerated salt spray test (CASS)

The solution for AASS test is prepared by lowering the pH of the previously mentioned sodium chloride solution to between 3 and 3.3 using acetic acid. In CASS test copper(II) chloride is added to the solution so that the concentration of the solution is  $0.26 \pm 0.02$  g/l. [13, p. 8.]

#### 4.6.3 Testing

Test pieces for neutral salt spray test were prepared by cutting 8 long cm pieces from coated objects. The test pieces are shown in Figure 13. The test pieces were then cleaned with ethanol. Areas of the specimens where the base material was exposed were sealed with duct tape.





Figure 13. Test pieces before exposure to salt spray.

Sodium chloride solution was prepared by dissolving sodium chloride in deionized water so that the concentration was 50 g/l. The pH of the solution was measured with pH paper and the measured pH was between 6.5 and 7 so no corrections had to be made.

Supports in the salt spray cabinet were not suitable for the test pieces so the test pieces were propped up against plastic sheets facing upwards at an angle of approximately  $15^\circ$  to the vertical. The plastic sheets were properly cleaned so that they would not introduce additional contaminants in the salt spray cabinet and were placed so that they did not block the specimens from being exposed to the salt spray.

Two plastic measuring cylinders with funnels placed on top were placed in the salt spray cabinet to function as collecting devices. The collection rate of the solution was checked daily and the average collection rate was 1.38 ml/h. The pH of the collected solution measured with pH paper and the measured pH levels were between 6.5 and 7.

The test pieces were removed from the salt spray cabinet after 170 hours and were allowed to dry for 45 minutes before the surfaces were cleaned under running water to remove remnants of salt spray solution. The coated parts of the test pieces did not show any corrosion defects. Duct tape that was used to seal off exposed base material had peeled in some parts of the specimens which resulted in the steel getting exposed to corrosion. Some of the corrosion products had dripped on the coating. Figure 14 shows the test pieces after the end of the test and after rinsing.



Figure 14 Test pieces after testing and cleaning.

Masses of the specimens changed slightly after they were cleaned. The mass of the specimen coated with Cr(III) coating changed from 365.4 g to 365.3 g and mass of the specimen coated with Cr(VI) coating changed from 87.7 g to 87.6 g.

## **5 Conclusion**

Development and use of trivalent chromium-based chrome plating processes and the use of other alternatives for hexavalent chrome will likely become more relevant as the use chromium trioxide is restricted. Information presented about electroless plating, HVOF and vapor deposition methods was limited as the goal was to present relevant information about their applicability as replacements for hard chrome plating.

Some of the tests did not go as initially planned, but they provided some useful observations such as duct tape possibly being not as reliable as protection for uncoated parts of samples with curved surfaces as it is for flat surfaces in salt spray tests. The goal of getting familiar testing parameters was achieved and those parameters and observations made during testing were used in the testing guidelines.

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## Coating thickness

### Microscopic method

Equipment:

- Mounting press
- PolyFast mounting resin or equivalent
- Grinding and polishing system / rotating wheel
- 100 or 180, 240, 400, 800 and 1200 grit abrasive
- Polishing cloth
- DiaPro Allegro/Largo diamond suspension or equivalent
- Microscope with a measuring device
- nitric acid-ethanol solution that consists of 5 vol% of nitric acid and 95 vol% of ethanol (optional)

Sample preparation:

- a) Cut a small piece of a coated object from the section where the measurement is to be performed.
- b) Mount, grind and polish the sample so that the cross-section is perpendicular to the coating, all deformed material is removed, and the surface is flat. Grinding can be started with 100 or 180 grit abrasive on a rotating wheel. Following grinding phases should be done with 240, 400, 800 and 1200 grit abrasives with no more than 30 – 40 seconds of grinding on each abrasive. Water is a suitable lubricant. Direction of the scratches should be adjusted by 90° every time the abrasive is changed and grinding pressure should be kept to a minimum. Polish the cross-section for 2 – 3 minutes using polishing cloth charged with 9 or 6 µm diamond paste or suspension on a rotating wheel.
- c) The cross section can be etched with a nitric acid-ethanol solution that consists of 5 vol% of nitric acid and 95 vol% of ethanol until the coating and the base material are clearly distinguishable from each other.

Measuring:

- a) Calibrate the microscope and measuring device.
- b) Measure the thickness of the coating on at least five points along the cross-section.

Test report:

- a) Identity of the sample.
- b) Results of the test including the location of the specimen on the coated object, measured thicknesses in micrometers or millimeters and the mean of the measured thicknesses.
- c) Unusual features that were observed during the test.
- d) Testing date.

Reference:

EN ISO 1463:2021. Metallic and oxide coatings. Measurement of coating thickness. Microscopical method.

### **Measurements with magnetic coating thickness gauge**

Equipment:

- Magnetic coating thickness gauge.

Shape and dimensions of the substrate can have an effect on reliability of the measurements.

Measuring:

- a) Calibrate the coating thickness gauge on an uncoated base material with a calibration foil that has a thickness that is close to, but greater than the expected thickness of the coating.
- b) Place the probe of the instrument perpendicularly to the coating so that the coating thickness is calculated.
- c) It is recommended to take at least 3 measurements that are spread evenly on each test area.

Test report:

- a) Identity of the tested product.
- b) Individual measurements and their mean.
- c) Any deviations from the procedure.
- d) Unusual features that were observed during the test.
- e) Testing date.

Reference:

EN ISO 2080:2019. Paints and varnishes. Determination of film thickness.



## Surface roughness

Equipment:

- Surface profilometer with a stylus that conforms to standard EN ISO 3744.

Sample preparation:

- a) The measured surface should be flat.
- b) Clean the surface of any defects that may have an effect on measurements.

Measurement:

- a) Select the surface roughness parameters that will be measured.
- b) Follow the conditions of Table 2.1. Application Group 1 refers to products that are mainly used in automotive industry and Application Group 2 refers to products that are mainly used in other applications. Cut-off  $\lambda_c$  that is used is important since if it is set too low the measured surface roughness values could be lower than the actual values. If it is set too high the measured values could be higher than the actual values.

Table 2.1 Measuring conditions [EN ISO 10049 Measurement of roughness average Ra and peak count R<sub>Pc</sub> on metallic flat products.]

Field of application	Unit	Application group 1 <sup>a</sup>	Application group 2 <sup>a</sup>
Cut-off $\lambda c$	mm	2,5	0,8 <sup>b</sup>
Section line $c_1$	$\mu\text{m}$	+ 0,5	<sup>c</sup>
Section line $c_2$	$\mu\text{m}$	- 0,5	<sup>c</sup>
Transmission band $\lambda c/\lambda s$	-	300/1	300/1
Evaluation length $l_m$	mm	5 $\lambda c$	5 $\lambda c$
Maximum sampling spacing	$\mu\text{m}$	1,6	0,5
Maximum measuring force	mN	1	1
Maximum measuring speed	mm/sec	1	1
Measuring direction for stochastic surfaces	-	The recommended direction is perpendicular to the rolling direction.	
Measuring direction for non-stochastic (or non-isotropic) surfaces	-	The measuring direction should be different from the principle direction of the texturing process, but can vary as agreed on between parties.  EXAMPLE A hexagonal pattern has 30° or 60° as principal directions, depending on the position of the hexagon relative to the rolling direction. In this case a measuring direction of 45° ± 5° is recommended.	
Number of measurements for averaging over the surface	-	Minimum 3 <sup>c</sup>	
Specification	-	R <sub>a</sub> and R <sub>Pc</sub> By exception R <sub>a</sub> <sup>c</sup>	R <sub>a</sub> or (R <sub>a</sub> and R <sub>Pc</sub> ) <sup>c</sup>
<sup>a</sup> For the definitions, see 3.1.			
<sup>b</sup> For certain specific applications, it may be necessary to use a different $\lambda c$ .			
<sup>c</sup> To be agreed between parties and included in the test report.			

Test report:

- a) Type of the used measuring head.
- b) Cut-off  $\lambda c$ .
- c) Measuring direction.
- d) Measurements and their mean.

e) Measuring conditions other than those presented in Table 1.

Reference:

EN ISO 10049. Measurement of roughness average Ra and peak count RPc on metallic flat products.

## Microhardness

### Equipment:

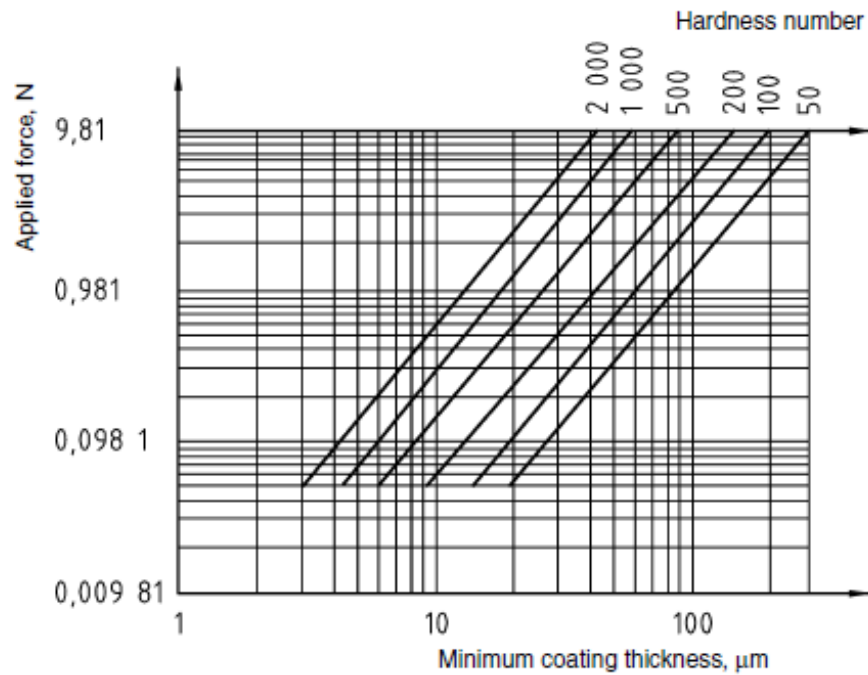
- Vickers or Knoop microhardness tester
- Mounting press
- PolyFast mounting resin or equivalent
- Grinding and polishing system / rotating wheel
- 100 or 180, 240, 400, 800 and 1200 grit abrasive
- Polishing cloth
- DiaPro Allegro/Largo diamond suspension or equivalent

### Cross-section preparation:

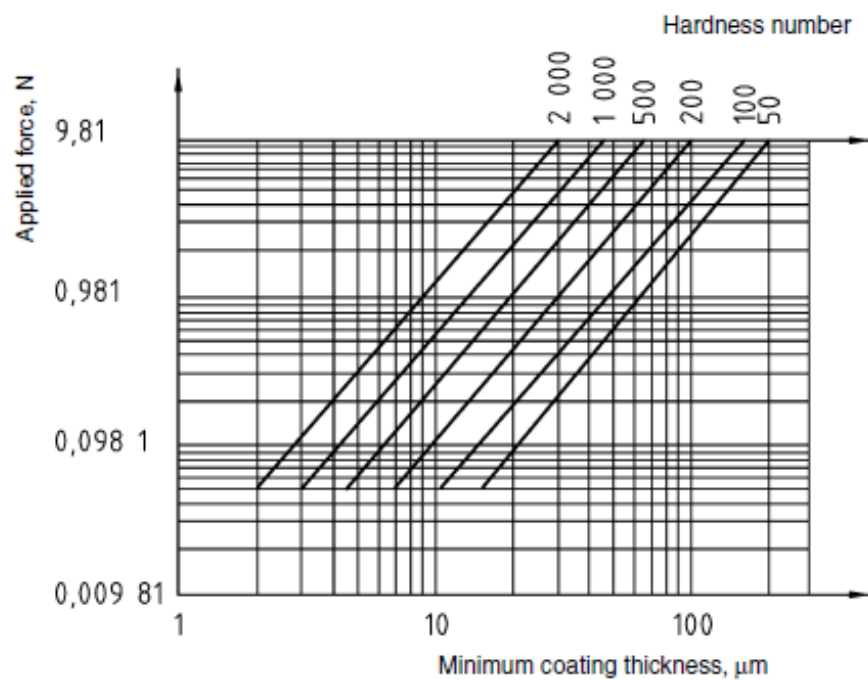
- a) Cut small piece of a coated object from the section where the measurement is to be performed.
- b) Mount, grind and polish the sample so that the cross-section is perpendicular to the coating, all deformed material is removed and the surface is flat. Grinding can be started with 100 or 180 grit abrasive on a rotating wheel. Following grinding phases should be done with 240, 400, 800 and 1200 grit abrasives with no more than 30 – 40 seconds of grinding on each abrasive. Water is a suitable lubricant. Direction of the scratches should be adjusted by 90° every time the abrasive is changed and grinding pressure should be kept to a minimum. Polish the cross-section for 2 – 3 minutes using polishing cloth charged with 9 or 6 µm diamond paste or suspension on a rotating wheel.
- c) The cross section can be etched with a nitric acid-ethanol solution that consists of 5 vol% of nitric acid and 95 vol% of ethanol until the coating and the base material are clearly distinguishable from each other.

### Testing:

- a) Select the maximum compatible force for the coating using the graphs shown in figure 3.1.



a) Vickers hardness



b) Knoop hardness

Figure 3.1 [EN ISO 4516. Metallic and other inorganic coatings. Vickers and Knoop microhardness tests.]

- b) Determine the correct velocity of the indenter for the test by gradually decreasing the velocity with which the indenter is brought on the test surface until the test results no longer vary.
- c) The force application time should be set to 10 – 15 s.
- d) The test should be done at  $23\text{ °C} \pm 5\text{ °C}$  temperature.

Test report:

- a) Measured microhardness values.
- b) Coefficient of variation.
- c) Force application time.
- d) Location on the sample measurement was performed. Surface of the coating or cross section.
- e) coating thickness.
- f) Surface roughness.
- g) Surface curvature.
- h) Temperature during measurement.
- i) Test date.

Reference:

EN ISO 4516. Metallic and other inorganic coatings. Vickers and Knoop microhardness tests.

## **Adhesion**

### **Quantitative single point scratch test**

#### Equipment:

- Material tester with scratch test configuration.
- Microscope with a measuring device.

#### Sample preparation:

- a) Remove any contamination that might have an effect on the test results.
- b) The test piece should be flat, level and uniform across its surface area

#### Testing:

- a) Specify which coating damage features are used to define critical scratch load or loads for coating that is being tested.
- b) Check the calibration of the system and the condition of the indenter.
- c) Select maximum load for the test. It should be selected so that it produces desired coating damage features without exceeding critical load too much.
- d) Select preload. In progressive load tests it should produce a recognizable opening indentation without visible local damage. In constant load tests preload can be selected so that it is 20 % of maximum load. Subsequent test can then increase the load in 20 % increments until maximum load is reached. Other increments can also be used.

- e) Other parameters should be set according to values provided in table 4.1.

Table 4.1 Scratch test parameter values.

Parameter	Values
Displacement Rate	10 mm/min
Loading Increment in constant load tests	1/5 of maximum load
Loading Rate in progressive load tests	100 N/min
Scratch length	≤ 10 mm
Minimum spacing between scratches	1 mm

- f) At least 5 tests should be made to determine the critical scratch load.
- g) Analyze the scratch track and damage features with microscope. In constant load tests the critical load is the force used in the test that caused desired damage features. Critical load for progressive load tests can be calculated with equation 1.

$$L_{CN} = \left[ L_{rate} \cdot \left( \frac{l_n}{x_{rate}} \right) \right] + L_{start}, \quad (1)$$

Where  $L_{CN}$  = critical scratch load for a defined type of damage,  $L_{rate}$  = rate of force application (N/min),  $l_n$  = Distance between the start of the scratch track and start point of defined type of damage (mm),  $x_{rate}$  = rate of horizontal displacement (mm/min) and  $L_{start}$  = Preload (N).

Test report:

- a) Identity of the sample
- b) Thickness of the coating



- c) Sample preparation and cleaning method
- d) Number of samples and tests for each sample.
- e) Maximum load, preload, loading rate, scratch length and horizontal displacement rate
- f) Critical scratch load of each test, coefficient of variation and mean.
- g) Distance between each scratch
- h) Description of the test system.

Reference:

ASTM C1624 – 05. Standard test method for adhesion strength and mechanical failure modes of ceramic coatings by quantitative single point scratch testing.

## Porosity

### Ferroxyl test

Equipment and reagents:

- Two beakers
- Glass plate (optional)
- Test paper. Filter paper for an example.
- Potassium hexacyanoferrate(III)
- Sodium chloride
- pH meter or pH paper
- Distilled or deionized water
- Or a ferroxyl test kit

Solution preparation:

Sodium chloride solution.

- a) Dissolve sodium chloride in distilled or deionized water so that the concentration is 50 g/l .

Potassium hexacyanoferrate(III) solution.

- a) Dissolve potassium hexacyanoferrate(III) in distilled or deionized water so that the concentration is 10 g/l.
- b) Measure the pH of the solution. The pH should be  $6 \pm 0.2$ .

If the test area is not very large 100 ml or less of each solution should be enough.

Sample preparation:

- a) Clean the surface of the sample.

Testing:

- a) Make sure the test paper is free of iron contamination by immersing it first in the sodium chloride solution and then in the potassium hexacyanoferrate(III) solution. Possible iron contamination will be revealed by blue markings developing on the paper.
- b) Immerse strips or pieces of test paper in the sodium chloride solution. Using multiple smaller pieces rather than fewer larger pieces can be helpful if the tested surface is round or irregular.
- c) Drain the pieces of paper of excess solution for 1 minute. Glass plate can be helpful.
- d) Apply the test paper on the surface of the coating so that it is firmly in contact. If the paper starts to dry, apply additional solution without moving the paper.
- e) After 10 minutes, place the paper in the potassium hexacyanoferrate(III) solution where blue markings develop in spots where cracking in the coating reaches the base material.
- f) Count the blue markings without the aid of magnification.

Test report:

- a) Area of the tested surface
- b) Number of pores per  $\text{cm}^2$  or maximum count of pores per  $\text{cm}^2$

Reference:

EN ISO 10309. Metallic coatings. Porosity tests. Ferroxy test.

## Corrosion

### Neutral salt spray test

#### Equipment:

- Salt spray cabinet
- At least 2 collecting devices. Measuring cylinder with a funnel placed on top for an example.
- Sodium chloride
- Distilled or deionized water
- pH meter or pH paper
- Something to protect uncoated parts of the test samples. Tape, paint or wax for an example.

#### Sample preparation:

- a) Type and the number of samples should be agreed between interested parties.
- b) Carefully clean the samples.
- c) Protect the uncoated parts of the sample. Duct tape can be useful if the samples are flat. Paint or wax might work better for round or irregular samples.

#### Salt solution preparation:

- a) Dissolve sodium chloride in distilled or deionized water so that the concentration of the solution is between 45 – 55 g/l
- b) Measure the pH of the solution. The pH should be 6.5 – 7.2. Adjust the pH with analytical grade hydrochloric acid, sodium bicarbonate or sodium hydroxide if necessary.

Select the criteria on the basis of which the performance of the coating is evaluated. These can include:

- a) Appearance after testing
- b) Appearance after removal of superficial corrosion products
- c) Time until first signs of corrosion appeared
- d) Change in mass
- e) Amount and distribution of corrosion defects
- f) Changes in the coating's properties
- g) Changes in the coating revealed by examination with a microscope.

Select the duration of the test. Recommended durations are 2 h, 6 h, 24 h, 48 h, 96 h, 168 h, 240 h, 480 h, 720 h, and 1008 h.

Select how to treat the samples after testing. For an example after removing the samples from the salt spray cabinet, let them to dry for 30 – 60 minutes. After drying the samples can be carefully cleaned under running water to remove remnants of the salt solution before examination.

Testing:

- a) Set the temperature to 35 °C
- b) Place the collecting devices in areas of the cabinet where the samples are located so that only mist and not liquid falling off other parts is collected
- c) Check the collection rate and pH of the collected solution. Collection rate should be 1.5 ml/h  $\pm$  0.5 ml/h and the pH of the collected solution should be 6.5 – 7.2.
- d) Place the samples in the salt spray cabinet so that they are exposed to the circulation of the salt spray and facing up as close to an angle of 20° to the vertical as possible. Supports for the samples should be inert.

- e) The test should only be interrupted for examination of the samples, replenishing the salt solution and checking the collection rate. It is recommended to check the collection rate daily.

Depending on the selected testing criteria, the test report can include:

- a) Test results based on the selected criteria.
- b) Amount and identity of the samples.
- c) Sample preparation method.
- d) Angle of the samples during testing.
- e) Treatment of samples after the test.
- f) Collection rate and pH of the salt solution.
- g) Temperature.
- h) Time between inspections.
- i) Duration of the test.

Reference:

EN ISO 9227:2022. Corrosion tests in artificial atmospheres. Salt spray tests.

