MĀPAN - Journal of Metrology Society of India, Vol. 23, No. 3, 2007; pp. 167-175

A 3D-Calibration Method for the Quantitative SPM Measurement of Hardness Intenders

MARTIN RITTER[1], THORSTEN DZIOMBA[2], MATTHIAS HEMMLEB[3]
LUDGER KOENDERS[2] and AXEL KRANZMANN[1]
[1]Federal Institute for Materials Research and Testing (BAM), Unter den Eichen 87, 12205 Berlin, Germany
[2]Physikalisch-Technische Bundesanstalt Braunschweig und Berlin (PTB),
Bundesallee 100, 38116 Braunschweig Germany
[3]m2c GbR, Alt Nowawes 83a, D-14482 Potsdam
e-mail: martin.ritter@bam.de

[Received : 21.06.2007]

Abstract

We introduce the application of a new 3D calibration method for the calibration of scanning probe microscopes (SPM) in order to be able to obtain faster and better quantitative topography measurements for the determination of technical, dimensional and geometrical surface parameters. The applied 3D-calibration routine is based on spatially distributed reference landmarks (nanomarkers), and, not only determines the scale factors in all dimensions but also the coupling factors as shear between all coordinate axes in one step. We show that the 3D-calibration method is a valuable alternative and enhancement to existing calibration strategies that conventionally apply two calibration steps, i.e., separate determinations of the calibration parameters for lateral and vertical scales. As an application example to technical surfaces, a hardness micro-indenter measured by SPM is discussed. The uncalibrated data, the conventionally calibrated data, and, the 3D-calibrated data are compared and analyzed.

1. Introduction

Spatial coordinate measurements, e.g., by scanning probe microscopy (SPM) are necessary to obtain the micro-topography, and, therefore, the geometrical functionality of technical surfaces at different scales. The determination of these parameters of either technical, processed or machined surfaces is a basic requirement for the analysis, interpretation and understanding of their durability, or, of the mechanical processes that have formed the geometry of the surface structures, respectively.

The classical technique for the quantitative analysis of topographies and technical parameters of surfaces is the stylus profilometer [1-2]. In production control, it is the main internationally standardized method for the determination of, e.g., surface roughness parameters. These parameters can be crucial in quality control, for example, if linked to the durability of adhesive layers of multi-component materials. However, the stylus technique has various drawbacks. According to the guidelines, this technique only permits the evaluation of 2D parameters based on profiles. Additionally, structures with high aspect ratio are a problem due to the apex angle of the diamond tip of the stylus profilometer, and, samples of susceptible materials are subject to scratches due to the contact pressure of the diamond tip. For applications at the lateral micrometer and the vertical nanometer range, other methods have to be considered, as scanning probe microscopy (SPM) or, e.g., confocal laser scanning microscopy (CLSM) or other.

Consequently it is crucial to ensure comparability of results obtained by different techniques and to

develop methods for correlative analysis, in order to obtain additional information. However this is often not sufficiently fulfilled up to now. For example, comparison measurements with stylus instruments and confocal scanning laser microscopy [3] as on ion-exchange membranes for gas separation, or, on ceramic layers of spray deposits that serve as thermal insulation (barrier) coatings (TBC) on gas-turbine blades have shown that both methods are insufficient to answer specific questions, such as to the real surface area. Already a decade ago the influence of the surface topography of adhesive substrates on the durability of TBCs has been investigated [4]. However, both articles provide an indication of the possibility to correlate the surface topography parameters to the durability of the entire layer system.

This paper describes the realization of a different approach. Together with the Physikalisch-Technische Bundesanstalt (PTB), the Federal Institute for Materials Research and Testing (BAM) started a DFG project within the main frame programme SPP1159 [5]. The goal of the project is the survey of the micro-topography of a variety of technical surfaces, e.g., membranes, reaction interfaces, boundary layers as well as friction surfaces with several 3D measurement methods in order to achieve a comprehensive analysis of the technical and geometrical parameters, and, to be able to exploit the final results for installing a quality control protocol. Hardness indenters and indentations as analyzed in this work mark a good and simple possibility to evaluate the specific calibration strategies and 3D measurement methods. In the long term, we plan to establish a standardized 3D-method for the evaluation of technical and machined surfaces.

Recently, we presented a new spatial reference standard and a 3D- calibration strategy based on spatially distributed landmarks (nanomarkers) for the geometrical calibration of scanning probe microscopes in one step [6]. Here, the new 3D-calibration method is exemplarily presented by its application to the determination of the geometry, i.e., especially the height and apex angle of a Vickers diamond tip indenter. The results are compared to the measurement data of the uncalibrated and conventionally calibrated SPM. The measured apex angles are inserted in the formulas for the determination of the apex angle dependent Vickers and Martens hardness factors c and A_s.

2. Hardness Testing

2.1 Hardness Testing According to the Vickers and the Martens Method

The determination of Hardness according to Vickers (HV) is regulated by ISO documents [7-8]. It is calculated by the relation of the testing force to the surface of the indentation Eq. (1) :

$$\text{Hardness} = \frac{\text{Test force}}{\text{Surface of indentation}} \tag{1}$$

The test procedure consists of three steps: the step method, the verification and the calibration of the hardness testing machine and the calibration of hardness reference blocks. For all steps, measurement uncertainties have been evaluated [9]

As hardness indenter, a diamond pyramid with a square base-area is used. The hardness after testing can be calculated by Eq. (2)

$$HV = c \cdot \frac{F}{d^2} \tag{2}$$

In Eq. (2), F is the test force and d is the mean of the two diagonals of the pyramidal shaped indentation.

$$c = 1.102 \cdot 2 \cdot \sin\left(\frac{\alpha}{2}\right) \tag{3}$$

The constant c is derived from Eq. (3) with α being the apex angle of the diamond pyramid. If the apex angle α of the diamond pyramid is exactly 136°, then the constant c is 0.1891. Because of the investigated indenter was intended for use in the macro-range of Martens hardness [10] the relationship between Vickers and Martens hardness is explained. In contrast to the Vickers hardness, for Martens hardness (HM) the indentation depth h is measured under test force instead of the diagonal length d. HM is defined as shown in Eq. (4):

$$HM = \frac{F}{A_s \cdot h^2} \tag{4}$$

The factor A_s is explained in Eq. (5). At an apex angle α of exactly 136°, the value of A_s equals 26.43. Both factors used in the determination of the Vickers or Martens hardness - c and A_s - depend on the apex angle a of the macro-indenter diamond tip used. Here, the apex angle is determined by scanning probe microscopy (SPM).

$$A_s = 4 \cdot \frac{\sin\left(\frac{\alpha}{2}\right)}{\cos\left(\frac{\alpha}{2}\right) \cdot \cos\left(\frac{\alpha}{2}\right)} \quad (5)$$

3. 3D-Calibration of Scanning Probe Microscopes

3.1 Conventional Calibration of Scanning Probe Microscopes

The geometric calibration of scanning probe microscopes is necessary to achieve accurate dimensional measurements. The SPM allows direct spatial coordinate measurements, however, the measurements are usually performed on microscopes without direct traceability to the meter definition. Therefore, the actually accomplished scanning measurement is usually distorted, and, a great deal of the distortions is produced because of limitations of the scan-generator, but also due to external factors such as thermal drift [11-13]. In order to achieve a proper scale definition, and, to overcome imperfections in the scanning movement, such microscopes have to be calibrated. Except for a few so called Metrology SPM (mainly of National Metrology Institutes) equipped with laser-interferometry for traceable position control, physical transfer standards need to be used for SPM calibration. SPM calibration by physical standards is described by guideline VDI / VDE 2625 Part 1 [14]. This guideline currently serves as a basis for international standardization (ISO201SC9).

The development and fabrication of highly accurate dimensional standards has been and still is being pursued by commercial companies and research projects [15-16]. Currently, the calibration of SPM is performed by separate calibration measurements for the lateral xy-plane, and, the z-axis, in order to obtain scale, linear and non-linear correction parameters. For each of these procedures - the horizontal and the vertical calibration - two separate kinds of reference standards have to be applied. The main drawback of such a separated 2-step calibration process is that orthogonality between each of the lateral coordinate axes x and y with respect to the z-axis is always assumed in the model, but not proven. Additionally, by separating the calibration procedure into two steps, the determination of the z-scale factor for the calibration measurement is an averaging process not depending on the lateral sampling coordinates, a situation that is not given in actual measurements. Therefore, local correlations of the vertical measurement height with respect to the lateral position cause residual position errors that cannot, or, at least, are difficult to be detected [17].

3.2 Landmark Based Calibration

For the calibration of SPMs we use an alternative approach with pyramidal reference structures that carry spatially distributed landmarks (Fig. 2). Landmarks are also called control points or fiducial marks. Here, they are referred to as nanomarkers due to their sub-micrometer size. Landmarks in general, are unambiguous geometrical features on real objects that can be detected by image processing methods. Landmarks define discrete positions, and, therefore, are widely used for coordinate measurements or calibration purposes in geoinformation sciences, but also for the trace of body movements in sport sciences or in car-safety testing (Fig. 1). We established the use of landmarks for the micro-range, by applying them on 3D-reference structures that have been fabricated

Fig. 1. Reference marks at close-range

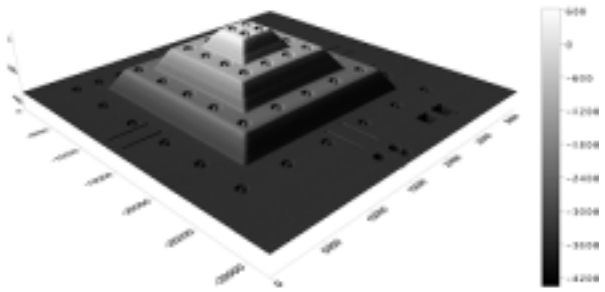

Fig. 2. 3D Calibration structure with reference marks at micro- and nano-range (scale in nm)

by focused-ion beam technology (FIB), using platinum deposition for the build-up of the pyramidal reference structures, and, milling for etching the landmarks [18].

Several geometries of such features are possible [19-20]. We chose a ring-shaped variant with a radius of roughly 200 nm for stable identification in the measurement of image data (Fig. 2). Besides the substrate, the nanomarkers are only applied to the flat regions of the pyramidal structure. In this way the z-coordinate of the nanomarkers is well defined. The distribution of the nanomarkers on the substrate level of the reference structure is non-symmetrical, in order to be always informed of the orientation of the pyramid. This feature also allows for a clear mapping of the nanomarkers to a predefined arrangement pattern.

3.3 3D Calibration

The geometric calibration of scanning microscopes is necessary to achieve accurate dimensional measurements. A great deal of systematic errors introduced into the measurement comes from imperfections of the scanning system [12]. An ideal scanning movement (Fig. 3) forms a coordinate system x (XYZ) with orthogonal scanning axes and constant step sizes in all directions. The actually accomplished, usually non-ideal scanning movement forms the measurement coordinate system w ($w_x w_y w_z$) that is only an approximation to that ideal, simply because of limitations of the hardware performing the scan.

In order to restore the orthogonal coordinate system x a 3D-correction of the measurement coordinate system by a 3D-affine transformation is necessary. This is achieved by a least-squares parameter estimation where measured coordinates wi (w_{xi}, w_{yi}, w_{zi}) are registered to the "known" object coordinates x_i ($X_i Y_i Z_i$) [6]. In order to be able to perform the parameter estimation, corresponding points in the reference coordinate system and the measurement coordinate system have to be available. These homologous points are provided by the nanomarkers.

(6)

After this linear 3D calibration in one step, the estimated affine transformation parameters (listed in Table 1) can then be used for the correction of the measurement data by applying the transformation shown above in Eq. (6).

4. Results

4.1 Calibration of SiS SPM

4.1.1 Conventional calibration

The measurements of the micro-hardness

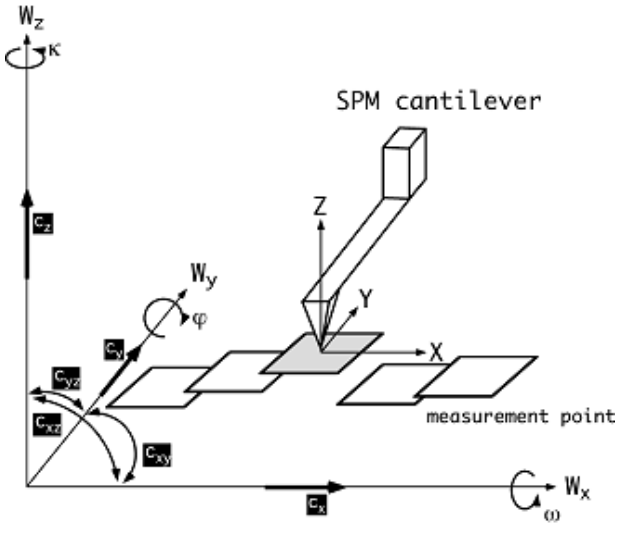

Fig. 3. Linear distortion of coordinate systems

Table 1
All linear rigid (position) and affine (distortion) parameters of the 3D-calibration

Rigid Parameters	
x_θ	Translation in x-direction
y_θ	Translation in y-direction
Z_θ	Translation in z-direction
φ	Rotation around y-axis
ω	Rotation around x-axis
κ	Rotation around z-axis
Affine Parameters	
C_x	Scale in x-direction
C_y	Scale in y-direction
C_z	Scale in z-direction
C_{xy}	Couplaing of x - and y - axis
C_{xz}	Couplaing of x - and z - axis
C_{yz}	Couplaing of y - and z - axis

Table 2
Scale factors obtained by conventional calibration of SiS nc-SFM

Conventional Calibration	c_x	c_y	c_z
Value	0.999	1.013	0.9905
Uncertainty	0.002	0.003	0.007

indenter were performed with an optimized commercial scanning force microscope (SiS nc-SFM), that is a modified NANO Station II (SIS GmbH, Herzogenrath, Germany), in the PTB clean-room centre. The instrument does not belong to the highest traceability class of laser-interferometer metrology SPMs, but it is equipped with a closed-loop capacitive position controlled xy-piezo scanstage (PI Physik Instrumente, Karlsruhe, Germany) and a piezostack for z-scanning with a strain gauge as a z-sensor. While this instrument has proven to be very stable and suitable for high-accuracy measurements, its potential can only be exploited by a careful fine-calibration [16].

The instrument has been first conventionally calibrated by transfer standards that have been used in international comparisons, or, that have been certified by PTB's metrology large range SPM (Met. LR-SPM) based on the nano-measuring machine (NMM, SIOS GmbH, Ilmenau, Germany) [21] . The metrological properties of the SiS nc-SFM have been characterized in detail [22]. The set of scale correction parameters of all axes was determined by transfer standards and is shown in Table 2.

4.1.2 3D-calibration

For the determination of the reference coordinates for the 3D calibration, the pyramidal reference structures were measured with the Met. LR-SPM. In order to obtain the affine calibration factors for the SiS nc-SFM, the same structures were measured with the device in non-contact mode. The total number of SiS measurements analyzed was 15, the total number of Met. LR-SPM measurements was 8. Two typical Met. LR-SPM and SiS nc-SFM measurements are shown in Figs 4 and 5.

The nanomarker reference coordinates of the Met. LR-SPM measurement, and the SiS nc-SFM measurement coordinates were then automatically detected by "m2c microCal" software (m2c company, Potsdam, Germany), as shown in Figs 6 and 7. The

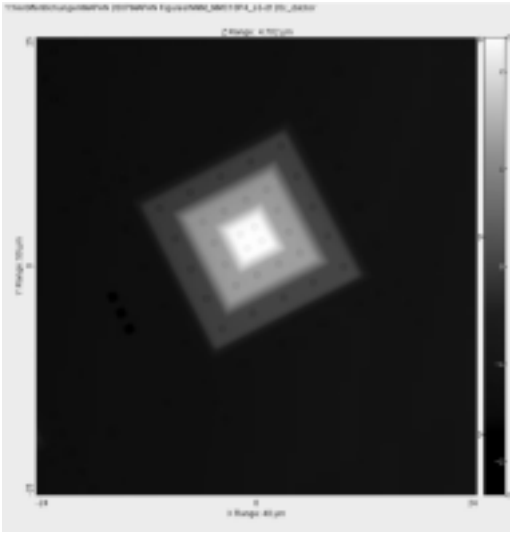

Fig. 4. Pyramidal 3D reference structure measured by Met. LR-SPM (ROI = 48 µm x 50 µm)

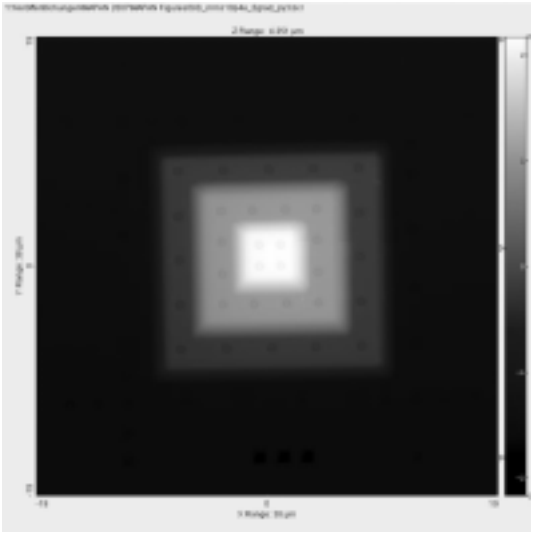

Fig. 5. Identical pyramidal 3D reference structure measured by SiS nc-SFM (ROI = 38 µm x 38 µm)

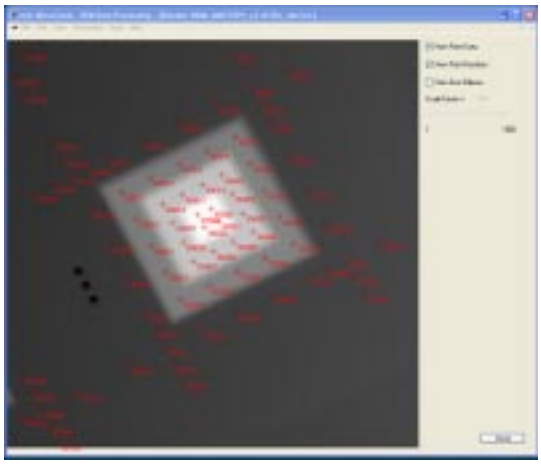

Fig. 6. Automatic detection and allocation of nanomarkers in NMM measurement data

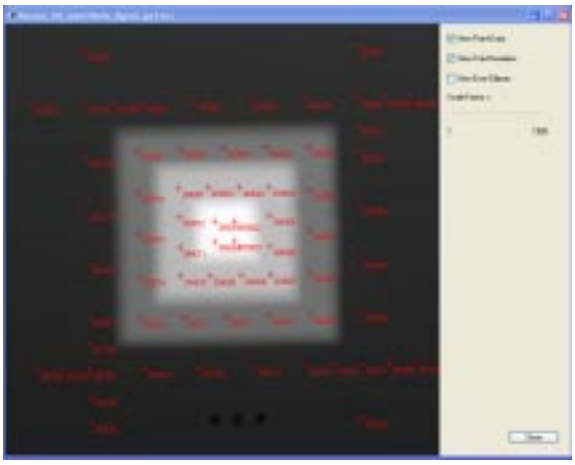

Fig. 7. Automatic detection and allocation of nanomarkers in the SiS nc-SFM measurement data

Table 3
Scale and coupling factors for SiS nc-SFM obtained by 3D calibration method

3D Calibration	c_x	c_y	c_z	c_{xy}	c_{xz}	c_{yz}
Value	0.9976	1.0111	0.9913	0.0019	-0.0119	-0.1325
Standard Deviation	0.0006	0.0020	0.0034	0.0010	0.0048	0.0111

software also automatically performs the allocation of the homologous points in each of the measurement sets, and, it carries out the least-squares parameter estimation to determine the affine correction factors. In our case, all twelve parameters were estimated (Table 1). The results are shown in Table 3.

The significance of the coordinate registration can very nicely be demonstrated by the resulting mean point error xp, that is a by-product of the parameter estimation, usually derived from the square root of the weighted residues (Eq. (7)). It can be seen that a decrease in xp indicates smaller residual distances, a proof for the accuracy of the model that the calibration is based on. In the calibration experiments performed here, the mean point error of the affine 3D registration of the homologous points did not exceed 13 nm in measurement data, where the pixel size was more than 100 nm (i.e. a mean point error of 0.13 pixel), and, it did not exceed 5 nm in measurement data, where the pixel size was more than 38 nm (0.13 pixel). We therefore state an uncertainty in the determination of the coordinate position of 0.1 to 0.2 pixel.

$$\xi_p = \frac{1}{n}\sum_{i-1}^{n}\sqrt{(x_i - w_{xi})^2 + (y_i - w_{yi})^2 + (z_i - w_{zi})^2} \qquad (7)$$

Table 3 also reveals a good agreement of the scale factors, if compared to the results of the conventional SPM calibration (Table 2). But, it also shows a larger distortion of the measurement coordinate system by a strong negative coupling between the y- and the z-axis. This coupling may be attributed to the hardware, i.e., to the design of the SiS nc-SFM. Either the mounting of the piezostack can only be achieved within a certain accuracy, or, it might behave asymmetrically so that a lateral bending of its bottom end, where the cantilever holder is mounted, is induced whenever the piezostack expands in z-direction.

4.2 Hardness Indenter Measurements

The Vickers diamond indenter was measured by SiS nc-SFM. The z-offset of the PI scanstage was carefully adjusted in order to allow the measurement of the indenter tip. The upper 13 µm of the diamond could be imaged. The scan range was set to 100µm x 100µm, digitized at 1024 x 1024 pixel with a scan rate of 0.1 lines. In Figs 8 and 9, the height as measured by the strain gauge is shown. All image processing and

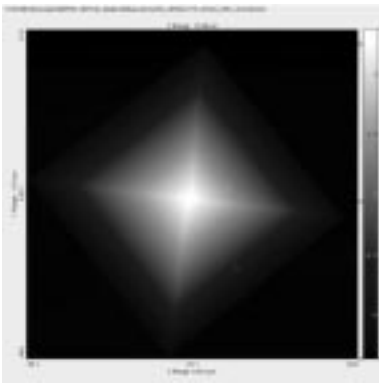

Fig. 8. Height coded SiS nc-SFM measurement of indenter diamond tip

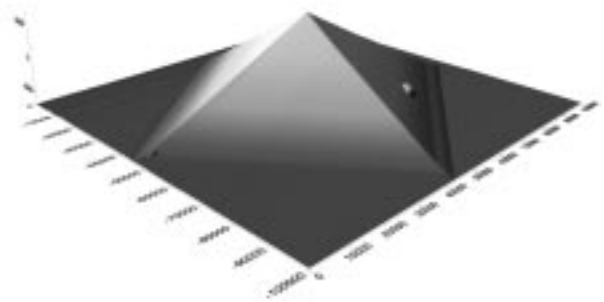

Fig. 9. 3D representation of SiS nc-SFM height measurement of indenter diamond tip

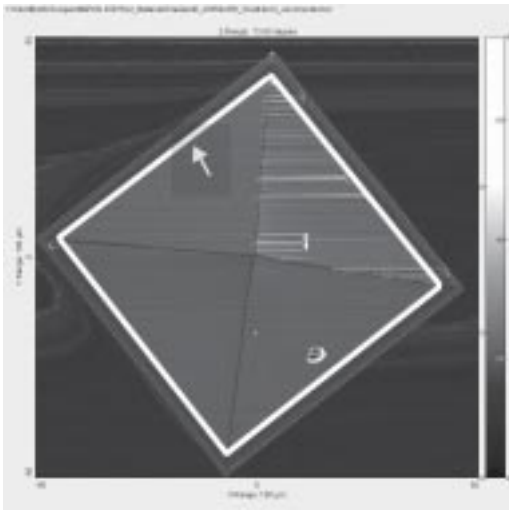

Fig. 10. Normal gradient within marked area (white line with arrow) was used for gradient histogram analysis

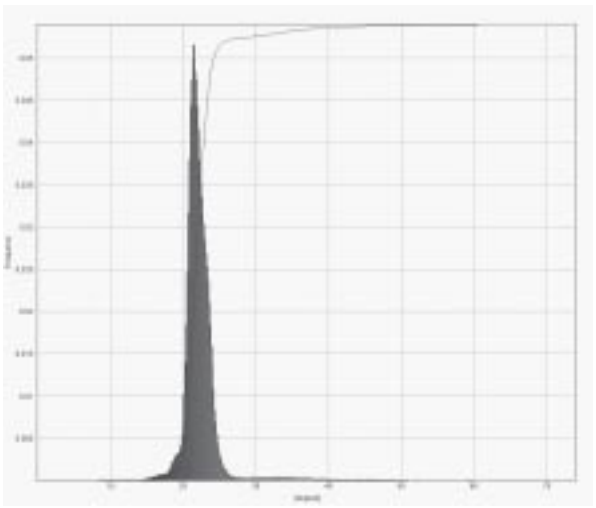

Fig. 11. Histogram of gradient SiS nc-SFM measurement data (frequency plot)

histogram analysis was performed with SPIP Software v. 4.4.11 (Image Metrology, Lyngby, Denmark).

Fig. 8 shows the indenter diamond tip measurement in a color-coded height representation. The same data were used to produce a 3D representation for a better visual impression (Fig. 9). The edge length was measured 68.3 µm, and the height was determined 13.66 µm after 3D calibration (13.76 µm in the uncalibrated measurement data).

SPIP software was also used to determine the normal gradient of the uncalibrated, conventionally calibrated, and 3D calibrated indenter measurements. In Fig. 10, the uncalibrated map of the normal gradients is shown. In Fig. 9 the area used for establishing the gradient histogram is marked by the red rectangle. Gradient histograms of the gradient map is shown in Fig. 11. The histograms of uncalibrated, conventionally calibrated and 3D calibrated normal gradient maps were calculated. All histograms show a maximum frequency peak around a gradient angle γ of 22 degrees. In order to determine the exact gradient angle of the uncalibrated, conventionally and 3D calibrated histogram data, the centroid γ_c of each frequency plot as in Fig. 11 was calculated according to Eq. (8).

Table 4
Results of gradient histogram analysis of uncalibrated, conventionally calibrated and 3D calibrated SiS nc-SFM measurement data

Parameter	Uncalibrated	Conventionally Calibrated	3D calibration
γ_c	22.3807°	22.1240°	22.0947°
α	135.2386°	135.7520°	135.8107°
c	0.18863	0.18898	0.18902
A_s	25.51224	26.12497	26.19639

$$\gamma_c = \frac{\sum_i (\gamma_i \cdot f_i)}{\sum_i f_i} \quad (8)$$

The results of the centroid determination of the gradient angle γ_c, and, the subsequent calculation of the apex angle α as well as the Vickers factor c and the Martens factor A_s can be taken from Table 4.

5. Discussion

We have shown that Vickers hardness intender diamond tips that are used for determination of the macro-range of Martens hardness can be calibrated by scanning probe microscopy (SPM), i.e. the SiS SPM used in this project. At the macro- and micro-range, tip blunting of such diamond tips can be neglected, as well as the intender area function for indentation depths larger than 6 µm [23]. Because we were using a macro-range hardness indenter as an example tool, we focused on the tip geometry, specifically the apex angle a of the diamond tip, due to its impact on the calculation of the hardness. The results of the determination of the apex angle a of the hardness indenter diamond tip suggest that a previous dimensional calibration of the SPM device is necessary. We compared uncalibrated, conventionally calibrated and 3D-calibrated SiS SPM data, and, the affect of the measured apex angle a on the Vickers factor c and the Martens factor As which is clearly visible. We therefore state as well that the applied 3D calibration is a valuable addition or enhancement to existing calibration strategies.

Acknowledgments

We thank G. Dai for the Met. LR-SPM measurements and K. Herrmann for discussion of the indenter tip measurements. This work is supported by the DFG (SPP1159).

References

[1] ISO 3274:1996: Geometrical Product Specifications (GPS) - Surface Texture: Profile Method - Nominal Characteristics of Contact (Stylus) Instruments (German Version EN ISO 3274:1997)

[2] H. J. Warnecke, and W. Dutschke, (Hrsg.): Fertigungsmesstechnik - Handbuch für Industrie und Wissenschaft. Springer-Verlag, Berlin, S., (1984) 431-448.

[3] C. Prüfer: Neue Kennwerte zur Bewertung Von Thermisch Gespritzten Schichten. Diplomarbeit FH Osnabrück (2003).

[4] A. G. Evans, D. R. Mumm, J. W. Hutchinson, G.H. Meier and F.S. Pettit: Mechanisms Controlling the Durability of Thermal Barrier Coatings. Prog. Mater. Sci., **46** (2001)) 505-553.

[5] http: / / www.stramnano.de

[6] M. Ritter, T. Dziomba, A. Kranzmann and L. Koenders : A Landmark-Based 3D Calibration Strategy for SPM. Meas. Sci. Technol., **18** (2007) 404-414.

[7] ISO 3878:1983: Hardmetals - Vickers Hardness Test.

[8] ISO 6507:2005: Metallic Materials - Vickers Hardness test - Part 1: Test Method; Part 2: Verification and Calibration of Testing Machines; Part 3: Calibration of Reference Blocks.

[9] T. Polzin, D. Schwenk, H. Frenz, and C. Vogt, : Determination of Uncertainty for Hardness Testing, Proposed Methods for the Standards of Hardness Testing. Prakt. Metallogr., **41** (2004) 506-516.

[10] ISO 14577-1:2002: Metallic Materials -

Instrumented Indentation Test for Hardness and Materials Parameters - Part 1: Test Method.

[11] C.T. Gibson, G. S. Watson and S. Myhra, : Scanning Force Microscopy - Calibrative Procedures for Best Practice Scanning, **19** (1997) 564-581.

[12] K. Henriksen and S.L.S. Stipp, : Image Distortion in Scanning Probe Microscopy American Mineralogist, **87** (2002) 5-16.

[13] X. Zhao : PTB-Report F-20 (Bremerhaven) (1995).

[14] VDI/VDE 2656 Part 1 : 2006-11 (Draft) Determination of Geometrical Quantities by Using Scanning Probe Microscopes - Calibration of Measuring Systems. (Expected Berlin 2007).

[15] L. Koenders, T. Dziomba, P. Thomsen-Schmidt and G. Wilkening : Standards for the Calibration of Instruments for Dimensional Nanometrology In: G. Wilkening and L. Koenders, Nanoscale Calibration Standards and Methods: Dimensional and Related Measurements in the Micro- and Nanometer Range, 245-257 (Weinheim 2005)

[16] T. Dziomba, L. Koenders and G. Wilkening : Standardization in Dimensional nanometrology : Development of a Calibration Guideline for Scanning Probe Microscopy. SPIE Europe Optical Design - Optical Fabrication, Testing and Metrology II, 5965 (12) (2005).

[17] M. Ritter : A Landmark-Based Method for the 3D Calibration of Scanning Microsopes, Dissertation, BAM-Dissertationsreihe Band, **21** (2007).

[18] O. Sinram, M. Ritter, S. Kleindiek, A. Schertel, H. Hohenberg and J. Albertz, : Calibration of an SEM, Using a Nano Positioning Tilting Table and a Microscopic Calibration Pyramid. ISPRS Commission V Symposium 2002, Close-Range Vision Techniques, Corfu, Greece, (2002) 210-215.

[19] M. Ritter, M. Hemmleb, O. Sinram, J. Albertz, and H. Hohenberg : A Versatile 3D Calibration Object for Various Micro-Range Measurement methods. Proceedings XXth ISPRS Congress Istanbul, **25** (2004) 696-701.

[20] M. Ritter, M. Hemmleb, B. Lich, P. Faber and H. Hohenberg : SEM / FIB Stage Calibration with Photogrammetric Methods. ISPRS Commission V Symp. 2006 (Int. Archives of Photogrammetry, Remote Sensing and Spatial Information Sciences) Vol. XXXVI Part 5 (2006).

[21] G. Dai, F. Pohlenz, H.-U. Danzebrink., M. Xu, K. Hasche and G. Wilkening : Metrological Large Range Scanning Probe Microscope. Review of Scientific Instruments, **75** (2004) 962-969.

[22] T. Dziomba, L. Koenders, H.-U. Danzebrink, G. Wilkening : Lateral & Vertical Calibration of Scanning Probe Microscopes and their Measurement Uncertainty. In: M. Dietzsch (Editor): Proceed. of the XIth Int. Colloq. on Surfaces, Chemnitz, Germany, 117-128 (Aachen 2004).

[23] Ch. Ullner : Development of the Instrumented Indentation Test. J. of Metrol. Soc. of India MAPAN, **20** (2005), 43-52.