THESIS ON MECHANICAL AND INSTRUMENTAL ENGINEERING E44

Calibration Methods of Coating Thickness Gauges

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INTRODUCTION

Background

In industry and agriculture, a wide range of various coverings is used, especially in radio technology and electronic industry, aviation, car industry, machine industry, etc. The steep growth of metallic, dielectric and special coverings in machine and apparatus industry, space technology and other fields of technology have created an increased demand for precise and reliable measurement of the latter. The thickness of a coating is its basic parameter, on which such indicators of machines in general, as well as of their separate parts, like wear resistance, resistance to external conditions, depend. It has been figured out that approx. 10 per cent of the annual steel production is destroyed as a result of corrosion. Another example is from electronic industry, in which about 90 per cent of the hidden defects have been detected in the initial stage of production and as a result of precise measurement of the covering of microcircuits.

That is why more and more of various coating thickness gauges are produced for non-destructive measurement of coatings. All of them rely upon the mutual connection between the coating thickness and underlying material and allow coating measurement without destroying or scratching it. The number of coating measurers is increasing year by year, and so is their quality.

From the industrial point of view, it is essential that all the coating thickness measurements should be connected with the international primary standard of length. It is possible to guarantee this requirement in case calibrated measurers are used. The latter allow correct measurement when using a variety of measurers for the same coating thickness. Hence, there arises the necessity of using such metrological methods, which would accelerate the checking and calibrating of the measurers and enhance their reliability. Therefore, the fact that in general the coating thickness measuring is carried out in unstable circumstances. Enhancing precision of measurement in the conditions requires usage of special equipment as well as providing the corresponding methods, to guarantee precision of coating thickness gauges in their working sphere, irrespective of the external conditions.

The above-mentioned fact imposes greater expectations for adjustment and other auxiliary measures, which find use when measuring coating thickness. To guarantee a close connection between primary and working standards and transition of their size to the coating thickness gauges, the following tasks should be solved:

- simultaneous measurement of the various characteristics of the coating and its thickness (electric conduction, magnetic penetrability, dielectric constant, porosity, etc);
- concentration of coating thicknesses, which have been obtained in points considerably distant from each other, their relation to coating thickness measuring results ;

- registration, maintenance and reproduction of coating thickness measurements and treatment of the results of the above;
- presenting the coating thickness measurement results in a way suitable for decisions regarding the state of coating thickness gauges.

It follows from the facts mentioned above that the task of measuring coating thickness is relatively complicated. The solution of the latter requires a multiple analysis of the methods given, coating thickness gauges and conditions as well as a search for new perspective methods and means, which allow to increase the efficiency of measuring procedure.

Problem setting

In connection with the rapid industrial increase, a demand regarding the quality of the industrial production has increased as well. The latter, in its turn, has raised a necessity for new and modern measuring devices. The measuring methods of coating thickness have undergone a rapid development during the last few decades. Companies are constantly launching to the market new models of coating thickness gauges, which have a modern design and allow more and more precise and convenient measuring of coating thicknesses used in industry. All gauges require regular calibrating. In order to increase the reliability of calibrating, new high-quality working standards of coating thickness are required. Until now, Prof. R. Laaneots from Tallinn University of Technology, a well-known specialist both here and in the spheres of metrology in Europe, has taken an active part in dealing with this issue. His research on the subject above was already published in the 1980s [1].

Rein Laaneots began his activity as an inventor when studying in D.I.Mendelejev's Institute of Scientific Research of Metrology, in which, as a result of research, he devised the first constructive solutions for coating thickness standards along with methods, how to produce them. In addition to developing the coating thickness standards, he invented new techniques for calibrating the latter. Calibrating coating thickness standards were to be performed relying on contact, pneumatic or interferential methods worked out by Rein Laaneots.

The first standards were produced, instructed by Rein Laaneots, basically from precious metals (gold, platinum, silver, etc) in the military plants of the former Soviet Union. In the years 1980 to 1985, he improved on the constructive approaches of coating thickness standards and samples of the latter were produced in the Experimental Laboratory of Tallinn Polytechnic Institute in 1988. Rein Laaneots, the author, and TPI as the applicant, were awarded more than 30 certificates of authorship of the USSR for the new technical developments, i.e. constructions of coating thickness standards, methods of production of the latter, imitators of standards, methods of calibration and measuring devices of the standards. Since the technical level of the Estonian SSR of that time did not allow industrial production of coating measuring standards, the technical solutions could not be applied or the corresponding coating thickness standards produced. Around

ten of the inventions, however, found application in the USSR. The scientific research association NPO “Isari“ was appointed as the producer and primary calibrator and coating thickness standards and calibration methods devised by Rein Laaneots came to be applied in the military industry, rocket and spaceship technology of the USSR.

Since measurers of coating thickness are in a constant process of development and improvement, the coating thickness standards and methods of calibration have also to be developed. The current paper is further development of the work and studies of Prof. R. Laaneots, considering the new trends in developing coating thickness gauges as well as their production.

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ABBREVIATIONS AND SYMBOLS

BIML	International Bureau of Legal Metrology
BIPM	International Bureau of Weights and Measures
CGPM	General Conference on Weights and Measures
DIS	Draft International Standard
EA	European co-operation of Accreditation
EAC	European Accreditation of Certification
EAL	European co-operation for Accreditation of Laboratories
EUROMET	European Collaboration in Measurement Standards
GOST	Russian Organization for Standardization
IMEKO	International measurement Confederation
ISO	International Organization for Standardization
IU	International unit
LSQ	Least squares
MI	Measuring instrument
NMI	National Metrology Institute
PTB	Physikalich-Technische Bundesansalt
SI	Systeme International d'Unites (International System of Units)
TUT	Tallinn University of Technology
D,d	Diameter
F_M	Attractive force of permanent magnet
F_V	Reaction, needed to lift the magnet
G	Maximum permissible error
h_{m}	Mean coating thickness
$h_{\max}$	Maximum coating thickness
$h_{\min}$	Minimum coating thickness
K	Correction to the indication
K_1	Factor, describing the magnetic characteristics of permanent magnet
K_2	Factor, describing the geometrical characteristics of permanent magnet
K_3	Factor, describing the sensitivity of permanent magnet (measuring instrument) against the coating thickness
k	Coverage factor
l	Length of permanent magnet
M	Magnetic moment of permanent magnet
$R_{\mathrm{max.}}$	Surface roughness parameter
R_{z}	Surface roughness parameter
r	Curved radius of permanent magnet tip
U,u	Expanded uncertainty, standard uncertainty
X	Measurand
x	Indication
ζ	Measurement signal

1 CALIBRATION OF MEASURING INSTRUMENTS

1.1 General aspects

Calibration is defined as the set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument (or a measuring system), or values represented by a material measure or a reference material, and the corresponding values realised by measurement standards [3]. The objective of calibration of measuring instruments is to ensure traceability (see *Figure 1.1*) of results of measurement.

The relationship between input quantities (measurands, stimuli) and corresponding output quantities (indications, responses) obtained in the course of calibration can be presented in the form of a table, graph, or an equation. Calibration yields an estimate of the error of indication, together with an estimate of its uncertainty. This enables us to determine the correction that should be applied to indications obtained when using the calibrated measuring instrument. The convention is that a correction is equal to the negative of the estimated error of indication. It has the same uncertainty as the estimated error of indication. Sometimes the compensation for an error of indication is through a numerical factor (correction factor) by which the indication has to be multiplied. The result of a calibration is usually recorded in a document, sometimes called a *calibration certificate* or a *calibration report*.

Before ordering calibration of a measuring instrument, it should be established that the calibration laboratory intended to be employed for the task is capable of providing traceability, i.e. that its measurement standards are regularly calibrated by other bodies whose measurement standards in their turn provide results that are traceable to stated references. This traceability chain should make it possible to trace the calibration result back to a standard that is acceptable for the customer. This task can be quite difficult for an ordinary owner of a measuring instrument. However, by using an accredited calibration laboratory, the traceability issues is solved automatically. *Accreditation* of a laboratory means in general a formal recognition is granted by an *accreditation body* that is specially appointed to run a laboratory accreditation system in a particular state.

1.2 Traceability

All efforts to obtain a valid estimate of the measured quantity and its associated uncertainty may turn out to be futile, if it becomes evident that the result is not traceable. According to its definition [2], traceability is a property of the results of a measurement or the value of a standard whereby it can be related to stated references through an unbroken chain of comparisons, all having stated uncertainties. It is stated that these references are usually national or international standards.

The metaphor of a chain means that there is a hierarchy of comparisons, where the measurement standard used for calibration of a measuring instrument at one level will be calibrated against a measurement standard of higher quality at the next higher level (see *Figure 1.1*). Starting out from an international standard, it is possible to draw up a traceability chain to be used for relating the metrological properties of measuring instruments to the primary measurement standard of the quantity concerned. Depending on the needs and the economic potential of a state, the competent metrology body of the state draws up such a traceability chain for each of the quantities that are of interest for the state. This chain has to reflect the traceability of the national standards of the country to corresponding international standards. The scheme in *Figure 1.1* illustrates the principle of the traceability chain for an arbitrary quantity in a state that is at an average level of economic development. The arrows in the scheme indicate the flow of traceability from standards of higher levels down to results obtained at laboratory and workshop level.

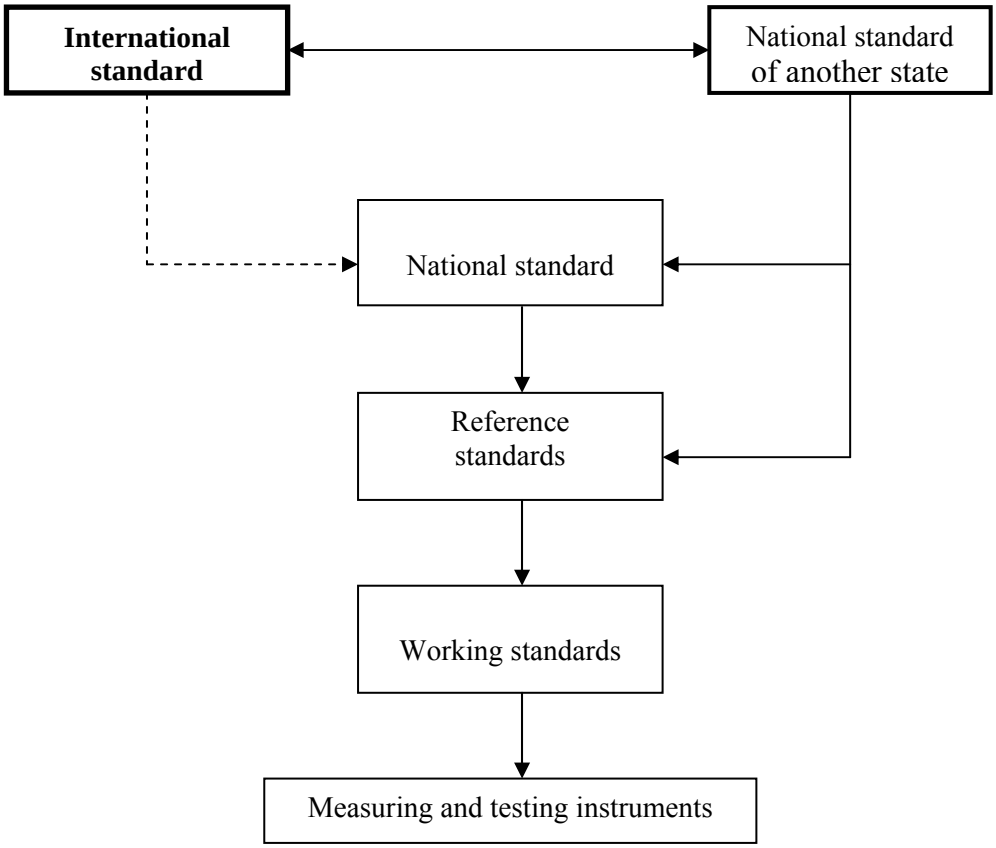

Figure 1.1 Traceability chain of measurement standards and working instruments

1.3 Measurement standards

A *measurement standard* is a device that is intended to define, realise, conserve, or reproduce a unit or more values of a quantity, to serve as a reference [3]. A material measure, a measuring instrument, a reference material or a measuring system can be a measurement standard. For example, a mass standard of 1 kg and a 100 Ω standard resistor are material measures, reproducing the mass unit and a value of electric resistance respectively. A standard ammeter is a displaying measuring instrument; it reproduces values of electric current. A caesium frequency standard can be characterised as a measuring system; it realises the unit of time interval. A less common alternate term to measurement standard is *etalon*.
A set of similar material measures or measuring instruments that, through their combined use, constitutes a standard is called a collective standard. A set of standards of chosen values that, either individually or in combination, provides a series of values of quantities of the same kind is called a group standard.

An international measurement standard is a standard that is recognised by an international agreement to serve internationally as the basis for assigning values to other standards of the quantity concerned. International measurement standards are usually at the level of primary standards (see below). There is only one international standard in the world that complies strictly with the definition: the mass standard that is kept at the BIPM and which, since the 1889 resolution of the CGPM, realises the mass unit of 1 kg.

Measurement standards within a state can be divided into national, reference and working standards (see *Figure 1.1*). A measurement standard is given the status of a national standard by a national decision that is reflected in suitable legislation and recognises the standard to serve in that state as the basis for assigning values to other standards of the quantity concerned. This means that there are no special quality requirements for a standard to be identified as a national standard. Its metrological level depends on the needs of science and industry and on the economic potential of the state concerned. Metrologically, a national standard can be a primary or a secondary standard or even of still more inferior quality.

A *primary standard* is defined as a standard that is designated or widely acknowledged as having the highest metrological qualities. Its value is accepted without references to other standards of the same quantity, while a *secondary standard* has its value assigned in comparable to base quantities as well as to derived quantities. In both cases, the aim is to use physical phenomena that can be reproduced with a high degree of accuracy or even be considered invariant, cf. the definitions of all SI base units except the unit of mass [3].

The term primary standard is another that is often misused: in this case to designate the standard that has the highest metrological quality available at a given location or in a given organisation. According to [3], such a standard should instead be called a *reference standard*. As shown in *Figure 1.1*, a reference standard can be traceable to the national standard of the state concerned or of another state. As a rule, reference standards are not used for calibration of

measuring instruments. For that purpose, *working standards* are used as intermediaries. In large organisations, there may be differ considerably from those of the corresponding reference standard. Working standards that are used routinely to ensure that measurements are carried out correctly are sometimes called *check standards*.

1.4 Measuring instruments

A measuring instrument is a technical device with defined metrological characteristics, intended to make measurements, either alone or in conjunction with supplementary devices [3]. Measuring instrument is a general term that includes sensors, transducers, indicating, and integrating instruments, as well as material measures, measurement standards, reference materials and complete sets of equipment that constitute measuring systems or measuring installations.

Every measuring instrument includes an element that is directly affected by the measurand. This element is called the sensor or the primary element of a measuring instrument (see *Figure 1.2*).

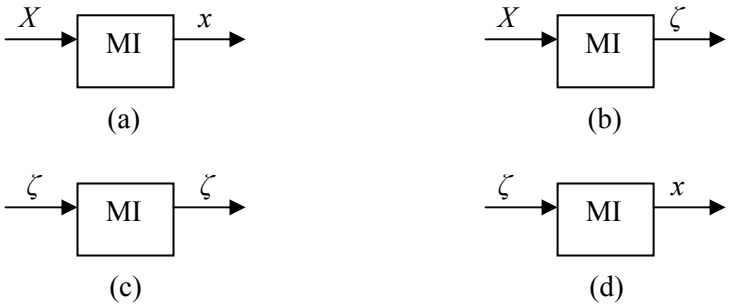

Figure 1.2 Possible designation of measuring instruments in structural schemes of measuring system: (a) - *measurand at input and indication at output,* (b) - *measurand at input and measurement signal at output,* (c) - *measurement signal at both input and output*, (d) - *measurement signal at input and indication at output*

1.5 Main objectives of the thesis

Since the issue raised above needs to be dealt with, the basic goal of the given doctoral thesis is **research on the precision characteristics of modern coating thickness gauges, development of the working standards of coating thickness measurement, development of calibration method of coating thickness gauges and standards as well as elaboration of a new technique of calculation of uncertainty of calibration results.**

The chief goals set by the author are as follows:

- research into precision of modern coating thickness gauges,

- elaboration of measurement model for magnetic coating thickness gauges,

- elaboration of new method for determining coating thickness and make its mathematical analysis,

- development of new uncertainty calculation method for determining coating thickness indeterminacy,

- elaboration of method for measuring the contour of the working standard of coating thickness,

- development of calculation method for the uncertainty of the given method,

- elaboration of new calibration method for coating thickness gauges, directly applying a device for measuring length as a working standard.

2 CALIBRATION OF COATING THICKNESS STANDARDS

2.1 Coating thickness standards. General description

Coating thickness standards represent devices for measuring coating thickness, which are designated for representing and maintaining the coating thickness required [1].

Coating thickness standards may be natural and equivalent. Coating thickness standards representing combination of the coating and the underlying material and which have been made from the same materials the measurement of which they have been designed are called natural coating thickness standards. Those coating thickness standards, however, which are to determine the required thickness ratio between coatings of a certain group and underlying materials, are called equivalent coating thickness standards. Take, for example, the copper-on-steel coating thickness standards, which are applied for calibrating coating thickness measuring devices with the help of which the thickness of nonmagnetic surfaces on magnetic foundations can be measured.

Sometimes, however, coating thickness is measured applying indicators of thickness, instead of coating thickness standards. An indicator is a device for transmitting signals to the measurer of coating thickness, which are identical to the initially obtained coating thickness.

Depending on the number of coating thicknesses, the coating thickness standards can be divided into single-value and multi-value groups. Single-value standards reproduce coating thickness of an individual value, whereas the multi-value standards represent coating thicknesses of the same type with a variety of values.

According to the field of application, the coating thickness standards can be divided into working standards and sustaining standards. The working standards are the coating thickness standards, which are used for calibrating the coating measuring devices. The sustaining standards, however, are those applied to calibrating the working standards. In addition, adjustment standards, which can in certain conditions applied as working standards, are used.

All the coating thickness standards used have a working surface, i.e. a part of the coating, to which certain norms apply. The value, attributed to the coating thickness standards after employment test, is the nominal value of the coating thickness standard and recorded on it.

2.2 Single-value coating thickness standards

Prof. R.Laaneots has dealt with a variety of coating thickness standards and the things connected with them [1]. He has described the first coating thickness standards, which are quadrangular steel plates measuring 30 mm x 30 mm x 10 mm, the centre of which was covered with a coating of 12 mm x 12 mm. Guaranteeing the parallel nature of all the three major technical requirement. The

three surfaces above incorporated the topmost and bottom part of the steel plate and the surface of the covering.

The graded coating thickness standards elaborated are spherical or quadrangular in shape. The latter is more preferable and widespread since, during its employment test, it is comfortable to connect it to the coordinate system.

The basic technical requirements for producing such coating thickness standards are a plane working surface of the underlying material and the smoothness of the top surface of the underlying material as well as that of the covering. A single-value coating thickness measuring standard has been represented in *Figure 2.1*.

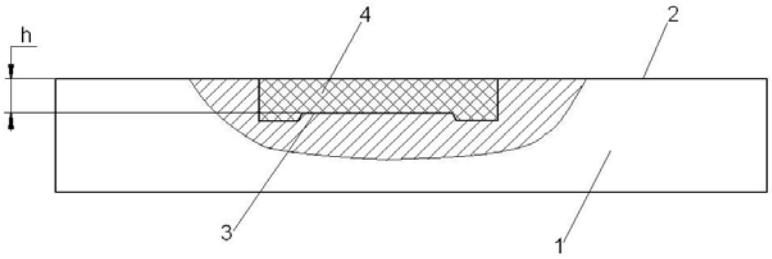

Figure 2.1 Single-value coating thickness standard: 1 – *the underlying material,* 2 – *the top surface of the underlying material,* 3 – *the base surface;* 4 – *the coating surface*

In addition to the requirements above, the coating thickness standards have to be devoid of any cracks, furrows, traces of corrosion and other surface defects.

2.3 Multi-value standards of coating thickness

To calibrate the whole scale of the coating thickness measurers, as many as 8 to 20 coating thickness standards with a variety of nominal values are applied, depending on the type of coating thickness measurer. The latter makes the calibrating a complicated and labour-consuming process. In order to avoid it, multi-value coating thickness measurers are used instead of the single-value standards. As it has been mentioned before; multi-value coating thickness standards represent the same type of coating thicknesses with different values. They may be either wedge-shaped or multi-step. A wedge-shaped coating thickness standard has been presented in *Figure 2.2*.

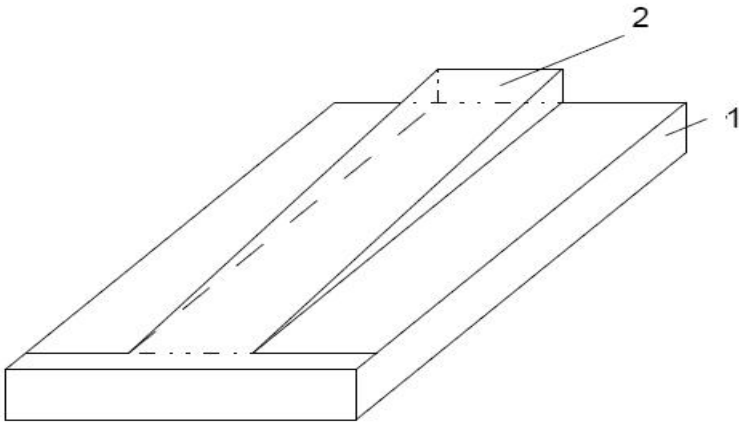

Figure 2.2 A wedge-shaped multi-value coating thickness standard: 1 – *underlying material,* 2 – *wedge-shaped covering*

In addition, the coating thickness measurers of the so-called alternating thickness are used (*Figure 2.3*) that type of a standard consists of the foundation material 1 and surface 2. The foundation material under the covering has a cylindrical or spherical shape with an approximate radius R. The central part of the underlying material may be level in order to allow adjustment of the neutral point (zero) of the coating thickness measurer. In the case above, the coating is placed on the underlying material so that their surface levels off with the top surface of the underlying material (*Figure 2.3*), where l_0 – starting distance, l_i – measurement distance, h_i – coating thickness, R – radius of underlying material.

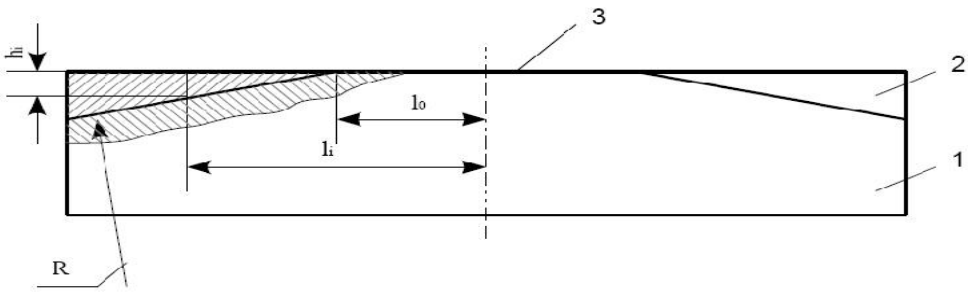

Figure 2.3 Coating thickness standard with an alternating coating thickness: 1 – *foundation material,* 2 – *coating,* 3 – *central part of the underlying material*

2.4 Research results of the coating thickness standards

The coating thickness standards will be used to represent, hold and reproduce the certain value of the coating thickness. The available coating thickness standards

were worked out by prof. R. Laaneots and taken into use in the former Soviet Union in 1971-1972.

In general the coating thickness standards look like cuboids made of steel, where in the middle of the surface, there is a galvanic ally covered or dispersed with a coating. Our research object is the set of coating thickness standards "Nickel-Steel 20" made in 1988 by NPO "Isari". These coating thickness standards have been used in Tallinn University of Tecnology as working standards for calibration of the coating thickness gauges. The abovementioned standards have been inspected from time to time and the changing of qualities has been analysed.

In the presentation, the research results of the mentioned coating thickness standards have been expressed. The results show us that developed coating thickness standards have not remarkably changed their metrological qualities.

As mentioned above, the coating thickness standards have been worked out to represent, hold and reproduce the certain value of the coating thickness. The first coating thickness standards in the world were worked out by Rein Laaneots and were first used in the former Soviet Union in the early seventies [1]. Those were the cuboids made of steel, where, in the middle of the surface, there is a galvanized or dispersed coating. The coating measurements were 10 mm x 10 mm or 20 mm x 20 mm. The important criterion was, that the non-flatness and parameter of the surface roughness R_z couldn't exceed 0,05 μm to 0,1 μm. The coating thickness of these standards was from 1,5 μm up to 100 μm. The coating thickness was measured using the contact, pneumatic or interference method so. that the expanded uncertainty of measuring results not exceed (0,1 + 0,05h) μm, where h is coating thickness in micrometers measured in the standard. There were also coating thickness standards with coatings made from precious metal (platinum, gold, silver etc.) Hereafter these coating thickness standards were standardized (GOST 25177-82). According to the abovementioned standard has made also the TUT set of coating thickness standards "Nickel-Steel 20" (10 different coating thicknesses). These coating thickness standards were made in 1988 and the manufacturer and first calibrator was NPO "Isari" in Tbilisi [4].

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

2.4.1 Theory

In this research we have studied the case where a coating is formed on a surface with realistic detail. In this case the boundary surfaces can be expressed as follows,

$$z_1 = f_1(x, y)\,;\; z_2 = f_2(x, y)\,, \tag{2.1}$$

where $f_1(x,y)$ is the equation describing the surface of the outer layer of the coating and is contiguous with the ambient medium; $f_2(x,y)$ is the equation describing the substrate surface. The local film thickness h_i at the standardized patch Δ of the outer coating layer at a point with coordinates x_i and y_i is in agreement with [5]. The latter can be stated in the following form,

$$h_i = \min_{x,y\in\Delta} \sqrt{\left[f_2(x,y) - f_1(x_i,y_i)\right]^2 + (x-x_i)^2 + (y-y_i)^2} \qquad (2.2)$$

Local film thickness is the minimum spacing between the two surfaces which bound the layers of coating material from a particular point on one of the boundary surfaces to the other surface. Since the outer surface of the coating is described by the function $f_1(x,y)$ in equation (2.1) and the substrate surface by $f_2(x,y)$, this allows describing coating thickness with the aid of a coating thickness function. Under the conditions where coating thickness can be represented as the spacing between two surfaces bounding the layers of the coating material, along the normal to one of the bounding surfaces [in this case along the normal to surface $f_1(x,y)$], the mathematical function for coating thickness is described by the equation (2.3),

$$h(x,y) = \sqrt{\left|\nabla f_1\right|^2 + 1} \cdot F(x,y), \qquad (2.3)$$

where $h(x,y)$ is the coating thickness function; ∇f_1 is the gradient of the function $f_1(x,y)$, which is defined by the relation $\nabla f_1 = (\partial f_1/\partial x, \partial f_1/\partial y)$; $F(x,y)$ is a function that satisfies the constraining equation:

$$f_1(x,y) - F(x,y) = f_2\left[x + \frac{\partial f_1}{\partial x}F(x,y), y + \frac{\partial f_1}{\partial y}F(x,y)\right] \qquad (2.4)$$

The introduction of the coating thickness function actually simplifies the mathematical description of the minimum thickness $h_{\min}$, the maximum coating thickness $h_{\max}$ and the mean coating thickness h_m:

$$h_{\min} = \min_{x,y\in\Delta} h(x,y);$$

$$h_{\max} = \max_{x,y\in\Delta} h(x,y);$$

$$h_m = \frac{1}{S}\iint_{\Delta} h(x,y)dxdy\,. \qquad (2.5)$$

Here Δ is the standardized patch, where the thickness function $h(x, y)$ is defined; S is the area of the standardized patch.

The coating thickness standards reproduce the mean coating thickness h_m.

2.4.2 Previous calibration results and analysis

Coating thickness standards with nickel coating were calibrated in 1988 in NPO "Isari".

Measurement of the coating thickness proceeded by the contact method and there was used a coating thickness measurement station KSP MTP-2 [6]. Mentioned coating thickness standards were calibrated 7 items in 1997 in TUT chair of metrology and measurement technique using the magnetic measuring principle and the coating thickness gauge MIKROTEST III/IV NiFe50 made in Germany by company "ElektroPhysik". With reference to the research of the TUT and PTB (Physikalisch-Technische Bundesansalt) the set of coating thickness standards "Nickel-Steel 20" was sent to the PTB, where these standards were calibrated in 1999. In PTB there were calibrated four coating thickness standards using the x-ray measuring station "X-RAY 1600" made by company "Fischer". The whole set was calibrated in PTB using the surface roughness measuring device "Perthometer S8P" made by company "Perthen" [4]. The average coating thickness h_m 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. The expanded uncertainty U of the measuring result h_m has been calculated using the method described in instruction EA-4/02 [7]. The expanded uncertainty U of the measuring result h_{m} has been calculated from the equation $U = k \cdot u(h_m)$, expecting the confidence level of 95 %, when coverage factor k = 2 and $u(h_m)$ is combined standard uncertainty of measurement. Calibration results have presented in *Table 2.1*.

Table 2.1. Calibration results of the coating thickness standards

Calibration standard No	NPO "Isari" in 1988 contact method		TUT in 1997 magnetic method		PTB in 1999 X-Ray method		PTB in 1999 contact method		TUT in 2005 with laser probe LS10	
	h_m/μm	U/μm	h_m/μm	U/μm	h_m/μm	U/μm	h_m/μm	U/μm	h_m/μm	U/μm
MO nr.1055	5,4	0,16	5,0	0,25	5,34	0,20	5,45	0,15	5,5	0,20
MO nr.1054	11,5	0,20	11,3	0,60	10,8	0,30	11,0	0,30	11,1	0,40
MO nr.1021	16,2	0,30	16,0	0,80	15,8	1,20	16,0	0,90	16,1	0,80
MO nr.1001	27,4	0,50	24,0	1,20	27,1	1,90	27,1	1,60	26,9	1,60
MO nr. 832	38,4	0,60	34,0	1,70			37,9	1,20	36,8	2,10
MO nr.1049	50,0	0,70	44,0	2,10			49,6	0,80	48,0	1,60
MO nr.1016	54,0	0,70	49,0	2,50			52,2	0,80	51,8	1,90
MO nr. 860	73,5	1,00					72,8	2,20	72,4	1,90
MO nr. 816	90,2	1,20					90,4	2,30	87,8	2,50

2.4.3 Measurement of the coating thickness standards of TUT

The abovementioned coating thickness standards were measured using the surface texture measuring system Perthometer Concept made by company MAHR [8]. The Laboratory of Metrology of Tallinn University of Technology owns this system about one year. Perthometer Concept (see *Figure 2.7*) is a modular computer-controlled station for measuring and analysing roughness, contour and topography. The Perthometer Concept software runs under the worldwide Windows user interface. Operation is therefore quickly learned, easy to understand, and compatible with other Windows applications. We used for our research the PGK 120 drive unit 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 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 work on the principle of dynamic focussing (see *Figure 2.6*). 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.

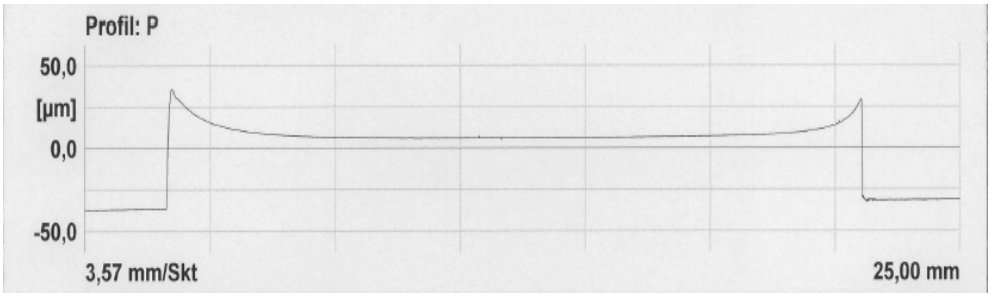

Figure 2.4 Profile P of the measured coating thickness standard by Perthometer Concept

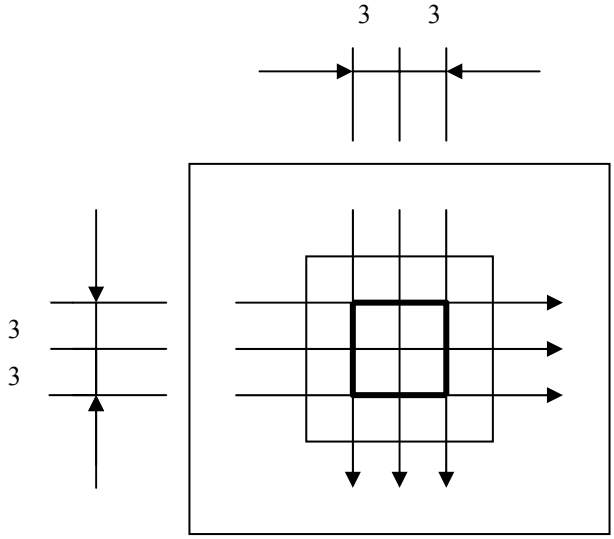

Figure 2.5 Measurement scheme used with Perthometer Concept

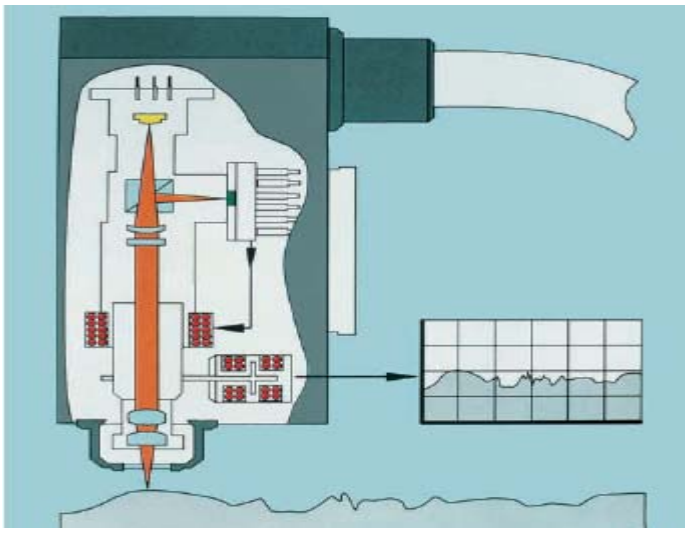

Figure 2.6 LS10 laser pick-up

Figure 2.7 Perthometer Concept

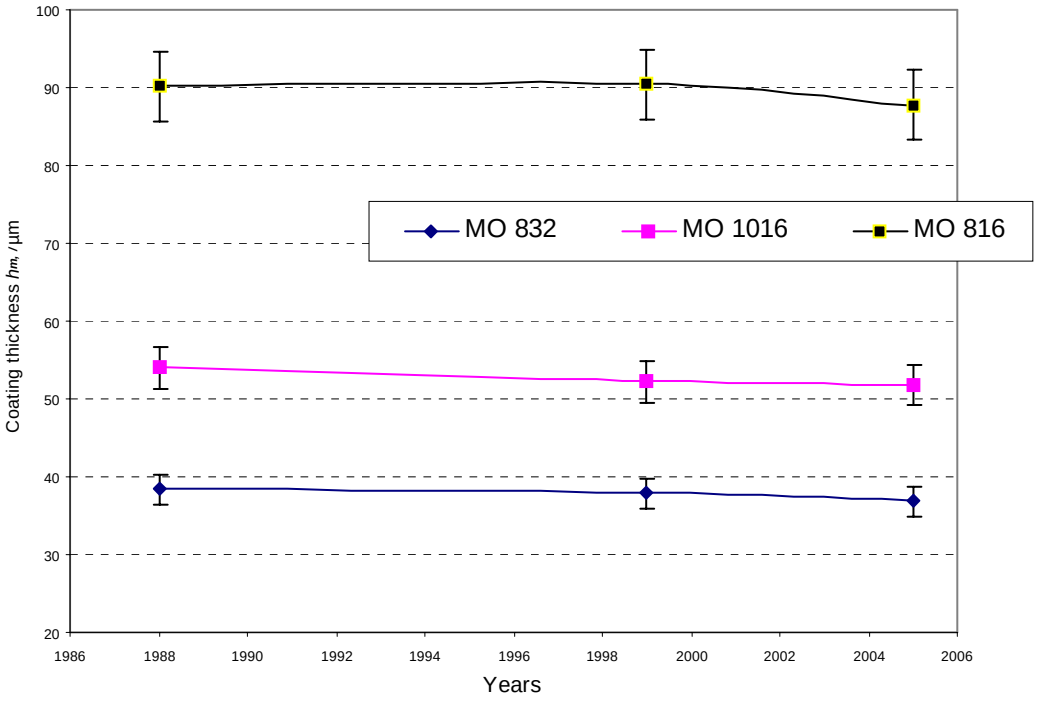

Figure 2.8 Stability of coating thickness of the coating thickness standards

2.4.4 Conclusions to the subchapter 2.4

Results of long-lasting research show us, that the mentioned coating thickness standards are stable (see *Figure 2.8*). They have been used for almost thirty years. In spite of this time the coating thickness is still nearly stable. We recommend using this type of coating thickness standards in every calibration application. They are most suitable working standards for calibration of the coating thickness gauges because they are natural and imitate the coating in the base material better.

Also we can see from the research results, that the magnetic method used in 1997 is inappropriate for calibrating the coating thickness gauges. This method magnetized the nickel coating of the standards (see *Table 2.1*). Since the calibration results had a relatively high expanded uncertainty *U* and we had to give this method up.

2.5 The coating thickness and its definition

Coating on the certain object will be formed between the boundary surface of surrounding environment and coating and between the boundary surface of object base and coating.

Really, the boundary surfaces are not parallel with each other, but, depending on production technology, have deviations in geometry as well as in roughness. For that reason, in defining the surface coating thickness, terms shall be used: local thickness (in a fixed point), and maximal and minimal thickness.

The paper presents the definition for surface coating thickness between the real coating surfaces. A mathematical model, which using Monte-Carlo iteration method, gives statistical distribution description for the coating surface, proves the definition. The definition proposed for surface coating thickness is checked through practical tests, which allows evaluating appropriateness of theoretical considerations elaborated.

For the surface coating the thickness is one of the most important characteristics. However, there does not exist any internationally accepted definition for the surface coating thickness up the present time. Considering basic principles of metrology, the surface coating thickness can be defined as the interval 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 [9]. Above definition is valid, however, in case of perfectly plane and parallel boundary surfaces. Really, the boundary surfaces are not parallel with each other, but, depending on production technology, have deviations in geometry as well as in roughness. For that reason, in defining the surface coating thickness, terms shall be used: local thickness (in a fixed point), and maximal and minimal thickness [10].

2.5.1 Theory

2.5.1.1 Coating thickness of a plane object of measurement

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. 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 2.12*), the random function of the covering can, in general, be represented (according to [11]) as follows:

$$h = Z_g - Z_s = f_1(X,Y) - f_2(X,Y), \tag{2.6}$$

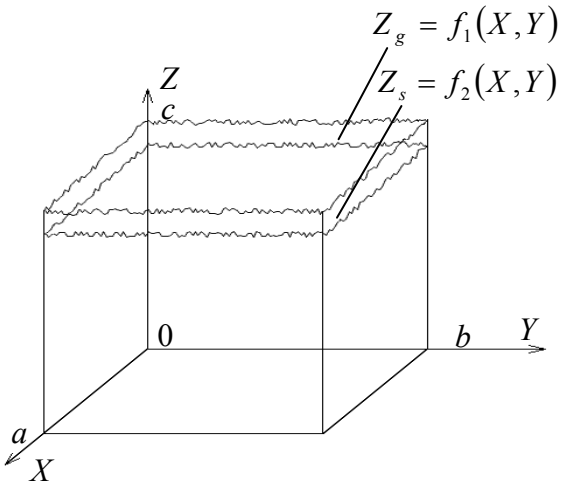

Figure 2.9 Covered element

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 2.10*), the mean thickness of coating in the intersection from y_1 to y_2 can be determined in the following relation

$$\underset{X=X_0}{h_m} = \frac{1}{y_2 - y_1} \int_{y_1}^{y_2} [f_1(X_0, Y) - f_2(X_0, Y)] \mathrm{d}y \tag{2.7}$$

In intersection 0*XZ*, in which the shape of the element of the object of measurement is analogous to the one presented in *Figure 2.13*, the mean coating thickness of the object in the intersection from a_1 to a_2 can be determined similarly:

$$\underset{Y=Y_0}{h_m} = \frac{1}{x_2 - x_1} \int_{x_1}^{x_2} [f_1(X, Y_0) - f_2(X, Y_0)] \mathrm{d}x \tag{2.8}$$

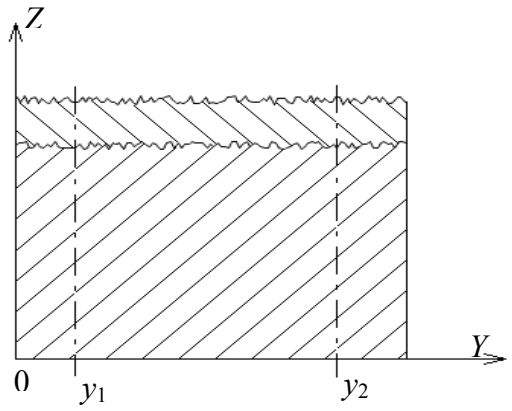

Figure 2.10 Element of the object

2.5.1.2 Coating thickness of a coating thickness standard

For determining, sustaining and reproducing a certain value of coating thickness, coating thickness standards are applied [12, 13]. 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 2.11*).

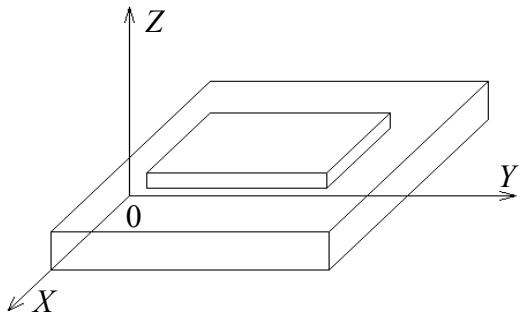

Figure 2.11 Coating thickness standard

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 2.12*.

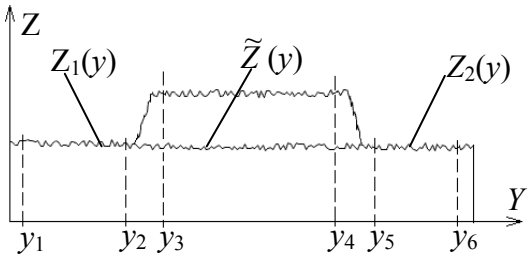

Figure 2.12 Random functions characterizing the top surface

When observing this coating thickness standard in an intersection parallel to axis *X*, the obtainable shape is analogous. The problem here underlies 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 2.12*). 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. 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. 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 $\widetilde{Z}(y)$ 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 $Z_1(y)$ and $Z_2(y)$, or their

estimates, which characterize profiles in the range from y_1 to y_2 and y_5 to y_6, and, secondly, what to do to determine the coating thickness

$$\widetilde{Z}(y)=\frac{y_4-y}{y_4-y_3}\cdot Z_1\left(\frac{y_2-y_1}{y_4-y_3}(y-y_3)+y_1\right)+ \\ +\frac{y-y_3}{y_4-y_3}\cdot Z_2\left(\frac{y_6-y_5}{y_4-y_3}(y-y_3)+y_5\right), \tag{2.9}$$

which has been obtained by shifting the functions $Z_1(y)$ and $Z_2(y)$ into the intersection y_3 to y_4. Initially, moving from y_3 to y_4, function $Z_1(y)$ dominates, and, afterwards, $Z_2(y)$, i.e. a linear change takes place.

Functions $Z_1(y)$ and $Z_2(y)$ are random function, the values of which can be obtained when measuring the surface of the base of the covering thickness standard by means of groping

$$Z_1(y)=\widetilde{Z}_1+a_1y+b_1 \\ Z_2(y)=\widetilde{Z}_2+a_2y+b_2 \tag{2.10}$$

in which $\widetilde{Z}_1$ and $\widetilde{Z}_2$ have random values according to the normal distribution N(0, σ_1) and N(0, σ_2). In the given case, the functions of the mean value of functions $Z_1(y)$ and $Z_2(y)$ are the following:

$$m_{Z_1}(y)=a_1y+b_1, \quad y_1\le y\le y_2 \\ m_{Z_2}(y)=a_2y+b_2, \quad y_5\le y\le y_6 \tag{2.11}$$

Based on the functions of mean value represented in formula (2.11), the function of the mean value of the assumed profile of the boundary surface between the coating and base (in the range from y_3 to y_4) can be expressed as follows

$$m_{\widetilde{Z}}(y)=\frac{y_4-y}{y_4-y_3}\cdot\left\{a_1\left(\frac{y_2-y_1}{y_4-y_3}(y-y_3)+y_1\right)+b_1\right\}+ \\ +\frac{y-y_3}{y_4-y_3}\cdot\left\{a_2\left(\frac{y_6-y_5}{y_4-y_3}(y-y_3)+y_5\right)+b_2\right\} \tag{2.12}$$

The dispersion of the function of the mean value represented in formula (2.12), however, can be determined in the following relation

$$D_{\tilde{Z}}(y) = \left(\frac{y_4 - y}{y_4 - y_3}\right)^2 \sigma_1^2 + \left(\frac{y - y_3}{y_4 - y_3}\right)^2 \sigma_2^2 + \\ + 2\frac{y_4 - y}{y_4 - y_3} \cdot \frac{y - y_3}{y_4 - y_3} \operatorname{cov}(\tilde{Z}_1, \tilde{Z}_2) \tag{2.13}$$

The coating thickness at a certain value of y can, in the case given (see *Figure 2.15*), be calculated in the following relation:

$$h(y) = Z_3(y) - \tilde{Z}(y), \tag{2.14}$$

in which $Z_3(y)$ 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 given by the following relation

$$Z_3(y) = \tilde{Z}_3 + a_3 y^2 + b_3 y + c_3 \tag{2.15}$$

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

$$m_h(y) = m_{Z_3}(y) - m_{\tilde{Z}}(y) = \\ = a_3 y^2 + b_3 y + c_3 - m_{\tilde{Z}}(y) \tag{2.16}$$

Distribution of coating thickness (dispersion), obtainable through relation (2.14), can be estimated relying on the following dispersion:

$$D_h(y) = \sigma_3^2 + \left(\frac{y_4 - y}{y_4 - y_3}\right)^2 \sigma_1^2 + \left(\frac{y - y_3}{y_4 - y_3}\right)^2 \sigma_2^2 - \\ - 2\frac{y_4 - y}{y_4 - y_3} \operatorname{cov}(\tilde{Z}_1, \tilde{Z}_3) - 2\frac{y - y_3}{y_4 - y_3} \operatorname{cov}(\tilde{Z}_2, \tilde{Z}_3) + \\ + 2\frac{y_4 - y}{y_4 - y_3} \cdot \frac{y - y_3}{y_4 - y_3} \operatorname{cov}(\tilde{Z}_1, \tilde{Z}_2) \tag{2.17}$$

2.5.2 Measurement of coating thickness and analysis of results

Coating thickness standards as a link in the traceability chain of coating thickness measurement are calibrated [4, 14]. 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 it will be placed on the working table of measuring device Perthometer Concept [8]. The stylus of the measuring instrument will be taken to the contact with the base surface of the coating thickness standard. The *y*-directional movement will be performed and the stylus tracing the measured surface. The computer screen of measuring device gives us a true surface profile of traced length (see *Figure 2.14*). It is quite similar represented in *Figure 2.13*. According to the true surface profile we get implementations of random functions $Z_1(y)$, $Z_2(y)$ and $Z_3(y)$ in range chosen in *y* axis (y_1 to y_2, y_5 to y_6 and y_3 to y_4, see *Figure 2.13*).

On the basis of these implementations regarding to *y* values we can get according to relations (2.10) and (2.11) using a Monte-Carlo method [15] possible estimates of the random functions $Z_1(y)$ and $Z_2(y)$. It means the estimates of mean values of these functions $z_1(y)$ and $z_2(y)$ and experimental variances s_{z1}^2 and s_{z2}^2. According to the *z* values of receiving profile in intersection of tracing length the estimates are as follows (*y* − mm; $z_1(y)$, $z_2(y)$, u_{z1}, u_{z2} − μm):

$$z_1(y) = -0{,}02y + 0{,}09; \qquad s_{z1} = u_{z1} = 0{,}07$$
$$z_2(y) = 0{,}036y - 0{,}569; \qquad s_{z2} = u_{z2} = 0{,}08$$

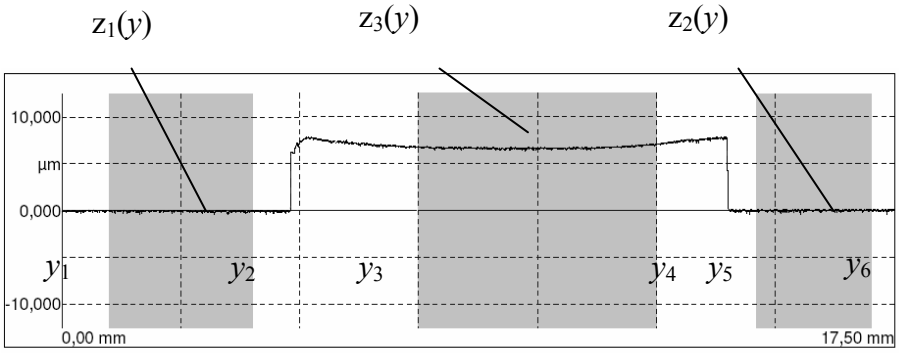

Figure 2.13 Surface profile cross section

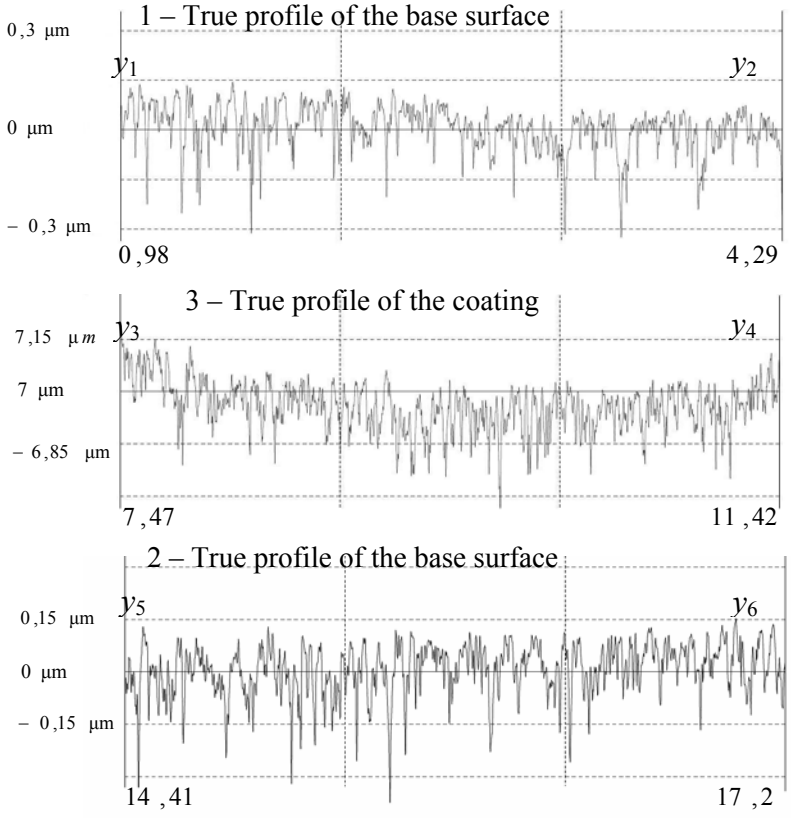

Figure 2.14 True surface profiles

Analogically we can calculate in the range from y_3 to y_4 the equation of parabola proper to relation (2.15) and its variance and standard uncertainty, using a Monte-Carlo method (y – mm; $z_3(y)$, u_{z3} – µm)

$$z_3(y) = 0{,}0385y^2 - 0{,}7517y + 10{,}57$$

$$s_{z3} = u_{z3} = 0{,}05$$

We can calculate using equations (2.12) and (2.13) the mean value function of the assumed profile of the boundary surface between the coating and base and its estimate of standard uncertainty, which are by the calibration results as follows (y – mm; $u_{m\tilde{Z}}$ – µm) (see *Figure 2.15*):

$$m_{\tilde{z}}(y) = \frac{y_4 - y}{y_4 - y_3} \cdot \left\{ a_1 \left(\frac{y_2 - y_1}{y_4 - y_3} (y - y_3) + y_1 \right) + b_1 \right\} +$$

$$+ \frac{y - y_3}{y_4 - y_3} \cdot \left\{ a_2 \left(\frac{y_6 - y_5}{y_4 - y_3} (y - y_3) + y_5 \right) + b_2 \right\} =$$

$$= 0{,}0015\, y^2 - 0{,}0346\, y + 0{,}1274$$

$$u_{m\tilde{Z}} \cong \sqrt{D_{\tilde{Z}}(y)} \cong \sqrt{u_{z1}^2 + u_{z2}^2} =$$

$$= \sqrt{0{,}07^2 + 0{,}08^2} \cong 0{,}11$$

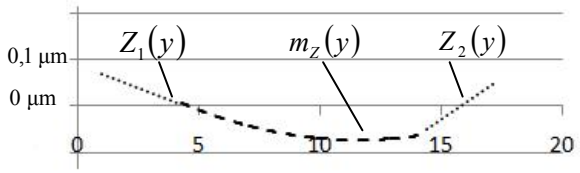

Figure 2.15 Function of the assumed profile

The coating thickness measurement result of coating thickness standard 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):

$$h(y) = z_3(y) - m_{\tilde{z}}(y) =$$

$$= 0{,}04\, y^2 - 1{,}097\, y + 10{,}697$$

The combined standard uncertainty of the coating thickness measurement result, using relation, can be seen below. We assume, that estimates of the random functions $z_1(y)$ $z_2(y)$ and $z_3(y)$ are independent.

$$u\left[h(y)\right] \cong \sqrt{D_h(y)} \cong$$

$$\cong \sqrt{u_{z3}^2 + u_{z1}^2 + u_{z2}^2} \cong 0{,}12\ \mu\text{m}$$

2.5.3 Conclusions to the subchapter 2.5

As the procedure of metrology control of the coating thickness measuring instruments can be verified, enhancing the accuracy of coating thickness standards proves vitally 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 the coating thickness. It gives increase of reliability of the calibration of coating thickness standards compared with the method, which consider only profiles of the upper boundary surface of the base material adjoining the coating.

2.6 Research into uncertainty of measurement in the surface measurement procedure

Modern manufacturing presses quite high demands on the accuracy of surfaces of the parts of machinery with a complicated form. Whether the produced parts of machinery comply with the demands listed in the technical specifications can only be determined in the course of the corresponding measurements. One of the possibilities of measuring the surface contour of the parts with a complicated form in the laboratory of metrology is by using the inductive surface roughness and form measurement instrument Perthometer Concept [8]. Although the above mentioned measuring instrument had previously been calibrated, to confirm the reliability of the measurement results characterizing the surface contour, a special research was carried out in the laboratory of metrology. 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 basing on expanded uncertainty.

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 [3, 7]. 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 an rough estimate of the true value of the measurand. For this reason, each measuring result should be associated with information about the uncertainty, 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 research papers [6, 16, 17, 18], surface roughness was measured by a roughness measuring instrument. The uncertainty of measurement results could be estimated only by the uncertainty contribution of the measuring instrument. This forms about (10–15) % of the total indication. Besides that, no other uncertainty contributors were used to estimate the measurement results. In the research works [4, 13], step height was measured by a surface roughness measuring instrument. To evaluate the measurement results, in addition to the uncertainty contributed by the

measuring instrument, the uncertainty 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.

2.6.1 Evaluation methods of uncertainty in measurement

Uncertainty in measurement comprises, in general, many components. Some of these components may be evaluated from the statistical distribution of the results of series of measurements and can be characterized by experimental standard deviations (type A evaluation of uncertainty). The other components, which can also be characterized by standard deviations, are evaluated from assumed probability distributions based on experience or other information (type B evaluation of uncertainty).

According to the reference document [7], the first step in determining the uncertainty of a measurement is to calculate the model function f that shows the relationship between the input quantities (X_1, X_2,…, X_N) and the quantity to be measured

$$Y = f(X_1, X_2, ..., X_i, ..., X_N) \tag{2.18}$$

Model function f represents the procedure of measurement and the method of evaluation. It describes how values of the output quantity Y are obtained from values of the input quantities X_i. In most cases it will be an analytical expression, but it may also be a group of such expressions which include corrections and correction factors for systematic effects, thereby leading to a more complicated relationship that is not written down as one function explicitly. Further, f may be determined numerically, or it may be a combination of all of these.

An estimate of the measurand Y, the output estimate denoted by y, is obtained from equation (2.6) using input estimates x_i for the values of the input quantities X_i

$$y = f(x_1, x_2, ..., x_i, ..., x_N) \tag{2.19}$$

In first place, standard uncertainties $u(x_i)$ of all input estimates x_i should be evaluated. For an input estimate of X_i, obtained from the statistical analysis of series of observations (type A evaluation of uncertainty), standard deviation of the mean value X_i is calculated as follows:

$$s(\bar{x}_i) = \frac{s(x_{i,j})}{\sqrt{n_i}} \tag{2.20}$$

Variance $s^2(x_{i,j})$ for non-correlated input values is calculated as follows:

$$s^2(x_{i,j}) = \frac{1}{n_i - 1}\sum_{i=1}^{n_i}(x_{i,j} - \bar{x}_i)^2 \tag{2.21}$$

If some of the input quantities are correlated, the correlation should be considered in equation 2.21.

Standard uncertainty $u(x_i)$ is equal to the standard deviation of the mean:

$$u(x_i) = s(\bar{x}_i) \tag{2.22}$$

As it can be seen, input values are the best estimates that were corrected in terms of all effects significant for the model. Where as, if it was not managed to do so, the necessary corrections were introduced as separate input quantities.

Due insufficient knowledge, the estimations of the input quantities are not exact, leading, therefore, to a concept of uncertainty, characterized by standard deviation of the output quantity *Y*. The calculation of the output quantity is performed applying the law of propagation of variances to equation 2.18, which is its equation, if the input quantities are independent:

$$u(y) = \sqrt{\sum_{i=1}^{N}\left(\frac{\partial f}{\partial x_i}\right)^2 \cdot u^2(x_i)} \tag{2.23}$$

It is, therefore, necessary to know the standard deviations, called standard uncertainties, of each of the input quantities ($u(x_i)$). Depending on how the standard uncertainty is estimated, the set of input quantities may be divided into two categories [2, 22]:

- *Evaluation Type A*

Standard uncertainty of input quantities can be evaluated in the course of a statistical analysis of a series of observations.

- *Evaluation Type B*

The standard uncertainty of input quantities is evaluated by means of tools different from the statistical analysis of a series of observations. In this case, the information can come from the following sources: calibration certificates, handbooks, producer's specifications and hypotheses on the function of density of

the input quantity. Calculating standard uncertainty of the output quantity on the basis of the law of propagation of variances, the expanded uncertainty of measurement U can be obtained when multiplying the standard uncertainty by a coverage factor k

$$U = k \cdot u(y) \tag{2.24}$$

The value of k depends on the probability distribution of the output quantity y and on the level of confidence. The assigned expanded uncertainty corresponds to a coverage probability of approximately 95 %. In most of the calibrations the output distribution can approach a normal distribution, where $k = 2$.

2.6.2 Measurement methods of contour measuring equipment

The above-mentioned surface contours with a complicated form were measured using the surface texture measuring system Perthometer Concept produced by company MAHR [8].

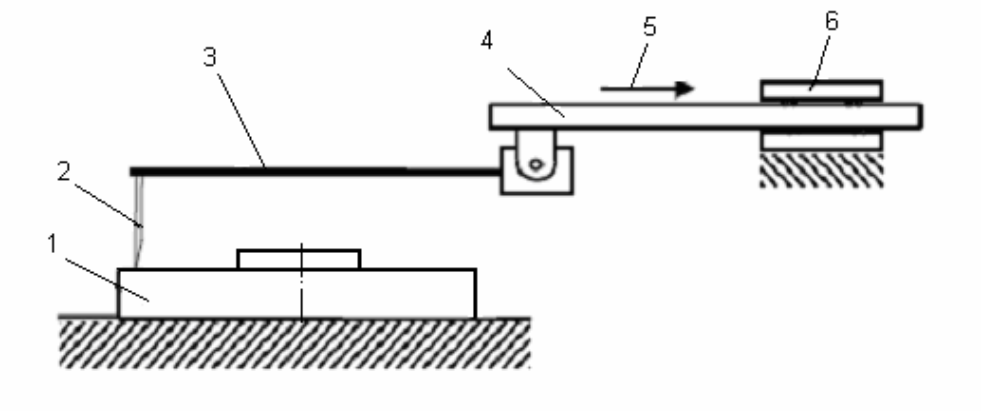

Figure 2.16 Measurement scheme: 1 – *measuring object;* 2 – *stylus;* 3 – *tracing arm;* 4 – *drive unit;* 5 – *measuring direction;* 6 – *calibrated support*

The Laboratory of Metrology has owned this system for about one year. Perthometer Concept is a modular computer-controlled station for measuring and analysing roughness, contour and topography. Perthometer Concept software runs under the worldwide Windows user interface. Operation is therefore quickly learned, easy to understand, and compatible with other Windows` applications. PCV 200 contour drive unit with an exchangeable tracing arm was used in our research. The high-precision PCV 200 contour drive unit is a long-distance instrument for the assessment of radii, distances, angles and straightness deviations. 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. Rigid design and unique material provide highly dynamic construction. Drive unit has programmable measuring routines including lowering, lifting and positioning of the tracing arm and selectable measuring speeds.

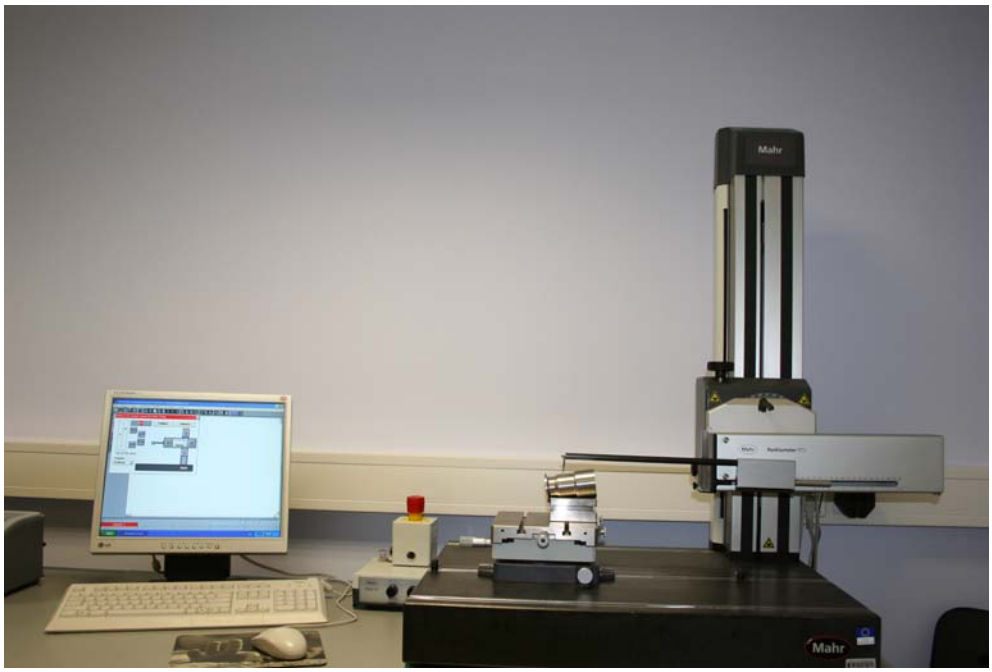

Figure 2.17 Surface texture measurement system Perthometer Concept

2.6.3 Measurement model

Proceeding from equation (2.19), express the measurement model can be expressed as follows:

$$y = x + \sum_{i=1}^{N-1} \delta x_{N-1} \tag{2.25}$$

where x is the measurement value

$$\sum_{i=1}^{N-1} \delta x_{N-1} = \delta x_{\mathrm{MI}} + \delta x_{\mathrm{r}} + \delta x_{\mathrm{F}} + \sum_{j=1}^{J} \delta x_{\mathrm{obj,\ j}} \tag{2.26}$$

where $\sum_{j=1}^{J} \delta x_{\text{obj, j}} = \delta x_{\text{cv}} + \delta x_{\text{cc}} + \delta x_{\text{ang}}$ (2.27)

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

$$y = x + \delta x_{\text{MI}} + \delta x_{\text{r}} + \delta x_{\text{F}} + \delta x_{\text{cv}} + \delta x_{\text{cc}} + \delta x_{\text{ang}} \tag{2.28}$$

where δx_{MI} – correction from the measuring instrument,
δx_{r} – correction from the stylus radius,
δx_{F} – measurement force correction,
δx_{cv} – surface curvature correction,
δx_{cc} – surface concavity correction,
δx_{ang} – correction from the surface angle.

2.6.4 Combined uncertainty of a result of measurement

The standard uncertainty to be ascribed to the estimate *y* of an output quantity *Y*, which is evaluated from the estimates of a number of input quantities, is termed [3, 7] *combined standard uncertainty.* By introducing this concept, it is possible to discern the uncertainty of the output quantity from the uncertainty of other quantities that occur in the measurement model. However, the uncertainty of an input quantity is, in its turn, often obtained from a relevant measurement model, which means that during the evaluation process it was itself termed 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 covariance's of all input quantities considered in the measurement model. The experimental variances and covariance's are obtained from the experimental standard deviations $u(x_i)$ associated with the estimates x_i of the input quantities X_i. In our case combined standard uncertainty is determined as follows:

$$u(y) = \begin{bmatrix} u^2(x) + u^2(\delta x_{\text{MI}}) + u^2(\delta x_{\text{r}}) + u^2(\delta x_{\text{F}}) + \\ + u^2(\delta x_{\text{cv}}) + u^2(\delta x_{\text{cc}}) + u^2(\delta x_{\text{ang}}) \end{bmatrix}^{1/2} \tag{2.29}$$

2.6.5 Research results

The standard uncertainties of input quantities from the different sources were determined. The following results were obtained experimentally and their standard uncertainties were calculated applying type B method of uncertainty evaluation.

Indication (contour) x.

Indication in this case is a contour we can see on the screen of the computer (see *Figure 2.18*)

Standard uncertainty of the indication can be determined according to the printer resolution. The current printer resolution $\Delta x = \pm 1$ μm

$$u(x) = \frac{\Delta x}{\sqrt{3}} = 0{,}6 \text{ μm}$$

Measuring instrument correction δx_{MI} .

The correction mentioned was not found in the calibration certificate, it was noted, however, that the indication could change within the limits of $\Delta_{\text{MI}} = \pm 0{,}5$ μm

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

Stylus radius correction δx_{r} .

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

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

Measuring force correction δx_{F} .

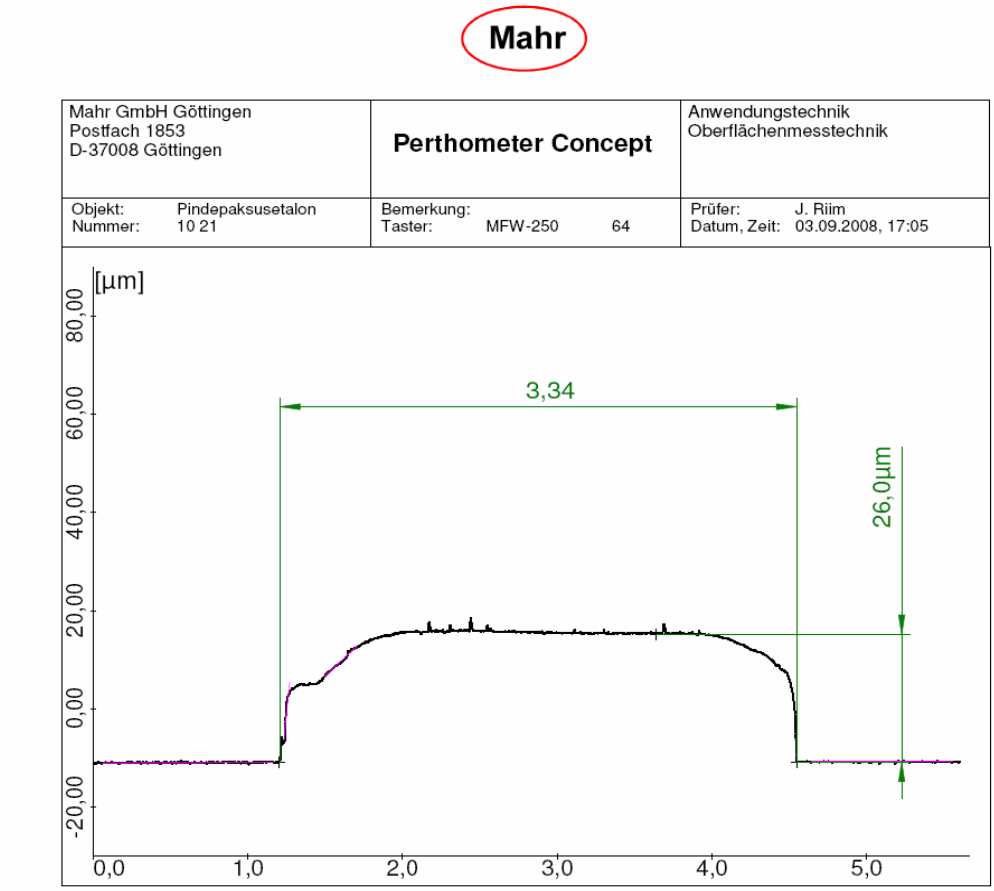

Figure 2.18 The coating thickness standards contour measured with Perthometer Concept

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

$$u(\delta x_F) = \frac{\Delta_F}{\sqrt{3}} \approx 0{,}3\ \mu\text{m}$$

In which $\Delta_F = \pm 0{,}6\ \mu\text{m}$

Surface complexity correction δx_{cv}, δx_{cc}.

The correction due to the surface curvature and concavity is assumed to be equal to zero.

$$\delta x_{cv} \approx 0 \text{ and } \delta x_{cc} \approx 0$$

The standard uncertainty of these corrections can be calculated from the following equation:

$$u(\delta x_{cv}) = \frac{\Delta_{cv}}{\sqrt{3}} \approx 0{,}9\ \mu m$$

$$u(\delta x_{cc}) = \frac{\Delta_{cc}}{\sqrt{3}} \approx 0{,}9\ \mu m$$

where Δ_{cv} and Δ_{cc} have found experimentally.

$$\Delta_{cc} = \Delta_{cv} = \pm 1{,}5\ \mu m$$

Correction of the surface angle δx_{ang}.

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

$$u(\delta x_{ang}) = \frac{\Delta_{ang}}{\sqrt{3}} \approx 1{,}7\ \mu m$$

where Δ_{ang} was experimentally determined during the research applying the angle standards

$$\Delta_{ang} = \pm 2{,}9\ \mu m$$

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

Table 2.2. Estimates of the input quantities and their uncertainties

Quantity X_i	Estimate x_i	Standard uncertainty $u(x_i)$/μm	Disper-sion $u^2(x_i)$/μm
x	contour	0,6	0,36
δx_{MI}	0	0,3	0,09
δx_{r}	0	0	0
δx_{F}	0	0,3	0,09
δx_{cv}	0	0,9	0,81
δx_{cc}	0	0,9	0,81
δx_{ang}	0	1,7	2,89
		$\sum$	5,05

From the equation (2.23), the following can be calculated:

$$u(y) = \sqrt{\sum_{i=1}^{N} u^2(x_i)} = \sqrt{5{,}05}\ \mu\mathrm{m} \approx 2{,}3\ \mu\mathrm{m}$$

Hence, the expanded uncertainty can be given according to the equation (2.24):

$$U = k \cdot u(y) = 2 \cdot 2{,}3\ \mu\mathrm{m} = 4{,}6\ \mu\mathrm{m} \approx 5\ \mu\mathrm{m}$$

2.6.6 Conclusions to the subchapter 2.6

As a result of the current research the measurement model and method for calculating the expanded uncertainty of the surface measurement was worked out. It is possible to give estimation to the surface elements obtained in printout after measuring the contour of complicated surface. It was shown how to estimate the variation range and analyse the limits within which the numerical values of the surface contour can change. Finally, the quality of the measuring values can be evaluated, and when applying this method, the measurement uncertainty of the measuring values can be estimated.

3 CALIBRATION OF COATING THICKNESS GAUGES

3.1 Measurement methods of coating thickness

There are several different possibilities and measurement methods to measure the thickness of wet or dry coatings like paint, varnish, chrome, zinc etc. on different base materials. But most used methods in industry are two methods – magnetic inductive method and eddy current method. Most coating thickness gauges are grounded on these two methods.

3.1.1 Magnetic measurement method

Magnetic method using in coating thickness instruments of the magnetic type for non-destructive measurements of the thickness of non-magnetic coatings on magnetic basis metals [19]. Coating thickness instruments of the magnetic type (see *Figure 3.1*) measure either the magnetic attraction between a permanent magnet and basis metal, as influenced by the presence of the coating, or the reluctance of a magnetic flux path passing through the coating and the basis material.

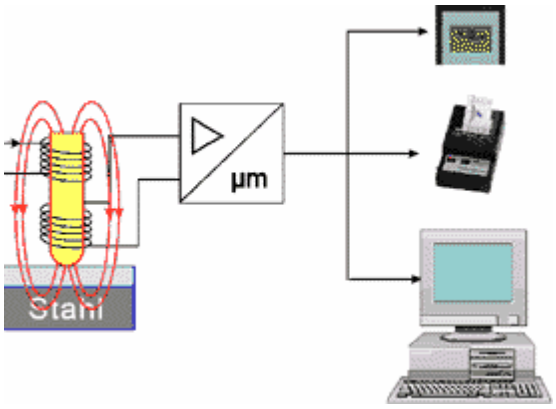

Figure 3.1 Scheme of the magnetic inductive method

3.1.2 Factors affecting the measuring accuracy

The following factors may affect the accuracy of measurements of coating thickness. The precision of a measurement changes with coating thickness depending on the instrument design. For thin coatings, the precision is constant, independent of the thickness. For thick coatings, the precision is an approximately constant fraction of the thickness.

Thickness measurements by the magnetic methods are affected by variations in the magnetic properties of the basis metal. For practical purposes, magnetic variations in low carbon steels can be considered to be insignificant. To avoid the influences of several or localized, heat treatments and cold working, the instrument should be calibrated using calibration standard having a basis metal with the same properties as that of the test specimen or, preferably, and if available, with a sample of the part to be tested before application of the coating.

For each instrument, there is a critical thickness of basis metal above which measurements will not be affected by an increase in thickness. Since it depends on the instrument probe and the nature of the basis metal, its value should be determined experimentally, unless the manufacturer specifies it.

The method is sensitive to abrupt changes in surface contour of the test specimen. Therefore, measurements made too near an edge or inside corner will not be valid unless the instrument is specifically calibrated for such measurements. The effect may extend up to about 20 mm from the discontinuity, depending on the instrument. Measurements are also affected by the curvature of the test specimen. The influence of curvature varies considerably with the make and type of instrument, but always becomes more pronounced as the radius of curvature decreases. Instrument with two-pole probes may also produce different readings if the poles are aligned in planes parallel or perpendicular to the axis of a cylindrical surface. Similar effect can occur with a single-pole probe if the tip is unevenly worn. Measurements made on curved test specimens may not, therefore, be valid unless the instrument is specifically calibrated for such measurements.

If the range of series of measurements, made within the same reference area on a rough surface, substantially exceeds the inherent repeatability of the instrument, the number of measurements required should be increased to at least five.

Measurements made by an instrument having a two-pole probe or an unevenly worn single-pole probe may be influenced by the direction in which the magnetic basis metal has been subjected to mechanical working, the reading changing with the orientation of the probe on the surface

Residual magnetism in the basis metal affects measurements made by instruments, which employ a stationary magnetic field. Its influence on measurements made by reluctance instruments employing an alternating magnetic field is much smaller.

Strong magnetic fields, such as those produced by various types of electrical equipment, can seriously interfere with the operation of instruments, which employ a stationary magnetic field.

The probes of the instruments have to make physical contact with the test surface because these instruments are sensitive to foreign material that prevents intimate contact between the probe and the surface of the coating. The probe tip should be checked for cleanliness.

The poles of the test probe have to be applied at a constant but sufficiently high pressure, such that no deformation of the coating occurs, even if the coating material is soft. Alternatively, soft coatings may be covered with foils, and the

thickness of the foils subtracted from the test results. Such considerations are also necessary if measuring the thickness of phosphate coatings. The readings of instruments using the magnetic attraction principle may also be affected by the orientation of the magnet in relation to the field of gravity of the earth. Thus, the operation of an instrument probe in a horizontal or upside-down position may require a different calibration, or may be impossible.

3.1.3 Eddy current method

Eddy current method using for non-destructive measurements of the thickness of non-conductive coatings on non-magnetic, electrically conductive (generally metallic) basis materials, using amplitude-sensitive eddy current instruments [20]. The method (see *Figure 3.2*) is particularly applicable to measurements of the thickness of most oxide coatings produced by anodising, but is not applicable to all conversion coatings, some of which are too thin to be measured by this method.

The methods principle is as follows. An eddy current probe (or integrated probe/instrument) is placed on the surface of the coating to be measured, and the thickness is read from the instrument's readout.

Probe, containing an eddy current generator and detector linked to a system capable of measuring and displaying the changes in amplitude, normally as a direct readout of coating thickness. The system may also be able to measure phase changes.

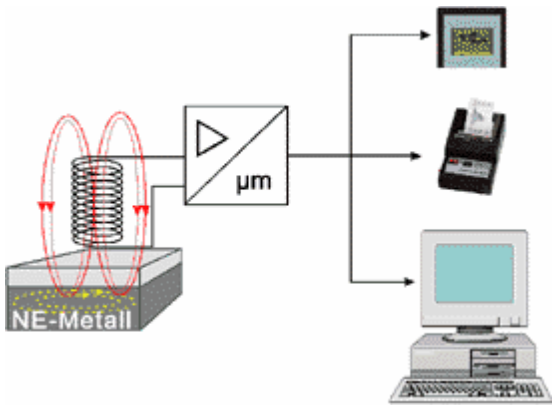

Figure 3.2 Scheme of the eddy current method

3.1.4 Factors affecting measurement uncertainty

A measurement uncertainty is inherent in the method. For thin coatings, this measurement uncertainty is constant, independent of the coating thickness and, for

single measurement, is at least 0,5 μm. For coatings thicker than 25 μm, the uncertainty becomes relative to the thickness and is approximately a constant fraction of the thickness. Measurements using eddy current instruments can be affected by the electrical conductivity of the basis metal, which is a function of the composition and heat treatment of the material. The influence of electrical conductivity on the measurement varies considerably with the make and type of instrument.

For each instrument there is a critical minimum basis metal thickness above which measurements are not affected by an increase in thickness. Since this thickness depends on both the eddy current generation frequency of the probe system and the electrical properties of the basis material its value should be determined experimentally, unless otherwise specified by the manufacturer.

Eddy current instruments can be sensitive to abrupt changes in surface contour of test specimen. Therefore measurements made too near to an edge or corner may not be valid unless the instrument has been specifically calibrated for such measurements.

Measurements are affected by the curvature of the test specimen. This influence of curvature varies considerably with the make and type of instrument and probe, but always becomes more pronounced as the radius of curvature decreases. Measurements made on curved test specimens might not, therefore, be valid unless the instrument is specifically calibrated for the surface curvature in question, or a special probe, which compensates for surface influence, is used.

Measurements are influenced also by the surface topography of the basis material and of the coating. Rough surfaces can cause both systematic and random errors. Random errors can be reduced by making multiple measurements, each measurement being made at a different location, and then calculating the average value of that series of measurements. If the basis material is rough, the zero of the instrument shall be checked at several locations on a typical sample of the uncoated, rough, basis material. If no typical uncoated basis materials available, the coating of the test specimen shall be stripped; at least over part of its area, with a chemical solution that does not attack the basis material.

If the probe is not placed directly on the coating, the gap between probe and coating affects the measurement of the coating thickness. The measured thickness will be equal to the coating thickness plus the additional “lift-off” gap. Lift-off can be produced unintentionally, e.g., by the presence of foreign particles between the probe and the coating.

The pressure with which the probe is applied to the test specimen affects the instrument readings and shall therefore be made constant. This pressure effects is more noticeable when the coating are soft. Most commercially available instruments are supplied with constant pressure probes.

Unless otherwise instructed by the manufacturer, the probe shall be applied perpendicularly to the coating surface as tilting the probe away from the perpendicular can cause measurement errors. The possibility of tilt occurring inadvertently can be minimized by probe design or by the use of a probe holding.

Because the temperature changes affect the characteristics of the probe, it should be used approximately the same temperature conditions as those used for calibration unless the probe has built-in temperature compensation. Most metal change their electrical conductivity with temperature. Because the measured coating thickness is influenced by changes in the electrical conductivity of the basis metal, large temperature changes should be avoided.

The presence of an intermediate coating can affect the measurement of the coating thickness if the electrical characteristics of that intermediate coating differ from that of the coating or basis material. If a difference does exist then the measurements will, in addition, be affected by an intermediate coating thickness of less than $h_{\min}$. If the thickness is greater than $h_{\min}$ then the intermediate coating, if non-magnetic, can be treated as the basis material. It has been found, that some instruments having probe systems operating with multiple frequencies can measure both top and intermediate coatings.

3.2 Comparison of measuring results of different MIKROTEST gauges

One of the simplest and most used method to measure the non-magnetic coatings as varnish, paint, plastic, electroplating, phosphate, nickel etc. on steel or cast iron basis is magnetic attraction principle. The measurement of the coating thickness is dependent on magnetic attraction. The attractive force F_M (see *Figure 3.3*) is related to the distance between a permanent magnet and a steel or cast iron substrate h, magnetic piercing ness of substrate material μ, permanent magnet magnetic field intensity H.

If the surface is wide and flat enough and with enough thickness, then the dimensions of surface don't change particularly the magnetic attractive force F_M and the expression can be written as follows:

$$F_M = f(h, H, \mu) \tag{3.1}$$

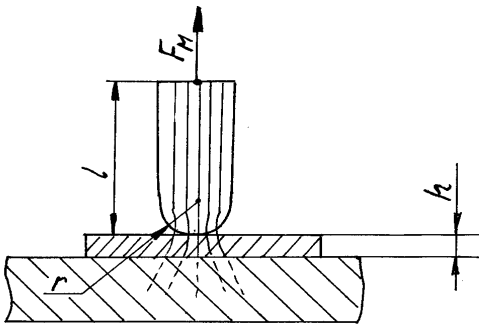

Figure 3.3 Magnetic attraction principle

3.2.1 The coating thickness measurement model function

The abovementioned measuring method is used for the practical coating thickness measurements in a slightly different form. Measuring the coating thickness we do not fix the attractive force of permanent magnet F_M , but the reaction F_V needed to lift the magnet from the surface.

This reaction will be fixed at the moment of lifting the permanent magnet and $F_M = -F_V$. The magnet is lifted from the surface by means of a spring connected to the magnet arm. The spring is tensioned by means of the thumb wheel and the coating thickness is shown directly on the scale (*Figure 3.2*).

The coating thickness can be calculated from the equation:

$$F_M = -F_V = K_1 \frac{M^2}{r^4} K_2 \frac{r}{l} \exp K_3 \frac{h}{r}, \qquad (3.2)$$

where M – magnetic moment of permanent magnet,
r – curved radius of permanent magnet tip,
l – length of permanent magnet,
K_1 – factor, describing the magnetic characteristics of permanent magnet,
K_2 – factor, describing the geometrical characteristics of permanent magnet,
K_3 – factor, describing the sensitivity of permanent magnet (measuring instrument) against the coating thickness.

3.2.2 Research object and experiment technique

The magnetic attraction principle and the measurement model function are achieved in coating thickness gauges type MIKROTEST produced in company ElektroPhysik GmbH. (*Figure 3.4*)

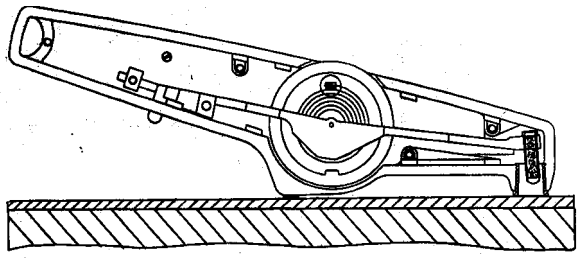

Figure 3.4 Coating thickness gauge "MIKROTEST"

These coating thickness gauges are specified to measure the non-magnetic coating (also nickel) thickness on steel. As in the MIKROTEST type of gauges the geometrical and magnetic characteristics are same for all produced gauges, the expression (3.2) can be rewritten as follows:

$$F_M = a \cdot \exp b \cdot h, \tag{3.3}$$

where a – factor, taking into consideration the geometrical and magnetic characteristics of permanent magnet,

$$a = K_1 \frac{M^2}{r^4} K_2 \frac{r}{l}, \tag{3.4}$$

b – factor, taking into consideration the sensitivity of attractive force towards the coating thickness,

$$b = \frac{K_3}{r} \tag{3.5}$$

From the expression (4.3) the coating thickness h can be expressed and the measurement model function for MIKROTEST type coating thickness gauges is:

$$h = \frac{\ln F_M}{b} - \frac{\ln a}{b} \tag{3.6}$$

In this model function the measured value is an attractive force F_{M}, the uncertainty of measurement of which depends of the permanent magnet used in coating thickness gauge, measurement object and the measuring mechanism of attractive force. This value has a expanded uncertainty U_{F_M} .

Factor a, characterising the coating thickness gauge, depends of the geometrical and magnetic characteristics and can vary within $\pm\delta a$.
Second factor b characterising the coating thickness gauge sensitivity toward the coating thickness and the magnetic piercingness of coating material. This factor can vary within $\pm\delta b$.

On the basis of measurement model (3.6) the scales of abovementioned coating thickness gauges have been graduated.

Indication of the gauge has a combined standard uncertainty expressed by equation:

$$u(h) = \sqrt{c_a^2 \cdot u^2(a) + c_b^2 \cdot u^2(b) + c_{F_V}^2 \cdot u^2(F_V)}, \tag{3.7}$$

where sensitivity coefficients c_a, c_b, c_{F_M} are calculated taking partial derivatives from the equation (3.6):

$$\begin{aligned} c_a &= \frac{\partial h}{\partial a} = -\frac{1}{a \cdot b}, \\ c_b &= \frac{\partial h}{\partial b} = \frac{\ln a}{b^2} - \frac{\ln F_V}{b^2}, \\ c_{F_M} &= \frac{\partial h}{\partial F_V} = \frac{1}{b \cdot F_V}. \end{aligned} \tag{3.8}$$

As a measurement object, a magnetic coating thickness gauges MIKROTEST had been taken to measure the thickness of nickel on steel. This selection was made because the measuring of nickel coatings is important and poses a problem in today's industry. Most of the known measuring methods are not useful for non-destructive nickel coating measurement. The reason is, that physical characteristics of nickel coating are close to the steel substrate characteristics. Also nickel coating has specific magnetic characteristics. To research the reliability of nickel coating thickness gauges were used for a long time and also the brand new gauges, calibrated since year 1997 in different places with calibration standards of Tallinn University of Technology. These standards were last calibrated in 1999, PTB (Physikalisch-Technische Bundesansalt) laboratory.

3.2.3 Research results

There were analysed six different nickel coating thickness gauges MIKROTEST NiFe50.

The deviation of measuring instrument indication was intended by calibration with coating thickness standards. The thickness of standards h_S are as follows: 0 μm; 5,45 μm, 11,0 μm; 16,0 μm; 27,1 μm; 37,9 μm; 49,6 μm.

The abovementioned coating thickness gauges were attributed following signs.
No.1 – the nickel coating thickness gauge using in company AS NORMA was calibrated in the end of 1997, because there was a suspicion, that the reliability of this instrument was small. I. Abiline calibrated the measuring instrument in Tallinn Technical University in 15th of September 1997.

No.2, No.3, No.4 – New nickel coating thickness gauges, which were calibrated in Berlin PTB 11th of February 2000 by the PTB laboratory "Schichtdicke" Dr. K. - P. Hoffmann and representative of company "ElektroPhysik" Mr. Liebl.

No.5 – three years used nickel coating thickness gauge, which was calibrated in abovementioned place and by the same persons.

No.6 – in December of 1999 purchased nickel coating thickness gauge, which was calibrated in Tallinn Technical University 13th of March 2000 by as.

All these calibrations were made using the same calibration standards.

The expanded uncertainty was calculated according to [21]. The value of correction is the difference between the thickness of standard and mean value of three indications got during the measuring procedure.

Three indications have been taken because of the rules in user's manual.
The calibration results have presented in *Table 3.1*. The expanded uncertainty for the corrections *K* was also calculated .

All processing of measurement results and presenting in *Table 3.1* went on according to the technique given in [3, 21].

The correction to the indication of coating thickness gauge calculated by equation (3.9):

$$K = h_S - h, \tag{3.9}$$

where h_S – calibrated coating thickness of standards,
h – mean value of three indications of coating thickness gauge, measured the calibration

The expanded uncertainty of calibration results is calculated by equation (3.10):

$$U = K\sqrt{u^2(h_S) + u^2(h)}, \tag{3.10}$$

where $u(h_S)$ – standard uncertainty of coating thickness,
$u(h)$ – standard uncertainty of calibration method.

Calculating expanded uncertainty *U* has been shown, and the value is presented in *Table 3.1*, which is connected with rectangular distribution and coverage factor $k = 2$. It gives a probability level 95 %.

The standard uncertainty $u(h_S)$ has been obtained from the calibration results of calibration standards, standard uncertainty of calibration method $u(h)$ has been calculated by the standard deviation s_P.

In the specification of coating thickness gauge MIKROTEST NiFe50 type 80-648-00-00 have given maximum permissible error $G = 2\ \mu m + 0{,}08h\ \mu m$, where h – indication of the coating thickness gauge.

For the evaluation of coating thickness gauge the following expression is used:

$$\left|\pm K_i \pm U_i\right| \le G, \tag{3.11}$$

where *i* – index, showing the number of coating thickness gauge.

Research results, given in *Table 3.1,* show that the studied coating thickness gauges lose the stability of indication during the long-range using, and it is eligible to calibrate them every year. The results show, that in case of the thicker nickel coatings (30 μm and more) the indications of coating thickness gauges, if they are not calibrated, are not reliable.

The construction of MIKROTEST type of coating thickness gauges doesn't intend the adjustment of the gauge. The zero point adjustment was performed by the producer "ElektroPhysik". Researching results reveal that for the measurements of nickel coatings in the range from 0 μm to 30 μm the gauges are reliable even without the calibration. However, for the measurements of nickel coatings, more than 30 μm, the coating thickness gauges must be calibrated for performing the relevant measurements.

Table 3.1. Calibration results with six different nickel coating thickness gauges MIKROTEST NiFe50

Calibr. standard coating thickness h_S/μm	The indication of coating thickness gauge K/μm						Expanded uncertainty U/μm	Gauge limits of error G/μm
	No.1	No.2	No.3	No.4	No.5	No.6		
0	0	−0,7	−0,5	−0,2	0,2	0,5	0,3	±2,0
5,5	0,5	−0,8	−0,5	−0,3	1,1	0,7	0,4	±2,4
11,0	0,3	−0,3	−0,2	0,3	1,9	0,1	0,7	±2,9
16,0	0	−1,3	0	−0,2	0,5	0,9	1,3	±3,3
27,1	2,9	−0,9	−1,1	−1,1	0,3	1,6	2,2	±4,2
37,9	3,9	−7,1	−6,8	−6,1	6,9	4,1	2,5	±5,0
49,6	5,6	−2,0	−2,0	−2,7	14,3	0,9	3,0	±6,0

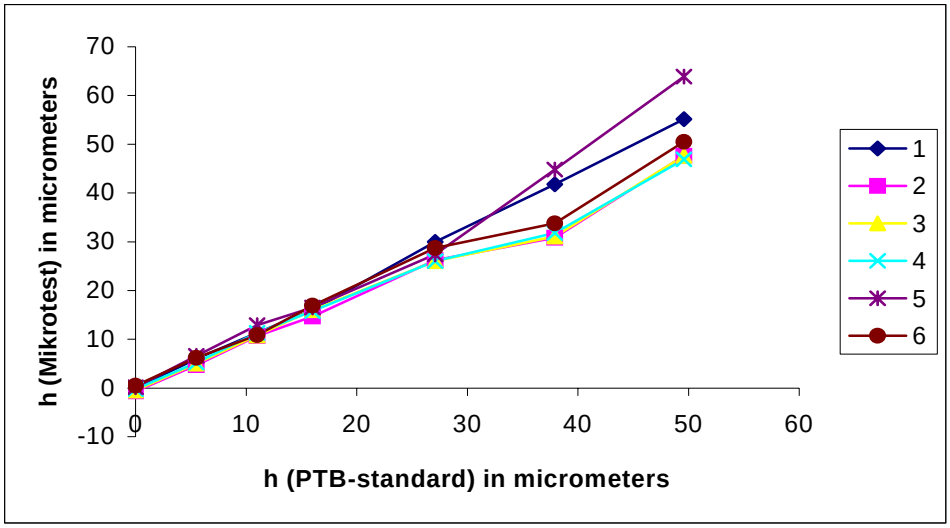

Figure 3.5 Calibration results in chart

3.3 The calibration method of coating thickness gauges

To this day, in the field of coating thickness gauges the calibration method has been used, in which, with the help of the coating thickness gauge, the coating thickness, reproduced by the coating thickness standard, was measured. In this case, the difference between the coating thickness value reproduced by the coating thickness standard and the reading of the calibrated coating thickness gauge would give us the correction in the mentioned calibration point.

In the presentation a new calibration method of the coating thickness gauges has been described. A special measuring object with the changeable coating thickness has been measured simultaneously with the standard measuring instrument and the calibrated thickness gauge. The calibration device worked out for applying the proposed measuring method will be analysed and examined. The accuracy characteristics of the above-mentioned device have been given and measurement uncertainty of the calibration results have been analysed. The researching results show us that using the new method we can substantially increase the calibration accuracy of coating thickness gauges.

Practically no modern product has any part or element without a coating. Coatings are widely used to cover and decorate the parts and products composed from the parts. The product life period and qualities (parameters) directly depend on the coating thickness. Without the accurate measuring of the coating thickness it would not be possible to produce most of the products, especially electronic products.

As the coating in general cannot be separated from its base material for measurement, its measurement by a direct method is not possible. By non-

destructive methods, the coating thickness can only be determined from one side (from the free side of the coating). Thus, only indirect methods are available for the determination of the coating thickness.

The need to measure the thickness of several kinds of coatings has led to the design and use of a series of gauges, applying the measuring principle, where the dimension (coating characteristic) ,for example, the magnetical or electrical resistant,depending on the coating thickness, is measured nondestructively. The received result is transformed to the coating thickness value.

However, in connection with these measuring instruments, another problem has arisen: to assure the traceability of the coating thickness measuring results. It means that to make the right decisions, the coating thickness measuring results must be reliabile. To achieve this, the coating thicness measuring result must be related to the appropriate base value. In general, it is a state or international standard [12]. Hence, the scientific goal is to establish uniformity and traceability in the measurement of coating thickness through the calibration of relevant measuring instruments. The calibration of the coating thickness measuring instruments gives us the relation between the reading of the gauge and the coating thickness value, presented for measurement. To achieve the above-mentioned goal, it is necessary to work out modern and scientifically well founded calibration methods as well as measurement standards for their implementation.

In this work we have observed a calibration method worked out in Tallinn University of Technology. In addition, we describe the standard instrument and research results applied to carrying out the calibration method.

3.3.1 The nature of the calibration method of coating thickness gauges

In Tallinn University of Technology a calibration method of coating thickness gauges has been worked out, where the special coating, created for the calibration procedure and to be kept stable during a certain period has been measured at the same time by the standard gauge and the calibrated gauge [12].

The scheme of the calibration of the coating thickness gauge is given in *Figure 3.4.*

By the mentioned calibration scheme the calibration model of the coating thickness gauge is given by:

$$y = x_{\mathrm{E}} - x + \sum_{i=1}^{N-2} \delta x_i \tag{3.12}$$

where y – the estimate of the correction to the coating thickness gauge indication,
x_{E} – the coating thickness value realized by the measurement standard,
x – the indication of the calibrated coating thickness gauge,
$\sum_{i=1}^{N-2} \delta x_i$ – the amount of the corrections considered in the calibration procedures.

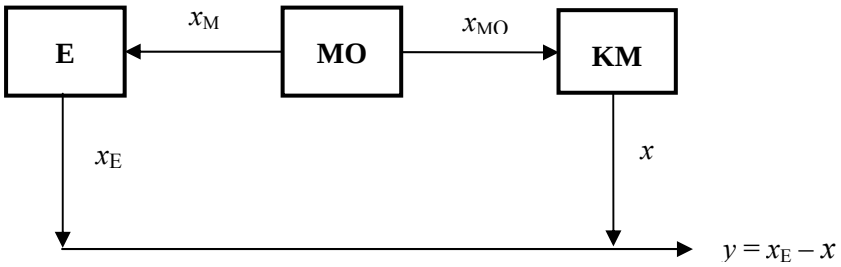

Figure 3.6 The measuring of the coating thickness by the standard instrument and the calibrated gauge at the same time: E – *standard instrument;* MO – *special coating (measuring object);* KM – *calibrated coating thickness gauge*

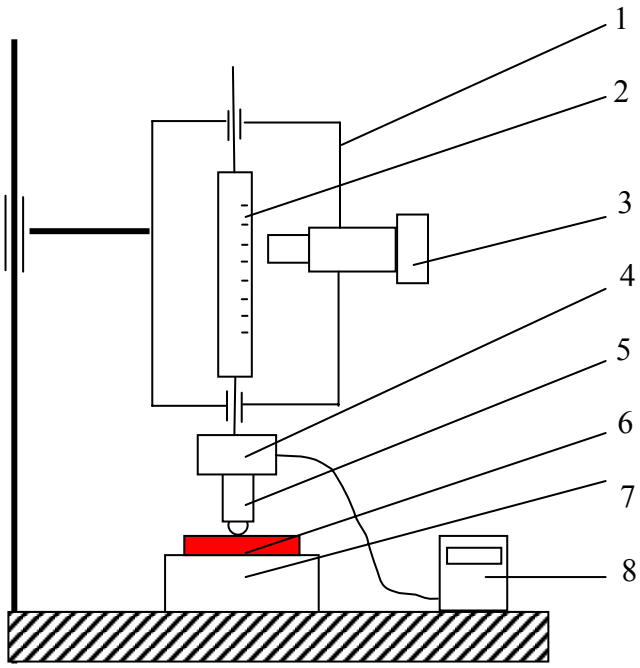

Figure 3.7 Standard measuring instrument with the coating thickness gauge to be calibrated: 1 – *Abbe linear gauge,* 2 – *linear gauge stock,* 3 – *reading device,* 4 – *fixture,* 5 – *probe of the coating thickness gauge,* 6 – *film,* 7 – *base material,* 8 – *indication device of the coating thicknes gauge*

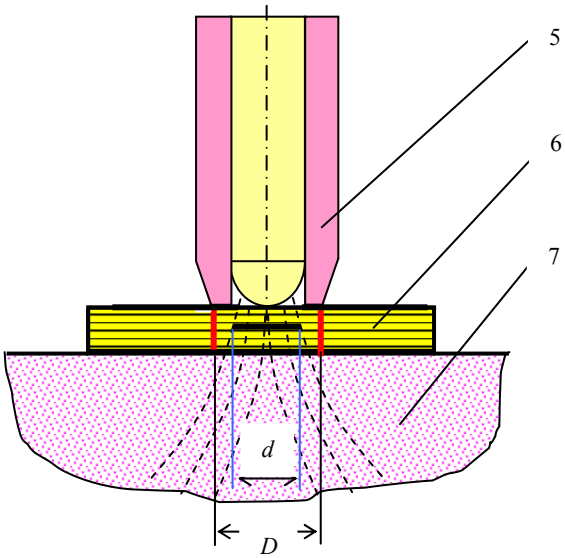

Figure 3.8 Measuring principle: 5 – probe of the coating thickness gauge, 6 – film, 7 – base material, D – the surface diameter measured by the calibrated coating thickness gauge, d – the diameter of the contact circle

To carry into effect the abovementioned calibration method of coating thickness gauges, a standard measuring instrument has been worked out, which scheme of principle is given in *Figure 3.7* and *3.8*.

Coating thickness measuring instruments with the indication previously adjusted and fixed will be calibrated by the following procedure:

The standard measuring device and the coating thickness gauge to be calibrated are simultaneously measuring the thickness of the same coating. The developed standard measuring instrument is shown in *Figure 3.9*. The coatings with different coating thicknesses were measured.

3.3.2 Selection of the number of calibration points

In the calibration of the coating thickness gauge on the whole measuring range, the selected number of calibration points is rather important. Generally the number of the calibration points must be optimal. All possible systematical effects, conditioned by the linearization of the indication function of the coating thickness gauge, must be estimated. However, in case of the coating thickness gauge, its systematical effect by the linearization of the indication function is the continuously volatile dimension and it can be determined as:

$$e(y) = (A_0 + A_1x) - (a_0 + a_1x + a_2x^2 + \ldots + a_mx^m) \qquad (3.13)$$

where $A_0 + A_1x$ – linearisated indication function according to the coating thickness x ,
$a_0 + a_1x + a_2x^2 + \ldots + a_mx^m$ – theoretical indication function according to the coating thickness x,
$A_1, a_1, a_2, \ldots, a_m$ – constants,

$$K = -e(y) \tag{3.14}$$

where K – correction value.

Then the equation (3.13) derivation by the x was calculated and equalized to zero,

$$\frac{\mathrm{d}e(y)}{\mathrm{d}x} = A_1 - a_1 - 2a_2x - \ldots - ma_mx^{m-1} = 0 \tag{3.15}$$

x_i ja x_j values can be calculated, where the function of the systematic function determined by the equation (3.13) has a maximum and minimum value. The origin by the determination of the optimal calibration points is the maximum value $\left|e(y)_{\max}\right|$. Hence the selection of the calibration points must be performed so, that it is maximum in the calibration of the coating thickness gauge. To determine the optimal number of calibration points, the methods described in [22] can be used as well.

3.3.3 Research results

The developed standard measuring instrument and the calibrated coating thickness gauge are shown in *Figure 3.7*.

During the research the universal coating thickness gauge MINITEST 4100 by ELEKTRO-PHYSIK was calibrated and two different probes F2 and N2 were used. Probe F2 is used to measure the non-magnetic coatings on steel and on other ferrous metals. The measuring principle is called “Magnetic induction principle” and it can be described as follows: The measurement works on the transformer principle. The inductive coupling between the primary (induction) coil and the secondary (measuring) coil is influenced by the thickness of the non-magnetic coating on the magnetic (ferrous) substrate. With increasing of the coating thickness the strength of the measuring signal diminishes. Probe N2 is used to measure all insulating coatings on non-ferrous metals (aluminium, copper, brass, zinc etc.). The principle called “Eddy current principle”. A high frequency, electro-magnetic field is induced into the non-ferrous metal. Thus, an eddy current is produced which size serves to measure the thickness of the insulating coating. The feedback of the eddy current on the probe results in a thickness value.

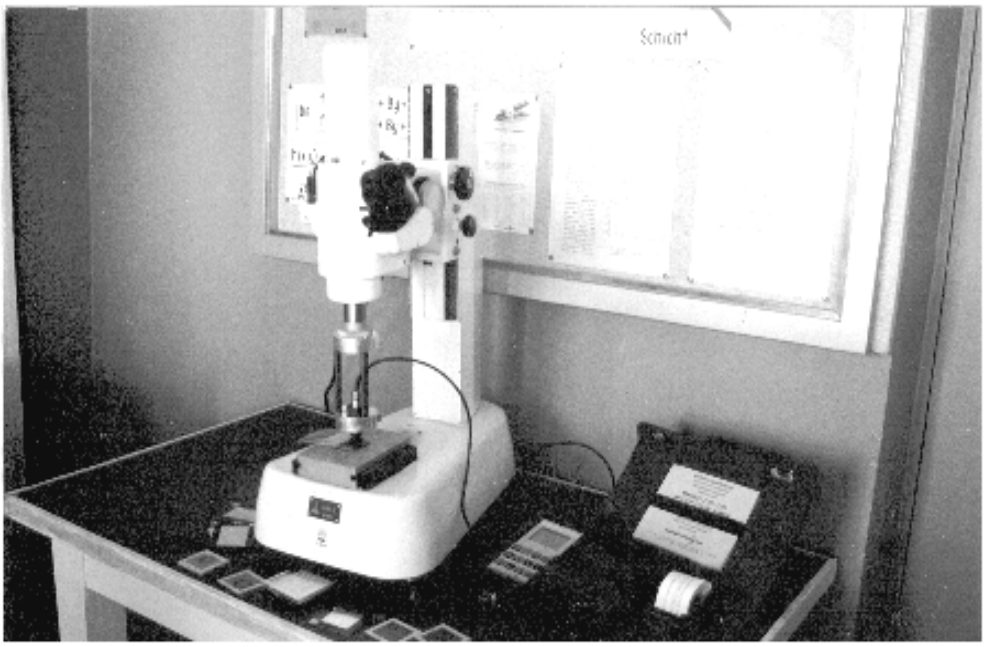

Figure 3.9 Coating thickness standard measuring instrument

In this research the coating thickness gauge was used at first with the probe F2. The foils with different coating thicknesses in steel plate were measured and was the single readings of the coating thickness gauge (the measuring instrument to be calibrated) and the length measuring instrument (standard instrument) were fixated. Analogically the coating thickness measuring gauge with the probe N2 was calibrated.

The research (calibration) results are given in *Figures 3.10* and *3.11*. To calculate the measuring uncertainty an appropriate program has been worked out.

3.4 Conclusions to the chapter

Research results show us, that the developed coating thickness gauges calibration method and the standard device developed would allow calibrating the coating thickness gauges in the arbitrary desired indication points. It provides an opportunity for us to increase significantly the accuracy of the calibration, as in case of the calibration method based on the Abbe measuring principle.

The pressure of the probe to the surface can also be taken into consideration. This calibration method reproduces quite precisely the coating thickness measuring scheme and procedure, which would significantly increase the reliability of the calibration results.

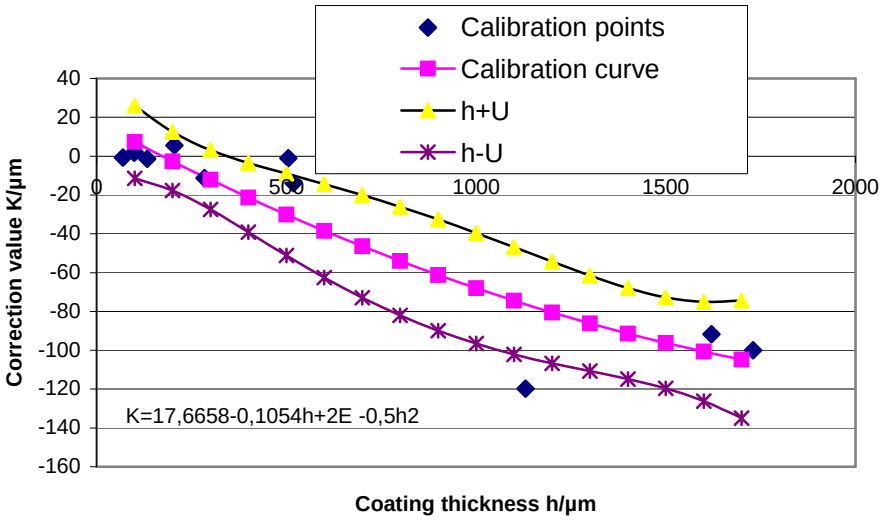

Figure 3.10 Calibration curve of the coating thickness gauge. Probe F2

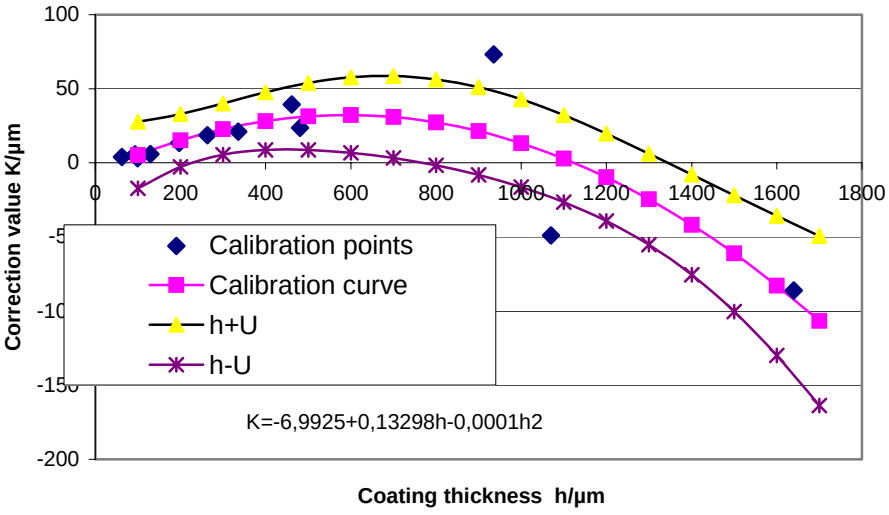

Figure 3.11 Calibration curve of the coating thickness gauge. Probe N2

CONCLUSIONS

The generalised conclusions of the work are as follows:

1. Applying the Monte-Carlo method known from the theory of probability and mathematical statistics, a new method for determining coating thickness has been developed. The method allows better determining (defining) the size of the measurand, i.e. coating thickness, which is especially necessary for calibrating coating thickness standards.
2. In addition to a new measuring method of the contour of coating thickness standard, methods for calculating the indeterminacy of the results obtained when measuring the coating thickness standard. This method has already been applied when calibrating one of the sets of working standards of coating thickness (nickel coating on steel).
3. Based on the universal conception of indeterminacy of measurement assessment, mathematically grounded theoretical bases for indeterminacy of calculations applicable when using magnetical coating thickness measuring instruments. The latter have also found proof in the course of practice.
4. The characterictics of precision of the magnetic coating thickness gauges have been studied, relying on the measuring model devised in this thesis. As an outcome of the latter, new solutions for producing new calibration models and techniques were worked out.
5. A new calibrating technique has been devised and taken into practical use when calibrating coating thickness measuring instruments, in which case a calibrated length measuring device is used as a calibrated length measuring device and, in the calibration procedure, the coating is imitated by an air-slot or films of plastic and as the result of which the indeterminacy of calibrating measures diminishes to a large extent (about twice).
6. On the basis of the results of the current research, a new patentworthy method for calibrating coating thickness standards was worked out, an application for the patent of which has been applied for in Germany.
7. For futher plans the theoretical as well as applicational research on the current method, however, is still to be continued.

ABSTRACT

Calibration methods of coating thickness gauges

Along with the rapid development of industry, quality requirements for the production have also increased. The latter, in its turn, has created the necessity for new and modern equipment. The technology of measuring coating thickness has been developing at a remarkable speed, in the last few decades. Every year, companies launch new models of coating thickness gauges to the market, all of them having a state of the art design, and allow more and more precise and comfortable measurement of the thickness of the various coatings applied in industry. All of the gauges, however, require regular calibration. In order to increase the reliability of calibration, high-quality working standards of coating thickness are required.

The goals of the thesis are the following - research of the precision characteristics of coating thickness gauges, development of the working standards and calibration methods of coating thickness gauges and standards as well as elaboration of a new technique of calculation of uncertainty of calibration results.

In the current research, bearing in mind the development of technology as well as the growing demand for the coating thickness standards and coating thickness gauges, the theoretical bases of calculating the indeterminacy of the method of coating thickness measurement was elaborated. Practical experiments have been carried out, in the course of which the theory of uncertainty calculation has been verified. Methods developed as a result of the current work have been adopted in the Laboratory of Metrology of Tallinn University of Technology, where the latter have also found practical application.

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

KOKKUVÕTE

Pindepaksuse mõõtevahendite kalibreerimismeetodite uurimine

Seoses tööstuse kiire arenguga on kasvanud ka nõudmised tööstustoodangu kvaliteedile. See omakorda on tinginud vajaduse uute ja kaasaegsete mõõte-vahendite järele. Pindepaksuse mõõtetehnika on viimastel aastakümnetel läbi teinud väga kiire arengu. Firmad toovad igal aastal turule uusi purustusvabasid pindepaksuse mõõtevahendite mudeleid, mis on kaasaegse disainiga ja võimaldavad üha täpsemalt ja mugavamalt (pinnet purustamata) mõõta tööstuses kasutatavate erinevate pinnakatete paksusi. Kõik need mõõtevahendid vajavad aga regulaarselt kalibreerimist. Kalibreerimistulemuse usaldusväärsuse tõstmiseks vajatakse kvaliteetselt kalibreeritud pindepaksuse tööetalone ja kaasaegseid pindepaksusmõõturite kalibreerimismetoodikaid.

Kuni tänaseni on selle teemaga aktiivselt tegelenud prof. R. Laaneots Tallinna Tehnikaülikoolist, kes on tunnustatud spetsialist nii meil kui ka Euroopa metroloogiaringkondades. Tema teemakohaseid uurimistöid avaldati juba 80-ndatel aastatel.

Kuna pidevalt arendatakse välja uusi pindepaksusmõõtureid, siis see on tinginud vajaduse nii pindepaksusetalonide konstruktsiooni kui ka pindepaksusmõõturite ja pindepaksusetalonide kalibreerimismeetodite edasiarendamiseks. Käesolev uurimistöö ongi prof. R. Laaneotsa seniste teadusuuringute edasiarendus silmas pidades uusi suundi pindepaksusmõõturite väljaarendamisel ja nende tootmises.

Doktoritöö põhieesmärk lähtuvalt ülalnimetatud probleemi püstitusest on kaasaegsete pinnet mittepurustavate pindepaksusmõõturite täpsuskarakteristikute uurimine, nende kalibreerimiseks kasutatavate pindepaksuse tööetalonide väljaarendamine, pindepaksusmõõturite ja pindepaksusetalonide uudse kalibreerimismetoodika ja saadavate kalibreerimistulemuste määramatuse arvutusmetoodika väljatöötamine.

Peamised sihid, mida autor endale eesmärgi saavutamiseks seadis, olid järgmised:

- Kaasaegsete pindepaksusmõõturite täpsuskarakteristikute uurimine.
- Magnetiliste pindepaksusmõõturite mõõtmise mudeli väljaarendus.
- Pindepaksuse määratluse uue meetodi väljaarendus ja selle matemaatiline analüüs.
- Väljaarendatud pindepaksuse määratluse kohta määramatuse arvutusmetoodika väljatöötamine.
- Pindepaksuse tööetaloni kontuuri uue mõõtemeetodi väljaarendus ja selle meetodikohasel kontuuri mõõtmisel saadava mõõtetulemuse määramatuse arvutusmetoodika.
- Pindepaksusmõõturite uue kaasaegse kalibreerimismeetodi väljaarendus.

Doktoritöö koosneb sissejuhatusest, kolmest põhipeatükist ja järeldustest. Esimeses peatükis kirjeldatakse põhitõdesid, mis on seotud mõõtevahendite kalibreerimise ja tööetalonidega. Esitatakse üldistatud kujul seni kasutusel olevad mõõtmise ja kalibreerimise mudelid ja vastavad mõõtmise ja kalibreerimise metoodikad. Samuti on esimeses peatükis välja toodud olulisemad pindepaksuse mõõtmise ja pindepaksusmõõturite kalibreerimise alased määratlused.

Teises peatükis vaadeldakse erinevaid pindepaksusetalonide tüüpe, kirjeldatakse lähemalt TTÜ pindepaksusetalonide konstruktsioone ja kalibreerimismeetodeid ning esitatakse nende uurimistulemused. Põhitähelepanu teises peatükis on osutatud pindepaksuse määratluse uuele väljaarendatud meetodile ja selle matemaatilisele analüüsile, aga ka selle meetodi määramatuse väljatöötatud arvutusmetoodikale. Esitatakse ka selle uue meetodi rakendusuuringu tulemused, mis kinnitavad väljaarendatud pindepaksuse määratluse õigsust nii pindepaksusetaloni kui ka pinde kontuuri mõõtmisel.

Kolmas peatükk keskendub pindepaksusmõõturite kalibreerimismeetodite uurimisele. Tutvustatakse kahte enamlevinud pindepaksusmõõturite tüüpi – magnetinduktiivsed ja pöörisvoolude meetodil töötavad mõõturid. Selles peatükis on kirjeldatud ka MIKROTEST tüüpi mõõtevahendite mõõtmise mudel ja selle alusel saadavate mõõtetulemuste määramatuse väljatöötatud arvutusmetoodika. On esitatud MIKROTEST tüüpi pindepaksusmõõturite kalibreerimistulemused, mis on teostatud nii Eestis kui ka Saksamaal. Tulemused kinnitavad saadava mõõtetulemuse määramatuse arvutusmetoodika õigsust. Samuti on kirjeldatud pindepaksusmõõturite kalibreerimise uus meetod, mille puhul kalibreerimisel on kasutatud pikkusmõõturit ja pindepaksuse imitaatoreid.

Töö järeldustes on kirjeldatud uurimistöö tulemused, mis on järgmised:

1. Tõenäosusteooriast ja matemaatilisest statistikast tuntud Monte-Carlo meetodit kasutades on välja arendatud uus pindepaksuse määratluse meetod. See meetod võimaldab täpsemalt määratleda (defineerida) mõõtesuurust, st. pindepaksust, mis on eriti vajalik pindepaksusetalonide kalibreerimisel.
2. On välja arendatud uus pindepaksusetaloni kontuuri mõõtemeetod ja selle meetodi kasutamisega etaloni pindepaksuse mõõtmisel saadavate mõõtetulemuste määramatuse arvutusmetoodika. Seda meetodit on juba kasutatud ühe komplekti Tallinna Tehnikaülikooli pindepaksuse tööetalonide (nikkelpinne terasel) kalibreerimisel.
3. Lähtudes universaalsest mõõtemääramatuse hindamise kontseptsioonist on välja arendatud magnetiliste pindepaksusmõõturite abil teostatava pindepaksuse mõõtmise meetodi matemaatiliselt põhjendatud määramatuse arvutamise teoreetilised alused, mis praktiliselt läbi viidud katsete käigus on ka kinnitust leidnud.
4. Uurimistöös väljaarendatud mõõtmise mudeli abil on uuritud magnetiliste pindepaksusmõõturite täpsuskarakteristikuid, mille tulemused olid lähteks pindepaksusmõõturite uute kalibreerimismudelite ja -meetodite välja-töötamisel.

5. On välja töötatud ja igapäevasesse kalibreerimistegevusse rakendatud uus pindepaksusmõõturite kalibreerimismeetod, mille puhul tööetalonina kasutatakse kalibreeritud pikkusmõõtevahendit ning kalibreerimis-protseduuris imiteeritakse pinnet õhupilu või kilede abil ja mille tulemusel tunduvalt (umbes kaks korda) väheneb kalibreerimistulemuse määramatus.

Uurimistöö tulemused olid aluseks uue patentse pindepaksusetalonide kalibreerimise meetodi väljaarendamiseks, mille kohta on esitatud patenditaotlus Saksamaal. Selle meetodi teoreetilised ja rakendusuuringud jätkuvad.

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2. Abiline, I., Laaneots, R., Leibak, A., Riim, J. The coating thickness and its definition. In: Proceedings of the 6th International Baltic Conference Industrial Engineering: Ed. R. Kyttner. Tallinn: DAAAM Baltic, Tallinn University of Technology, DAAAM International Vienna, 2008, 13 – 18
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4. Abiline, I., Laaneots, R., Nanits, M., Riim, J. Measurement uncertainty of surface contour with complicated form. In: Proceedings of the 5th International Conference of DAAAM Baltic Industrial Engineering – Adding Innovation Capacity of Labour Force and Entrepreneur: Ed. R. Kyttner. Tallinn: DAAAM Baltic, Tallinn University of Technology, DAAAM International Vienna, 2006, 101 – 105
5. Abiline, I., Laaneots, R., Nanits, M. Research results of the coating thickness standards. In: Proceedings OST-05 Symposium on Machine Design. Stockholm: KTH – the Royal Institute of Technology, 2005, 5 p
6. Abiline, I., Laaneots, R., Nanits, M. Calibration of thickness gauges. In: Proceedings 10th IMEKO TC7 International Symposium on Advances of Measurement Science: Ed. Sergey V. Muravyov. Saint-Petersburg: IMEKO, 2004, 372 – 373
7. Abiline, I., Laaneots, R., Nanits, M. Calibration of coating thickness gauges. In: Proceedings of the 4th International Conference Industrial Engineering – New Challenges to SME: Ed. J. Papstel/B. Katalinic. Tallinn: DAAAM Estonia, Tallinn University of Technology, DAAAM International Vienna, 2004, 117 – 119
8. Abiline, I., Laaneots, R., Nanits, M. The calibration method of coating thickness gauges. In: OST-03 Symposium on Machine Design. Oulu: Oulun Yliopisto Konetekniikan Osasto, 2003, 169 – 176
9. Abiline, I., Laaneots, R. Magnetic measuring method of coating thickness. In: Proceedings of the 2nd International Conference: Ed. J. Papstel/B. Katalinic. Tallinn: DAAAM Estonia, Tallinn University of Technology, DAAAM International Vienna, 2000, 19 – 22
10. Abiline, I., Laaneots, R. Calibration of thread limit plug gauges. In: Proceedings of the Third National DAAAM Conference in Estonia. Science' 98. Tallinn: Tallinn University of Technology, 1998, 30 – 32

ELULOOKIRJELDUS

1. Isikuandmed

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2. Kontaktandmed

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3. Hariduskäik

Õppeasutus (nimetus lõpetamise ajal)	Lõpetamise aeg	Haridus (eriala/kraad)
Tallinna Tehnikaülikool	1999	Metroloogia/ tehnikateaduste magister
Tallinna Tehnikaülikool	1984	Masinaehitustehnoloogia/ mehaanikainsener
Tallinna 2. Keskkool	1979	Keskharidus

4. Keelteoskus

Keel	Tase
Eesti	emakeel
Vene	kesktase
Soome	kesktase
Inglise	kesktase

5. Täiendusõpe

Õppimise aeg	Õppeasutus või muu org.-ni nimi
22-24. september 2004	MIKES, Soome
8-12. oktoober 2004	MIKES, Soome

6. Teenistuskäik

Töötamise aeg	Tööandja nimetus	Ametikoht
1995-...	PRENAR OÜ	juhataja

1999-2003	Tallinna Tehnikaülikool	assistent
1991-1995	AS “Viro Tools”	müügijuht
1987-1991	vabrik “Salvo”	tehnoloogiainsener
1885-1987	PI “EKE Projekt”	insenerkonstruktor

7. Kaitstud lõputööd

„Keermekaliibrite kalibreerimine” M. Sc. (Metroloogia), Tallinna Tehnikaülikool, 1999.
„Termiliselt töödeldud kõvasulamite kulumiskindluse uurimine” Dipl. Ins. (Mehaanika), Tallinna Tehnikaülikool, 1984.

8. Teadustöö põhisuunad

Mõõtevahendite täpsuskarakteristikute uurimine sõltuvalt mõjuritest ja mõõtevahendite kalibreerimine

CURRICULUM VITAE

1. Personal data

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Date and place of birth:	18.01.1961 Tallinn

2. Contact information

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3. Education

Educational institution	Graduation year	Education (field of study/degree)
Tallinn University of Technology	1999	Metrology/M.Sc Eng.
Tallinn University of Technology	1984	Production engineering/ Mechanical Engineer
Tallinn 2nd Secondary School	1979	High school

4. Language skills

Language	Level
Estonian	mother tongue
Russian	average
Finnish	average
English	average

5. Special Courses

Period	Educational or other organisation
22-24. September 2004	MIKES, Finland
8-12. Oktober 2004	MIKES, Finland

6. Professional employment

Period	Organisation	Position
1995-...	PRENAR LLC	manager
1999-2003	Tallinn University of Technology	assistent

1991-1995	“Viro Tools”	sales manager
1987-1991	“Salvo” factory	technology engineer
1885-1987	PI “EKE Projekt”	engineer

7. Defended thesis

„Calibration of thread limit gauges” M. Sc. (Metrology), Tallinn University of Technology, 1999.
„Research of wear resistance of hard metals” Dipl. Eng. (Mechanics), Tallinn University of Technology, 1984.

8. Main research interest

Research of accuracy characteristics of the measuring instruments depending from different factors and the calibration of measuring instruments

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.
4. **Paul Kallas**. Abrasive erosion of powder materials. 1996.
5. **Jüri Pirso**. Titanium and chromium carbide based cermets. 1996.
6. **Heinrich Reshetnyak**. Hard metals serviceability in sheet metal forming operations. 1996.
7. **Arvi Kruusing**. Magnetic microdevices and their fabrication methods. 1997.
8. **Roberto Carmona Davila**. Some contributions to the quality control in motor car industry. 1999.
9. **Harri Annuka**. Characterization and application of TiC-based iron alloys bonded cermets. 1999.
10. **Irina Hussainova**. Investigation of particle-wall collision and erosion prediction. 1999.
11. **Edi Kulderknup**. Reliability and uncertainty of quality measurement. 2000.
12. **Vitali Podgurski**. Laser ablation and thermal evaporation of thin films and structures. 2001.
13. **Igor Penkov**. Strength investigation of threaded joints under static and dynamic loading. 2001.
14. **Martin Eerme**. Structural modelling of engineering products and realisation of computer-based environment for product development. 2001.
15. **Toivo Tähemaa**. Assurance of synergy and competitive dependability at non-safety-critical mechatronics systems design. 2002.
16. **Jüri Resev**. Virtual differential as torque distribution control unit in automotive propulsion systems. 2002.
17. **Toomas Pihl**. Powder coatings for abrasive wear. 2002.
18. **Sergei Letunovitš**. Tribology of fine-grained cermets. 2003.
19. **Tatyana Karaulova**. Development of the modelling tool for the analysis of the production process and its entities for the SME. 2004.
20. **Grigori Nekrassov**. Development of an intelligent integrated environment for computer. 2004.
21. **Sergei Zimakov**. Novel wear resistant WC-based thermal sprayed coatings. 2004.
22. **Irina Preis**. Fatigue performance and mechanical reliability of cemented carbides. 2004.

23. **Medhat Hussainov**. Effect of solid particles on turbulence of gas in two-phase flows. 2005.

24. **Frid Kaljas**. Synergy-based approach to design of the interdisciplinary systems. 2005.

25. **Dmitri Neshumayev**. Experimental and numerical investigation of combined heat transfer enhancement technique in gas-heated channels. 2005.

26. **Renno Veinthal**. Characterization and modelling of erosion wear of powder composite materials and coatings. 2005.

27. **Sergei Tisler**. Deposition of solid particles from aerosol flow in laminar flat-plate boundary layer. 2006.

28. **Tauno Otto**. Models for monitoring of technological processes and production systems. 2006.

29. **Maksim Antonov**. Assessment of cermets performance in aggressive media. 2006.

30. **Tatjana Barashkova**. Research of the effect of correlation at the measurement of alternating voltage. 2006.

31. **Jaan Kers**. Recycling of composite plastics. 2006.

32. **Raivo Sell**. Model based mechatronic systems modeling methodology in conceptual design stage. 2007.

33. **Hans Rämmal**. Experimental methods for sound propagation studies in automotive duct systems. 2007.

34. **Meelis Pohlak**. Rapid prototyping of sheet metal components with incremental sheet forming technology. 2007.

35. **Priidu Peetsalu**. Microstructural aspects of thermal sprayed WC-Co coatings and Ni-Cr coated steels. 2007.

36. **Lauri Kollo**. Sinter/HIP technology of TiC-based cermets. 2007.

37. **Andrei Dedov**. Assessment of metal condition and remaining life of in-service power plant components operating at high temperature. 2007.

38. **Fjodor Sergejev**. Investigation of the fatigue mechanics aspects of PM hardmetals and cermets. 2007.

39. **Eduard Ševtšenko**. Intelligent decision support system for the network of collaborative SME-s. 2007.

40. **Rünno Lumiste**. Networks and innovation in machinery and electronics industry and enterprises (Estonian case studies). 2008.

41. **Kristo Karjust**. Integrated product development and production technology of large composite plastic products. 2008.

42. **Mart Saarna**. Fatigue characteristics of PM steels. 2008.

43. **Eduard Kimmari**. Exothermically synthesized B_4C-Al composites for dry sliding. 2008.