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TOPOGRAFICAL CALIBRATION METHOD OF COATING THICKNESS STANDARDS

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Abstract: *The paper presents a new method for calibration of coating thickness standards which was developed in Tallinn University of Technology. The new method allows us to measure the coating thickness of thickness standards by way of measuring the area between the upper and the inner boundary surface of the surface coating, in other words to calibrate the thickness standards according to the definition of the thickness and thus reduce the uncertainty component of the definition.*
The paper presents the coating and its thickness between real coating surfaces according to the definition for surface coating thickness. The definition is proved by mathematical model, which gives statistical distribution description for the coating surface. The definition proposed for surface coating thickness is checked through practical tests, which allows to evaluate the appropriateness of theoretical considerations elaborated.
Keywords: coating thickness, definition of coating thickness, measurement of coating thickness, coating thickness standard, uncertainty of results of coating thickness

1. INTRODUCTION

Coating thickness standards are used for determining, sustaining and reproducing a certain value of a coating thickness. Such standards are by their nature cuboids (that is the bases) which are made of a specific material and which are covered with a specific material in the middle of the top surfaces of the cuboids.

Coating thickness standards are used as working measurement standards for the purpose of calibrating the non-destructive principle based coating thickness measurement instruments. Thickness standards which are as links ensuring the metrological traceability need to be calibrated as well. Rather substantial methods are used for calibration of the coating thickness standards.

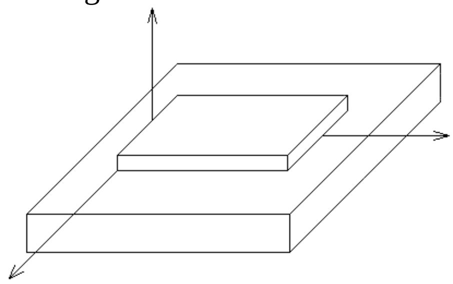

Fig.1. Coating thickness standard

All those methods use the measurements of the quantities describing the geometrical surface of the base before coating and the measurements quantities describing the geometrical surface of the coating after the coating process. But 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.
Therefore methods, which are widely used, measure the coating thickness by evaluating the height from the surface of the coating down to the surface of the base aside which is not under the coating. That means that the calibration is not done according to the definition of coating

thickness. The disadvantage of such calibration methods is their relatively low accuracy and these methods do not allow carrying out the comparison-calibration of the thickness standards. Naturally there are non-destructive measurement methods (ultrasound, X-ray, etc.) which enable to measure coating thickness according to its definition [1]. However, such methods do not allow the direct measurement of coating thickness and cannot thus be used for the initial calibration of coating thickness standards [2].

2. THICKNESS MEASUREMENT OF COATING THICKNESS STANDARDS

Let us relate the coating thickness standard to the cross coordinate system 0XYZ so that the plane of the cross coordinate system 0XY 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 Fig. 1).

It is clear that if we want to measure coating thickness non-destructively on the basis of its definition, we must be able to measure the lower and upper boundary surface of the coating. The only option in use is the measurement of surfaces before and after the coating process. The top surface of the coating thickness standard is measured in several sections before coating (see Fig. 2 for the measuring lines). Thereby we get data on the base of the top surface of the coating thickness standard, a part of which will be under the coating in future. Thereafter the surface of the base of the coating thickness standard is covered with a coating using required technology. The whole measurement process is repeated. Since by the definition of coating thickness it is necessary to determine the distance between the lower and upper boundary surface, the measurement data in each section must also characterise the lower and upper boundary surface [2,3]. Whichever technology is used, the measurement results will never characterise the total boundary surface, but only a certain part of it, i.e. the measurement results in those coordinates where the measurement was carried out.

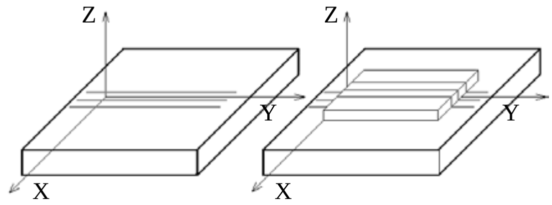

Fig. 2. Measurements before and after the coating process

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

The problem would not be there, if the base surface was made absolutely flat or at least flat enough to prevent the deviations related to the XY coordinate from having an effect on the measurement results. That, however, would require standard bases of very high quality. Even the end faces of gauge blocks are made by the use of modern technology in such a way that the flatness deviation of the end faces does not exceed the value of 0.05 µm. This means that if the described method is not used, that component has to be included in uncertainty calculations to its full extent. The double measurement is needed in order to prevent it and allow cheaper and faster manufacture of coating thickness standards.

3. POSITIONING

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

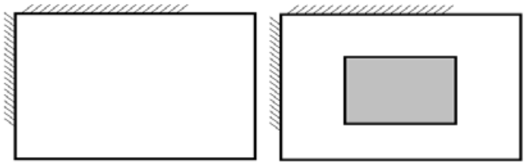

Fig. 3. The positioning of a coating thickness standard using base surfaces

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

The Figure 4 and 5 shows an example how positioning influences measurement results. The black line depicts the measurement line before the coating application and the grey line stands for measurements that were taken after the coating application.

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

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

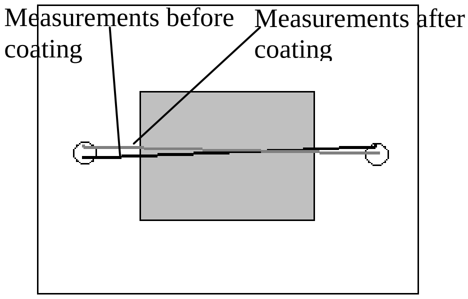

Fig. 4. Drift caused by positioning

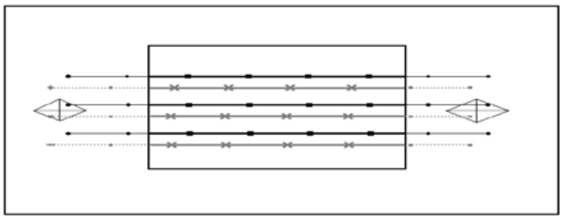

Fig. 5. Positioning related drift towards the X and Y axis

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

The rougher and more deviated (the more curved) both of the surfaces are, the more precise must the position be. Or in other words, the more the positioning in the XY direction influences the results of coating thickness measurement in the Z direction, the more precisely must the standard be positioned for both measuring operations. Figure 6 shows how the deviation of positioning has a direct effect on the result of coating thickness measurement.

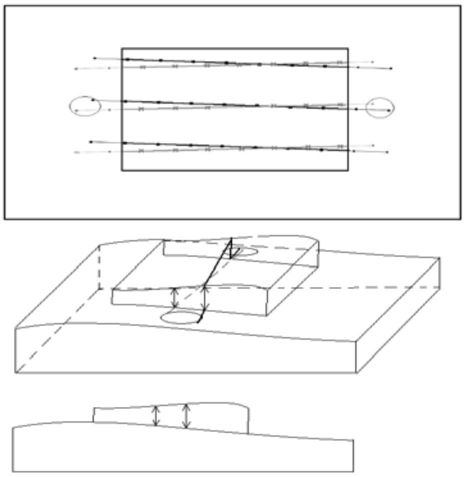

Fig. 6. The effect of positioning on the results of coating thickness measurement.

4. CALCULATING OF MEASUREMENT RESULTS

The measuring of the coating thickness standard gives a random function $Z_1(y)$ characterising the base surface in the intersection from y_1 to y_2.

After the coating application and second measurement we get a random function $Z_2(y)$ characterising the top surface of the coating in the same intersection from y_1 to y_2 [4].

$$Z_{i1}(y) = \tilde{Z}_{i1} + a_{i1}\, y^3_3 + a_{i1}\, y^2_2 + a_{i1}\, y + b_{i1}$$
$$Z_{i2}(y) = \tilde{Z}_{i2} + a_{i2}\, y^3_3 + a_{i2}\, y^2_2 + a_{i2}\, y + b_{i2} \qquad (1)$$

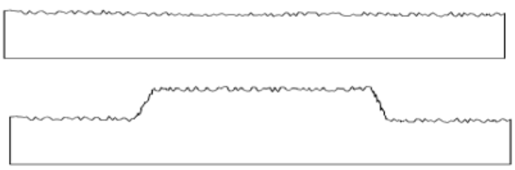

Fig. 7. Surface profiles in the section parallel to the XZ plane

The means of those functions can be expressed as follows

$$m_{Z_1}(y) = a_1\, y_3^3 + a_1\, y_2^2 + a_1\, y_1 + b_1$$
$$m_{Z_2}(y) = a_2\, y_3^3 + a_2\, y_2^2 + a_2\, y_1 + b_2 \qquad (2)$$

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

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

$$h_i(y) = Z_{i2}(y) - Z_{i1}(y) \qquad (3)$$

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

$$m_{ih}(y) = m_{Zi2}(y) - m_{Zi1}(y) = a_{i13}y^3 + a_{i12}y^2 + a_{i11}y + b_{i10} - a_{i23}y^3 - a_{i22}y^2 - a_{i21}y - b_{i20} \qquad (4)$$

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

$$H = \frac{\sum_{i=1}^{n} m_{ih}}{n} \qquad (5)$$

5. MEASUREMENT UNCERTAINTY

The uncertainty of the mean coating thickness of the coating thickness standard can be calculated from the relation

$$u(H) = \frac{\sum_{i=1..n} \sqrt{D_{i\,h}(y) + u^2(z) + u_{pos}^2 \left(\frac{\partial h}{\partial x \partial y}\right)^2}}{i} \qquad (6)$$

If we assume that the functions $Z_1(y)$ and $Z_2(y)$ are independent, the dispersion of the function characterising the coating thickness is:

$$D_H(y) = D_{Z2}(y) + D_{Z1}(y) \qquad (7)$$

The standard uncertainty component in the direction of the Z-axis can be found from the relation [5]

$$u(z) = \sqrt{u_{cal}^2 + u_{envi}^2 + u_{Ra}^2} \qquad (8)$$

where u_{cal} is the standard uncertainty of the calibration of the measurement instrument, u_{envi} is the standard uncertainty of the environment (noise) and u_{Ra} is the standard uncertainty on the surface roughness. The positioning related combined standard uncertainty component in the direction of the X- and Y-axis appears twice, thus

$$u_{pos} = 2\sqrt{\left(u_X^2 + u_y^2 + u_{resY}^2 + u_{resX}^2\right)} \qquad (9)$$

where u_X is the uncertainty of the positioning in the direction of the X-axis of the profile, u_Y is is the uncertainty of the positioning in the direction of the Y-axis of the profile, u_{resY} is the uncertainty component in the direction of the X-axis caused by the position of the device, and u_{resX} is the uncertainty component in the direction of the Y-axis caused by the position of the device. For calculating the uncertainty caused by the flatness deviation of the base of the coating thickness standard and uneven distribution of the coating thickness. I have used the assumption that the value of $\frac{\partial h}{\partial x \partial y}$ is approximately equal to the standard uncertainty $u(\Delta_{XY})$ caused by the unevenness of the base of the standard and coating thickness

$$u(\Delta_{XY}) = \sqrt{u^2(\Delta_{SX}) + u^2(\Delta_{SY}) + u^2(\Delta_{CTX}) + u^2(\Delta_{CTY})} = \sqrt{\left(\frac{\Delta_{SX}}{2\sqrt{3}}\right)^2 + \left(\frac{\Delta_{SY}}{2\sqrt{3}}\right)^2 + \left(\frac{\Delta_{CTX}}{2\sqrt{3}}\right)^2 + \left(\frac{\Delta_{CTY}}{2\sqrt{3}}\right)^2} \qquad (10)$$

where Δ_{SX} is the flatness deviation of the base in the X direction, Δ_{Sy} is the flatness deviation of the base in the Y direction, Δ_{CTX} is the change of coating thickness in the direction of the X-axis, and Δ_{CTY} is the change of coating thickness in the direction of the Y-axis.

6. EXPERIMENTAL RESEARCH RESULTS

The measurements were performed in the Coating Thickness Laboratory PTB (Physikalich-Technische Bundesanstalt), Braunschweig. The measuring instrument Tencor P-11 was used. The sample was a specially manually micropolished sample of nickel that was galvanised in copper. The flatness deviation of the base surface is within the limits from 3 µm per millimetre to 5 µm per millimetre (in the middle of the measuring area within the limits from 1 µm per millimetre to 2 µm per millimetre). The change of coating thickness in the centre of the standard was within the limits from 0,2 µm per millimetre up to 0,3 µm per millimetre.
Thus

$$u(\Delta_{XY}) = \sqrt{\left(\frac{2}{2\sqrt{3}}\right)^2 + \left(\frac{2}{2\sqrt{3}}\right)^2 + \left(\frac{0,3}{2\sqrt{3}}\right)^2 + \left(\frac{0,3}{2\sqrt{3}}\right)^2} \approx 0,8\,\mu m/mm$$

For double measurement of the sample (before and after coating), special marks (Knoop microhardness tester)were used for positioning. Upon measuring, we tried to hit as accurately as possible the centre of the marks. The hit uncertainty in the X direction has been calculated on the basis of a half of the mark width (Δ_x = 15 µm), and in the Y direction the hit uncertainty has been calculated on the basis of the triple distance between measurement points ($\Delta_y = 3 \cdot 8 = 24$ µm).

$$u_{pos} = 2\sqrt{\left(\left(\frac{1}{2\sqrt{3}}\right)^2 5 + \left(\frac{2}{2\sqrt{3}}\right)^2 4 + 1^2\right)} \approx 1,4\mu 6$$

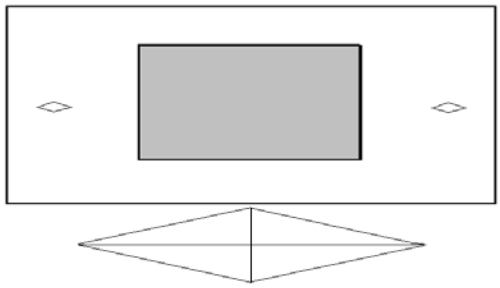

Fig. 9. Marks on the coating thickness standard made by the Knoop microhardness tester

In the case in question the worst possible measurement conditions have been taken into account, i.e. as if the largest deviations were in the middle of the measuring area. Therefore the first profile before coating should be for example as much as possible on the upper right area (in Figure 5 the line marked 1), and on the second profile that is

measured after coating should be on the lower left area (in Figure 5 the line marked 2).
Thus the value of the combined uncertainty component in the direction of the Z-axis is $u(z)=\sqrt{0.07^2+0.005^2+1^2}=0.3\,\mu\text{m}$. The dispersions of the functions have been found experimentally and the standard uncertainty of the function characterising coating thickness is $D_H(y)=0.01\,\mu\text{m}$.

The expanded uncertainty of the mean coating thickness in that case is

$$U(H)=2\cdot\frac{\sum_{i=1}^{n}\sqrt{D_{ih}(y)+u^2(z)+u^2_{pos}\left(\frac{\partial h}{\partial x\partial y}\right)^2}}{i}\approx 0.41\,\mu\text{m}$$

The largest uncertainty component in the combined uncertainty equation is the component caused by surface roughness (*R*a = 0.2 µm). If that component is reduced twice, the expanded uncertainty that characterises the measurement result is approx. four times smaller. The measurement results of a *i*-th profile have been presented in Figure 10, where the profile marked 1 stands for the measurement of the base surface before coating and the profile marked 2 represents the measurement result after coating. The defect with the depth of 5 µm visible on the left is the trace of the positioning mark.

Figure 11 presents the mean functions $m_{ih}(y)=m_{Zi2}(y)-m_{Zi1}(y)$ characterising the mean coating thickness of a coating thickness standard, indicating also the mean value of coating thickness in the middle of the standard *H* = 8.8 µm.
Figure 11 below presents the mean functions $m_{ih}(y)=m_{Zi2}(y)-m_{Zi1}(y)$ characterising the mean coating thickness of a coating thickness standard, indicating also the mean value of coating thickness in the middle of the standard *H* = 8.8 µm.

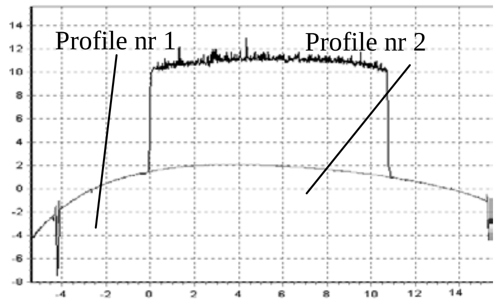

Fig. 10. The surface profiles in the section parallel to the XZ plane

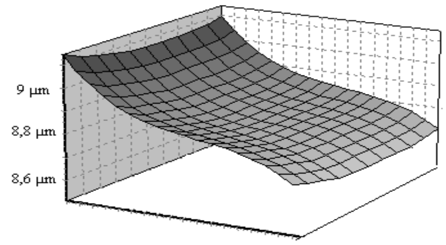

Fig. 11. Functions characterising coating thickness

7. CONCLUSIONS

As the procedure of metrology control of the coating thickness measuring instruments can be assured only with a calibrated coating thickness standard, it is very important to increase the accuracy of coating thickness standards.
This method enables to calibrate coating thickness standards considerably cheaper and more accurately, since the calibration of the coating thickness standard is done on the basis of the definition of coating thickness. That increases the accuracy of calibration in comparison to the calibration methods known so far and also allows to carry out the recalibration of the standards in the future.

8. REFERENCES

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