THESIS ON MECHANICAL AND INSTRUMENTAL ENGINEERING E59

# Calibration Methods of Coating Thickness Standards

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Declaration:
Hereby I declare that this doctoral thesis, my original investigation and achievement, submitted for the doctoral degree at Tallinn University of Technology has not been submitted for any academic degree.

/Jürgen Riim/


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# CONTENTS

# INTRODUCTON

The history of using coatings is fairly long. Goldsmiths' techniques which enabled to make approximately 100 nm thin gold foils were known long ago, whereas a scientific approach to the study of thin layers began in the 17th century when Boyle, Hooke and Newton conducted optical experiments which resulted in the first artificially created coating by M. Faraday. Greater development started between the two world wars and was made even more intensive by the rise of microelectronics. The term „thin film" (German *dünne Schicht*) also comes from that time, having been introduced by H. Mayer in 1950s [25, 57, 71, 72].

Nowadays it is probably impossible to find a scientific or technical discipline where thin coatings are not used. Industry is forced into using various combinations of materials primarily because of stronger competition, decreasing raw material resources and ever-increasing customer demands. Although in everyday life we see coatings mainly in the use of paints and lacquers, coatings are even more used in electronics, mechanical engineering and aviation. A wide range of use in its turn means very different combinations of bases and coatings from insulating, conductive, ferromagnetic, non-ferromagnetic and other materials.

Since the purpose of coatings is very wide, a number of various parameters are assessed at coatings. For example, zinc film as a coating may have a function of preventing corrosion on car body, while elsewhere its purpose could be to function as a conductor or an interference filter. H. Mayer has described thin coatings as **a special state of matter** (German *Besondere Zustandsform der Materie*) whose characteristics differ from those of a bigger body of the same material [57]. The value of such physical quantities as density, refractive index, Hall number, permittivity, spectral resistance and others depends on coating thickness. Therefore the thickness of coatings mostly has two meanings:

1. To ensure an optimum coating thickness, for example, durability of galvanic surface to environmental factors, or saving on expensive materials**,** like gold, silver or platinum for a minimum coating thickness.
2. To create a coating of certain thickness resulting in new physical properties of the material [25].

Nowadays a number of various methods are used to evaluate coatings. The working principles of the methods depend on the place where the coating is used, combination of materials, accuracy, and the physical quantity evaluated. The estimated number of methods based on different working principles is more than one hundred [25]. Based on working principles, the following methods of measurement are used, some of which are even standardized: **mechanical methods** (*e.g* measurement by means of length measuring instrument [60], stylus instruments [60], wet film meters; evaluation of coating thickness according to the mass of coating or spraying power; evaluating decrease in difference of atmospheric pressure of the coating thickness by means of absorbing layer of gas or pneumatic method of measurement; ultrasound method [74]; measuring coating thickness by measuring the mass of material evaporated or coated [13, 61**]**); **electric and magnetic methods** (*e.g.* measurement of electric resistance, capacity as well as ionic emission and discharge voltage; HALL voltage-, Eddy Current [69], magnetic force, magnetic

induction [70] and electrolysis properties [62]; **thermal methods** (method of thermoelectric effect–Seebeck-effect); **measurement by means of emission** (Scanning [63], Fluorescence and Beta backscatter [64] and Trace method); **optical methods** (methods applying microscopic enlarging [59], interference [65], light intensity, ellypsometric, spectroscopic or absorption methods).

Most industrial measurement methods are non-destructive and indirect measurement methods. This means that upon the evaluation of coating thickness a physical quantity length is not measured, but the quantity is evaluated in another manner, for example by the value of magnetic force or eddy currents. In order to express the results of measurement received by using indirect measurement methods in units of length, measuring instruments have to be calibrated by using coating thickness standards.

A coating thickness standard is a detail, the thickness of coating(s) on which has been mostly measured by a direct length-measuring instrument [87, 88]. Whereas in case of majority of non-destructive measurement methods the results of measurement are in addition to coating thickness influenced by a number of other parameters, such as selection, cleanliness and dimensions of the base material, surface roughness, geometrical deviations and shape of the surface, etc., the coating thickness standards should be as similar as possible to objects to be measured. For example, if a magnetic force meter is used to measure the thickness of a zinc layer applied on a thin steel detail, a thin coating thickness standard made from similar steel should be used to calibrate such a measuring instrument. Which coating thickness standard should be used, is naturally greatly dependent on the technique used, range of measurement, accuracy of measurement and other parameters. Nowadays, a number of different standards are used. Based on the number, thickness and shape of coatings and other parameters, they can be classified as follows: **single value-**, **multi value-**, **multi base-**, **multi coatings-**, **stair-shaped coating thickness standards** and imitators. A better overview has been presented in [88] and the more important patents in the field are [7–10, 42–56].

Several measurement methods are used nowadays to calibrate coating thickness standards, principal methods still being direct mechanical or optical methods. Modern standards of coating thickness, by which coating thickness is directly measured by the value length , have been created since the 1970s, when Prof. Rein Laaneots from Tallinn University of Technology started research into this field. As a matter of fact, standards for measuring coating thickness according to mass had already been created before, for example, by the company KOCOUR since 1952, NPO “Isari” in Georgia [33, 82]. Outstanding work has been done by NIST and PTB, where uncertainty of calibration of coating thickness standards has been reached even up to nanometers [4, 5, 11, 27–31, 67, 85].

## Problem settings

Measuring devices used to evaluate coatings have undergone major development in last twenty years. Both the sensitivity of sensors (nowadays approximately (0,05 – 0,1) μm) and measurement software used for analysis have been improved. NIST and PTB have offered both calibration service and standards to manufacturing companies [15, 67, 86]. In using such working standards manufacturers of different

coating thickness standard gauges have expressed “the accuracy” of coating thickness (not the measurement uncertainty of measurement result) in the range from 0,5 µm + 1 % to 2,5 µm + 3 % [6, 14, 15, 17, 18, 20, 24, 73]. It is still questionable, because it is unknown what is meant under accuracy. At present the expanded uncertainty of coating thickness measurement result of working standards is within (0,5 – 2) µm [15, 67, 86]. On the one hand, it is possible to make very accurate standards, but their price, measuring range and combinations of materials do not meet market expectations. In order to produce accurate standards, it is necessary to know the shape of both boundary surfaces of the coating. In case of common calibration methods, boundary surfaces are not evaluated directly but by means of other planes or similar methods. Such an approach creates a situation were a very accurate coating thickness standard is also very expensive or a less expensively made coating thickness standard is of lower accuracy.

## Main objectives of the thesis

Because of the above mentioned problem, the objective of the doctoral thesis is **to find an alternative to calibrate coating thickness standards of various types and various materials more accurately and less expensively, so that the result of measurement is in accordance with the definition of coating thickness**. On this basis the following tasks need to be solved to accomplish the doctoral thesis:

- Updating of the existing coating thickness calibration methods
- Development of a new measurement method for direct calibration of coating thickness standards
- Application of measurement uncertainty theory to measurement models
- Verification of theoretical models in the course of practical measurements

## Acknowledgements

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# ABBREVATIONS

| | |
|---|---|
| BIML | International Bureau of Legal Metrology |
| BIPM | International Bureau of Weights and Measures |
| EURAMET | European Association of National Metrology Institutes |
| ISO | International Organization for Standardisation |
| LNE | Laboratoire national de métrologie et d'essais (National Metrology Institute, France) |
| NIST | National Institute of Standards and Technology, USA |
| NPO | Scientific Industrial Association in Georgia |
| PTB | Physikalisch-Technische Bundesanstalt (National Metrology Institute, Germany |

# SYMBOLS

| | |
|---|---|
| $h_c$ | Coating thickness |
| $h_{max}$ | Maximum coating thickness |
| $h_{mean}$ | Mean coating thickness |
| $h_{min}$ | Minimum coating thickness |
| $h_p$ | Coating thickness at certain point |
| $h_t$ | True coating thickness |
| *Ra* | Surface roughness parameter |
| *Rz* | Surface roughness parameter |
| *S* | Cross section |
| *s* | Variance |
| *U, u* | Expanded uncertainty, standard uncertainty |
| $Z_i$ | Boundary surface |
| $\delta x$ | Correction factor |

# 1 DEFINITION OF COATING THICKNESS

The surface of every physical substance (solid or liquid) forms a boundary/marginal surface with its surrounding environment. Therefore, as can also be seen in Figure 1.1, coating can be defined as an amount/volume of liquid or solid material, which has been protracted in at least two dimensions and which borders on two parallel boundary surfaces. Coating thickness $h_c$ is defined as a coating normal (a line crosswise to the base) and a distance between the intersections of the boundary surfaces. [25, 58, 68, 88]

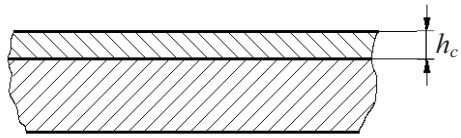


*Figure 1.1 Theoretical coating thickness*

That kind of geometrical positioning, however, never occurs in practice. Even highly polished surfaces or even crystal surfaces have a certain surface roughness. That is why further it is possible to speak about coating thickness at a certain point $h_p$, which often is also referred to as local coating thickness [25]. In essence, that is proximity of a true coating thickness $h_t$. At the current level of technology, the closest measuring method for assessing $h_t$ is the x-ray interference method, which allows coating thickness to be measured on an area of a few square nanometres [25]. As a result, the function of coating thickness is obtained depending on location.

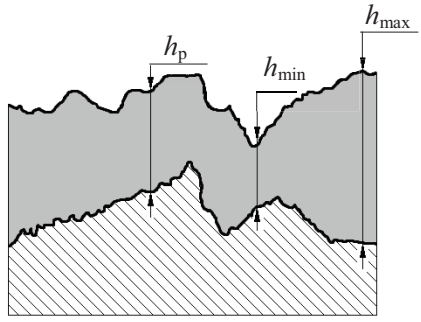


*Figure 1.2 Various coating thicknesses*

The other extreme is assessment of coating thickness according to the mass. Coating thickness calculated relying on this method is also referred to as mass thickness and it describes the mean thickness of the coating referred to as mean thickness $h_{mean}$ the best.

The majority of other methods lie, essentially, in between the above two methods and, in general, coating thicknesses measured by different methods are incomparable with each other. For example, such terms are used as optical coating thickness and obstructional/resistance coating thickness. In addition to the above-mentioned terms,

there are word combinations used the most, such as minimal coating thickness $h_{min}$ and maximum coating thickness $h_{max}$, the essence of which is clearly easy to understand. Since the expected properties of a coating vary to a great extent, there is a need for coating thicknesses, which are differently defined. However, there has to be an opportunity provided to compare them with each other or measure the value of coating thickness.

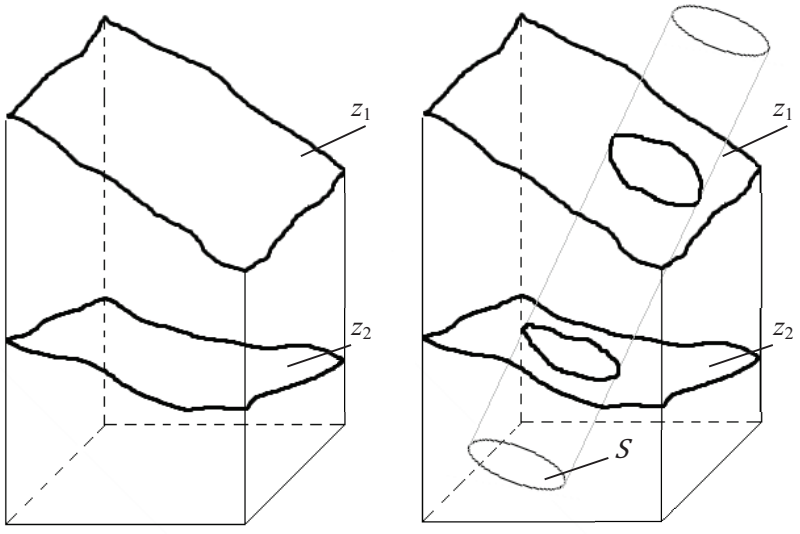


*Figure 1.3 The illustration of a coated element: $z_1$ – surface between the coating and exterior environment, $z_2$ – surface between the coating and the base*

Figure 1.3 shows an element which has been coated. The topmost and bottom boundary areas can, according to [12] be described as follows:

$$z_1 = f_1(x, y);\ z_2 = f_2(x, y), \tag{1.1}$$

in which $f_1(x, y)$ is an equation between the coating and the exterior environment and $f_2(x, y)$ is an equation for the boundary area between the coating and the base material. According to the definition of coating thickness it is impossible in this situation to find the value $h_c$ since there is no possibility of the coating normals of the topmost and bottom boundary areas to conform. In order to simplify the situation, let us select a part of the element $A$ with a cross-sectional area $S$ (Figure 1.3). According to [12] the *mean surface* has to be found to the bottom boundary surface,

$$a_2x + b_2y + c_2z + d_2 = 0 \tag{1.2}$$

in which the sum of square distances of all the points of boundary surface $z_2$ from the *mean surface* would be minimal.

The plane of the *mean surface* is combined with the coordinate system 0*XYZ* so that the coordinate plane 0*XY* would be congruent with the *mean surface*, as can be seen in Figure 1.4.

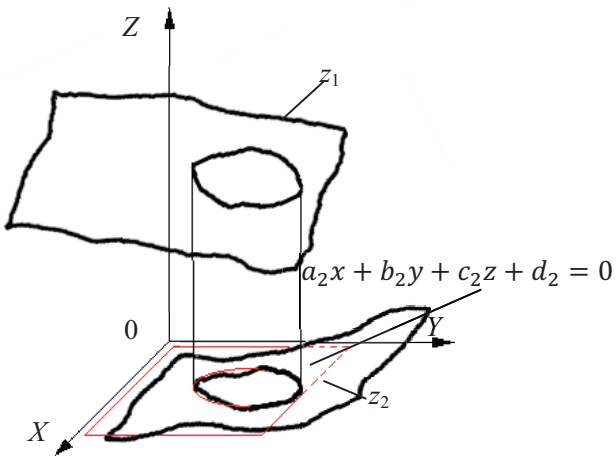


*Figure 1.4 The coating thickness from the mean surface: $z_1$ – surface between the coating and exterior environment, $z_2$ – surface between the coating and the base*

In that case, coating thickness $h_p$, can be found according to [37] at any point $i$, which is located on the plane in the coordinate system $0XY$, using the following equation:

$$h_{pi} = z_{i1} - z_{i2} = f_1(x_i, y_i) - f_2(x_i, y_i) \tag{1.3}$$

The minimal and maximum coating thicknesses $h_{min}$ and $h_{max}$ are correspondingly defined by the minimal and maximum values of $h_{pi}$ in section $A$ (with an area $S$). The mean coating thickness $h_{mean}$, between the bottom and topmost boundary surface can be found according to the integral:

$$h_{mean} = \frac{1}{S}\iint_A [f_1(x_i, y_i) - f_2(x_i, y_i)]\, dx\, dy \tag{1.4}$$

At the same time, when issuing from the surface thickness definition, it is possible to calculate the coating thickness of the element in between the $h_{pi}$ coating and a certain point $j$ of the boundary surface from the following equation:

$$h_{pij} = \min_{x_j, y_j \in A} \sqrt{\begin{matrix}[f_1(x_j, y_j) - f_2(x_j, y_j)]^2 + \\ +(x_j - x_j)^2 + (y_j - y_j)^2\end{matrix}} \tag{1.5}$$

in which $A$ is the area within which the coating thickness is studied. The equation essentially finds out the shortest spatial distance between two boundary areas, applying the sum of the squares of the three legs.

Since both the upper and the lower boundary surface of the coating can be described by functions $f_1(x, y)$ and $f_2(x, y)$, the solution mentioned also allows finding the function characterizing the coating thickness. If coating thickness can be expressed as a minimal distance between two planes in a room in relation to a

boundary surface, *e.g.* to the normal of surface $f_1(x,y)$, it follows that, on the basis of [38] the mathematical function describing the coating thickness is:

$$h(x,y) = \sqrt{|\nabla f_1|^2 + 1} \cdot F(x,y) \tag{1.6}$$

in which $\nabla f_1$ is gradient $\left(\nabla f_1 = \left(\frac{\partial f_1}{\partial x}, \frac{\partial f_2}{\partial y}\right)\right)$ of the function $f_1(x,y)$ and $F(x,y)$ is the function, which satisfies the condition:

$$f_1(x,y) - F(x,y) = f_2\left[x + \frac{\partial f_1}{\partial x} \cdot F(x,y), y + \frac{\partial f_1}{\partial y} \cdot F(x,y)\right] \tag{1.7}$$

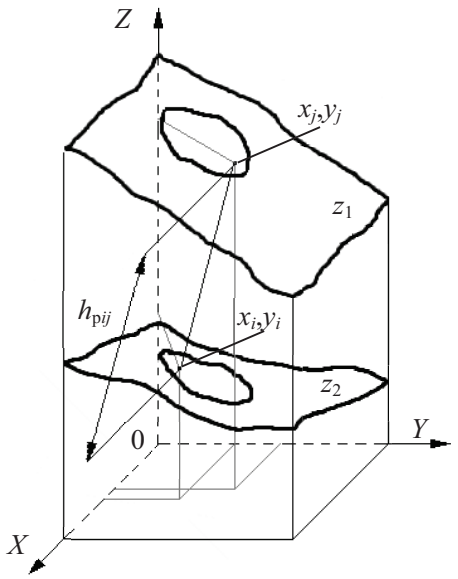


*Figure 1.5 The coating thickness located in between two boundary surfaces $z_1$ and $z_2$*

The function of coating thickness is simplified if a minimal, maximum or mean coating thickness is to be found. According to [38] the latter can mathematically be expressed as follows:

$$h_{\min} = \min_{x,y \in A} h(x,y) \tag{1.8}$$

$$h_{\max} = \max_{x,y \in A} h(x,y) \tag{1.9}$$

$$h_{\text{mean}} = \frac{1}{S} \iint_A h(x,y)dxdy \tag{1.10}$$

in which $A$ is the field, in which function $h(x,y)$, of the coating thickness is valid, and $S$ is the area of the field. Assuming that boundary surfaces $f_1(x,y)$ and $f_2(x,y)$ are parallel to each other, they can be described as:

$$f_1(x,y) = ax + by + c \tag{1.11}$$

$$f_2(x,y) = ax + by + d \tag{1.12}$$

The function of the coating thickness can thereby be considerably simplified

$$h(x,y) = \sqrt{a^2 + b^2 + 1}\frac{c+d}{a^2+b^2+1} \tag{1.13}$$

that from arithmetics is known as an expression of the addition of distances between the two parallel surfaces.

# 2 MEASUREMENTS OF A CONTOUR WITH STYLUS INSTRUMENT

The aim of this thesis is the direct measurement of coating thickness standards, for which various direct measurement instruments are used. In order to evaluate the quality of the measurement and the results, it is necessary to analyze possible uncertainty components. Depending on the coating thickness standard, the measurement model of assessing coating thickness is very different. In certain cases it is not required to consider geometrical deviations as well as surface roughness, but in the case of cost-effective standards, these are the main uncertainty components. Therefore, a general description of the measurement model has been given which is suitable for measuring other details (complex bodies) too.

This measurement model was prepared for the measurement system Perthometer Concept [75] located in the metrology laboratory of the Tallinn University of Technology. In the course of measurement of the surface, a measurement model was composed and the values of the input quantities as well as their distribution were experimentally determined. As a result of the research, the reliability of the measurement results of the surface measurement can be characterized based on expanded uncertainty.

The calculation example and the values in it depend to a great extent on the measurement device as well as the object, thus it should be considered as information and not to be used as a direct requirement.

*Uncertainty of measurement* is, by its definition a parameter, associated with the result of a measurement, and characterizing the dispersion of the values that can, in all probability, be attributed to the measurand [22, 35]. It reflects the lack of exact knowledge about the value of the measurand. Thus, owing to the uncertainty arising from random effects and from imperfect correction of the result for systematic effects, the result of the measurement after correction for recognized systematic effects is still only a rough estimate of the true value of the measurand. For this reason, each measuring result should be associated with information about the uncertainty evaluatin, identifying the possible dispersion of the true value of the measurand. In metrology laboratories, mostly standardized procedures are used in evaluating measuring uncertainty. However, these procedures require extended statistical and mathematical knowledge, the application of which, as a rule, cannot be found to the required extent in industry.

In published research results [79–81] surface roughness was measured by a roughness measuring instrument. The uncertainty of measurement results could be estimated by the uncertainty contribution of the measurement instrument. This forms about (10−15) % of the total indication. Besides that, no crucial uncertainty contributors were used to estimate the measurement results. What makes the situation more difficult is the fact that upon evaluating surface roughness, measurement uncertainty depends on many components. In addition, the uncertainty of the surface roughness value and its sensitivity coefficient (depends on the surface profile not on *Ra*, *Rz* or other values of unit of measurement) influence the coating thickness value. A thorough overview of these differences is demonstrated in [34, 83], where the importance of surface profile in various technical engineering

processes and upon measuring surface roughness is determined. As it was not the objective of this thesis to deal with surface roughness in great detail, the uncertainty measurement model does not pay close attention to it either, but the poorest incident has been chosen. If could be possible to create new 3D surface roughness units in the future and uncertainty measurement models depending on the surface profile were found, this component could be reduced upon measuring coating thickness.

In research works [27, 29], step height was measured by a surface roughness measuring instrument. To evaluate the measurement results, in addition to the uncertainty evaluation contributed by the measuring instrument, the uncertainty evaluation caused by the measurer was considered. As a result, the measurement results became more reliable. In the above-mentioned papers, however, other contributions – the ones made by the stylus radius, measurement force, surface angle, were ignored. The current research has attempted at considering all the possible uncertainty contributors essential in estimating the measurement result. The current research aimed at studying measurement uncertainty of a surface contour with a complicated form as well as applying the results of the research [2].

## 2.1 Measurement instruments

The surface contours with a complicated form were measured using the surface texture measuring system Perthometer Concept produced by company MAHR [75].

Perthometer Concept is a modular computer-controlled station for measuring and analyzing roughness, contour and topography. Upon creating the measurement and uncertainty model, the best occasion has been considered, where the tracing arm is the shortest and the precisest contour instrument with drive unit PCV200. The high-precision PCV 200 contour drive unit is a long-distance instrument for the assessment of radii, distances, angles and straightness deviations, so actually this is not the best method for two-dimensional assessment of coating thickness. However, as mentioned earlier, the measurement model can be later simplified to a large extent. The smooth traverse and the computer-assisted error correction guarantee reproducible measurements with utmost vertical and horizontal resolution in a measuring field of 200 mm x 50 mm. PCV 200 contour drive unit allows automatic lowering and lifting of the tracing arm with programmable speed and quick positioning. The measuring force can be adjusted from 2 mN to 120 mN. Measurement system was calibrated by using different steph height-, angle-, profile- and special weight standards.

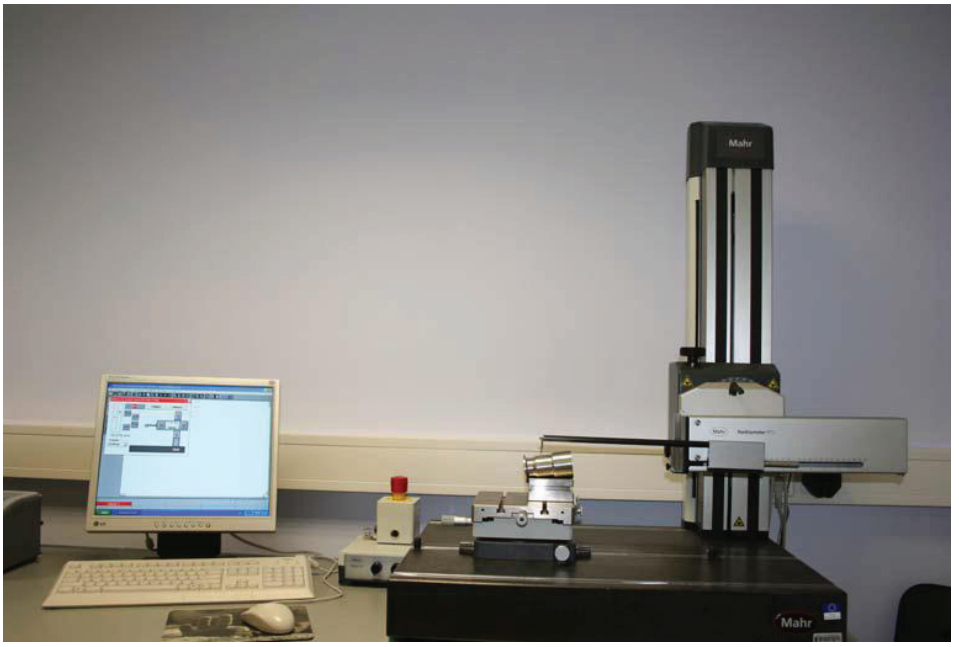


*Figure 2.1 Surface profile measurement system Perthometer Concept*

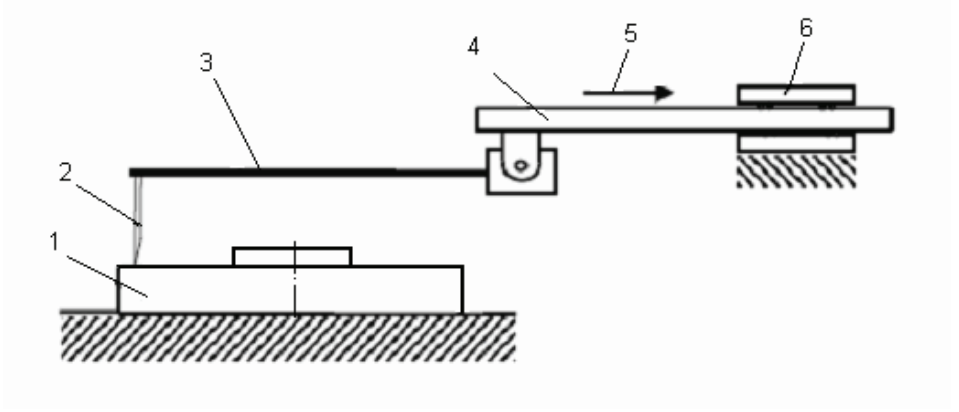


*Figure 2.2 Schematic lay-out of the measurement principle:* 1 – *test object;* 2 – *stylus;* 3 – *tracing arm;* 4 – *drive unit;* 5 – *measuring direction;* 6 – *calibrated support* [3]

## 2.2 Measurement model

According to [3], the measurement model can be expressed as follows:

$$y = x + \sum_{i=1}^{N-1} \delta x_{N-1} \tag{2.1}$$

where $x$ is the measurement value.

$$\sum_{i=1}^{N-1} \delta x_{N-1} = \delta x_{\mathrm{MI}} + \delta x_{\mathrm{r}} + \delta x_{\mathrm{F}} + \sum_{j=1}^{J} \delta x_{\mathrm{obj},j} \tag{2.2}$$

where,

$$\sum_{j=1}^{J} \delta x_{\mathrm{obj},j} = \delta x_{\mathrm{cv}} + \delta x_{\mathrm{cc}} + \delta x_{\mathrm{ang}} + \delta x_{\mathrm{en}} \tag{2.3}$$

Now we can express the measurement model by the following equation:

$$y = x + \delta x_{\mathrm{MI}} + \delta x_{\mathrm{r}} + \delta x_{\mathrm{F}} + \delta x_{\mathrm{cv}} + \delta x_{\mathrm{cc}} + \delta x_{\mathrm{ang}} + \delta x_{\mathrm{en}} \tag{2.4}$$

where, $\delta x_{\mathrm{MI}}$ – correction from the measuring instrument,
$\delta x_{\mathrm{r}}$ – correction from the stylus radius,
$\delta x_{\mathrm{F}}$ – measurement force correction,
$\delta x_{\mathrm{cv}}$ – surface curvature correction,
$\delta x_{\mathrm{cc}}$ – surface concavity correction,
$\delta x_{\mathrm{ang}}$ – correction from the surface angle,
$\delta x_{\mathrm{en}}$ – correction from the measuring environment.

## 2.3 Uncertainty estimation and research results

The standard uncertainty to be associated with an estimate y of an output quantity *Y*, which is evaluated from the estimates of a number of input quantities, is defined as *combined standard uncertainty* [36, 22]. The uncertainty of an input quantity *X* is, in turn, often obtained based on relevant measurement model. This means that during the evaluation process the input quantity itself can be characterized with a combined uncertainty. Similarly, we can use the output from the measurement model as an input for a coming measurement task. The concept of combined standard uncertainty is therefore of only limited use. The symbol $u(y)$ is used for the standard uncertainty to be ascribed to the estimate $y$, regardless of the way in which the uncertainty has been evaluated. The combined standard uncertainty is the positive square root of the combined variance, which is the weighted sum of the experimental variances and covariances of all input quantities considered in the measurement model [19]. The experimental variances and covariances are obtained from the experimental standard deviations $u(\mathrm{x_i})$ associated with the estimates $x_i$ of the input quantities $X_i$. In our case combined standard uncertainty is determined as follows:

$$u(y) = \begin{bmatrix} u^2(x) + u^2(\delta x_{\mathrm{MI}}) + u^2(\delta x_{\mathrm{r}}) + u^2(\delta x_{\mathrm{F}}) + \\ u^2(\delta x_{\mathrm{cv}}) + u^2(\delta x_{\mathrm{cc}}) + u^2\left(\delta x_{\mathrm{ang}}\right) + u^2(\delta x_{\mathrm{en}}) \end{bmatrix}^{1/2} \tag{2.5}$$

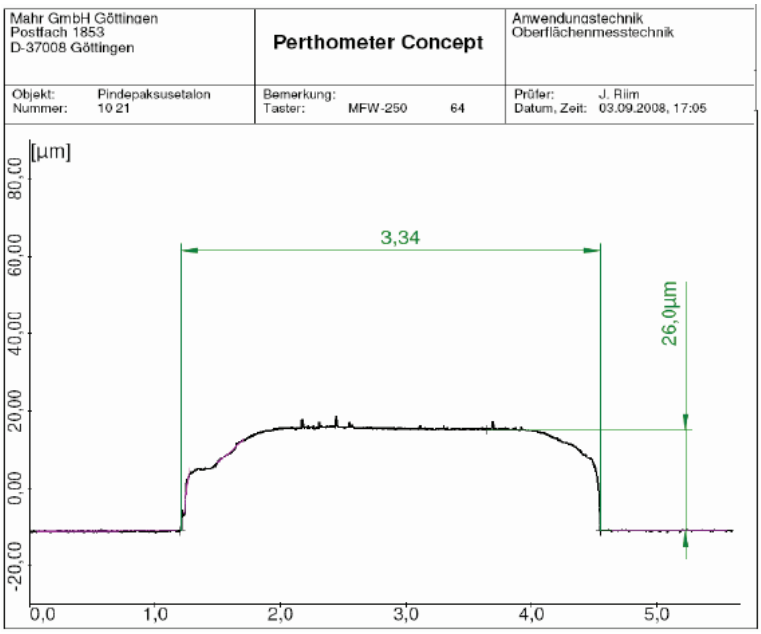


*Figure 2.3 The coating thickness standards contour measured with Perthometer Concept* [2]

The standard uncertainties of input quantities from the different sources were determined. The following results were obtained experimentally and their standard uncertaintie evaluations were calculated applying type B method [19, 22].

Indication in this case is a contour we can see on the screen of the computer (see Figure 2.3). Standard uncertainty of the indication can be determined according to the pointer resolution (digitalisation). The current printer resolution $\Delta x = \pm 0{,}5$ µm gives us the standard uncertainty

$$u(\mathrm{x}) = \frac{\Delta x}{2\sqrt{3}} \approx 0{,}15 \text{ µm}$$

The device's calibration directed at the *Z*-axis, the flatness of the standard's surface as well as the sensor's sensitivity have been considered here. The corrections have been taken into account in the device's software, but it can be seen on the basis of [76] that the uncertainty components exist. The sensor's sensitivity is also added. Uncertainty evaluation, determined from measurement devices on the basis of [75] and [76] is:

$$u(\delta x_{\mathrm{MI}}) = \frac{\Delta_{\mathrm{MI}}}{2\sqrt{3}} \approx 0{,}15 \text{ µm}$$

Research results indicated that stylus radius correction did not remarkably affect the contour measurements. So the following can be assumed:

$$\delta x_{\mathrm{r}} \approx 0 \text{ µm and } u(\delta x_{\mathrm{r}}) \approx 0 \text{ µm}$$

Measuring force correction and its standard uncertainty can be calculated as follows. From the Hertz formula [26] the elastic deformation can be calculated. The worst situation − sphere-sphere − was observed. The correction value is to be considered equal to zero [76] and its standard uncertainty evaluation can be calculated according to the following equation:

$$u(\delta x_{\mathrm{F}}) = \frac{\Delta_{\mathrm{F}}}{2\sqrt{3}} \approx 0{,}03\ \mu\mathrm{m}$$

This component is the most difficult one to find, as it depends to a large extent on the measurer and their experience with such measuring devices. In the event of simple measurements (coating thickness in one direction), we presume that the value is zero $\delta x_{\mathrm{cv}} \approx 0\ \mu\mathrm{m}$ and $\delta x_{\mathrm{cc}} \approx 0\ \mu\mathrm{m}$, but their standard uncertainty has a value and can be found with the equation:

$$u(\delta x_{\mathrm{cv}}) = \frac{\Delta_{\mathrm{cv}}}{2\sqrt{3}} \approx 0{,}5\ \mu\mathrm{m}$$

$$u(\delta x_{\mathrm{cc}}) = \frac{\Delta_{\mathrm{cc}}}{2\sqrt{3}} \approx 0{,}5\ \mu\mathrm{m}$$

where $\Delta_{\mathrm{cv}}$ and $\Delta_{\mathrm{cc}}$ have been found experimentally.

Correction of the surface angle $\delta x_{\mathrm{ang}} = 0\ \mu\mathrm{m}$ and its standard uncertainty evaluation can be calculated from equation:

$$u\left(\delta x_{\mathrm{ang}}\right) = \frac{\Delta_{\mathrm{ang}}}{2\sqrt{3}} \approx 1\ \mu\mathrm{m},$$

where $\Delta_{\mathrm{ang}}$ was experimentally determined during the research applying the angle standards and different tests.

Correction of the environment noise $\delta x_{\mathrm{en}} = 0\ \mu\mathrm{m}$ as well as its uncertainty evaluation $u(\delta x_{\mathrm{en}}) = 0\ \mu\mathrm{m}$. Upon measuring the contour, regression curves and lines are calculated and based on that estimates are found. We may presume that due to this, noise does not have a particular effect on the measurement results. What definitely affects measurement results is the changing of parameters characterising the environment during the measuring process and when the maximum dimensions of details are measured, but regression polynomials are not calculated.

The above-mentioned quantities and their values are presented in the following table.

*Table 2.1 Estimates of the input quantities and their uncertainties*

| Quantity $X_i$ | Estimate $x_i$ | Standard uncertainty $u(x_i)$ /µm | Dispersion $u^2(x_i)$ /µm |
|---|---|---|---|
| $x$ | Contour | 0,15 | 0,023 |
| $\delta x_{\mathrm{MI}}$ | 0 | 0,15 | 0,023 |
| $\delta x_{\mathrm{r}}$ | 0 | 0 | 0 |
| $\delta x_{\mathrm{F}}$ | 0 | 0,03 | 0,0009 |
| $\delta x_{\mathrm{cv}}$ | 0 | 0,5 | 0,25 |
| $\delta x_{\mathrm{cc}}$ | 0 | 0,5 | 0,25 |
| $\delta x_{\mathrm{ang}}$ | 0 | 1 | 1 |
| $\delta x_{\mathrm{en}}$ | 0 | 0 | 0 |
| | | $\sum$ | 1,24 |

Combined standard uncertainty can be calculated as follows:

$$u(y) = \sqrt{\sum_{i=1}^{N} u^2(x_i)} \approx 1{,}24\ \mu\mathrm{m} \tag{2.6}$$

Hence, the expanded uncertainty can be presented as follows:

$$U = k \cdot u(y) = 2 \cdot 1{,}24\ \mu\mathrm{m} = 2{,}5\ \mu\mathrm{m}$$

As it can be seen, the value of extended uncertainty is relatively high compared to the value of the coating thickness (Figure 2.3), but the measuring device used was meant for measuring larger objects. Thus, if we use a special nozzle, for example for surface roughness, the measuring uncertainty is significantly lower.

## 2.4 Conclusions of Chapter 2

Naturally, it must be considered for this general model that by using other measuring devices, some components may be excluded or included. The model assessment also depends on which measurement object is measured and how the measurement is performed. For example, upon two-dimensional measurement of a coating thickness standard, the parameters resulting from the measuring instrument can almost completely be excluded if the device is calibrated with a more precise standard directly before the measurement. However, the standard itself (its surface roughness, flatness *etc*.) may have a more significant role for the coating thickness standard. It all depends on the (*e.g.* mean coating thickness or minimum coating thickness) we wish to find.

# 3 MEASUREMENT OF COATING THICKNESS BY METHOD OF BASE SURFACE ESTIMATION

As described in Chapter 1, considering basic principles of metrology, the surface coating thickness can be defined as the distance along the normal line of surface coating between crossing points of this line with the upper and the inner boundary surfaces of the surface coating. The boundary surfaces are determined as surfaces between coating and surrounding gas or liquid environment, and between coating and base material, correspondingly. The above definition is valid, however, in case of perfectly plane and parallel boundary surfaces. Actually, the boundary surfaces are not parallel with each other, but, depending on production technology, have deviations in geometry as well as in roughness.

The problem is, it is difficult to predict the coating thickness of a coating thickness standard – specific methods are used for the indirect presentation of the parallelism and the full flatness of the surface coating and of the boundary surface.

The method presents the definition for surface coating thickness between real (estimated) coating surfaces as proposed on figure [38]. Therefore the contours of the boundary surfaces of the coating thickness standard before and after the coating and so the top surface of the coating thickness standard are measured.

The definition is proved by mathematical model, based on polynomial regression analysis and estimated profile of the boundary surface beneath the coating, which gives the statistical distribution description for the coating surface and therefore for the coating itself.

With mathematical model the local thickness (in a fixed point), maximal, minimal and the overall thickness can be calculated any time. Similarly, the data collected during the first calibration process can be used on the re-calibration of the same thickness standard. Besides, upon re-calibration there is no need of measuring the boundary surfaces, but only the top surface of the coating thickness standard. During the re-calibration it is possible to estimate the condition of the coating thickness standard – for instance the wear or the defect of the top surface and influence it may have on the measurement result.

With advantage of the above method, evaluation of the profile can be described under the coating. Using the developed method the coating thickness can be determined during the calibration procedure according to the definition of the coating thickness standard. Of course, it increases the reliability of the calibration of coating thickness standards.

The definition proposed for surface coating thickness is checked through practical tests, which allows validate applicability of theoretical considerations elaborated.

The method is suitable for thickness standards that cannot be calibrated during the manufacturing process, or for standards the boundary surfaces of which are not very plain due to cost effect.

## 3.1 Measurement model

There are two random functions which determine the coating thickness of a real plane object of measurement, and which characterize the boundary surface between

the coating and the surrounding environment, as well as the boundary surface between the coating and the base.

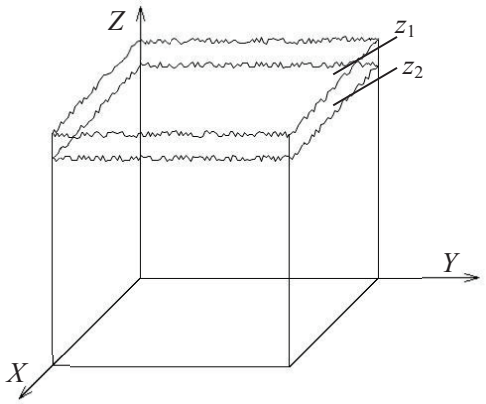


*Figure 3.1 The coated object: $z_1$ – surface between the coating and exterior environment, $z_2$ – surface between the coating and the base* [1]

The values of the above-mentioned random functions are restricted by the conditions proposed in the technical specifications, *i.e.* the tolerance limits of the shape deviations (usually tolerance of a plane surface) and the parameter of surface roughness $R_{max}$. Relating the covered element, measuring $x \times y \times z$ of a plane object of measurement, to the cross coordinate system $0XYZ$ in a way where the surface of the cross coordinate system $0XY$ is parallel to the mean plane surface (derived from random function $Z_s = f_2(X, Y)$, the boundary surface of the covering and base (see Figure 3.1 ). The random function of the covering can, in general, be represented as follows:

$$h_{\mathrm{p}} = Z_1 - Z_2 = f_1(x, y) - f_2(x, y) \tag{3.1}$$

Observing the coating of the element of the object of measurement with dimensions $x \times y \times z$ in the intersection $0YZ$ (presented in Figure 3.2), the mean thickness of coating in the intersection from $y_1$ to $y_2$ can be determined in the following relation:

$$h_{\underset{x=x_0}{\mathrm{mean}}} = \frac{1}{y_2 - y_1} \int_{y_1}^{y_2} [f_1(x_0, y) - f_2(x_0, y)] dy \tag{3.2}$$

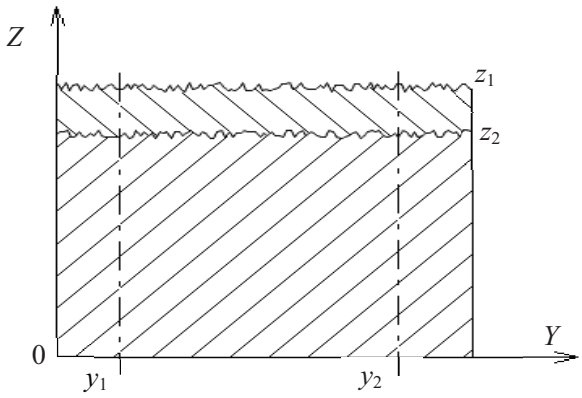


*Figure 3.2 Element of the object: $z_1$ – surface between the coating and exterior environment, $z_2$ – surface between the coating and the base* [1]

In intersection 0*XZ*, in which the shape of the element of the object of measurement is analogous to the one presented in Figure 3.2, the mean coating thickness of the object in the intersection from $x_1$ to $x_2$ can be determined similarly:

$$h_{\underset{y=y_0}{\text{mean}}} = \frac{1}{x_2 - x_1} \int_{x_1}^{x_2} [f_1(x, y_0) - f_2(x, y_0)] dx \tag{3.3}$$

For determining, sustaining and reproducing a certain value of coating thickness, coating thickness standards are applied. The latter are cuboids or bases made from a standard material, and the middle of the topmost surface of which is covered with a standard material, the thickness of which can be measured or calibrated.

Let us relate the coating thickness standard to the cross coordinate system 0*XYZ* so that the plane of the cross coordinate system 0*XY* is parallel to the foundation of the base, and the point of origin of the coordinates is in the middle of the intersectional line between the side and the foundation of the base (see Figure 3.3).

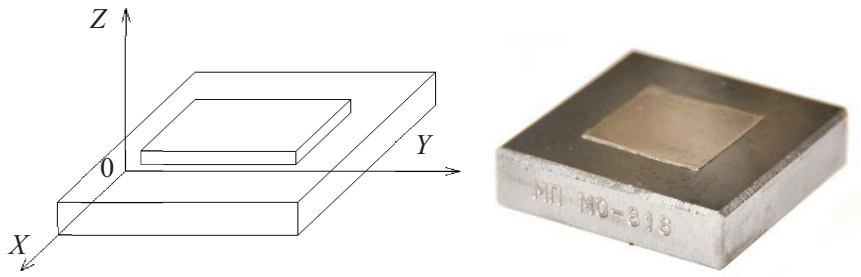


*Figure 3.3 Coating thickness standard*

In this case, the contours of the boundary surfaces of the coating thickness standard in intersection 0*YZ*, which are determined by random functions, take the shape provided in Figure 3.4.

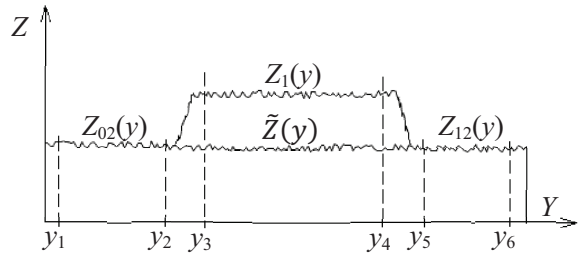


*Figure 3.4 Random functions characterizing the top surface* [96]

When observing this coating thickness standard in an intersection parallel to axis *X*, the obtainable shape is analogous. The problem here lies in the fact that the two random functions characterizing the top surface of the base in the range from $y_1$ to $y_2$ and from $y_5$ to $y_6$ can be determined by groping (see Figure 3.4). However, in the range from $y_3$ to $y_4$ of the random function of the boundary surface of the coating and the base, it proves impossible to determine the covering thickness through groping, since the coating is attached to the base/foundation. Therefore, within the range from $y_3$ to $y_4$, the thickness of the coating has to be determined based on the profiles of the surface of the base, which, in its turn, are determined by two random functions in the range from $y_1$ to $y_2$ and from $y_5$ to $y_6$ [77]. Those random functions, however, characterize the surface profile on both sides of the coating and not directly under it. The problem lies in, firstly, how to evaluate the random function $\tilde{Z}(y)$ of the boundary surface between the coating and the base in the range from $y_3$ to $y_4$, relying on the two random functions $Z_{02}(y)$ and $Z_{12}(y)$, or their estimates, which characterize profiles in the range from $y_1$ to $y_2$ and $y_5$ to $y_6$? Secondly, what to do to determine the coating thickness, which has been obtained by calculating the third-degree polynomial in the intersection $y_2$ to $y_5$? Functions $Z_{02}(y)$ and $Z_{12}(y)$ are random, the values of which can be obtained when measuring the surface of the base of the covering thickness standard by means of groping.

$$Z_{02}(y) = \tilde{Z}_{02} + a_{02}y + a_{00} \tag{3.4}$$

$$Z_{12}(y) = \tilde{Z}_{12} + a_{12}y + a_{10} \tag{3.5}$$

in which $\widetilde{Z}_{02}$ and $\widetilde{Z}_{12}$ have random values according to the normal distribution N(0, $\sigma_{02}$) and N(0, $\sigma_{12}$) [23]. In the current case, the functions of the mean value of functions $Z_{02}(y)$ and $Z_{12}(y)$ are the following:

$$m_{z02}(y) = a_{02}y + a_{00}, \quad y_1 \le y \le y_2 \tag{3.6}$$

$$m_{z12}(y) = a_{12}y + a_{10}, \quad y_5 \le y \le y_6 \tag{3.7}$$

It is clear the profile under the coating can have many different shapes in our case. In Figure 3.5 two surface profiles are on both sides of the coating and between them is the most probably appeared profile between the coating and base.

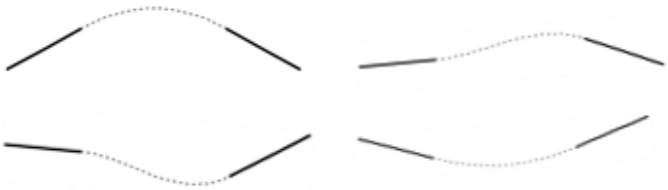

*Figure 3.5 Random functions characterizing the surface of the base (four different cases)* [77]

However, in case of coating thickness standards it can be presumed (and various practical measurements have also indicated) that the surface of the base is either convex or concave. This arises from the production process, in which the surface of the standard's base is burnished or polished. The most probable profile under the coating in the intersection $y_2$ to $y_5$ can be described by many different degrees of polynomials. Moreover, the surface of the thickness standard is very similar to the gauge blocks the surfaces of which are typically convex or concave [16]. And considering the complexity of calculation of uncertainty to the higher degree of polynomials and the relative flatness of the surface of the base, we can assume, that the base can be described with the third degree polynomials:

$$c_3 y^3 + c_2 y^2 + c_1 y + c_0 \tag{3.8}$$

The mathematical function of the polynomial must fill the conditions like positional continuous and tangential continuity.

Upon finding the profile, **positional continuous** is necessary due to the surface's actual continuity and we wish that the mathematical model would describe the situation analogically. Mathematically the continuity condition can be expressed as the random functions which characterize the surface profile on both sides of the coating:

$$c_3 y_2^3 + c_2 y_2^2 + c_1 y_2 + c_0 = a_{02} y_2 + a_{00} \tag{3.9}$$

$$c_3 y_5^3 + c_2 y_5^2 + c_1 y_5 + c_0 = a_{02} y_5 + a_{10} \tag{3.10}$$

where the left side indicates the average value of the random function of the boundary surface in points $y_2$ and $y_5$ and this has been equalised to the average values calculated for measured profiles in the same points.

**Tangential continuity** is at least as important as positional continuous. Tangential continuity is a mathematical representation of the condition that the whole base surface is level and has no grades and angles. In points $y_2$ and $y_5$, the first order derivatives of the functions describing the boundary surface as well as measured profiles must be equal, *i.e.* in points $y_2$ and $y_5$ end vectors of the surface profiles on both sides of the coating and the polynomial must be parallel (spline must be smooth):

$$3c_3 y_2^2 + 2c_2 y_2 + c_1 = a_{02} \tag{3.11}$$

$$3c_3 y_5^2 + 2c_2 y_5 + c_1 = a_{12} \tag{3.12}$$

Based on the functions of mean value represented in formula (3.8) and the necessary conditions in formulas (3.9, 3.10) and (3.11, 3.12) the function of the mean value of the profile of the boundary surface between the coating and base (in the range from $y_2$ to $y_5$) can be expressed as follows:

$$m_{\tilde{Z}}(y) = c_3 y^3 + c_2 y^2 + c_1 y + c_0 \tag{3.13}$$

According to [96], the constants can be calculated as follows:

$$\begin{aligned}
c_3 &= \frac{a_{02} - a_{12}}{(y_2 - y_5)^2} + 2\frac{Z_{II} - Z_I}{(y_2 - y_5)^3} \\
c_2 &= -\frac{a_{02}(y_2 + 2y_5) + a_{12}(2y_2 + y_5)}{(y_2 - y_5)^2} + 3\frac{(Z_I - Z_{II})(y_2 + y_5)}{(y_2 - y_5)^3} \\
c_1 &= \frac{a_{02}\left(2y_2 y_5 + y_5^2\right) + a_{12}(2y_2 y_5 + y_2^2)}{(y_2 - y_5)^2} + 6\frac{y_2 y_5 (Z_{II} - Z_I)}{(y_2 - y_5)^3} \\
c_0 &= -\frac{a_{02} y_2 y_5^2 + a_{12} y_2^2 y_5}{(y_2 - y_5)^2} + \frac{Z_I y_5^2 (3y_2 - y_5) + Z_{II} y_2^2 (y_2 - 3y_5)}{(y_2 - y_5)^3}
\end{aligned} \tag{3.14}$$

where $Z_I = a_{02} y_2 + a_{00}$ and $Z_{II} = a_{12} y_5 + a_{10}$.

It was pointed out earlier that the coating thickness is the shortest distance between two boundary surfaces.

Analogically, it is possible to find the coating thickness (in every $y$ point) in the given case (see Figure 3.4) with the following equation:

$$h_{\mathrm{p}}(y) = Z_1(y) - \tilde{Z}(y) \tag{3.15}$$

Analogically to functions $Z_{02}$ and $Z_{12}$, $Z_1$ is the random function of the top surface of the coating of the coating thickness standard in the range of $y_3$ to $y_4$ and can be presented by the following relation:

$$Z_1(y) = \tilde{Z}_1 + b_3 y^3 + b_2 y^2 + b_1 y + b_0 \tag{3.16}$$

in which $\widetilde{Z}_1$ has random value according to the normal distribution N(0, $\sigma_1$). In the current case, the function of the mean value of function $Z_1(y)$ is the following:

$$m_{z1}(y) = b_3 y^3 + b_2 y^2 + b_1 y + b_0 \quad y_3 \le y \le y_4 \tag{3.17}$$

The mean value of the coating thickness can be represented on the basis of the equations above as follows:

$$\begin{aligned}
m_h(y) &= m_{Z1}(y) - m_{\tilde{Z}}(y) = \\
&= b_3 y^3 + b_2 y^2 + b_1 y + b_0 - \\
&- c_3 y^3 - c_2 y^2 - c_1 y - c_0
\end{aligned} \tag{3.18}$$

## 3.2 Uncertainty estimation

The standard uncertainties of input quantities from the different sources were determined. The following results were obtained experimentally and their standard

uncertainty evaluations were calculated applying type B method [19, 22]. In our case we note that the total combined uncertainty $u_{\Sigma}(y)$ of $h(y_i)$ can be expressed as [76]

$$u_{\sum}(y) = \sqrt{D_h(y) + u^2(z) + \left(\frac{\partial m_{Z1}}{\partial y}\right)^2 u^2(y)} \tag{3.19}$$

The combined standard uncertainty of the estimated $z_i$ can expressed as follows:

$$u^2(z) = u_{\text{cal}}^2 + u_{\text{en}}^2 + u_{\text{ob}}^2 \tag{3.20}$$

where $u_{\text{cal}}$ is standard uncertainty of the calibration of measurement instrument, $u_{\text{en}}$ standard uncertainty of the environment (noise) and $u_{\text{ob}}$ is standard uncertainty of the surface of the base and of the surface of the coating.

The values of the given three components must be found as well. The estimate $u_{\text{cal}}$ can be found from the device's technical documentation or by calibrating the device - the component should include the z-axis resolution, linearity as well as other deviations.

The measuring environment's uncertainty component $u_{\text{en}}$ must be calculated experimentally and it depends on the room where the measuring is performed. It is logical to find it according to maximum vibration influencing the device and presume that the vibration occurs in a rectangular distribution.

It is undoubtedly most difficult to find the uncertainty component $u_{\text{ob}}$ arising from the object. It includes all kinds of geometrical deviations of the object of measurement which are not reflected in the dispersions $D_{Z1}(y)$ and $D_{\tilde{Z}}(y)$ and must therefore be considered separately. The geometrical deviations of high-quality coating thickness standards have a relatively small value as well as surface roughness. However, experiments showed that the polynomial dispersion (equation (3.26) does not consider the uncertainty caused by surface roughness for standards with relatively rough surfaces, so it must be separately inserted in the combined uncertainty equation. As upon later use of the coating thickness standard the sensor of the calibrated device might suffer from an arbitrary defect on the standard's surface, this must be taken into account also in the measurement uncertainty. Standard measurand *Ra* – the average surface roughness – suits according to experiments best for this purpose. Therefore it is recommended that $u_{\text{ob}} = Ra$.

The last component is an uncertainty directed at the y-axis. As the measuring was performed only once in one section, the uncertainty directed at the y-axis is directly in the combined uncertainty equation. The uncertainty $u^2(y)$ of the estimated $y_i$ is uncertainty caused by finite resolution.

At present it is possible to use very powerful calculation programs (even in Excel, for example). Therefore, the mean values given in equations (3.6, 3.7) and (3.17) can be found, figuratively speaking, with just one mouse click. As all random functions $Z_{02}(y)$, $Z_{12}(y)$ and $Z_1(y)$ have been found from the finite measuring points, their dispersions also have a calculable value in the calculations of measurement uncertainty. In order to find the dispersion of random functions' mean values $m_{z02}(y)$, $m_{z12}(y)$ and $m_{z1}(y)$ it is necessary to solve the regression analysis of polynomials. In the case of mean values $m_{z02}(y)$, $m_{z12}(y)$ the task is easier, as

these are lines, but $m_{z1}(y)$ requires the calculation of dispersion for a third order polynomial. According to [35] the resulting equations for a regression equation that is a polynomial of *m*-th order in $x_i$ is the following:

$$y_i = a_0 + a_1 x_i + \cdots + a_{m-1} x_i^{m-1} + a_m x_i^m \tag{3.21}$$

The condition for minimising the sum of the squares $R^2$ of the deviations between the observed results $\bar{y}_{ij}$ and the corresponding values obtained from equation (3.21) is

$$R^2 = \sum_{j=1}^{J} \begin{pmatrix} \bar{y}_{ij} - a_0 - a_1 x_1 - \cdots \\ -a_{m-1} x_i^{m-1} + a_m x_i^m \end{pmatrix} = \min \tag{3.22}$$

For $R^2$ to be minimum, the conditions $\partial R^2/\partial a_0 = 0$, $\partial R^2/\partial a_1 = 0$, ..., $\partial R^2/\partial a_m = 0$ have to be fulfilled. This yields the set of normal equations:

$$\begin{aligned} &a_0 J + a_1 \sum x_i + \cdots + a_m \sum x_i^m = \sum \bar{y}_i \\ &a_0 \sum x_i + a_1 \sum x_i^2 + \cdots + a_m \sum x_i^{m+1} = \sum x_i \bar{y}_i \\ &\ldots, \\ &a_0 \sum x_i^m + a_1 \sum x_i^{m+1} + \cdots + a_m \sum x_i^{m+m} = \sum x_i^m \bar{y}_i \end{aligned} \tag{3.23}$$

where the sum sign $\sum$ here and in the remainder of this subsection denotes summation of the *J* (*j*=1, 2 – *J*) values $\bar{y}_{ij}$ and $x_{ij}$ obtained in the experiment, or of their products, as displayed by the symbols that follow the summation sign; for example, $\sum x_i = \sum_{J=1}^{J} (x_{ij} \cdot \bar{y}_{ij})$. From the number of parameters to be estimated from the experimental data, we require $J > m(m+2) + 2$.

By solving the system of normal equations (3.23), we obtain the parameters of equations (3.21):

$$a_0 = \frac{\Delta_0}{\Delta}, \quad a_1 = \frac{\Delta_1}{\Delta}, \quad \ldots, \quad a_m = \frac{\Delta_m}{\Delta}, \tag{3.24}$$

where

$$\Delta_0 = \begin{bmatrix} \sum \bar{y}_i & \sum x_i & \cdots & \sum x_i^m \\ \sum x_i \bar{y}_i & \sum x_i^2 & & \sum x_i^{m+1} \\ \vdots & & \ddots & \vdots \\ \sum x_i^m \bar{y}_i & \sum x_i^{m+1} & \cdots & \sum x_i^{m+1} \end{bmatrix}, \quad \Delta_1 = \begin{bmatrix} J & \sum \bar{y}_i & \cdots & \sum x_i^m \\ \sum x_i & \sum x_i \bar{y}_i & & \sum x_i^{m+1} \\ \vdots & & \ddots & \vdots \\ \sum x_i^m & \sum x_i^m \bar{y}_i & \cdots & \sum x_i^{m+1} \end{bmatrix},$$

$$\ldots, \qquad \ldots,$$

$$\Delta_m = \begin{bmatrix} J & \sum x_i & \cdots & \sum \bar{y}_i \\ \sum x_i & \sum x_i^2 & & \sum x_i \bar{y}_i \\ \vdots & & \ddots & \vdots \\ \sum x_i^m & \sum x_i^{m+1} & \cdots & \sum x_i^m \bar{y}_i \end{bmatrix}, \quad \Delta = \begin{bmatrix} J & \sum x_i & \cdots & \sum x_i^m \\ \sum x_i & \sum x_i^2 & & \sum x_i^{m+1} \\ \vdots & & \ddots & \vdots \\ \sum x_i^m & \sum x_i^{m+1} & \cdots & \sum x_i^{m+m} \end{bmatrix}.$$

The variances (the independent components of the combined uncertainty) of parameters $a_0$, $a_1$, ..., $a_m$ are obtained from relationships

$$s^2(a_0) = \frac{\Delta_{00}}{\Delta} \cdot s^2, \quad s^2(a_1) = \frac{\Delta_{11}}{\Delta} \cdot s^2, \quad s^2(a_m) = \frac{\Delta_{mm}}{\Delta} \cdot s^2, \tag{3.25}$$

where

$$\Delta_{00}=\begin{bmatrix} \sum x_i^2 & \sum x_i^3 & \cdots & \sum x_i^{m+1} \\ \sum x_i^3 & \sum x_i^4 & \cdots & \sum x_i^{m+1} \\ \vdots & & \ddots & \vdots \\ \sum x_i^{m+1} & \sum x_i^{m+2} & \cdots & \sum x_i^{m+m} \end{bmatrix}, \quad \Delta_{11}=\begin{bmatrix} J & \sum x_i^2 & \cdots & \sum x_i^m \\ \sum x_i^2 & \sum x_i^4 & \cdots & \sum x_i^{m+2} \\ \vdots & & \ddots & \vdots \\ \sum x_i^m & \sum x_i^{m+2} & \cdots & \sum x_i^{m+m} \end{bmatrix},$$

$$\ldots, \qquad \ldots,$$

$$\Delta_{mm}=\begin{bmatrix} J & \sum x_i & \cdots & \sum x_i^{m-1} \\ \sum x_i & \sum x_i^2 & \cdots & \sum x_i^m \\ \vdots & & \ddots & \vdots \\ \sum x_i^{m-1} & \sum x_i^m & \cdots & \sum x_i^{m+m-1} \end{bmatrix}, \quad s^2=\frac{\sum(a_{0+}a_1x_i+\cdots+a_mx_i^m-\bar{y}_i)^2}{J-(m+1)}.$$

The covariances (the dependent components of the combined uncertainty) of the parameters $a_0$, $a_1$, ..., $a_m$ are obtained from relationships

$$s(a_0,a_1)=\frac{\Delta_{01}}{\Delta}\cdot s^2,\ \ldots,\ s(a_0,a_m)=\frac{\Delta_{0m}}{\Delta}\cdot s^2,\ \ldots,\ s(a_{m-1},a_m)=\frac{\Delta_{m-1,m}}{\Delta}\cdot s^2,$$

where

$$\Delta_{01}=\alpha\begin{bmatrix} \sum x_i & \sum x_i^3 & \cdots & \sum x_i^{m+1} \\ \sum x_i^2 & \sum x_i^4 & \cdots & \sum x_i^{m+2} \\ \vdots & & \ddots & \vdots \\ \sum x_i^m & \sum x_i^{m+2} & \cdots & \sum x_i^{m+m} \end{bmatrix},$$

$$\Delta_{0m}=\alpha\begin{bmatrix} \sum x_i & \sum x_i^2 & \cdots & \sum x_i^m \\ \sum x_i^2 & \sum x_i^3 & \cdots & \sum x_i^{m+1} \\ \vdots & & \ddots & \vdots \\ \sum x_i^m & \sum x_i^{m+1} & \cdots & \sum x_i^{m+m-1} \end{bmatrix}$$

...,

$$\Delta_{m-1,m}=\begin{bmatrix} J & \sum x_i & \cdots & \sum x_i^{m-1} \\ \sum x_i^{m-2} & \sum x_i^{m-1} & \cdots & \sum x_i^{m+m-3} \\ \vdots & & \ddots & \vdots \\ \sum x_i^m & \sum x_i^{m+1} & \cdots & \sum x_i^{m+m-1} \end{bmatrix}.$$

The constant α in front of the determinant in equation (3.25) is $\alpha=+1$ if the sum of the indices of the symbol Δ is even; otherwise, $\alpha=-1$. The experimental variance $s^2(y_i)$ associated with the variable $y_i$ defined by equation (*x*), is

$$s^2(y_i) = u^2(y_i) =$$

$$= \frac{s^2}{\Delta}\left\{\begin{array}{c}\left(\frac{\partial y_i}{\partial a_0}\right)^2 \Delta_{00} + \left(\frac{\partial y_i}{\partial a_1}\right)^2 \Delta_{11} + \\ \ldots + \left(\frac{\partial y_i}{\partial a_m}\right)^2 \Delta_{mm} + \\ +2\left(\begin{array}{c}\frac{\partial y_i}{\partial a_0}\frac{\partial y_i}{\partial a_1}\Delta_{01} + \cdots + \frac{\partial y_i}{\partial a_0}\frac{\partial y_i}{\partial a_m}\Delta_{0m} \\ + \cdots + \frac{\partial y_i}{\partial a_{m-1}}\frac{\partial y_i}{\partial a_m}\Delta_{m-1,m}\end{array}\right)\end{array}\right\} \quad (3.26)$$

In addition to the dispersion of functions' mean values $m_{z02}(y)$, $m_{z12}(y)$ and $m_{z1}(y)$, it is necessary to find the dispersion of mean value $m_{\tilde{z}}(y)$ of random function $\tilde{Z}(y)$ describing the estimated base surface.

Since the function describing the base layer has been derived directly from functions $m_{z02}(y)$, $m_{z12}(y)$ and their mean values, the task of calculating dispersion analytically is a very complex task. But the result is very presumable. The dispersions of mean values of random surfaces in front of and behind the coating can be calculated on the basis of equation (3.26). Since the mean values were shown as straight lines, the dispersion of the mean value is by one step/grade higher. In the case given, it is a polynomial of the second grade/power. The mean value of the random function and the dispersion of the axis are shown on Figure 3.6. This means that the straight line can be in any position between the two curves. The situation is similar both to the left and to the right of the coating. This means that the estimated profile under the coating may begin and end at those points. It is a polynomial of the third power, which is an extension to the polynomial of the second power. Its value, however, becomes apparent only after measurement

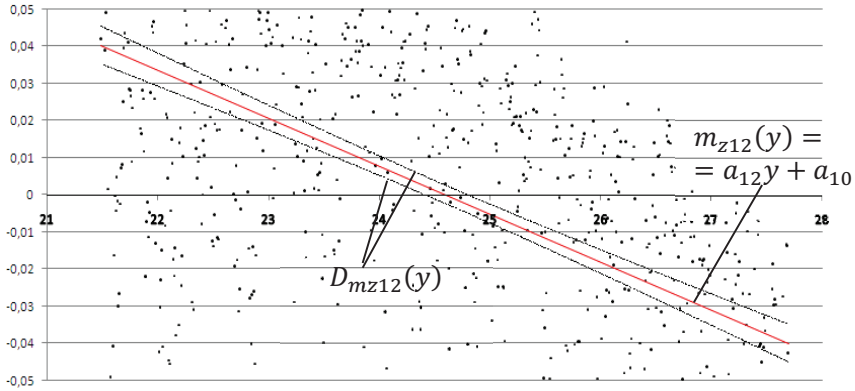


*Figure 3.6 Measured data, mean vales of random function with dispersion* [77]

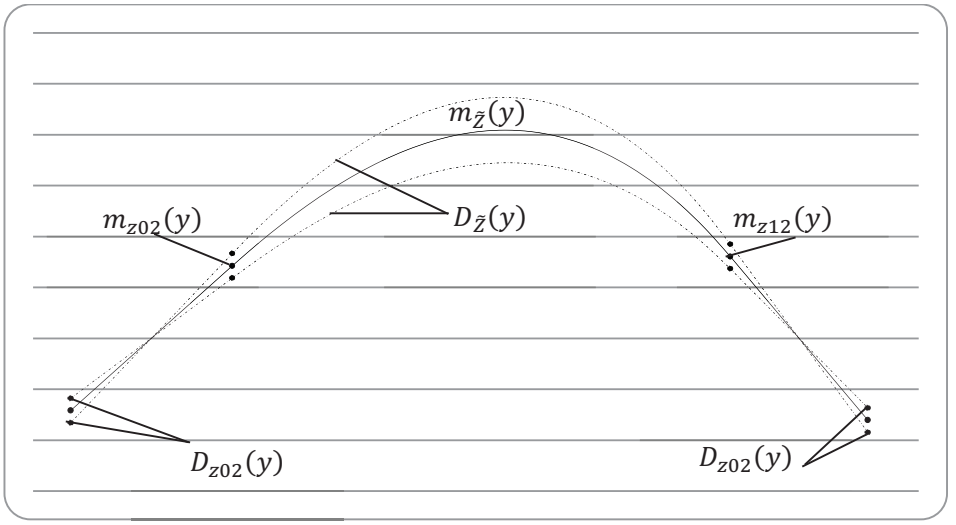


*Figure 3.7 Dispersions and mean values of random functions of surface profiles* [77]

Along with the finding of dispersions of the mean values of all random functions have been found, the dispersion of the function characterising the coating thickness has to be found too. As all profiles have been measured (groped) using the same measuring instrument, all measured values are definitely dependent. However, the uncertainty component conditioned from that (the mutual correlation of functions) is so minimal in comparison to other uncertainty values that the latter can be disregarded, *i.e.* the functions can be deemed as independent. Therefore if we assume, that functions $Z_{02}(y)$ and $Z_1(y)$, $Z_{02}(y)$ and $\tilde{Z}$ are independent, the distribution of coating thickness (dispersion), obtainable through relation (3.15), can be estimated relying on the following dispersion:

$$D_{\mathrm{h}}(y) = D_{\mathrm{Z1}}(y) + D_{\tilde{Z}}(y) \tag{3.27}$$

It is rather common that the correction factors and related uncertaintes may not be final in case of evalutation of the measurement models and their uncertainty and thus, if necessary, respective components may be added to the equation (3.19).

## 3.3 Experimental research results

The coating thickness standards are calibrated to provide unbroken traceability chain inb coating thickness measurements. In our case we observe how we can use the definition of coating thickness in a procedure of calibration of coating thickness standards. To calibrate the coating thickness standard (Figure 3.3) it will be placed on the working table of measuring device "Tencor P-11" [84]. The stylus of the measuring instrument will be put to the contact with the base surface of the coating thickness standard. The $y$-directional movement will be performed and the stylus will trace the surface under measurement. The computer screen of measuring device gives us the surface profile of the traced length (see Figure 3.9). Measurement data ($y_i$ and $z_i$) will be saved on the file which will be used for furthered calculations.

A special computer program was created in DELPHI programming environment for processing measurement data.

### 3.3.1 Developed measurement and calculation program

The objective of the program was to process measurement data and issue calculation results. For this purpose, the software had to be able to:

- calculate the mean values $m_{z02}(y)$ and $m_{z12}(y)$ of the functions characterising the base surface and their dispersions;
- calculate the mean value $m_{\tilde{Z}}(y)$ of the estimated base surface and its dispersion;
- calculate the mean value $m_{z1}(y)$ of the random function characterising the top surface of coating;
- remove local defects (dust, scratches *etc*.) from the measurement data;
- calculate the mean covering thickness $m_{hp}(y)$ in the specified area;
- use a flexible graphic interface.

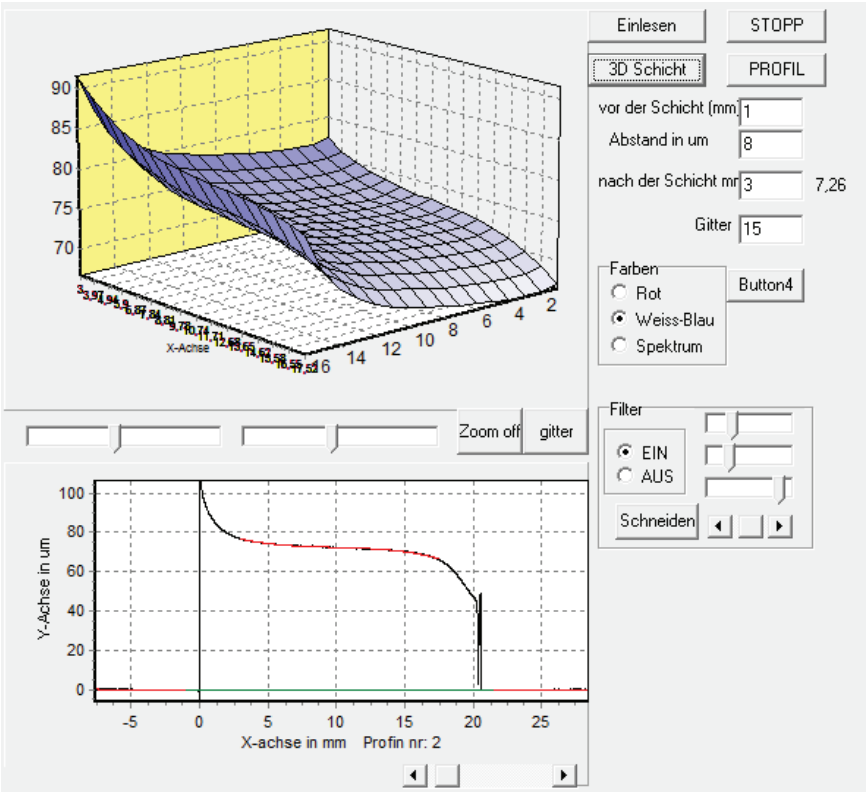


*Figure 3.8 Measurement and calculation program in DELPHI*

Proceeding from the established task, the program's order of tasks was the following. After inserting measurement data, the data is analyzed and new adjusted measurement values are saved. It is necessary for levelling the base surface and making it equal to the 0*XY* surface. Figure 3.9 depicts two illustrative images: initial measurement data on the left and adjusted data on the right. On the right it can be seen how the profile has been turned counter-clockwise so that $m_{z02}(y)$ and $m_{z12}(y)$ would be at the same level with the 0*XY* surface. The profile has also been

shifted so that the coating would start from point $y = 0$ mm and the measurable parts of the base surface would be on the average at the height level $z = 0$ µm.

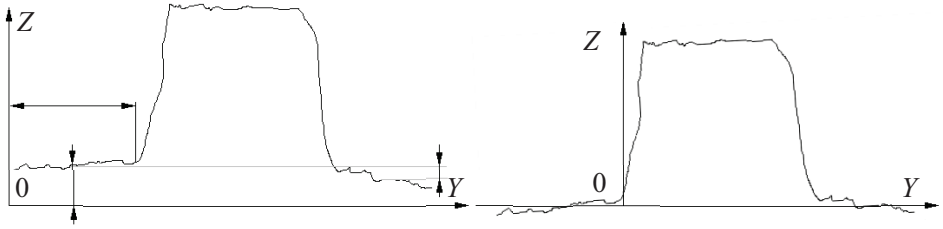


*Figure 3.9 Initial measurement data (on the left) and adjusted measurement data (on the right)*

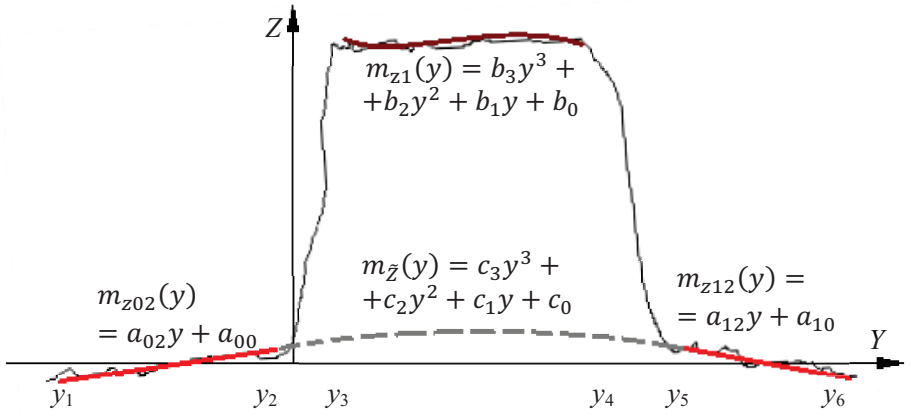


*Figure 3.10 Mean values of random functions*

The programme calculates from the measurement data both mean values $m_{z02}(y)$ and $m_{z12}(y)$ of the random function of the base surface and the mean values $m_{\tilde{z}}(y)$ and $m_{z1}(y)$of the random functions of the estimated profile and surface profile (see Figure 3.10). As specified in the established task, the program was supposed to filter all kinds of surface defects (scratches, unevenness) so that they would not affect the measurement results. A so-called filter was programmed adjustable by the user. All values above or below a certain level are cut off and given new values. Figure 3.11 depicts the print screen of the respective program component which shows the unadjusted as well as adjusted profile.

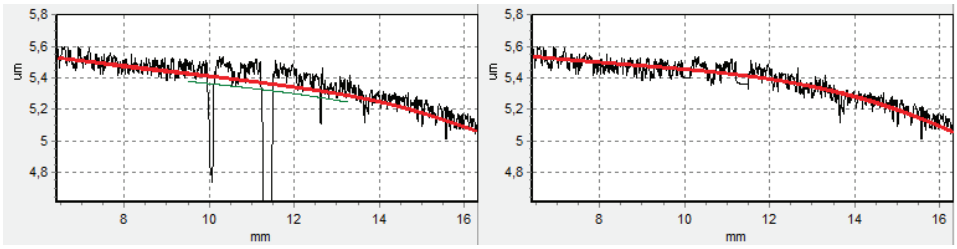


*Figure 3.11 Unfiltered profile (on the left) and filtered profile (on the right)*

After making these procedures and determining in which area (*e.g.* 10 mm x 10 mm) one wishes to examine the coating thickness, the program issues a surface characterising the average coating thickness and the mean value of the whole surface (see Figure 3.12).

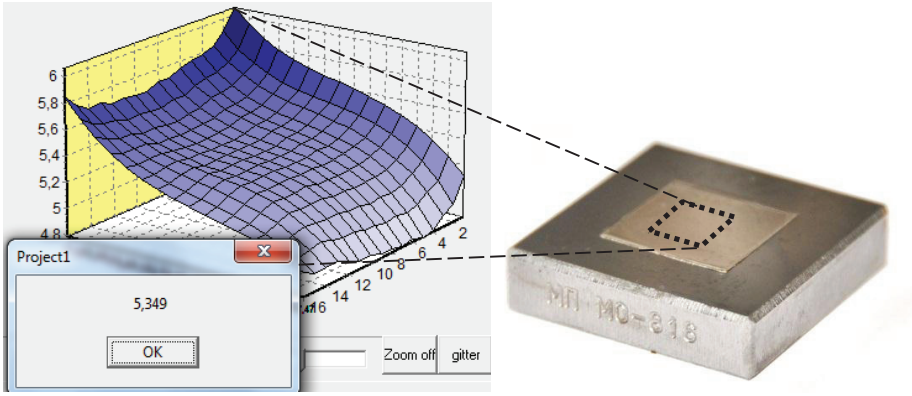


*Figure 3.12 Graphic depiction of the coating thickness of the coating thickness standard*

### 3.3.2 Analysis of measurement results

From January to April 2009, the measurement of several coating thickness standards took were conducted as PTB, Germany for the purpose of their calibration. The provided example is the analysis of the measurement and measurement results of the coating thickness standard of TUT (Nickel-Steel –No 1055).

It is quite similarly represented in Figure 3.10. According to the true surface profile we get implementations of random functions $Z_{02}(y)$, $Z_{12}(y)$ and $Z_1(y)$ in range chosen in $y$ axis ($y_1$ to $y_2$, $y_5$ to $y_6$ and $y_3$ to $y_4$).

On the basis of these implementations regarding $y$ values we can get possible estimates of the random functions $Z_{02}(y)$ and $Z_{12}(y)$ according to equations 3.4 and 3.5 using a polynomial regression analysis. It means the estimates of mean values of these functions $z_{02}(y)$ and $z_{12}(y)$ (equation 3.6 and 3.7) and experimental variances $s^2_{z02}$ and $s^2_{z12}$ (equation 3.13)

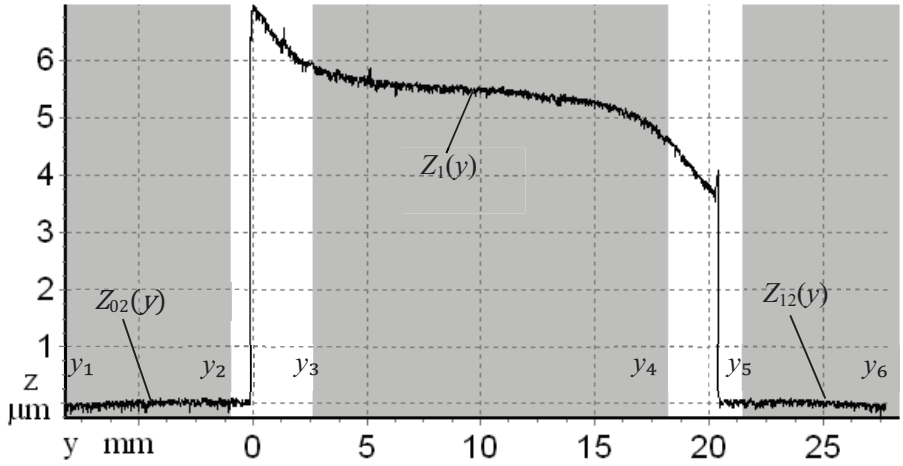


*Figure 3.13 Profile of the base (of coating thickness standard No* 1055*) and functions* $Z_{02}$, $Z_{12}$ *and* $Z_1$ [77]

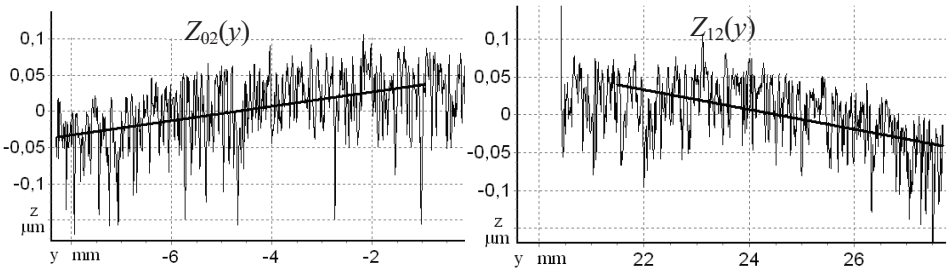


*Figure 3.14 True profile of the base surface and* $m_{z02}(y)$ *(left )* $m_{z12}(y)$ *(right) - mean value of function* $Z_{02}$(y) *and* $Z_{12}$(y) [77]

According to the $z$ values of receiving profile in intersection of tracing length the estimates of coating thickness standard (no 1055) are as follows ($y$ − mm):

$$z_{02}(y) = 9{,}737 \cdot 10^{-3} y + 0{,}045 \text{ µm}; \quad s_{z02} = u_{z02} = 0{,}005 \text{ µm}$$

$$z_{12}(y) = -1{,}281 \cdot 10^{-2} y + 0{,}315 \text{ µm}; \; s_{z12} = u_{z12} = 0{,}005 \text{ µm}$$

Analogically we can calculate in the range from $y_3$ to $y_4$ the equation of parabola proper to relation 3.16 and its variance and standard uncertainty, using polynomial regression analysis ($y$ − mm) (equation 3.26):

$$z_1(y) = -7{,}893 \cdot 10^{-4} y^3 + 2{,}201 \cdot 10^{-2} y^2 - 0{,}2241 y + 6{,}301 \text{ µm}$$

$$u_{z1} \cong \sqrt{D_{z1}} \cong 0{,}01 \text{ µm}$$

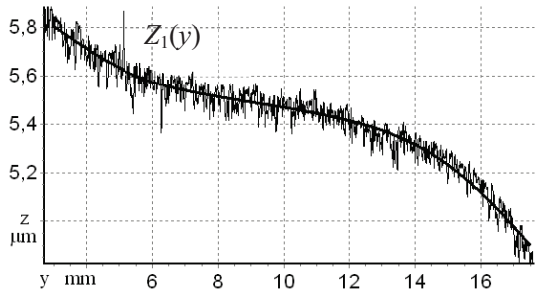


*Figure 3.15 Measured profile of the coating surface and* $m_{z1}(y)$ *- mean value of function* $Z_1(y)$ [77]

We can calculate using equations3.13 and 3.14 the mean value function of the profile of the boundary surface between the coating and base and its estimate of standard uncertainty, which are according to the calibration results as follows ( $y$ − mm) (see Figure 3.16):

$$m_{\tilde{Z}}(y) = -6{,}846 \cdot 10^{-6} y^3 - 2{,}915 \cdot 10^{-4} y^2 + \\ +9{,}174 \cdot 10^{-3} y + 0{,}045\ \mu m \qquad (3.28)$$
$$u_{m\tilde{Z}} \cong \sqrt{D_{\tilde{Z}}} \cong 0{,}016\ \mu\text{m}$$

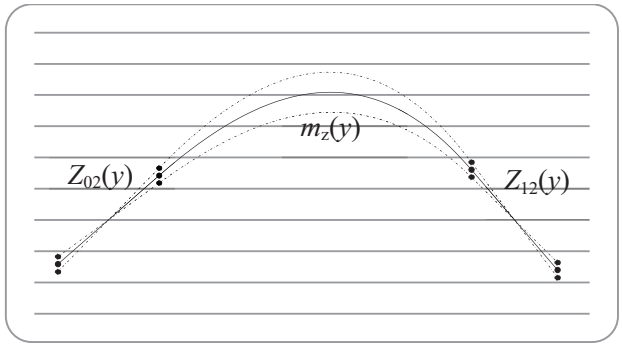


*Figure 3.16 Mean value of the function of estimated profile with the dispersion* [77]

The coating thickness measurement result of coating thickness standard (No 1055) obtained by calibration on the basis of the tracing profile of the surface on the section from $y_3$ to $y_4$ is ( $y$ − mm):

$$h(y) = z_1(y) - m_{\tilde{Z}}(y) = -7{,}825 \cdot 10^{-4} y^3 + \\ +2{,}231 \cdot 10^{-2} y^2 - 2{,}332 \cdot 10^{-1} y + 6{,}256\ \mu\text{m} \qquad (3.29)$$

The above-mentioned quantities and their values have been presented in the following table.

*Table 3.1 Estimates of standard uncertaintyes of input quantities*

| Uncertainty component | Value in µm |
|---|---|
| $u_{z1}$ | 0,01 |
| $u_{m\tilde{z}}$ | 0,016 |
| $u_{\text{cal}}$ | 0,07 |
| $u_{\text{en}}$ | 0,005 |
| $u_{\text{ob}}$ | 0,05 |
| $\left(\frac{\partial \text{m}_{\text{Z1}}}{\partial \text{y}}\right)u_{\text{y}}$ | 0,0178·1 |

The combined standard uncertainty of the coating thickness measurement result of coating thickness using relation 3.19 is as follows. Like described earlier we assume, that estimates of the random functions $z_{02}(y)$ $z_{12}(y)$ and $z_1(y)$ are independent ( $y$ – mm; $U[h(y)]$ – µm).

$$U[h(y)] \cong 2\sqrt{D_h(y) + u^2(z) + \left(\frac{\partial m_{Z1}}{\partial y}\right)^2 u^2(y)} =$$

$$= 2\sqrt{\begin{matrix}(0{,}01\ \mu\text{m})^2 + (0{,}016\ \mu\text{m})^2 + (0{,}07\ \mu\text{m})^2 + \\ +(0{,}005\ \mu\text{m})^2 + 0{,}05\ \mu\text{m}^2 + (0{,}0178)^2(1\ \mu\text{m})^2\end{matrix}} =$$

$$= 0{,}18\ \mu\text{m} \approx 0{,}2\ \mu\text{m} \qquad (3.30)$$

Based on the coating thickness of surface of the standard and its expanded measurement uncertainty by the coating thickness measured for an example at the point $y$ = 10 mm, we can express the final result (of the measurement) as follows:

$$h(10) = 5{,}4\ \mu\text{m} \pm 0{,}2\ \mu\text{m}$$

## 3.4 **Comparison measurement**

TUT owns the set of coating thickness standards „Nickel-Steel 20“. These coating thickness standards were created in 1988 and the manufacturer and first calibration laboratory was NPO “Isari” in Tbilisi. Those coating thickness standards have been used in TUT as working standards for calibration of the coating thickness gauges. The above mentioned standards have been inspected from time to time and changes in quality have been analyzed.

The standards (the cuboids 40 mm x 40 mm) were made of steel, where, in the middle of the surface, there is a galvanized or dispersed coating (20 mm x 20 mm). The coating thickness of these standards was from 1,5 µm up to 100 µm. These coating thickness standards were standardized (GOST 25177-82) and the important criterion was, that the non-flatness and parameter of the surface roughness *Rz* was not allowed to exceed 0,05 µm to 0,1 µm. By manufacturing the coating thickness was measured using the contact, pneumatic or interference method so that the expanded uncertainty of measuring results would not exceed (0,1 + 0,05·$h$) µm, where h is coating thickness in micrometers measured in the standard.

The abovementioned coating thickness gauges are in use in TUT and also in Metrosert Ltd. as working standards for calibration of the coating thickness gauges. As the represented and reproduced coating thickness value of the working standards can change, the standards need to be recalibrated after the certain time interval.

The mentioned coating thickness standards were calibrated (7 standards of set) in 1997 in TUT chair of metrology and measurement technique using the magnetic measuring principle. Coating thickness gauge MIKROTEST III/IV NiFe50 made in Germany by company "ElektroPhysik" was used.

With reference to the research of the TUT and PTB the set of coating thickness standards "Nickel-Steel 20" was sent to the PTB, where these standards were calibrated in 1999. In PTB four coating thickness standards were calibrated using the x-ray measuring station "X-RAY 1600" made by company "Fischer". The whole set was calibrated at PTB using the surface roughness measuring device "Perthometer S8P" made by company "Perthen". The average coating thickness $h_{mean}$ measured in NPO "Isari" and in TUT in the surface area of 10 mm x 10 mm and in the PTB in area of 6 mm x 6 mm. In 2005, the standards were calibrated again in TUT, using the MAHR GmbH measuring device Perthometer Concept with drive unit PGK120 and LS10 Laser pick-up. This type of laser pick-up is particularly suited for non-contact surface texture measurements of test pieces with plane or curved surfaces made from sensitive materials (*e.g.* surfaces of fluids, glass, rubber, plastics and soft metals) or elastic materials such as thin foils. LS10 laser pick-up is a skidless pick-up to be used in stylus instruments featuring a datum plane. It has a measuring range of ± 250 µm. Due to its larger measuring distance of 10 mm the LS10 pick-up is set up easily. It is also particularly suited for measuring recessed surfaces. The LS10 laser pick-up works on the principle of dynamic focusing. The infrared light of a laser diode is brought into a parallel beam and then guided to the objective. The objective focuses the beam in such a way that 10 mm respectively, below the outlet, it forms a measuring spot (focus) with a diameter of approx. 2 µm on the test piece surface [2].

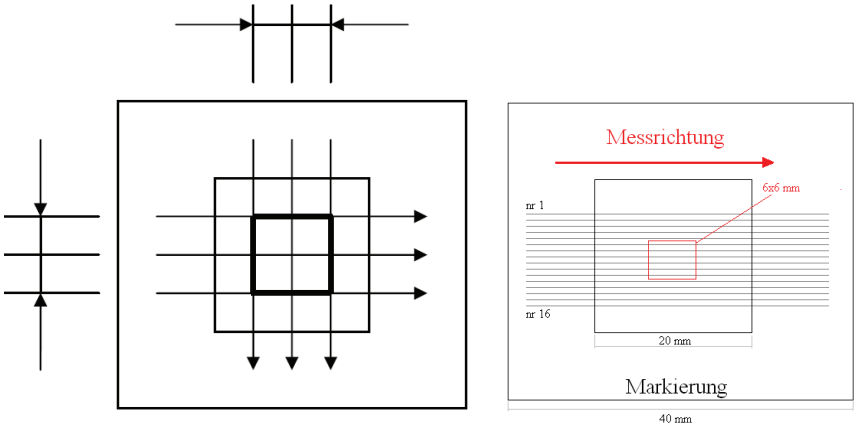


*Figure 3.17 Measurement scheme of thickness standard used in TUT* (1997, 2005), *PTB* (1999) *on the left and PTB* (2009) *on the right*

In addition to the aforementioned measurements, a repeated measuring was done in PTB. The measuring device used was Tencor-P11". In order to guarantee

traceability, the device was calibrated with different standards (see Table 3.2), the uncertainty of which was smaller than the device's sensitivity.

*Table 3.2 Calibration standards*

| Serial number | Nominal value in µm | Uncertainty *U* in µm | Calibration certificate |
|---|---|---|---|
| C02 R18 N361 | 1,0162 | 0,004 | 4451PTB 04 |
| C03 R16 N312 | 2,0902 | 0,006 | 4451PTB 04 |
| EN 802 R1 | 0,277 | 0,007 | 021 PTB 01 |
| EN 802 R2 | 0,571 | 0,008 | 021 PTB 01 |
| EN 802 R3 | 1,197 | 0,009 | 021 PTB 01 |
| EN 802 R4 | 2,609 | 0,021 | 021 PTB 01 |
| EN 802 R5 | 5,345 | 0,020 | 021 PTB 01 |
| EN 802 R6 | 8,36 | 0,025 | 021 PTB 01 |
| Id 2 | 46,346 | 0,001 | 4158 PTB 04 |
| Id 4 | 101,900 | 0,001 | 4158 PTB 04 |
| Id 8 | 189,965 | 0,001 | 4158 PTB 04 |
| Id 10 | 305,582 | 0,001 | 4158 PTB 04 |

Upon processing the readings, the measurement and calculation program described in subschapter 3.3.1 was used that considered the possible curvature of the base surface.

The differences between the measurement procedures conducted in 1997, 1999, 2005 and 2009 lied in the number of measurement profiles. In earlier years the measurements were made like depicted in *i.e.* 3+3 profile measurement (from left to right and from top down), but in 2009, 16 different profiles were measured for each standard with 1 mm distances, but only in one direction (from left to right). It means that almost the whole covered area of the standard was included in the measurement. With the measurement and calculation program it is possible to determine the average coating thickness in the specified area, for example 6 mm x 6 mm in the middle of the covered area as it is shown in Table 3.3.

Table 3.4 specifies the results of comparison calibration. The right column gives calibration results received upon a straight profile under the coating. The left column includes measurement results taking into account the estimated profile under the coating. It is evident that the measurement results of the two methods are not very different, except for four standards the nominal value of which is 27 µm, 38 µm, 52 µm and 73 µm. In case of these four standards, the curvature was the largest on the edge of the standards' base surface and thus the results were expected.

Obviously it must be remembered that the measurement result of all calibrations is the average coating thickness in the middle of the standard in an area of roughly 6 mm x 6 mm. But the measurement uncertainty is much smaller in the 2009 calibration. One of the reasons was the use of a more precise measuring device, but the most important aspect here was the measurement uncertainty and measurement model. Namely, the uncertainty conditioned by the coating's unevenness has not been considered.

*Table 3.3 Calibration results in μm of the coating thickness standards – $h_m$,U, in μm*

| | NPO „Isari“ 1988 contact method | | TUT 1997 magnetic method | | PTB 1999 X-Ray method | | PTB 1999 contact method | | TUT 2005 laser probe LS10 | | 2009 new method | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | $h_m$ | $U$ | $h_m$ | $U$ | $h_m$ | $U$ | $h_m$ | $U$ | $h_m$ | $U$ | $h_m$ | $U$ |
| 1055 | 5,4 | 0,16 | 5,0 | 0,25 | 5,34 | 0,20 | 5,45 | 0,15 | 5,5 | 0,20 | 5,35 | 0,2 |
| 1054 | 11,5 | 0,20 | 11,3 | 0,60 | 10,8 | 0,30 | 11,0 | 0,30 | 11,1 | 0,4 | 10,9 | 0,2 |
| 1021 | 16,2 | 0,30 | 16,0 | 0,80 | 15,8 | 1,20 | 16,0 | 0,90 | 16,1 | 0,8 | 16,1 | 0,2 |
| 1001 | 27,4 | 0,50 | 24,0 | 1,20 | 27,1 | 1,90 | 27,1 | 1,60 | 26,9 | 1,6 | 27,3 | 0,2 |
| 832 | 38,4 | 0,06 | 34,0 | 1,70 | | | 37,9 | 1,20 | 36,8 | 2,1 | 38,1 | 0,2 |
| 1049 | 50 | 0,70 | 44,0 | 2,10 | | | 49,6 | 0,80 | 48,0 | 1,6 | 49,7 | 0,2 |
| 1016 | 54 | 0,70 | 49,0 | 2,50 | | | 52,2 | 0,80 | 51,8 | 1,9 | 52,3 | 0,2 |
| 860 | 73,5 | 1,00 | | | | | 72,8 | 2,20 | 72,4 | 1,9 | 73,8 | 0,2 |
| 816 | 91,2 | 1,20 | | | | | 90,4 | 2,30 | 87,8 | 2,5 | 90,9 | 0,2 |

*Table 3.4 Calibration results of coating thickness standards with straight- and with estimated profile under the coating*

| With estimated profile (in μm) | Difference (in μm) | With straight profile (in μm) |
|---|---|---|
| 5,35 | -0,05 | 5,4 |
| 10,9 | -0,10 | 11,0 |
| 16,1 | 0,00 | 16,1 |
| 27,3 | -0,40 | 27,7 |
| 38,1 | -0,20 | 38,3 |
| 49,7 | 0,00 | 49,7 |
| 52,3 | -0,40 | 52,7 |
| 73,8 | 0,20 | 73,6 |
| 90,9 | 0,00 | 90,9 |

The coating thickness for certain standards was rather different also in the middle of the measuring area (see Figure 3.18). For example, for standard MO No 1001 (with nominal value 27 μm) the coating thickness in the measuring area ranged ca $h_c$= ±1,2 μm and thus the uncertainty of measurements by PTB in 1999 has an abnormally high value. But as the above mentioned measuring method gives the coating thickness value in each measuring point, this component is directly not necessary either, as it is already reflected in the measurement model. It is true that a later user will have to find the exact central point of the measuring area exceptionally and consider the resulting uncertainty component, but it is not related to a comparison calibration, but to the later use of the standards.

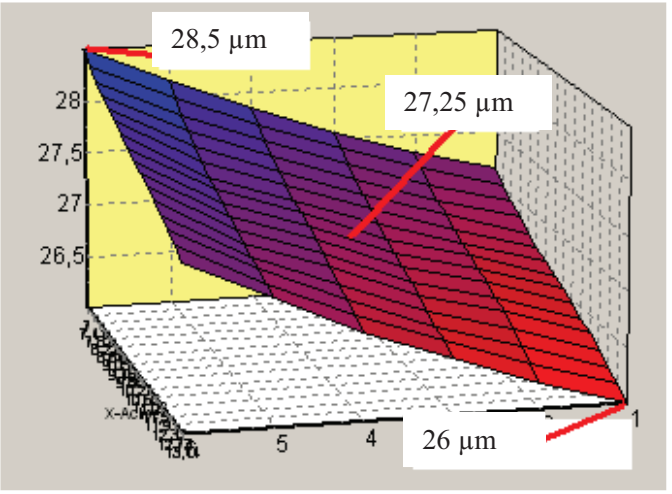


*Figure 3.18 Uneven division of coating of coating thickness standards Nickel-Steel No 1001 in the middle of the measuring area*

## 3.5 Conclusion of Chapter 3

The calibration results specified in Table 3.3 indicate that a developed method provides more trustworthy measurement results and is in more compliance with the definition of coating thickness. However, it cannot be completely confirmed that the estimated profile is actually identical under the covering. Depending on the object of measurement, the measurement model can naturally be improved. For example, it can be presumed that the profile next to the coating can be described with a second or third order polynomial and perhaps that will produce more trustworthy results. At the same time it cannot be controlled, as it is a non-destructive method.

The clear advantage of this method is indeed the smaller measurement uncertainty. Upon calibrating the standards of TUT, the level of uncertainty was even 5 times lower. As already mentioned, this is partially due to better and newer measuring devices, but mainly the calculation of coating thickness unevenness in the measurement model. Upon later use of the standards the component presumed to provide the user with information about the coating thickness in the middle of the measurement area has to be considered, as well. This can be a graphic image, such as Figure 3.12 or a mathematical model.

As the procedure of metrology control of the coating thickness measuring instruments can be assured only with calibrated coating thickness standard increasing the accuracy of coating thickness standards is very important.

On the basis of the described method we can evaluate the profile under the coating of the coating thickness standard. Using the developed method we can determine the coating thickness during the calibration procedure according to the definition of coating thickness. It increases reliability of the calibration of coating thickness standards compared to the method considering only profiles of the upper boundary surface of the base material adjoining the coating.

# 4 MEASUREMENT OF COATING THICKNESS BY SCANNING METHOD OF BASE SURFACE

Chapter 3 described the method suitable for calibrating coating thickness standards and although this is an innovative method, it did not entirely correspond to the definition of coating thickness. Naturally, there are non-destructive measurement methods (ultrasound, X-ray, *etc.*) that enable to measure coating thickness according to its definition. However, such methods do not allow the direct measurement of coating thickness and therefore cannot be used for the initial calibration of coating thickness standards.

It is clear that it is not possible to measure coating thickness standards under the coating without destruction, but the possibility arises upon including calibration in the production process of standards.

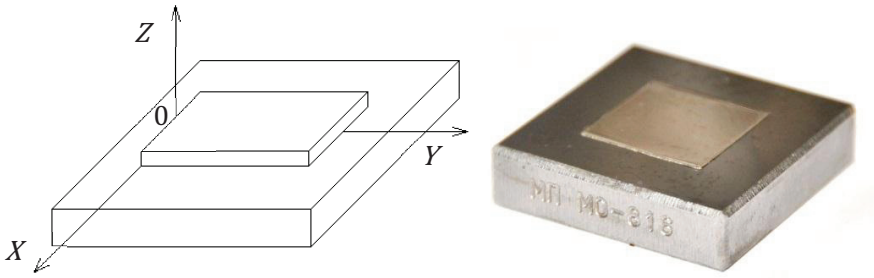


*Figure 4.1 Coating thickness standard*

Similarly to the previous method (described in Chapter 3) let us relate the coating thickness standard to the cross coordinate system 0*XYZ* so that the plane of the cross coordinate system 0*XY* is parallel to the foundation of the base, and the point of origin of the coordinates is in the middle of the intersectional line between the side and the foundation of the base (see Figure 4.1).

It is clear that if we want to measure coating thickness non-destructively on the basis of its definition, we must be able to measure the lower and upper boundary surface of the coating. The only option in use is the measurement of surfaces before and after the coating process. The top surface of the coating thickness standard is measured in several sections before coating. Thereby we get data on the base of the top surface of the coating thickness standard, a part of which will be under the coating in future. Thereafter the surface of the base of the coating thickness standard is covered with a coating by means of the required technology. The whole measurement process is repeated. Since by the definition of coating thickness it is necessary to determine the distance between the lower and upper boundary surface, the measurement data in each section must also characterise the lower and upper boundary surface. Whichever technology is used, the measurement results will never characterise the total boundary surface, but only a certain part of it, *i.e.* the measurement results in those coordinates where the measurement was carried out.

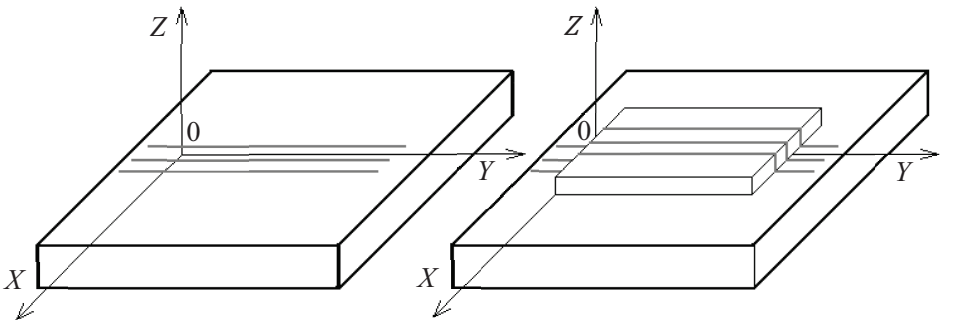


*Figure 4.2 Measurements before and after the coating process* [78]

Two measurement results enable to determine the coating thickness or by definition the distance between the lower boundary surface and upper boundary surface of the coating. The problem here is that firstly it has to be ensured that the base surface of the standard does not change during the coating application, and that in case of topographic measurements the measurement data are comparable to each other. This means that based on the definition of coating thickness the values characterising the surface have to come from the same *XY* coordinates in case of both measurements, because coating thickness is the distance between the lower and upper boundary surface of the coating measured crosswise to the boundary surface of the base.

The problem would not be there, if the base surface was made absolutely flat or at least flat enough to prevent the deviations related to the *XY* coordinate from having an effect on the measurement results. That, however, would require standard bases of very high quality. Even the end faces of gauge blocks are made by the use of modern technology in such a way that the flatness deviation of the end faces does not exceed the value of 0,05 μm. This means that if the described method is not used, that component has to be included in uncertainty calculations to its full extent. The double measurement is necessary in order to prevent it and allow cost-effective and faster manufacture of coating thickness standards.

## 4.1 **Positioning**

For the measurement results to be comparable in the *XY* coordinate system, the object of measurement must be positioned very precisely for measurements. Instead of reference points it is also possible to use base surfaces as shown in the figure below. That would however make the manufacturing of standards more difficult, because the base surfaces should be at an angle of 90 degrees as accurately as possible and the flatness of the base surfaces should be perfect. When handling the standard, damage to the base surfaces should also be avoided that is virtually impossible.

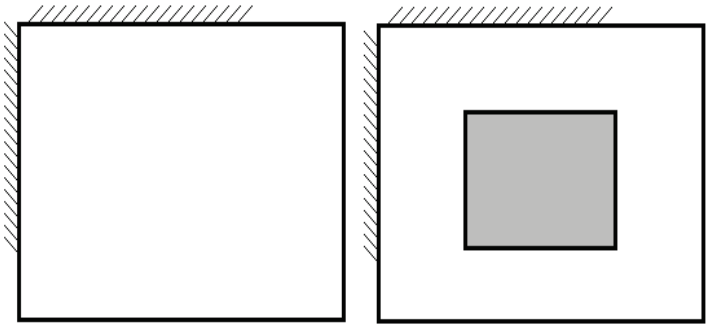

*Figure 4.3 The positioning of a coating thickness standard using base surfaces* [78]

The easiest way is to use special marks that are made on the top surface of the coating thickness standard.

The Figure 4.4 and Figure 4.5 shows an example how positioning influences measurement results. The black line depicts the measurement line before the coating application and the grey line stands for measurements taken after the coating application.

When measuring, there is always a drift towards both *X* and *Y* and therefore the points of the topographic network drift in relation to each other.

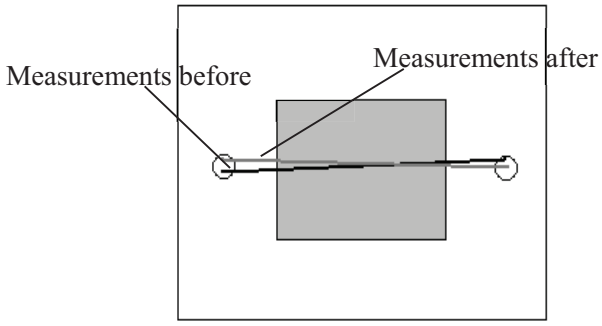


*Figure 4.4 Positioning related drift towards the Y axis* [78]

It can be seen from the Figure 4.5, how the measuring points drift mutually. When two topographic networks drift, it is necessary to differentiate between deviation in the *X* direction, deviation in the *Y* direction or deviation from turning around the *Z* axis. Subject to the directions of the deviations, they either merge or compensate each other.

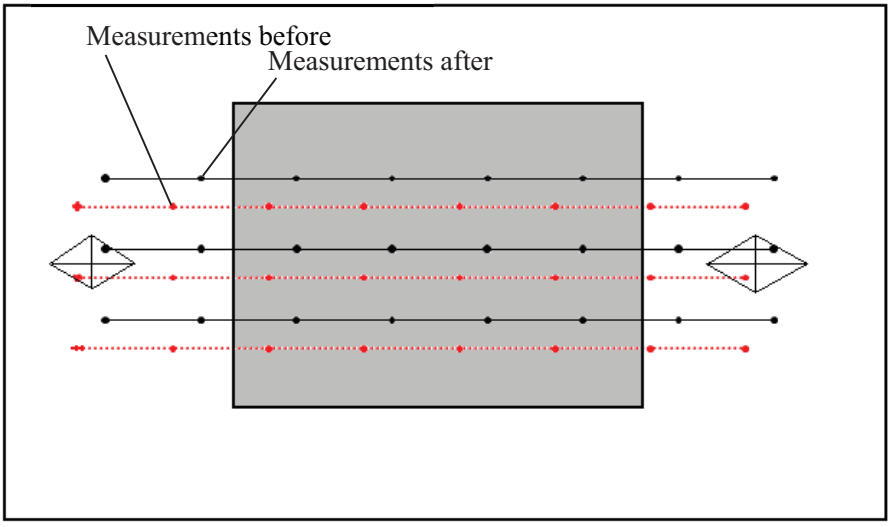


*Figure 4.5 Positioning related drift towards the X and Y axis* [78]

Because of its best quality, the middle surface of the coating thickness standard is used. Therefore, the reference points for centring should be selected in such a manner that the uncertainties caused by the position of the standard on the middle area of the coating would be as small as possible. The need for the preciseness of the position of the standard depends on the quality of the top surface of the base of the standard as well as on the quality of the top surface of the coating.

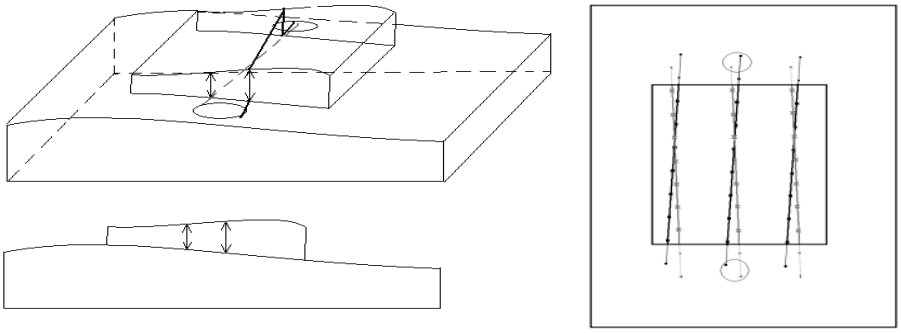

*Figure 4.6 The effect of positioning on the results of coating thickness measurement* [78]

The rougher and more deviated (more curved) both surfaces are, the more precise the position must be. In other words, the more the positioning in the *XY* direction influences the results of coating thickness measurement in the *Z* direction, the more precisely the standard must be positioned for both measuring operations. Figure 4.6 shows how the deviation of positioning has a direct effect on the result of coating thickness measurement.

## 4.2 Measurement model

Similarly to chapter 3, this section also describes surfaces with random functions. All other theories related to coating thickness definitions specified in chapters 1 and subchapter 3.1 are also valid here.

The measuring of the surface of the coating thickness standard by means of groping gives a random function $Z_{i1}(y)$ characterising the base surface in the intersection from $y_3$ to $y_4$. After the coating application and second measurement we get a random function $Z_{i2}(y)$ characterising the top surface of the coating in the same intersection from $y_1$ to $y_2$. Functions $Z_{i1}(y)$ and $Z_{i2}(y)$ are random functions,

$$Z_{i1}(y) = \tilde{Z}_{i1} + a_{i13}y^3 + a_{i12}y^2 + a_{i11}y + b_{i10} \quad (4.1)$$

$$Z_{i2}(y) = \tilde{Z}_{i2} + a_{i23}y^3 + a_{i22}y^2 + a_{i21}y + b_{i20} \quad (4.2)$$

where $\tilde{Z}_{i1}$ and $\tilde{Z}_{i2}$ have random values according to the normal distribution N(0, $\sigma_1$) and N(0, $\sigma_2$).

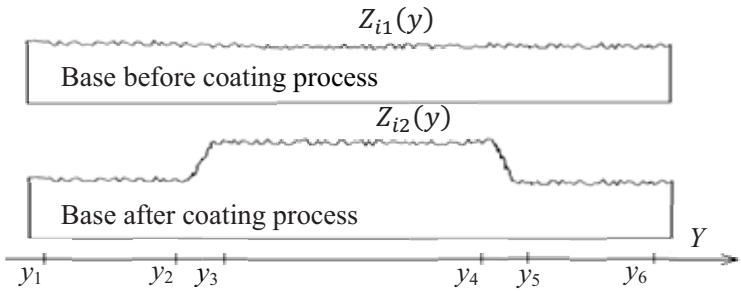


*Figure 4.7 Surface profiles in the intersection parallel to the XZ plane* [78]

The means of those functions can be expressed as follows:

$$m_{Z1}(y) = a_{13}y^3 + a_{12}y^2 + a_{11}y + b_{10} \quad (4.3)$$

$$m_{Z2}(y) = a_{23}y^3 + a_{22}y^2 + a_{21}y + b_{20} \quad (4.4)$$

where index $i = 1 \ldots n$ is the number of the topographic profile, which varies in the range from $x_1$ to $x_2$.

The coating thickness in a point $i$ can be calculated by the formula

$$h_i(y) = Z_{i2}(y) - Z_{i1}(y) \quad (4.5)$$

The mean value of coating thickness can be found by the formula:

$$m_{ih}(y) = m_{Zi2}(y) - m_{Zi1}(y) =$$
$$a_{i13}y^3 + a_{i12}y^2 + a_{i11}y + b_{i10} -$$
$$-a_{i23}y^3 + a_{i22}y^2 + a_{i21}y + b_{i20} \quad (4.6)$$

The mean coating thickness of the coating thickness standard in the intersection from $y_1$ to $y_2$ and from $x_1$ to $x_2$ can be found from the relation:

$$h_{\text{mean}} = \frac{\sum_{i=1}^{n} m_{ih}}{n} \tag{4.7}$$

It is evident that when proceeding from the coating thickness definition, the functions $Z_{i1}(y)$ and $Z_{i2}(y)$ must be located in the same reference systems. This condition must somehow be connected with the measurement model, but later also with the uncertainty calculation. Positioning does not add any value or correction to the measurement model $\Delta_{\text{pos}} = 0$ µm, but it must be considered in the uncertainty calculation model. Analogically to subchapter 0 the polynomial regression task could also be used for mutual positioning of two random functions $Z_{i1}(y)$ and $Z_{i2}(y)$ the reference system, but it adds unnecessary complexity. For the purpose of simplification and even increasing precision, the so-called discrete data (special markings in the profile) specified in subchapter 4.4 are used for positioning.

As the method is universal and describes different coatings, it can be used in addition to singe-value standards also in case of multi-value, stair-shape *etc* standards. The general measurement model does not change, because all the boundary surfaces are similarly described. Upon calculating the coating thickness or coatings thicknesses, it is relevant to choose proper boundary surfaces. For example in case of common coatings in industrial area where steel is covered with zinc and turn is covered with paint.

*Figure 4.8 Measuring the thickness of zinc and paint in VW factory* [24]

The commonly used measurement method is the following: at frst, the total thickness of coating (zinc+paint) is evaluated with the magnetic-induction method thereupon phase-sensitive Eddy-Current method is used to evaluate thickness of zinc coating. It can be seen from Figure 4.9 that the total thickness of the coating is $h_{\text{total}}$ and zinc $h_{\text{zinc}}$. The thickness of the paint can be calculated as follows:

$$h_{paint} = h_{total} - h_{zinc} \tag{4.8}$$

For such measurement instruments it is better if the coating thickness standard has a two-layer coating. This can be accomplished by using multi coatings standards [45, 46, 50–52, 55]. The method described above is most suitable for calibrating such standards. In that case, the top of the surface of the coating thickness standard is measured at first and thereafter the standard is covered with zinc. Next, surface measurement is carried out similarly by evaluating the top of the surface of zinc coating which forms the boundary surface between two coatings. The third measurement is carried out after covering the layer with the second coating (in this case with paint) by evaluating the profile of the surface of the coating.

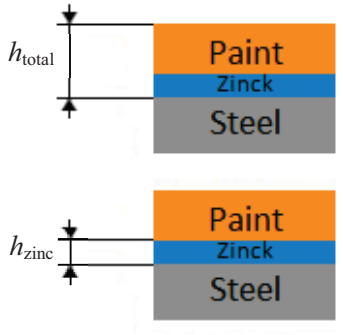


*Figure 4.9 Coating thicknesses $h_{total}$ measured with magnetic induction and $h_{zinc}$ measured with phase-sensitive Eddy-Current method* [24]

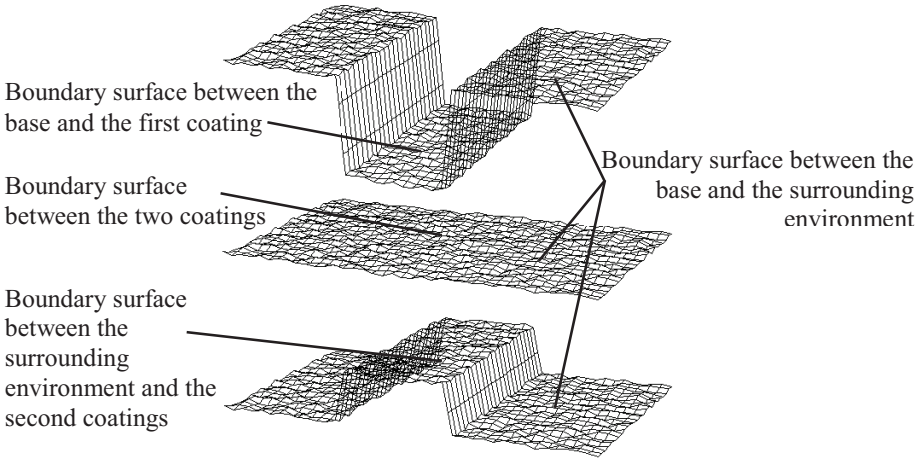


*Figure 4.10 Thee different profiles measured* [40, 41]

According to information about all boundary surfaces, it is possible to evaluate the thickness of zinc or paint or the total thickness of two layers. Figure 4.11 shows the boundary surfaces of two coatings: 1 – the boundary surface between the

steel-base and zinc, 2 – boundary surface of zinc and paint, 3 – boundary surface between the surrounding environment and paint.

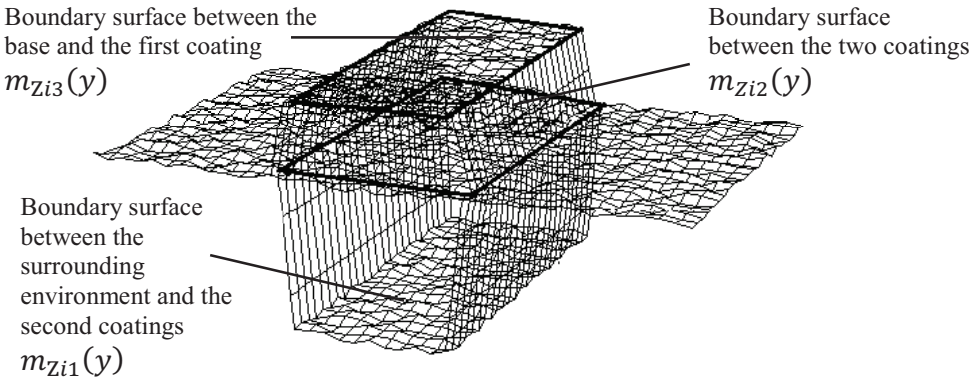


*Figure 4.11 Boundary surfaces of thickness standard with two coatings*

The are a clear advantages in comparison with common methods– the uncertainty of the total- or single coating thickness is lower, because the measurement model contains only two components or boundary surfaces.

Without this method the thickness of paint should be calculated as follows:

$$h_{\text{paint}} = \frac{\sum_{i=1}^{n} m_{i\text{h total}} - m_{i\text{h zinc}}}{n}, \tag{4.9}$$

where

$m_{i\text{h total}}(y) = m_{Zi3}(y) - m_{Zi1}(y)$

$m_{i\text{h zinc}}(y) = m_{Zi2}(y) - m_{Zi1}(y)$

$m_{i\text{h total}}$ – total thickness of coatings,

$m_{i\text{h zinc}}$ – thickness of zinc coating

$m_{Zi3}(y)$ – topmost boundary surface (Figure 4.11),

$m_{Zi2}(y)$ – boundary surface between two coatings (Figure 4.11),

$m_{Zi2}(y)$ – boundary surface of the base (Figure 4.11).

By using this new method, the thickness of upper coating as showed on Figure 4.11 (for example a paint) can be calculated based on two boundary surfaces:

$$h_{\text{paint}} = \frac{\sum_{i=1}^{n} m_{i\text{h}}}{n}, \tag{4.10}$$

where

$m_{i\text{h}}(y) = m_{Zi3}(y) - m_{Zi2}(y)$,

$m_{Zi3}(y)$ – topmost boundary surface (Figure 4.11),

$m_{Zi2}(y)$ – boundary surface between two coatings (Figure 4.11).

Similarly, the number of different coating layers can be added as much as necessary, whereas the uncertainty of the thickness is calculated with the same formula depending only on two measurement results.

## 4.3 Uncertainty estimation in scanning method

The measurement uncertainty calculation model is relatively similar to the model given in Chapter 3.2. According to the [78], uncertainty of the mean coating thickness of the coating thickness standard can be calculated from the relation:

$$u_i(h) = \sqrt{D_{i\mathrm{h}}(y) + u^2(z) + u_{\mathrm{pos}}^2 \left(\frac{\partial h}{\partial x \partial y}\right)^2} \tag{4.11}$$

where $D_{i\mathrm{h}}(y)$ is the dispersion of the coating thickness function, $u^2(z)$ is the uncertainty component in the direction of the *Z*-axis and $u_{\mathrm{pos}}^2$ is the uncertainty component arising from the positioning made due to two measurements. Index *i* determines the profile number.

Similarly to the calibration of estimated base surface (see chapter 3) we cannot presume that functions $Z_1(y)$ and $Z_2(y)$ are totally independent. It cannot be done already because for an example the measuring device is the same. Nevertheless, the uncertainty component resulting from that is relatively small and therefore we can make a compromise and consider these functions independently in the uncertainty model. If we assume that the functions $Z_1(y)$ and $Z_2(y)$ are independent, the dispersion of the function characterising the coating thickness is:

$$D_{i\mathrm{h}}(y) = D_{iz2}(y) + D_{iz1}(y) \tag{4.12}$$

The uncertainty calculation equation in the direction of *Z*-axis must include all important factors that can influence the measurement in the direction of Z-axis. Therefore we express the standard uncertainty component in the direction of the Z-axis as follows:

$$u(z) = \sqrt{u_{\mathrm{cal}}^2 + u_{\mathrm{en}}^2 + u_{\mathrm{ob}}^2} \tag{4.13}$$

where $u_{\mathrm{cal}}$ is the standard uncertainty of the calibration of the measurement instrument, $u_{\mathrm{en}}$ is the standard uncertainty of the environment (noise) and $u_{\mathrm{ob}}$ is the uncertainty of the surface of the base and of the surface of the coating (roughness).

The positioning related combined standard uncertainty component in the direction of the *X*- and *Y*-axis appears twice (we measure before and after coating process), thus

$$u_{\mathrm{pos}} = 2\sqrt{u_{\mathrm{x}}^2 + u_{\mathrm{y}}^2 + u_{\mathrm{resX}}^2 + u_{\mathrm{resY}}^2} \tag{4.14}$$

where $u_{\mathrm{x}}$ is the uncertainty of the positioning in the direction of the *X*-axis of the profile, $u_{\mathrm{y}}$ is the uncertainty of the positioning in the direction of the *Y*-axis of the profile, $u_{\mathrm{resX}}$ is the uncertainty component in the direction of the *X*-axis caused by the position of the device, and $u_{\mathrm{resY}}$ is the uncertainty component in the direction of the *Y*-axis caused by the position of the device.

The last component in the uncertainty equation is $\frac{\partial h}{\partial x \partial y}$ . It can be said that calculating this component is actually very difficult. The necessity arises from the fact that we measure the profile twice. It does not matter how precise the measurement is, measurement values are never taken from the same *XY* coordinates - there is always a shift. The uncertainty depending on positioning that can be found relatively easily is still in the direction of *XY*-axis. In order to transfer the uncertainty in the direction of *XY*-axis into the uncertainty in the direction of *Z*-axis (towards coating thickness), the component $\frac{\partial h}{\partial x \partial y}$ needs to be present. This component shows how much the coating thickness actually changes if we move towards the *XY* level. It is clear that the base surface's curvature as well as the unevenness of the coating thickness must be taken into account too. For calculating the uncertainty caused by the flatness deviation of the base of the coating thickness standard and uneven distribution of the coating thickness we must use the assumption that the value of $\frac{\partial h}{\partial x \partial y}$ is approximately equal to the standard uncertainty $u(\Delta_{XY})$ caused by the unevenness of the base of the standard and coating thickness [78]:

$$u(\Delta_{\mathrm{XY}}) = \sqrt{u^2(\Delta_{\mathrm{SX}}) + u^2(\Delta_{\mathrm{SY}}) + u^2(\Delta_{\mathrm{CTX}}) + u^2(\Delta_{\mathrm{CTY}})} \qquad (4.15)$$

where $\Delta_{SX}$ is the flatness deviation of the **base** in the *X* direction, $\Delta_{\mathrm{SY}}$ is the flatness deviation of the **base** in the *Y* direction, $\Delta_{\mathrm{CTX}}$ is the **change of coating thickness** in the direction of the *X*-axis, and $\Delta_{\mathrm{CTY}}$ is the change of coating thickness in the direction of the *Y*-axis. As it can be seen, this includes uncertainty components, whose value becomes evident only after measurements and is directly dependent on the standard's quality.

It is rather common that the correction factors and related uncertaintes may not be final in case of evalutation of the measurement models and their uncertainty and thus, if necessary, respective components may be added to the equation 4.11.

## 4.4 Experimental research results

The measurements were performed in the Coating Thickness Laboratory PTB on April 2009. The measuring instrument „Tencor P-11" was used. In order to guarantee traceability, the device was calibrated with different standards (see Table 4.1), the uncertainty of which was smaller than the device's sensitivity.

As the method presumes that the top surface of the standard is measured before and after the coating is spread, the standard had to be prepared manually, too. The usage of Ni base surface and Cu coating was chosen. A rotary table and various abrasive papers and diamond paste were used for polishing. The surfaces were cleaned and the first measurement was conducted. One-third of the top surface of the standard's base was covered on the left side and one-third on the right side with a tape with copper glued on it. The covering was spread on the standard with galvanic method and the second measuring was performed.

*Table 4.1 Calibration standards*

| Serial number | Nominal value in µm | Uncertainty *U* in µm | Calibration certificate |
|---|---|---|---|
| C02 R18 N361 | 1,0162 | 0,004 | 4451PTB 04 |
| C03 R16 N312 | 2,0902 | 0,006 | 4451PTB 04 |
| EN 802 R1 | 0,277 | 0,007 | 021 PTB 01 |
| EN 802 R2 | 0,571 | 0,008 | 021 PTB 01 |
| EN 802 R3 | 1,197 | 0,009 | 021 PTB 01 |
| EN 802 R4 | 2,609 | 0,021 | 021 PTB 01 |
| EN 802 R5 | 5,345 | 0,020 | 021 PTB 01 |
| EN 802 R6 | 8,36 | 0,025 | 021 PTB 01 |
| Id 2 | 46,346 | 0,001 | 4158 PTB 04 |
| Id 4 | 101,900 | 0,001 | 4158 PTB 04 |
| Id 8 | 189,965 | 0,001 | 4158 PTB 04 |
| Id 10 | 305,582 | 0,001 | 4158 PTB 04 |

After many tests, a standard with satisfactory characteristics was prepared. The whole process (polishing, micro-polishing, making the marking, measuring, surfacing, measuring) took 4–5 hours. As the polishing of the standard's top surface was performed manually, the curvature of the surface was rather great. The flatness deviation of the base surface is within the limits from 3 µm per millimetre to 5 µm per millimetre (in the middle of the measuring area within the limits from 1 µm per millimetre to 2 µm per millimetre).

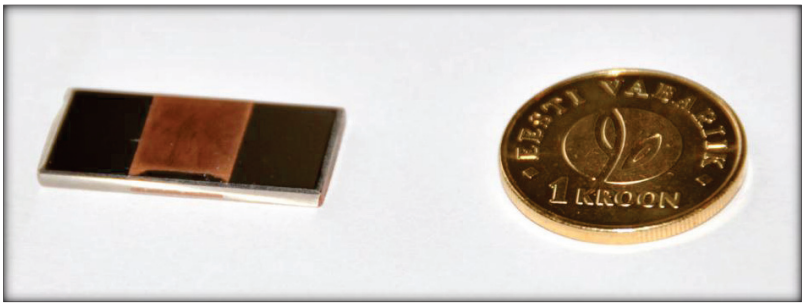


*Figure 4.12 Manually made coating thickness standard (Ni-Cu)*

Upon spreading the galvanic coating, the decisive aspect is the density of direct current on the whole top surface of the standard. Special screens and electrodes are necessary for receiving better results. The amount of current must be limited too in order to get a denser and smoother surface. The change of coating thickness in the centre of the standard was within the limits from 0,2 µm/mm up to 0,3 µm/mm.

For double measurement of the sample (before and after coating), special marks were used for positioning. The Knoop microhardness tester was used for that before coating which leaves a relatively small and well recognisable mark on the surface.

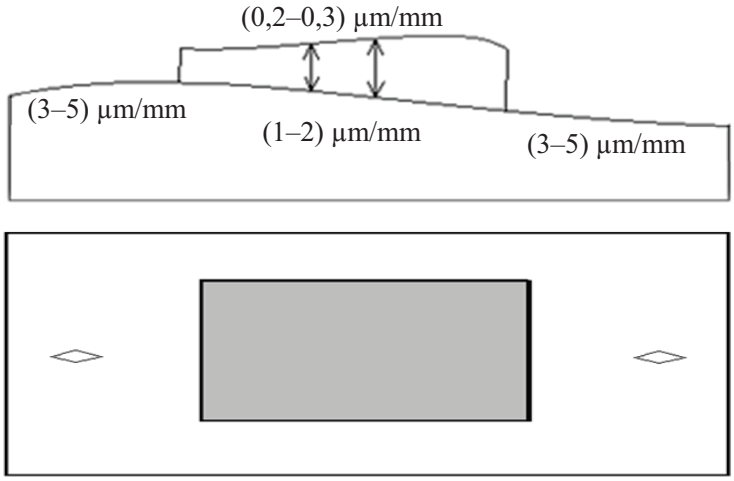


*Figure 4.13 Flatness deviation of the base surface and change of coating thickness in the centre of standard* [78]

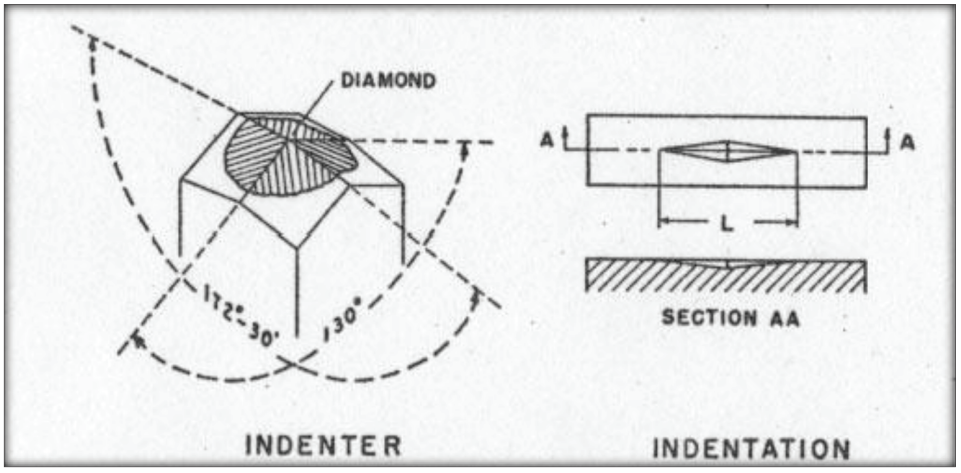


*Figure 4.14 The indenter of the Knoop microhardness tester and the image of indentation* [66]

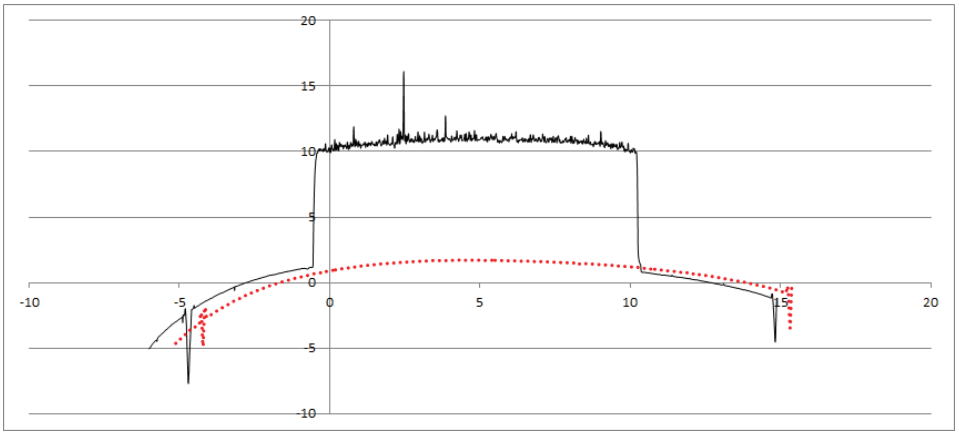


*Figure 4.15 Measurement profiles before and after coating*

Figure 4.15 shows that the real measurement profiles of measuring are not in the same reference system. Profiles must be shifted, turned, tilted in order to get measurement results - covering thickness in a certain point.

### 4.4.1 Developed measurement and calculation program

Analogically to the description in subchapter 3.3.1 a measurement and calculation program (in DELPHI programming environment) was created for processing also these measurement results. An automatic data processing had to be able to:

- Fix the markings used for positioning (made with the Knoop microhardness tester),
- Analyse measurement result and locate the measurement profiles of the base in the range from $y_1$ to $y_2$ and from $y_5$ and $y_6$ (measurement results before and after coating) to as the same level as possible,
- Calculate the mean value $m_{iz1}(y)$ of the random function characterizing the base surface and its dispersion in the range from $y_3$ to $y_3$,
- Calculate the mean value $m_{iz2}(y)$ of the random function of the coating's top surface and its dispersion in the range from $y_3$ to $y_4$,
- Remove local defects (dust, scratches *etc*.) from the measurement data
- Calculate the mean covering thickness $m_{\mathrm{hp}}(y)$ in the specified area,
- Use a flexible graphic interface.

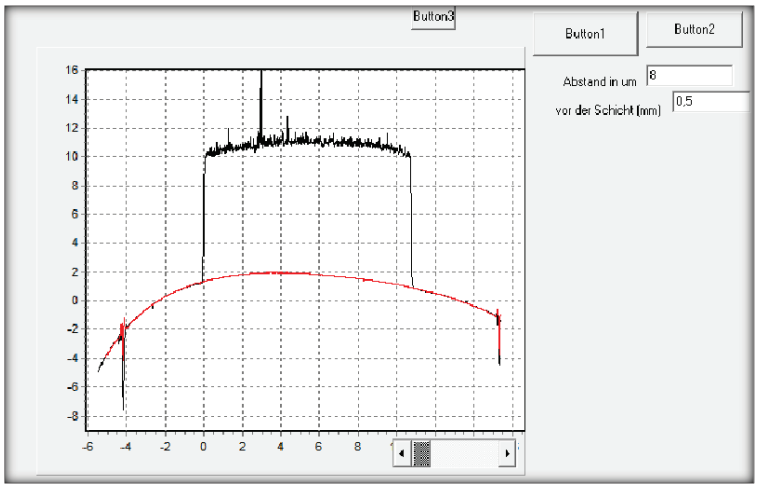


*Figure 4.16 Measuring and calculating program for measuring covering thickness*

Proceeding from the established task, the measurement program operates relatively similarly to that described in Subchapter 3.3.1. But here the program does not calculate the estimated profile between the coating and the base, but locates two measurement profiles (before and after coating) on top of each other as precisely as possible. The markings made with the Knoop microhardness tester are used for that. Figure 4.17 indicates how the program has placed the local minimum levels of both markings to the same Y-axis coordinate.

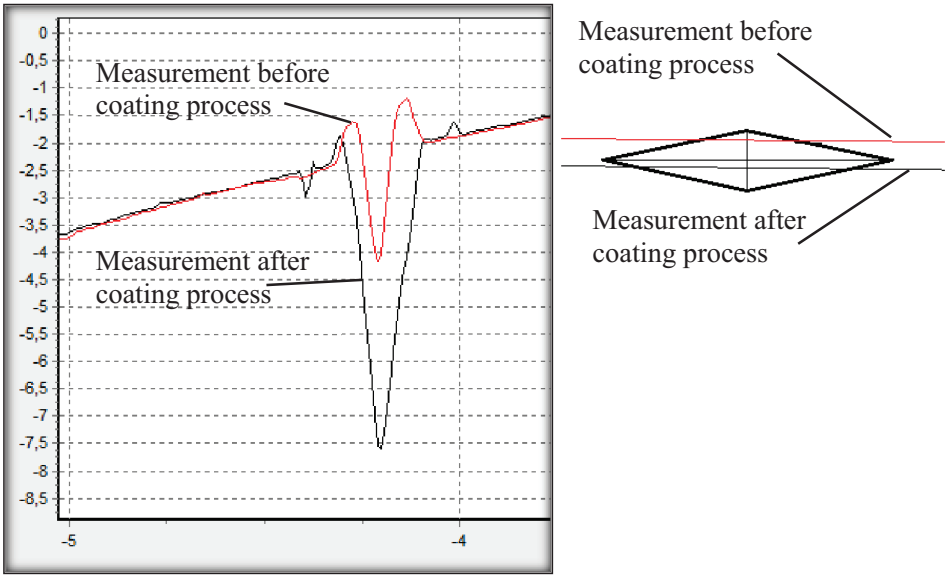


*Figure 4.17 Profile images of markings used for positioning*

It is as important to shift the two profiles into the same reference system on the Z-axis. For that purpose, the measurement program calculates the mean value in the ranges from $y_1$ to $y_2$ and $y_5$ to $y_6$ and places the $Y$ coordinates of the respective point

of the profiles at the same level on the *Z*-axis. It cannot be seen well on the left-side part in Figure 4.18, as the two profiles have been placed so well on top of each other. The right-side image has been magnified and there the two profiles can be well distinguished.

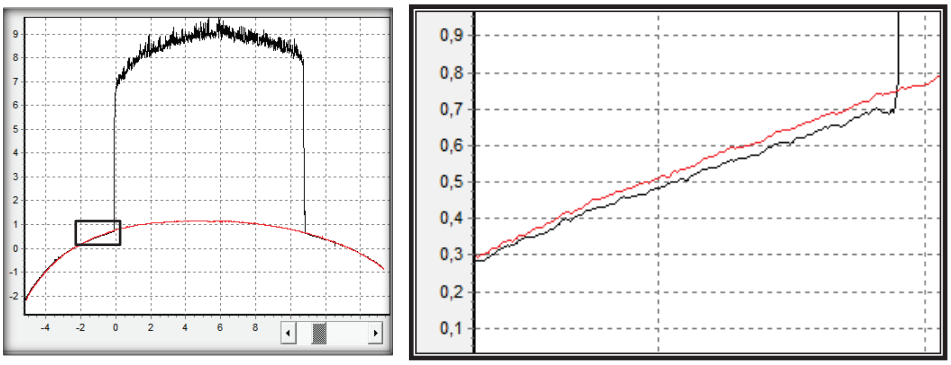


*Figure 4.18 Shifting of profiles toward the z-axis and the deviations resulting from that*

### 4.4.2 An analysis of measurement results

However, the uncertainty arising from positioning can be seen as well. As two measurements cannot be made at exactly the same point, the profiles from two different places cannot be placed in a way that they overlap completely. The approximate height difference was ca 30 nm, which is rather small considering other uncertainty components.

Proceeding from the prepared standard and its initial measurement results, the average standard uncertainty component resulting from the unevenness of the coating thickness and geometrical deviations of the base surface is (according to the equation 4.15):

$$u(\Delta_{XY}) = \sqrt{\left(\frac{2\ \mu m}{2\sqrt{3}}\right)^2 + \left(\frac{2\ \mu m}{2\sqrt{3}}\right)^2 + \left(\frac{0{,}3\ \mu m}{2\sqrt{3}}\right)^2 + \left(\frac{0{,}3\ \mu m}{2\sqrt{3}}\right)^2} =$$

$$= 0{,}8\ \mu m/mm = 8 \cdot 10^{-4}\,\frac{\mu m}{\mu m}$$

For double measurement of the sample (before and after coating process), special marks (Knoop microhardness tester) were used for positioning. Upon measuring, we tried to hit as accurately as possible the centre of the marks. The hit uncertainty in the *X* direction has been calculated on the basis of a half of the mark width ($\Delta_x$ = 15 µm), and in the *Y* direction the hit uncertainty has been calculated on the basis of the triple distance between measurement points ($\Delta_y$ = 3·8 = 24 µm). Thus, the value of the standard uncertainty component of two positioning is (according to the equation 4.14):

$$u_{pos} = 2\sqrt{\left(\frac{15\ \mu m}{2\sqrt{3}}\right)^2 + \left(\frac{24\ \mu m}{2\sqrt{3}}\right)^2 + (1\ \mu m)^2} = 16{,}4\ \mu m$$

In the case in question the worst possible measurement conditions have been considered, *i.e.* as if the largest deviations were in the middle of the measuring area. Therefore, the first profile before coating should be as much as possible on the upper right area and on the second profile that is measured after coating should be on the lower left area (Figure 4.5).

The uncertainty equation in the direction of the Z-axis proceeds from the principles specified in subchapter 3.2. Thus, the $u_{cal}$ estimate can be found from the technical documentation of the device or if the device is calibrated, the component should contain the *Z*-axis sensitivity (resolution), linearity as well as other deviations. The device was calibrated before measurement with three PTB standards (with different nominal values) and considering the value of linearity and sensitivity (resolution) given in the device's documentation, the value of $u_{\mathrm{cal}} = 0{,}07$ µm.

The measurement environment's uncertainty component $u_{en}$ must be found experimentally and depends on the room the measurement takes place in. It is logical to find it by using maximum vibration affecting the device and presuming that the vibration occurs in a rectangle shaped division. It is estimated to be $u_{\mathrm{en}} = 0{,}005$ µm.

It is undoubtedly difficult to find the uncertainty component $u_{ob}$ deriving from the object. It includes all kinds of geometrical deviations not reflected in dispersions$D_{iZ1}(y)$ and $D_{iZ2}(y)$ and thus it must be considered separately. The geometrical deviations of the given standard are rather great, but their influence to the measurement results have usually been taken into account already. Still, testing indicated that in the case of standards with a relatively rough surface, the uncertainty resulting from the surface roughness of the polynomial dispersion is not taken into account as much as it should be, so it must be considered separately. As upon later use of the coating thickness standard the sensor of the device to be calibrated may be located at any place on the standard's surface, this randomness must also be taken into account in the measurement uncertainty Ra, which is an averaged surface roughness parameter. Therefore the recommended value of it is $u_{\mathrm{ob}} = Ra = 0{,}2$ µm.

Thus the value of the combined uncertainty component in the direction of the Z-axis is (according to the equation 4.13):

$$u(z) = \sqrt{(0{,}07\ \mu\mathrm{m})^2 + (0{,}05\ \mu\mathrm{m})^2 + (0{,}2\ \mu\mathrm{m})^2} = 0{,}22\ \mu\mathrm{m}$$

The dispersions of the functions have been found with polynomial regression analysis if we assume that the functions $Z_1(y)$ and $Z_2(y)$ are independent, the dispersion of the function characterising the coating thickness is (according to the equation 4.12):

$$D_{ih}(y) = 0{,}01\ \mu\mathrm{m}$$

The above-mentioned quantities and their values have been presented in the table 4.1.

*Table 4.2 Estimates ofctandard uncertaintyes of input quantities*

| Uncertainty component | Value in µm |
|---|---|
| $u_{\mathrm{y}}$ | 0,1 |
| $u_{\mathrm{cal}}$ | 0,071 |
| $u_{\mathrm{en}}$ | 0,005 |
| $u_{\mathrm{ob}}$ | 0,2 |
| $\frac{\partial h}{\partial x \partial y} u_{\mathrm{pos}}$ | $8 \cdot 10^{-4} \cdot 16{,}4$ |

The expanded uncertainty of the mean coating thickness in that case is (according to the equation 4.11):

$$U(H_i) = 2\sqrt{\begin{matrix}(0{,}1\ \mu\mathrm{m})^2 + (0{,}071\ \mu\mathrm{m})^2 + (0{,}005\ \mu\mathrm{m})^2 + \\ (0{,}2\ \mu\mathrm{m})^2 + (16{,}4\ \mu\mathrm{m})^2 \left(8 \cdot 10^{-4} \frac{\mu\mathrm{m}}{\mu\mathrm{m}}\right)^2\end{matrix}} = 0{,}4\ \mu\mathrm{m}$$

The largest uncertainty component in the combined uncertainty equation is the component caused by surface roughness ($Ra$ = 0,2 µm). If that component is reduced twice ($Ra$ = 0,1 µm), the expanded uncertainty that characterises the measurement result is approximately twice smaller ($U(H_i) = 0{,}2$ µm).

It should also be mentioned that the value of the coating thickness of the given coating thickness standard (see in the middle of the coated area ($y_3$=2 mm; $y_4$=8 mm) was $H = (9{,}2 \pm 0{,}4)$ µm.

Considering the common measurement methods, which assume that the surface under the coating is straight, the level of uncertainty or even the measurement error could be up to 2 µm. It indicates clearly the very low quality of the standard but significantly better results due to the use of a new approach.

Since the value of surface roughness has a great impact on uncertainty, this method is applicable only in case the uppermost surface of the coating is ca 10 times smaller than the coating thickness. In addition, the roughness of the base should be below $Rz$ < 0,1 µm. It is only natural that in certain cases (*e.g.* when an especially rough base or topmost surface is required) the value of the surface roughness may be bigger, but in that case the factors of the sensibility in the mathematical equation have to be altered. Similarly, an uncertainty calculation model like this is valid only in case the value of waviness $W_{\mathrm{t}}$ is smaller than the dispersion of the mean value of the function characterising the surface. If the value of the waviness is bigger (for example lathed or grinded surface), the value of $W_{\mathrm{t}}$ has to be added to the uncertainty equation.

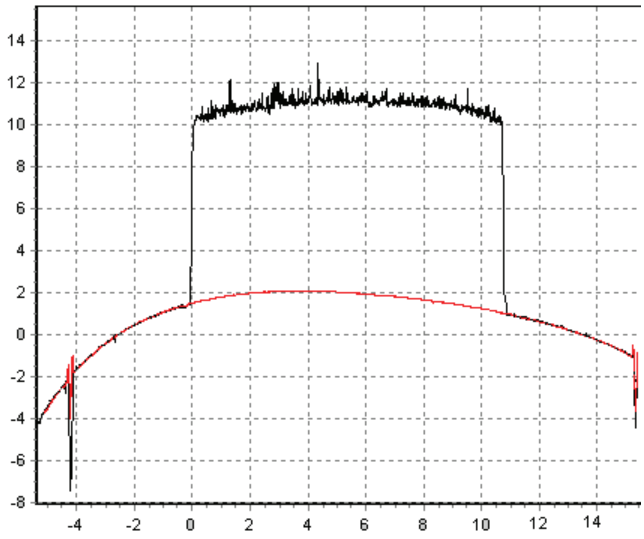


*Figure 4.19 Profile of thickness standard*

## 4.5 Conclusion of Chapter 4

The research, test measurements and their final results indicate that the developed method gives reliable measurement results and conforms to the definition of coating thickness. The measurement model can naturally be improved, depending on the measurement object. For example, we may presume that the mean values of the random functions characterising the surface of the coating thickness can be expressed with an n-th order polynomial.

The apparent advantage of this method is its great reliability and the relatively small measurement uncertainty. This partially results from the use of very good measurement devices, but mostly from the developed measurement model. Standards prepared and measured with this method should be more competitive as to their price, as they do not presume the use of very expensive equipment. It is possible to produce a more accurate standard with the same price or that the standards with the same accuracy can be produced with much cost-effective.

In addition, the method allows the usage of other non mechanical measurement methods. The markings made with the Knoop microhardness tester in the given case may be replaced with any other method (*e.g.* engraving with a laser *etc*.).

Standards prepared with this kind of a method do not require such a good testing device (polishing equipment). Their preparation is not that complex, but the precision and reliability are much greater.

In addition to calibration of single value standards, the method allows to calibrate also other well-known coating thickness standards such as multi-value, stair-shape *etc*. In principle, neither shape or proportions, nor the number of different coating layers are restricted. The main demand of the method is the presence of the surfaces without the coating during the whole measurement process.

A patent has been obtained from the Estonian Patent Office [40] and a patent has been applied for in the German Patent Office [41] for the measurement model and

measurement uncertainty theory specified in subchapter 4.2 included in a single coating thickness calibration method. The main difference with the aforementioned calibration method description is the fact that [40] and [41] do not define how positioning takes place and that measurements are performed only topographically or three-dimensionally.

As the procedure of metrology control of the coating thickness measuring instruments can be assured only with calibrated coating thickness standard, increasing the accuracy of coating thickness standards is very important.

This method enables to calibrate coating thickness standards in a considerably cost-effective and more accurate way, since the calibration of the coating thickness standard is performed on the basis of the definition of coating thickness. That increases the accuracy of calibration in comparison with the calibration methods known so far and also allows to carry out the recalibration of the standards in the future.

# CONCLUSIONS

Methods for calibration different coating thickness standards and their mathematical- and uncertainty models have been investigated. The generalized conclusions of the work are the following:

- A measurement model and the uncertainty calculation model were developed which can be used in case of measurement of complex forms (also complex coating thickness standards).

- A common measurement method was improved aiming to provide more accurate calibration of thickness standards considering the manufacturing process of standards. The model of mathematical measurement and a model of calculation of uncertainty were developed. Due to such models, the accuracy of the calibration increased up to 2 to 5 times depending on the object.

- A new method was developed for making the calibration more cost-effective, more accurate and time-efficient. The model of mathematical measurement and a model of calculation of uncertainty were developed. The price of manufacturing and calibrating the metrological traceable standard decreased significantly and its accuracy increased (depending on the object) up to 10 times. The method allows calibrating almost any standards irrespective of their construction and shapes.

## ABSTRACT

As new coating thickness meters are constantly developed, it has created a need for increasing the preciseness of coating thickness standards as well as further development of calibration methods. Proceeding from the problem presented above, the goals of the thesis are the following – to improve the methods of calibrating coating thickness standards. On the basis of the goals, the author has established the following tasks to be solved:

- Analysis of modern coating thickness calibration systems and coating thickness standards.
- Updating the existing coating thickness calibration methods.
- Development of a mathematical model for direct measurement of coating thickness.
- Development of a functional relation of calibration results on the basis of the measurement model and the implementation of this measurement uncertainty theory.
- Performing check-up procedures of the calibration methods for the developed coating thickness standards by using international comparative calibration.

In the current research a development for the calibration method of coating thickness with a new uncertainty calculation method was created, a new coating thickness calibration method was developed, comparative measurements were conducted which verified the greater credibility of the methods. A patent has been obtained from the Estonian Patent Office and a patent has been applied for in the German Patent Office for the new developed measurement model and measurement uncertainty theory included in a single coating thickness calibration method.



*Keywords*: coating thickness standard, calibration, uncertainty of measurement

# KOKKUVÕTE

Raske on leida teadus- või tehnikaala kus ei kasutatakse õhukesi pindeid. Kõige rohkem on teada-tuntud elektroonika, lennundus, masinaehitus, autotööstus aga ka mööblitööstus, kodumajapidamine, disain jne. Aina vähenevad toormaterjali ressursid, konkurents ja ühe suuremad nõudmised toodangu kvaliteedile sunnivad tööstust kasutama üha rohkem erinevate materjalide kooslusi – pindeid. Kuna pinnete otstarve on väga lai hinnatakse pinnete juures väga erinevaid parameetreid. Näiteks tsingi kihil võib pindena olla ülesanne toimida korrosiooni takistajana sõiduki kerel, samal ajal kui mujal võiks selle pinde eesmärgikas olla hoopis elektrijuht või hoopiski interferentsfilter. Üheks olulisemaks parameetriks pinnate juures on nende paksus.

Pinnete hindamiseks kasutataks tänapäeval väga erinevaid meetodeid, millede tööpõhimõte sõltub pinde kasutuskohast, materjalide kombinatsioonist, täpsusest ning hinnatavast füüsikalisest suurusest. Hinnanguliselt on erinevatel tööpõhimõtetel põhinevaid meetodeid üle saja [25]. Väga paljud pindepaksuse mõõtemeetodid on kaudsed meetodid. See tähendab, nende abil ei mõõdeta otseselt pinde paksust, vaid mõõdetakse mingit mud füüsikalist suurust, mille kaudu hinnatakse pinde paksust. Lähtudes mõõtmiste alustest, on vajalik, et kõik mõõtmised ja nende tulemused oleks jälgitavad SI ühikuni [32]. Sobivaim viis pindepaksuse mõõteseadmete kalibreerimiseks on pindepaksusetalonide kasutamine, mis on omakorda otseste mõõtemeetoditega kalibreeritud.

Pinnete hindamiseks kasutatavad mõõteseadmed on läbi teinud väga suure arengu viimasel paarikümnel aastal. Parendatud on nii andurite tundlikkust (tänapäeval ca (0,05 –0,1) μm) kui ka analüüsimiseks kasutatavat mõõtetarkvara. NIST ja PTB on pakkunud tootjafirmadele nii kalibreerimise teenust kui ka etalone [15, 67, 86]. Selliste tööetalonide kasutamisel on erinevate pindepaksusetalonide mõõturite tootjad väljendanud pindepaksuse täpsuseks (mitte mõõtetulemuse määramatuseks) 0,5 μm + 1 % kuni 2,5 μm + 3 %. Siiski on see küsitav, kuna puudub info mida täpsuse all mõeldakse [6, 14, 15, 17, 18, 20, 24, 73]. Ka tööetalonide valmistamise täpsus ei ole suurt arengut läbi teinud. Kasutatavate tööetalonide pindepaksuse laiendmääramatus on praegu piirides (0,5 – 2) μm [15, 67, 86]. Ühelt poolt on võimalik valmistada väga täpseid etalone, kuid nende hind, mõõtepiirkond, materjalide kombinatsioonid ei vasta turu ootustele. Täpsete etalonide tootmisel on vaja teada pinde mõlema piirpinna kuju. Levinud kalibreerimismeetodi juures ei saa piirpindade kuju mõõta otse, vaid selleks kasutatakse muid tasapindu oletades, et need ühitvad piirpindadega. Selline lähenemisviis loob olukorra kus väga täpne pindepaksusetalon on ka väga kallis või odavamalt valmistatud pindepaksusetalon on ka väiksema täpsusega.

Pindepaksusetalonidega on aktiivselt tegelenud ka professor R. Laaneots Tallinna Tehnikaülikoolist. Käesolev uurimustöö ongi professor R. Laaneotsa seniste teadusuuringute edasiarendus, kaasates sinna moodsat mõõtetehnikat ja arvutustarkvara.

Doktoritöö põhieesmärk lähtuvalt ülalnimetatud probleemi püstitusest oli **leida võimalus kalibreerida erinevaid tüüpi ning erinevatest materjalidest**

**pindepaksusetalone täpsemalt ja odavamalt**. Sellest tulenevalt on doktoritöö teostamiseks ette nähtud järgmiste ülesannete lahendamine:

- Olemasolevate pindepaksuse kalibreerimismeetodite uuendamine
- Uue mõõtemetoodika välja-töötamine pindepaksusetalonide otseseks kalibreerimiseks
- Kalibreerimise mõõtemudelite mõõtemääramatuse teooria rakendamine
- Teoreetiliste mudelite kontroll praktiliste mõõtmiste käigus

Antud ülesanded ja nende lahendused ongi doktoritöö peatükkides käsitletud. Töö esimene peatükk keskendub kahele töös väljaarendatud pindepaksusetalonide kalibreerimise meetodite aluseks olevale matemaatilisele teooriale. Lahti on seletatud pindepaksuse määratlus ja sellest tulenevad erisused pindepaksuse väärtuse hindamisel. Töös on toodud kahe juhusliku piirdepinna põhjal (nende vahel on pinne) matemaatilised avaldised, mis kirjeldavad pinde paksuse arvutusvalemeid, alates pindepaksusest mingis konkreetses punktis, lõpetades nii keskmise kui ka tõese pindepaksuse avaldisega ruumis.

Töö teises peatükis on vaatlusele võetud praegu pindepaksusetalonide kalibreerimisel kõige levinum puutekombitsaga mõõtemudel. Lähtutud on asjaolust, et pindepaksusetalon on keerukas detail. Välja on arendatud nii mõõtmise mudel kui ka selle põhjal mõõtetulemuse mõõtemääramatuse arvutusmeetod. Hinnatud on kõigi mõõte mudelis olevate komponentide standardmääramatust. Arvutusnäide on koostatud Tallinna Tehnikaülikooli Mehaanika ja Metroloogia Katselaboris kasutatava mõõteseadme Perthometer Concept baasil. Kuna pindepaksuse mõõtmiseks etalonil kasutati väga suure liikuvus- ning mõõteulatusega puutekombitsat, siis on ka mõõtevahendist tingitud määramatuse komponendi väärtus suhteliselt suur. Samas kehtib väljaarendatud mõõte mudel mistahes keerukate mõõteobjektide mõõtmiseks, milleks võivad olla hoopis erikujuga pindepaksuse etalonid – näiteks muutuva väärtusega kaldetalonid.

Kolmandas peatükis on võetud vaatluse alla tuntud pindepaksusetalonide kalibreerimismeetod ning on välja arendatud lahendus selle meetodi usaldusväärsuse tõstmiseks. Töös kirjeldatakse nii mõõtemudelit kui ka selle alusel saadavate pindepaksuse mõõtetulemuste määramatuse arvutusmetoodikat. Meetodi põhimõte seiseb juhuslike pindade kirjeldamises kolmanda astme polünoomidega. Pinde all olevat pinda, mida tavapäraste meetoditega arvatakse olevat tasapind, hinnatakse kahe baaspinna järgi. Sellisel moel on võimalik pindepaksust kirjeldada funktsioonina, mitte pelgalt minimaalse, maksimaalse või keskväärtusena. Lisaks on ära toodud ka väljaarendatud spetsiaalse mõõte- ja arvutusprogrammi lühike tutvustus. Meetodite näitlikustamiseks ja selgitamiseks on teostatud vastav analüüs ning arvutused on läbi viidud ühe reaalse pindepaksusetaloni mõõteandmete põhjal. Enne kokkuvõtavat osa on tutvustatud väljaarendatud meetodiga saadud võrdlusmõõtmiste tulemusi, mille aluseks on tavapärase meetodiga ning uuendatud meetodiga pindepaksuse mõõtmine. Võrdlusmõõtmised, mis mõlemad on teostatud Saksamaal PTB's (1990 ning 2009), kinnitavad nii mõõtemetoodika kui ka määramatuse aruvutusjuhendi õigsust ja usaldusväärsust.

Neljandas peatükis on kirjeldatud väljaarendatud uudset pindepaksuse kalibreerimismeetodit. Analoogselt kolmandale peatükile on ka siin ülesehitus sarnane. Arutlus hõlmab endas nii mõõtemeetodi, selle põhjal loodud määramatuse

arvutusmudeli tutvustust kui ka reaalsete mõõtetulemuste põhjal toodud näiteid. Meetodi põhimõte seiseb siinjuures vähemalt kahel mõõtmisel. Enne pindamisprotsessi viiakse läbi etaloni aluse pinna mõõtmine, misjärel kantakse etalonile pinne. Seejärel teostatakse uuesti pindade mõõtmine. Kahe topograafilise mõõtmise tulemusena on kirjeldatud pinde pealmist ja alumist juhuslikku pinda ristlõigetes kolmanda astme polünoomiga. Meetodi reaalseks kontrolliks valmistati proovi-katsekehad, mille pindepaksuse mõõtetulemus kinnitas meetodi sobilikkust ja usaldusväärsust. Väljaarendatud uue mõõtemudeli ning mõõtemääramatuse teooria kohta, mis on koondatud ühtsesse pindepaksuse kalibreerimismeetodisse, on Eesti Patendiamet väljastanud patendi ning Saksamaa Patendiametile on esitatud patenditaotlus.

Töö järeldustes on kirjeldatud uurimustöö tulemused mis on järgmised:

- Arendati välja mõõtemudel koos määramatuse arvutusmeetodiga keerulise kujuga detailide (ka pindepaksusetalonid) mõõtmiseks.

- Parendati tuntud mõõtemeetodit pindepaksusetalonide täpsemaks kalibreerimiseks arvestades nende töötlemist tootmisel. Loodi mõõtmise matemaatiline- ja selle põhjal määramatuse arvutusmudel mille tulemusena suurenes kalibreerimise täpsus sõltuvalt pindepaksusetaloni konstruktsioonist (2 – 5) korda

- Arendati välja uudne mõõtemeetod pindepaksusetalonide odavamaks, täpsemaks ja kiiremaks kalibreerimiseks. Loodi mõõtmise matemaatiline ja selle põhjal määramatuse arvutusmudel. Metroloogiliselt jälgitava pindepaksusetaloni hind (valmistamine + kalibreerimine) vähenes tunduvalt ning samuti suurenes täpsus (sõltuvalt objektist) kuni 10 korda. Meetod võimaldab kalibreerida peaaegu mistahes konstruktsiooniga pindepaksusetalone.

Uurimustöö eesmärkide täitmiseks:

- Teostati kaasaegsete pindepaksuse mõõtemeetodite, pindepaksusetalonide ja nende kalibreerimissüsteemide analüüs.
- Täiustati, eksperimentaalsete katsete põhjal, keerukate detailide mõõtmise mõõtemudelit.
- Loodi arvutustarkvarad kahele uudsele pindepaksusetalonide kalibreerimismeetodile.
- Viidi läbi TTÜ pindepaksusetalonide võrdlusmõõtmised Saksamaal PTB-s.
- Valmistati prototüüpetalonid väljaarendatud pindepaksusetalonide kalibreerimismeetodi usaldusväärsuse kontrolliks.
- Patenteeriti pindepaksusetalonide pindepaksuse mõõtemeetod Eestis ja esitati patenditaotlus Saksamaa Patendiametile
- Osaleti TTÜ Mehaanika ja Metroloogia Katselaboriga 2010. aastal rahvusvahelises pinna geomeetria võrdluskalibreerimisel (SIM-EURAMET L-K8 *Surface roughness comparison project No* 1003, projekti koordinaator Prantsusmaa rahvuslik metroloogiakeskus LNE).

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2. Abiline, I., Laaneots, R., Nanits, M., Riim, J. Measurement uncertainty of surface contour with complicated form. –*Proceedings of the 5th International Conference of DAAAM Baltic Industrial Engineering 2006, Tallinn, Estonia,* 101–105.
3. Kulderknup, E., Riim, J., Levandi, T. Uncertainty of road traffic safety measurements. – *Proceedings of the XIX IMEKO World Congress, 06-11.09.2009, Lisbon, Portugal*, 1439–1442.
4. Laaneots, R., Riim, J. Evaluation method of coating thickness of coating thickness standard. – *Proceedings of the 14 International Congress of METROLOGIE, Paris-France, 22–25 juin 2009.*
5. Laaneots, R., Abiline, I., Nanits, M., Riim, J. Method for the calibration of thickness standards : Estonian Patent. EE 05164 B1, 2006. [*Online*] esp@cenet (10.12.2010). (in Estonian)
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9. Riim, J. Topographical calibration method of coating thickness standards. – *Proceedings of the 7th International Conference of DAAAM Baltic INDUSTRIAL ENGINEERING, 22–24th April 2010, Tallinn, Estonia*, 2010 238–244.

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| Tallinna Tehnikaülikool | 2005 | Tootearendus/ bakalaureus |
| PTB - Saksamaa Riiklik Metroloogiakeskus | 2009 | |
| Fachhochschule Kiel | 2003 | |

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| Keel | Tase |
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| 2011- | Flexa Eesti AS | Kvaliteedijuht |
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| 2006-2008 | Qvalda Tools OÜ | Arendusjuht |

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7. Kaitstud lõputööd

„Pinna geomeetriat iseloomustavad suurused ja nende mõõtmine“ Magister (Mehhatroonika), Tallinna Tehnikaülikool, 2007

„Automatiseeritud mõõtesüsteem konstruktsioonielementide laboratooriumile“ Bakalaureus (Tootearendus), Tallinna Tehnikaülikool, 2005

8. Teadustöö põhisuunad

Pinna geomeetriat iseloomustavate suuruste mõõtmine, mõõtekompleksi arendus ja mõõtmiste jälgitavus

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| Tallinn University of Technology | 2007 | Mechatronics/ Master |
| Tallinn University of Technology | 2005 | Product development/ Bachelor |
| PTB German National Metrology Insitute | 2009 | |
| Kiel University of Applied Sciences | 2003 | |

4. Language skills

| Language | Level |
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| Estonian | Mother tongue |
| German | High level |
| English | Average |

5. Special Courses

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| Assessor training | Estonian Accreditation Centre |
| Legal protection of intellectual property rights and information as part of business development | Estonian Patent Information Centre |

6. Professional employment

| Period | Organisation | Position |
|---|---|---|
| 2011 | Flexa Eesti AS | Quality manager |
| 2008-2011 | Estonian Accreditation Centre | Lead auditor |

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| 2006-2008 | Qvalda Tools Ltd | Development manager |
| 2004-2006 | Estonian Accreditation Centre | Leading Auditor |

7. Defended thesis

„Quantities characterizing surface texture and their measurement“ M. Sc (Mechatronics), Tallinn University of Technology, 2007

„Automated measuring system for the laboratory of construction elements“ B. Sc (Product development), Tallinn University of Technology, 2005

8. Main research interest

Methods of measurement and estimation of accuracy of surface geometry characteristics

## DISSERTATIONS DEFENDED AT TALLINN UNIVERSITY OF TECHNOLOGY ON *MECHANICAL AND INSTRUMENTAL ENGINEERING*

1. **Jakob Kübarsepp**. Steel-bonded hardmetals. 1992.
2. **Jakub Kõo**. Determination of residual stresses in coatings & coated parts. 1994.
3. **Mart Tamre**. Tribocharacteristics of journal bearings unlocated axis. 1995.
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6. **Heinrich Reshetnyak**. Hard metals serviceability in sheet metal forming operations. 1996.
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