Physikalisch-Technische Bundesanstalt

EURAMET.L-S15.a

(EUROMET Project No. 925)

Supplementary Comparison According to the rules of CCL key comparisons

EUROMET PROJECT 925

INTERCOMPARISON ON STEP HEIGHT STANDARDS AND 1D GRATINGS

FINAL REPORT

Braunschweig, 17. September 2009 / H.U. Danzebrink

1 INTRODUCTION

In 2004 the CCL supplementary comparison CCL-S2 for step height (Nano2) as well as the Euromet comparison No. 707 (Interlaboratory Step Height comparison for SPM) had been finished. In the meantime additional NMIs requested to join a subsequent comparison. PTB initiated therefore this comparison to disseminate the obtained knowledge to additional labs. Among them is the National Metrology Centre of A*STAR Singapore (formerly part of SPRING). The participation from Singapore helps to establish a link between EURAMET and the Asian Pacific Metrology Programme (APMP).

Finally, the Istituto Nazionale di Ricerca Metrologica (INRIM), the Centre for Metrology and Accreditation (MIKES), the NMi Van Swinden Laboratorium (NMi-VSL), the National Metrology Centre of A*STAR (NMC/A*STAR) and the Physikalisch-Technische Bundesanstalt (PTB) agreed to put through this comparison. In contrast to CCL-S2 this comparison focuses on the scanning probe microscopy (SPM) method to measure the set of step height and 1D grating standards. The comparison should support the Mutual Recognition Arrangement (MRA). The pilot laboratory for this comparison is the Physikalisch-Technische Bundesanstalt (PTB).

In autumn 2008 CMI asked PTB to perform a bilateral comparison in the field of nanoscaled standards. Since the comparison reported here was started already, it was agreed that CMI should perform their measurements on the same set of samples as the other participants. However, the measurements should be done after measurements of this comparison had been completed. The results of CMI should be sent to PTB before Draft A of the comparison would have been sent to the participants. CMI's results (see Appendix B) would not be taken into account for the reference value of this comparison, however the values should be compared to the reference values. By this procedure the CMI could refer to these measurements in order to verify their measurement capabilities in the CMC list.

2 STANDARDS

Two different types of standards were to be measured within this comparison: step height standards on the one hand and 1D gratings on the other hand. Both types should be measured using SPMs.

2.1 STEP HEIGHT STANDARDS

The step height standards used for this comparison were manufactured by Fraunhofer Institute of Microelectronics, Stuttgart, for PTB. They are available with step heights between 7 nm and 2000 nm. The standard is a chip of approx. 5 mm x 5 mm in size (Fig. 1). The structures featuring the step height consist of SiO_2 on a silicon substrate. The surface of the standard is coated with Chromium.

The structure on the chip comprises a rectangular frame for alignment purposes. The individual serial number of the standard is displayed in the upper left corner just below the writing "PTB". As this outer frame may suffer from slight contaminations due to the cutting of the Si-wafer and contact with tweezers, only the four single lines in the centre of this rectangular frame are intended for calibration and measurement purposes.

The four single lines of approx. 20 µm (left one), 3 µm, 100 µm and 6 µm (right one) in width are interrupted in the middle to help to navigate to the reference area, which is located slightly above the interruption of the bar. The reference area for this comparison is defined on the **6 µm line** as indicated in Fig. 2. Test approaches of the tip as well as test measurements are to be performed at the other lines (or on the "southern" branch of the 6 µm line) in a sufficient distance to the reference area to avoid the risk of contamination and/or damage in the reference area or its vicinity.

Please note that orientation structures, logos and serial number may have a step height significantly different from that of the structure intended for the comparison.

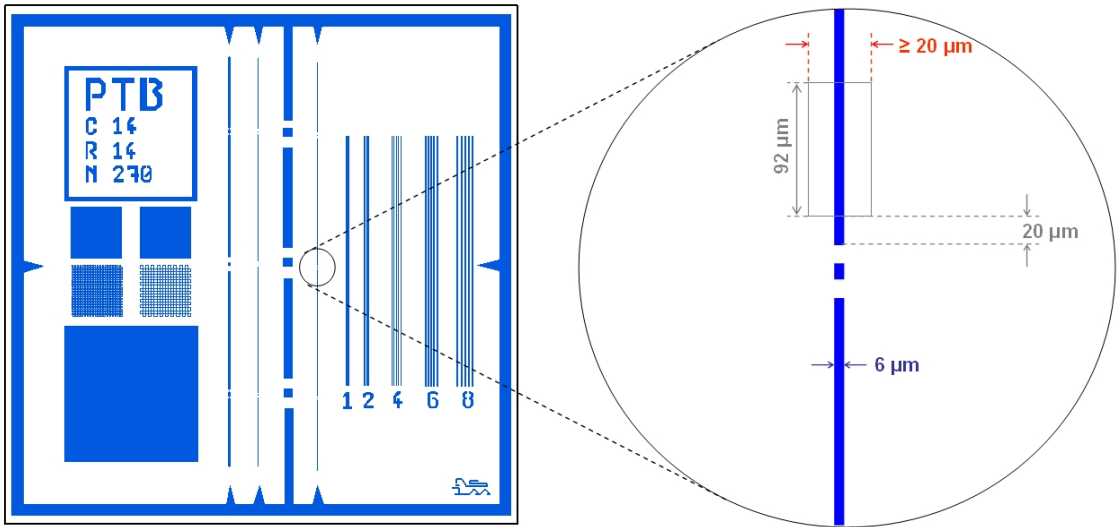


Fig. 1: Layout of the step height standards of 7 nm, 40 nm, 1000 nm, and 2000 nm step height. The substrate is silicon, the lines are silicon oxide, and the whole sample is covered by a chromium layer (not shown). There are four lines at the centre with widths of 20 µm, 3 µm, 100 µm and 6 µm. The line used for the comparison has a width of 6 µm and is located on the right side.

The five measurement positions MP1 to MP5 on the right bar (approx. 6 µm in width) are located at equidistant positions within a total area extending 92 µm along the bar, as indicated in Fig. 2. At each of these positions, a field of ≥20 µm x 12 µm is to be scanned. The lower end of MP1 is just 20 µm "north" of the beginning of the line, measured from its "northern" interruption. With each measurement field covering 12 µm of the bar, a gap of 8 µm is to be left between each subsequent pair of measurement fields, i.e. this the step height is not to be determined within this gap.

The measurements should have been repeated at least 5 times per MP and the step height values for the 5 individual MPs had to be reported.

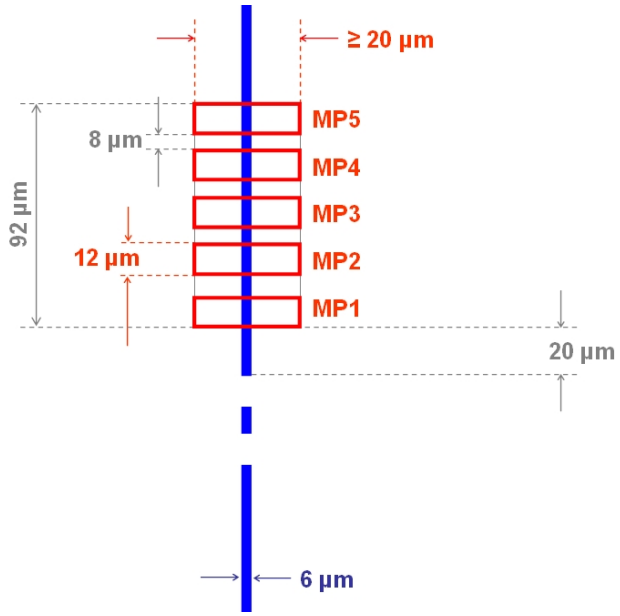


Fig. 2: Locations of the 5 measurement positions (of 20 µm x 12 µm each, with a spacing of 8 µm between neighbouring MPs). The whole area extends over 92 µm x 20 µm.

2.2 1D GRATING STANDARDS

The 1D gratings used in this comparison do have nominal pitches of 300 nm (called 1D300 hereafter for brevity) and 700 nm (1D700). The standards are identified by the manufacturer's (Moxtek Inc.) model code and serial number: 300-1D SN: 2330D084 and 700-1D SN: 2332F090, respectively.

Both standards consist of a silicon chip glued onto a metal disk of approx. 12 mm in diameter by the manufacturer (Fig. 3). The gratings cover the whole surface of the chip. According to the manufacturer, the grating structures are created by coating the silicon wafers with a polymer material and subsequent patterning of the photosensitive layer by laser-generated interference patterns. As a final step applied by the manufacturer, the lateral standards are coated by nominally 50 nm of tungsten.

Test measurements show that a step height of 120..140 nm (1D300) and 210..230 nm (1D700) is to be expected in the centres of the chips.

The silicon chips are in good approximation rectangular: The sizes are approx. 4 mm x 3 mm for the 1D300 and 3 mm x 4 mm for the *1D700.*

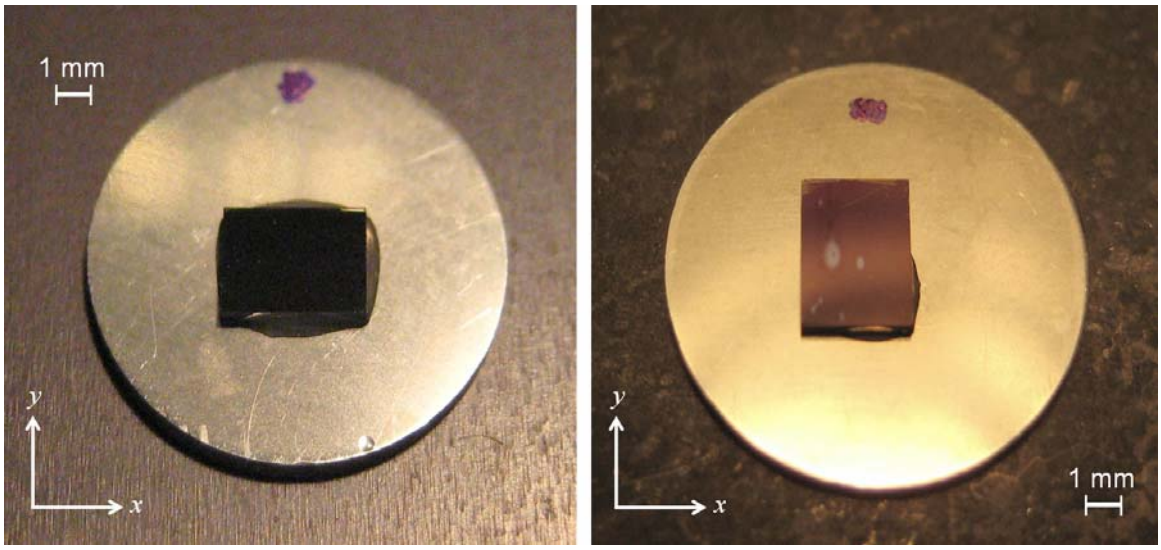


Fig. 3: Overview photographs of the lateral standards. Left: 1D300, right: 1D700.
Note: the grating's lines run from top to bottom in both images

The grating structure is well parallel to one of the chip edges, thus easing the mounting and pre-alignment. In the case of the 1D300, the lines are nearly parallel to the shorter edge, in the case of the 1D700, they are nearly parallel to the longer edge. The orientation and direction indications are defined as sketched in Fig. 3. Consequently, the lines run from the top to the bottom in both images shown in Fig. 3, and the fast scan axis measurement direction x is from left to right. Both samples are marked with a blue dot to indicate the "north" of the samples.

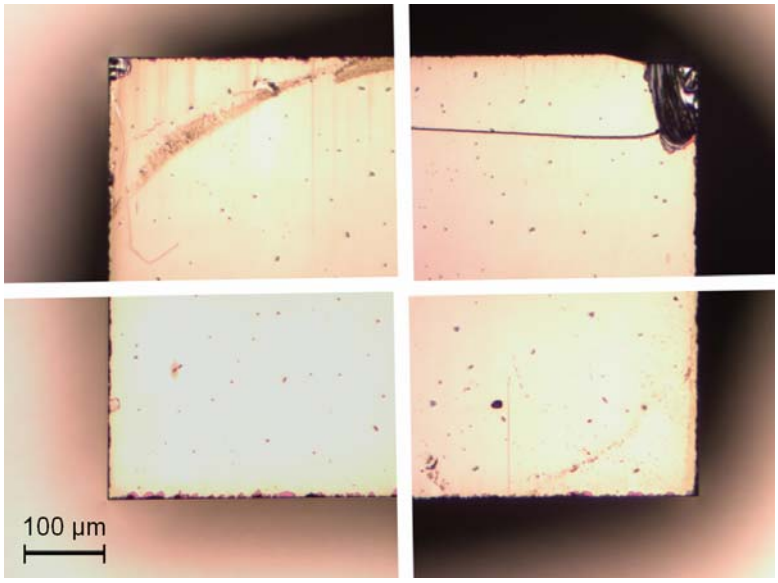


Fig. 4: Bright-field low magnification optical micrographs of the four corners of the 1D300.

Although these samples (bought in 2008) were taken out of the box only in the clean room centre, many small particles scattered all over the surface are visible (Fig. 4, with the 1D300 as example). These particles are so small and adhere to the surface so strongly that they cannot be easily blown off by ultra-pure nitrogen or any other clean room-compatible pressurized gas. So it was decided NOT to start any other attempts to remove these particles and also the participants are asked NOT to clean the samples.

Since there are no better or worse areas, with respect to contaminations, the measurement position is defined as the centre of the rectangular silicon chips for simplicity and practicability. The participants were asked to perform their measurements as closely to the centre as their positioning capabilities allow.

Test measurements showed that both centres are free of any major contamination particles or irregularities, but high resolution dark-field optical microscopy nevertheless did reveal many scattering centres. These tiny scattering centres have, however, turned out not to disturb scanning force microscopy significantly (Fig. 5) and can hardly be fully avoided when scanning images of several 10 µm in length and width. Nevertheless, it was recommended to try to avoid such regions and to acquire SFM images next to them at the nearest intact region instead. Larger particles are, furthermore, found in the vicinity of the centres and need to be avoided as well. Similarly, the participants were asked to move to the nearest cleaner region in case that the central region turns out to have become contaminated in the course of the comparison, e. g. due to wear or accidents.

No binding statement was made for the measurement range to be selected. The range should be chosen according to the participant's capabilities and his/her best instrument performance.

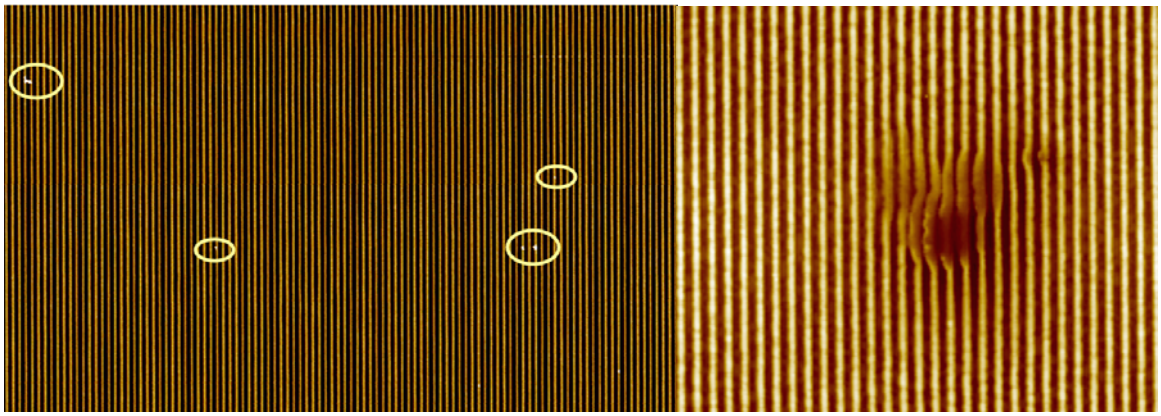


Fig. 5: Left: Minor particles on the 1D700 (SFM image); Right: Defect in the grating structure (here: SFM image of the 1D300)

3 PARTICIPANTS AND TIME SCHEDULE

3.1 ORGANISATION

The Technical Protocol of this comparison has been drafted following the rules set up by the BIPM[1]. Parts of it were taken from earlier protocols like "Nano4: 1D gratings" and "Euromet No. 707" (edited by F. Meli, METAS, and L. Koenders, PTB, respectively). By their declared intention to participate in this preliminary comparison, the participants accepted the general instructions and the technical protocol written down in the Technical Protocol document, which was sent to them and committed themselves to following the procedures.

[1] see http://www.bipm.org/utils/en/pdf/guidelines.pdf

3.2 REQUIREMENTS FOR PARTICIPATION

The participating laboratories should offer this measurement as a calibration service (now or in future), be willing to participate in a regional comparison in order to provide a link between the interregional and the regional comparisons.

3.3 PARTICIPANTS IN THE CIRCULATION

The participants of this comparison are listed in Table 1.

Table 1: List of Participants

Laboratory	***Responsible***	***Address***	***Phone:, Fax, e-mail***
INRIM	G. B. Picotto	Istituto Nazionale di Ricerca Metrologica (INRIM) Strada delle Cacce, 73 10135 - Torino ITALY	Phone: + 39 011 3919 969/973 Fax: + 39 011 3919 959 e-mail: g.picotto@inrim.it
MIKES	A. Lassila	Centre for Metrology and Accreditation (MIKES) P.O. box 9 (street address: Tekniikantie 1) FIN-02151 Espoo, FINLAND	Phone: + 358 10 6054413 Fax: + 358 10 6054 499 e-mail: Antti.Lassila@mikes.fi
NMC/A*STAR	S. H. Wang	Optical Metrology Department National Metrology Centre, NMC/A*STAR, Singapore 1 Science Park Drive Singapore 118221 SINGAPORE	Phone: +65 6279 1941 Fax: +65 6279 1994 e-mail: wang_shihua @nmc.a-star.edu.sg
NMi-VSL	R. Koops	Van Swinden Laboratorium (VSL) BV Department of Length Thijsseweg 11, 2629 JA Delft The NETHERLANDS	Phone: +31 15 269 1642 Fax: +31 15 261 2971 e-mail: rkoops@NMi.nl
Pilot laboratory			
PTB	H.U. Danzebrink	Physikalisch-Technische Bundesanstalt WG 5.25 Scanning Probe Metrology Bundesallee 100 D- 38116 Braunschweig GERMANY	Phone: +49 531 592 5136 Fax: +49 531 592 5205 e-mail: Hans-Ulrich.Danzebrink@ptb.de
Additional laboratory (see comment in chapter 1)			
CMI	P. Klapetek	Czech Metrology Institute Okruzni 31 638 00 Brno Czech Republic	Phone: +420 545 555 111 Fax: +420 545 555 183 e-mail: pklapetek@cmi.cz

3.4 TIME SCHEDULE

The comparison was carried out in circulation type. The period of time available to each laboratory was one month for calibration **and** transportation to the next participant. Each laboratory should have been capable of performing the measurement in the limited time allocated to it.

During the comparison a delay occurred owing to technical problems of the instrument at the NMi-VSL. Therefore, the NMi-VSL withdrew their participation.

Table 2: Time Schedule

Lab.	Country	Original schedule	Confirmation of reception	Shipment to the next partner
PTB	Germany	April 2008	-	29.04.2008
INRIM	Italy	May 2008	02.05.2008	06.06.2008
MIKES	Finland	June 2008	09.06.2008	04.07.2008
NMC/A* STAR	Singapore	July/August 2008	18.07.2008	28.08.2008
NMi-VSL	Netherlands	September 2008	September 2008	withdrawal of participation on 21.11.2008
PTB	Germany	October 2008	26.11.2008	final measurements to check the stability of the samples: February 2009
CMI	Czech Rep.	-	24.05.2009	05.06.2009

3.5 TRANSPORTATION AND HANDLING

The standards were stored in a special box (see Fig. 6). In the normal position the back of the sample holder was visible (see Fig. 7). A special tool (magnetic stick) was provided with the box to lift the sample holder with the standard. The samples should have been handled with care and under clean conditions, as stated in the Technical Protocol.

The box contains a set of 6 standards:

SH7_new	***7 nm***	***No. C 19 R 11 N 197,***
SH40_new	***40 nm,***	***No. C 05 R 14 N 261,***
SH1000_new	***1000 nm,***	***No. C 04 R 06 N 63,***
SH2000_new	***2000 nm,***	***No. C 06 R 06 N 65,***
1D300	***300 nm,***	***SN: 2330D084,***
1D700	***700 nm,***	***SN: 2332F090.***

Fig. 6: A photograph of the box with the standards and the tool for handling (magnetic stick). In the normal position the back of the sample holder (steel disk ∅~12mm) can be seen, while the standard's reference side faces to the bottom.

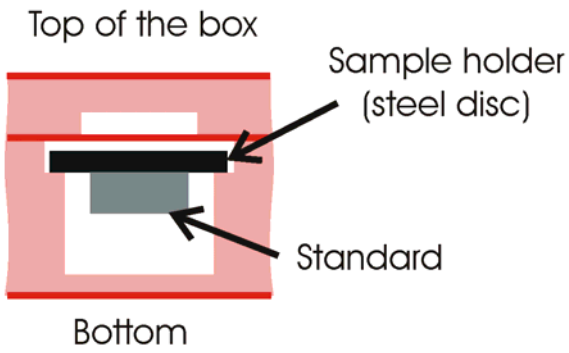


Fig. 7: Sketch showing the position of a standard in the box. Note: the structures are on the side facing downwards.

4 MEASURAND

4.1 MEASUREMENT AND EVALUATION OF THE STEP HEIGHT STANDARDS

POSITION OF THE MEASUREMENT

For the comparison the measurements were performed in the areas (MP1 to MP5) denoted in Fig. 2. The area MP1 is located 20 µm above the lower end of the line. The scanning fields should be 20 µm * 12 µm. At least 5 measurements should have been made in each area for the determination of the step heights.

DETERMINATION OF THE STEP HEIGHT

The step depth *h* is defined in analogy to ISO 5436. A continuous straight mean line is drawn over the line to represent the lower level of the surface and another representing the upper level, both lines extending symmetrically about the centre of the line (Fig. 8). The surface at the top of the line is assessed only over the

central part of its width. The step height *h* is defined as the perpendicular distance of the mean of the portion C to the line through the mean of portion A and the mean of portion B.

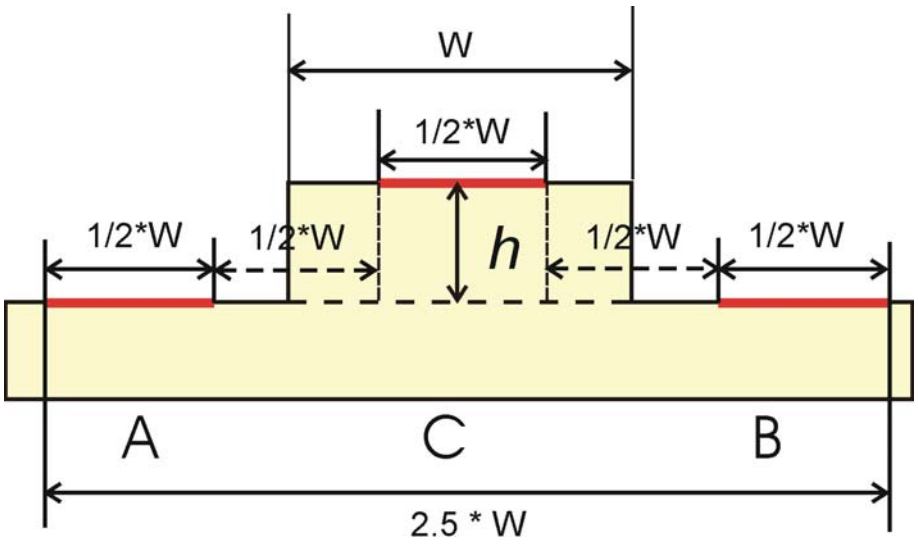


Fig. 8: Definition of step height h used in the comparison

The step height measurand to be used in this comparison is the average height obtained from measurements within the reference areas MP1 to MP5 as shown in Fig. 2. Additionally, the information from the individual 5 MPs should have been given.

All values had to be given for the reference temperature of 20° C.

4.2 MEASUREMENT AND EVALUATION OF THE 1D GRATING STANDARDS

POSITION OF THE MEASUREMENT

The pitch measurand to be used in this comparison is the average pitch from data recorded in the centre of the standard. No binding statement was made for the measurement range to be selected. The range should be chosen according to the participant's capabilities and his/her best instrument performance.

The direction of the pitch is orthogonal to the ribs of the grating. An indicator of the direction is marked as x-direction in Fig. 3.

DETERMINATION OF THE PITCH VALUES

The participants were free to choose their own method of evaluation of the pitch values. For each method applied a complete description of the method and a detailed estimation of the measurement uncertainty according to the ISO Guide to the Expression of Uncertainty in Measurement (GUM) had to be supplied.

All values had to be given for the reference temperature of 20° C.
Especially for the grating standards: it was recommended to keep the standards at lab. temperature for more than 2 days before the measurement is started (in order to let the temperature stabilise after transport).

4.3 REPORTING OF THE RESULTS

The participating laboratories sent their reports to the pilot laboratory. The reports contain:

- the measurement set-up and the conditions
- the result(s) of the measurements,
- the combined standard uncertainty
- the degrees of freedom,
- the complete uncertainty budget and the evaluation method.

The measured step height *h* and pitch values *p* have to be stated for the reference temperature at 20°C. The expansion coefficient of the amorphous silicon dioxide is $0.5*10^{-6}$/K. The uncertainty of the measurement has to be estimated according to *the Guide to the Expression of Uncertainty in Measurement (GUM)*.

5 MEASUREMENT INSTRUMENTS

Table 3 gives an overview about the instruments used and their traceability. The full description of the measurement methods and instruments by the participants can be found in Appendix A.

Table 3. Measurement instruments

No	Institute	Method	Instruments	Traceability
1	INRIM	AFM	Homebuilt SPM with plane mirror interferometer for x,y and capacitance transducer for z-direction	Laser traceable to INRIM standards
2	MIKES	AFM	Homebuilt SPM with plane mirror interferometer for x,y,z	Laser traceable to MIKES standards
3	NMC/A*STAR	AFM	Large Range SFM based on AFM head and Nano Measuring Machine (NMM).	Laser frequencies were calibrated by an iodine frequency stabilised He-Ne laser traceable to the SI unit for length
4	PTB	AFM	Large Range SFM based on AFM head and Nano Measuring Machine (NMM).	Lasers traceable to PTB standards

6 STABILITY OF THE STANDARDS

Each participant was asked to inspect the standards after reception (see Technical Protocol) and to send a report to the pilot laboratory. The reference areas on the standards remained almost unchanged. The stability of the standards was determined at the end of the comparison.

Table 4. Stability of the standards as measured by LR-SFM at PTB

	SH0007	SH0040	SH1000	SH2000
Date	***h* / nm**	***h* / nm**	***h* / nm**	***h* / nm**
April 2008	6,2	42,60	1014,10	2096,40
Febr. 2009	6,2	42,90	1014,50	2096,30
Difference	0	-0,30	-0,40	0,10

	1D300	1D700
Date	***p* / nm**	***p* / nm**
April 2008	287,603	700,763
Febr. 2009	287,589	700,776
Difference	0,014	-0,013

The results show that there are not any significant changes in step height values nor pitch values. Additional observation of the reference areas on the standards by optical microscope did not show any irregularities in the reference areas.

In the following, 3 photographs are shown from the SH0040, SH1000 and SH2000 (the step height feature of the SH0007 is nearly not visible – the image from the optical microscope shows a homogeneous area)

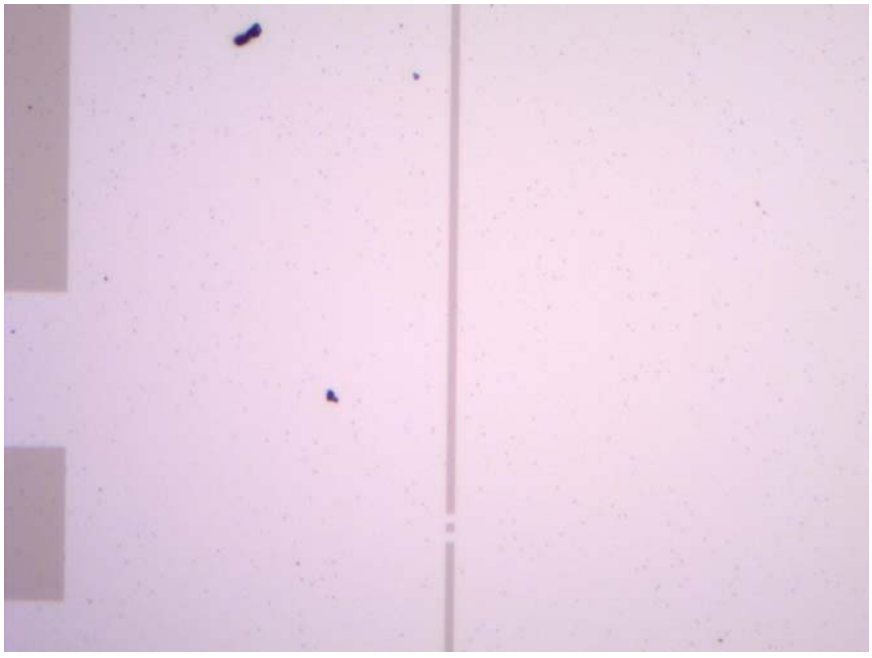


Fig. 9: Picture of the SH0040 obtained by optical microscope (10x objective).

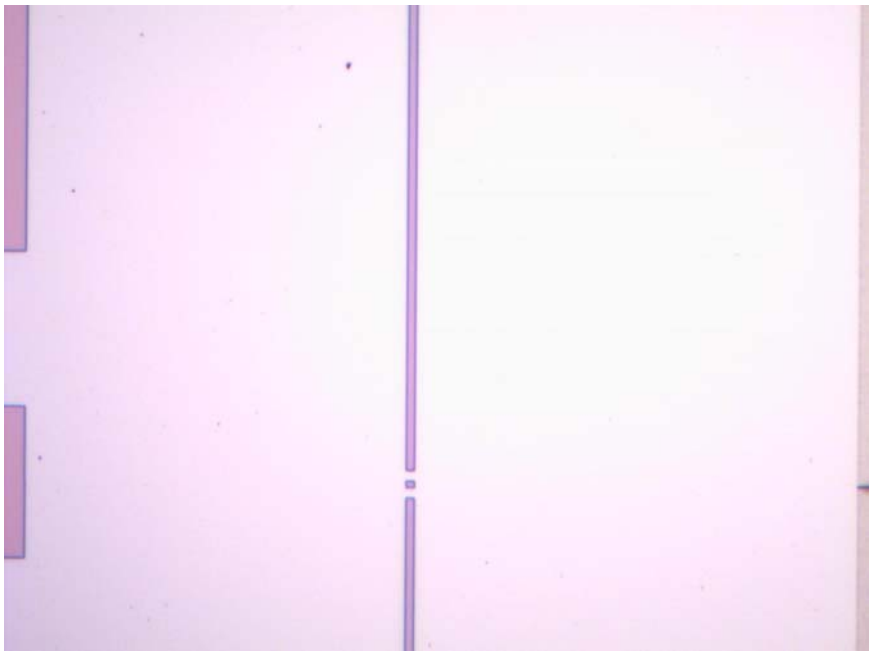


Fig. 10: Picture of the SH1000 obtained by optical microscope (10x objective).

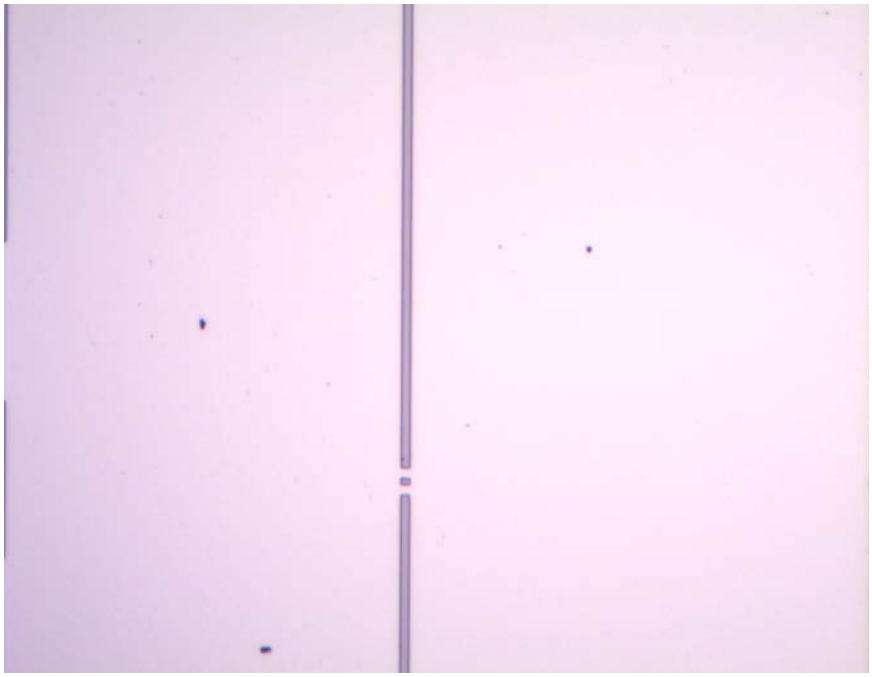


Fig. 11: Picture of the SH2000 obtained by optical microscope (10x objective).

7 MEASUREMENT RESULTS

The results received from all the participants are presented[2]. Besides the measured values for the step heights *h* and pitch values *p*, the combined standard uncertainties u_c, the degrees of freedom ν_{eff} and the expanded uncertainties *U*(k=2) are given. The other value E_n is explained below.

For some standards a deviation of reported values was observed. The participant was informed following the rules of the BIPM guidelines and was allowed to check the results before Draft A was distributed. In the case of correction the new value is given in the table, whereas the former results are indicated and listed below.

7.1 RESULTS ON STEP HEIGHT STANDARD SH0007

Table 5: Step height standard SH0007

SH0007 C19 R11 N197								
Institute	Country	Meas.	*h* / nm	u_c / nm	ν_{eff}(h)	k	*U*(k=2) /nm	E_n
PTB	DE	April 2008	6,2	0,5	50	2	1,0	0,10
INRIM	IT	May 2008	6,4	0,5	114	2	1,0	0,08
MIKES	FI	June 2008	6,14	0,35	17	2	0,70	0,20
NMC/A*STAR	Singapo-re	Aug. 2008	7,0	0,7	∞	2	1,4	0,47

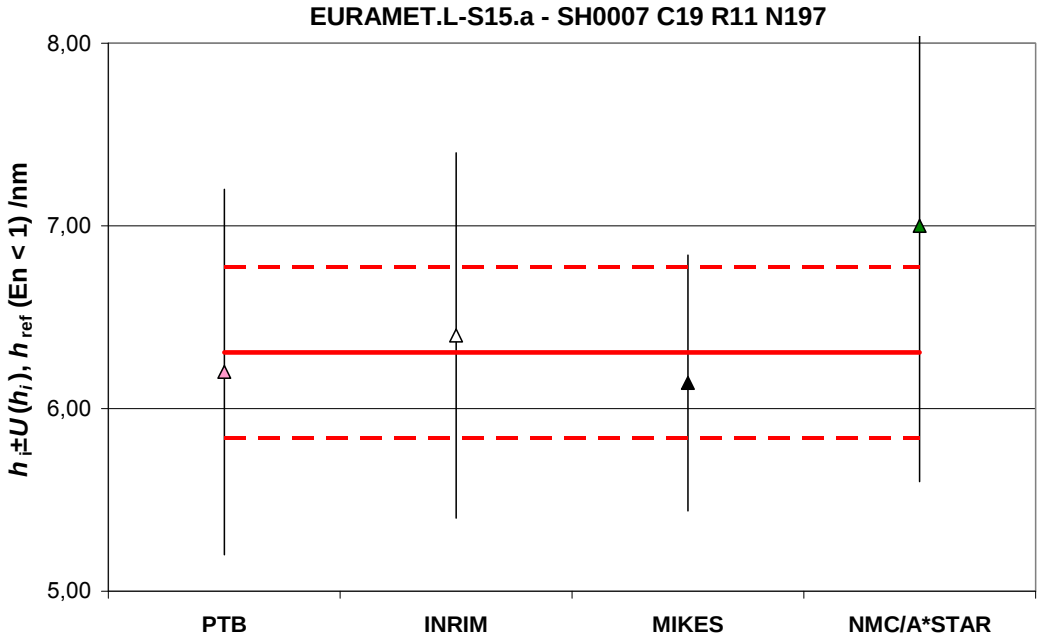


Fig. 12: Measured step heights h_i of the institutes as well as reference values h_{ref} (red line) and expanded uncertainties of the reference values (red dotted line) calculated from all values, since for all of them En ≤ 1.

[2] The results from CMI are presented in the Appendix B.

7.2 RESULTS ON STEP HEIGHT STANDARD SH0040

Table 6: Step height standard SH0040

SH0040 C05 R14 N261								
Institute	Country	Meas.	h / nm	u_c / nm	$\nu_{eff}(h)$	k	$U(k=2)$ /nm	En
PTB	DE	April 2008	42,6	0,6	50	2	1,2	0,25
INRIM	IT	May 2008	42,2	0,7	103	2	1,4	0,04
MIKES	FI	June 2008	41,52	0,62	18	2	1,24	0,53
NMC/A*STAR	Singapo-re	Aug. 2008	42,8	0,7	∞	2	1,4	0,35

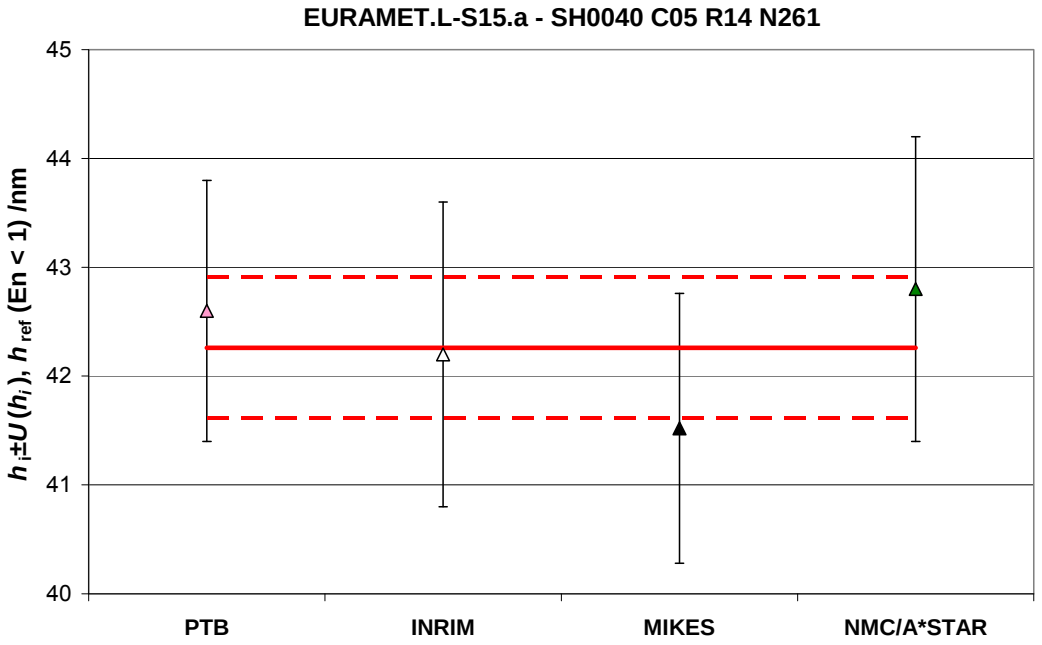


Fig. 13: Measured step heights h_i of the institutes as well as reference value h_{ref} (red line) and expanded uncertainties of the reference values (red dotted line) calculated from all values, since for all of them En ≤1.

7.3 RESULTS ON STEP HEIGHT STANDARD SH1000

Table 7: Step height standard SH1000

SH1000 C04 R06 N63								
Institute	Country	Meas.	*h* / nm	u_c / nm	$\nu_{eff}(h)$	k	*U(k=2) /nm*	En
PTB	DE	April 2008	1014,1	0,6	50	2	1,2	0,01
INRIM	IT	May 2008	1014,2*	1,3	63	2	2,6	0,03
MIKES	FI	June 2008	1014,05	0,55	19	2	1,10	0,05
NMC/A*STAR	Singapo-re	Aug. 2008	1014,3	0,9	∞	2	1,8	0,09

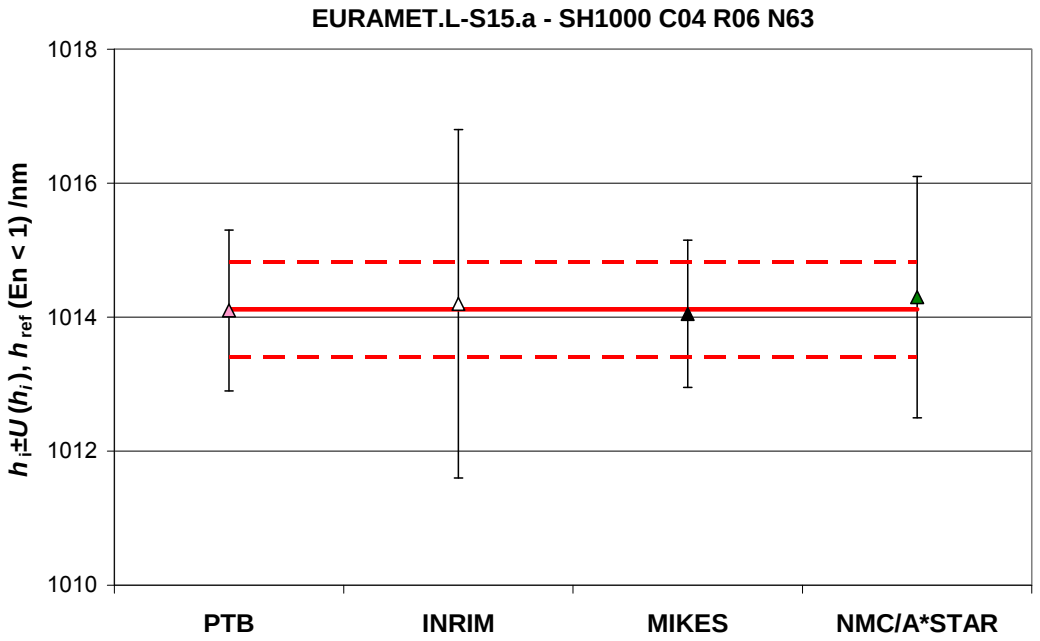


Fig. 14: Measured step heights h_i of the institutes as well as reference values h_{ref} (red line) and expanded uncertainties of the reference values (red dotted line) calculated from all values, since for all of them En ≤1.

* the initially reported value from INRIM was: *h* = 1013,3 nm, with u_c = 1,3 nm - this value was changed on INRIM's own initiative - see "Track of changes" in Appendix A

7.4 RESULTS ON STEP HEIGHT STANDARD SH2000

Table 8: Step height standard SH2000

SH2000 C06 R06 N65								
Institute	Country	Meas.	h / nm	u_c / nm	$\nu_{eff}(h)$	k	$U(k=2)$ /nm	En
PTB	DE	April 2008	2096,4	0,75	50	2	1,50	0,24
INRIM	IT	May 2008	2093,1*	1,9	77	2	3,8	0,95
MIKES	FI	June 2008	2097,92	0,64	24	2	1,28	0,71
NMC/A*STAR	Singapo-re	Aug. 2008	2095,2	1,3	∞	2	2,6	0,59

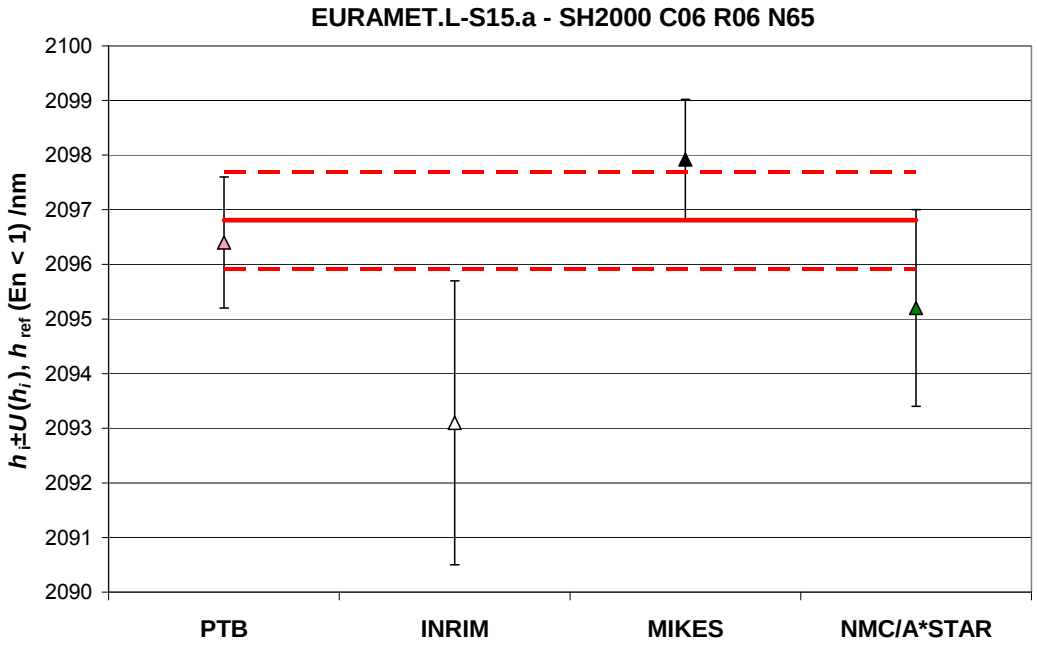


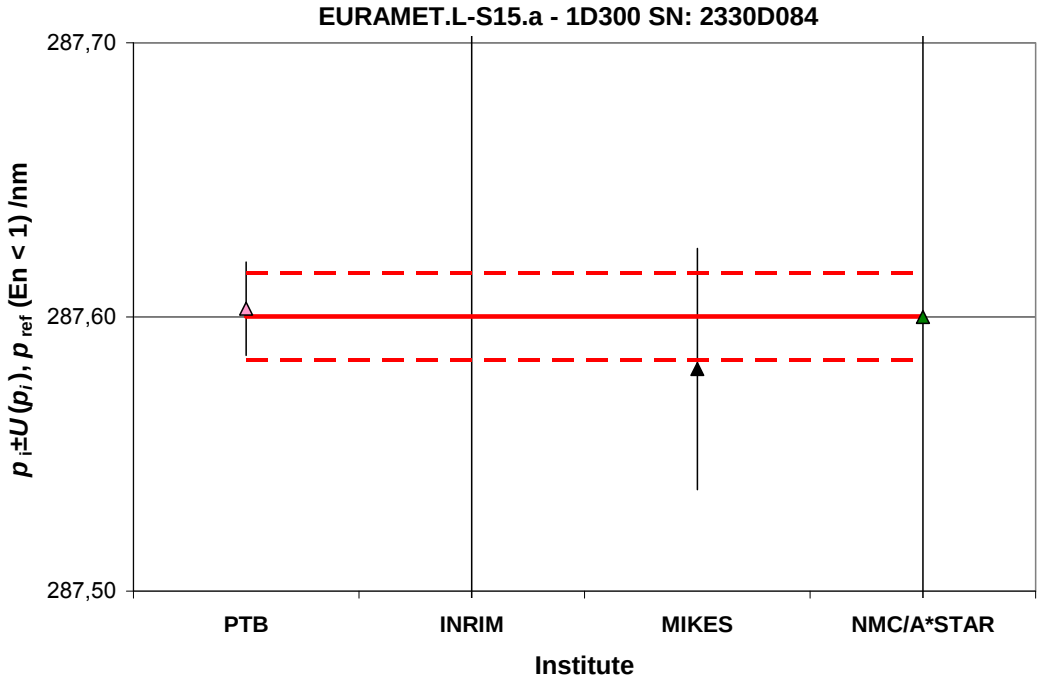
Fig. 15: Measured step heights h_i of the institutes as well as reference values h_{ref} (red line) and expanded uncertainties of the reference values (red dotted line) calculated from all values, since for all of them En ≤1.

* the initially reported value from INRIM was: h = 2089,0 nm, with u_c = 1,8 nm - see “Track of changes” in Appendix A

7.5 RESULTS ON 1D GRATING STANDARD 1D300

Table 9: 1D grating standard 1D300

1D300 SN: 2330D084								
Institute	Country	Meas.	p / nm	u_c / nm	$\nu_{eff}(p)$	k	U(k=2) /nm	E_n
PTB	DE	April 2008	287,603	0,0085	50	2	0,0170	0,12
INRIM	IT	May 2008	287,1	1,0	101	2	2,0	0,25
MIKES	FI	June 2008	287,581	0,022	25	2	0,044	0,41
NMC/A*STAR	Singapo-re	Aug. 2008	287,6	0,2	∞	2	0,4	0,00



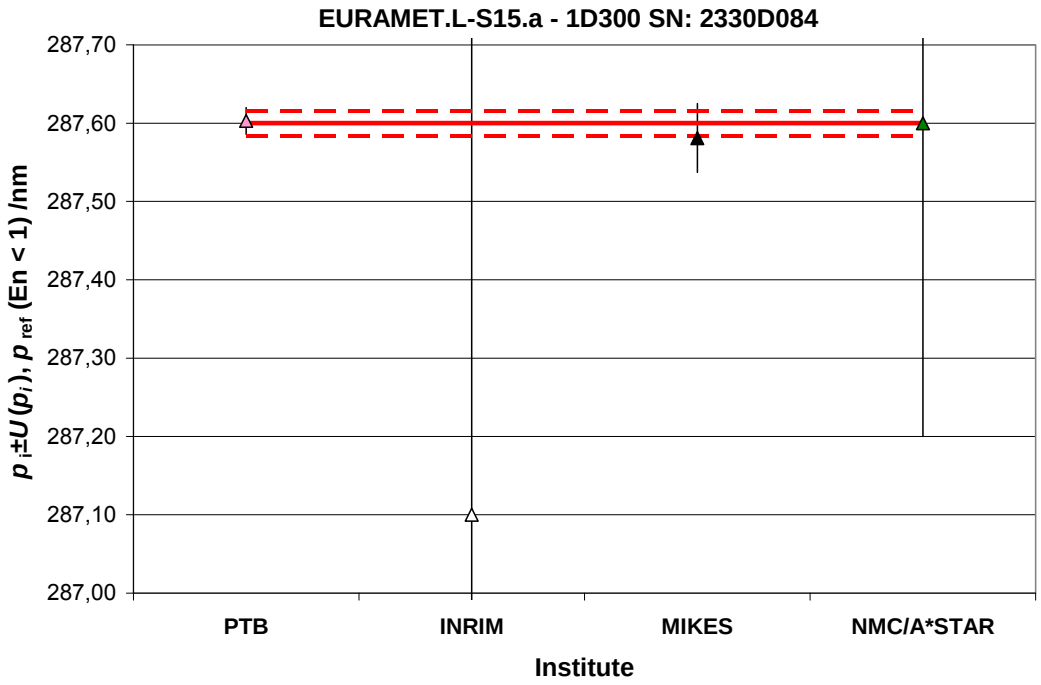
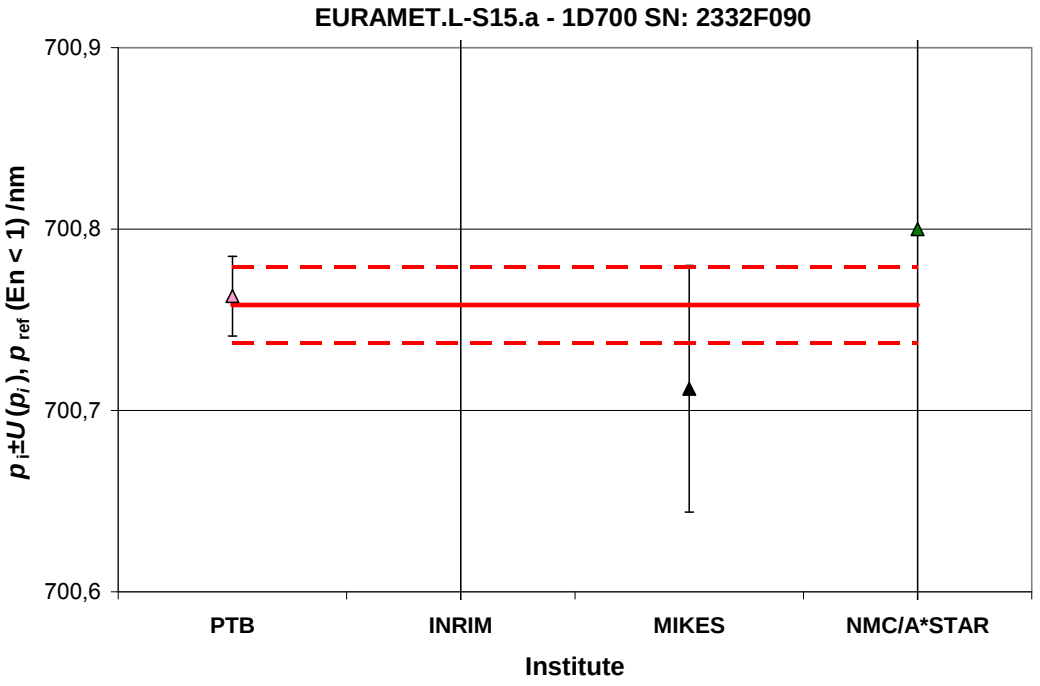


Fig. 16: Measured pitch values p_i of the institutes as well as reference values p_{ref} (red line) and expanded uncertainties of the reference values (red dotted line) calculated from all values, since for all of them En ≤ 1.

7.6 RESULTS ON 1D GRATING STANDARD 1D700

Table 10: 1D grating standard 1D700

1D700 SN: 2332F090								
Institute	Country	Meas.	p / nm	u_c / nm	$\nu_{eff}(p)$	k	$U(k=2)$ /nm	En
PTB	DE	April 2008	700,763	0,011	50	2	0,022	0,16
INRIM	IT	May 2008	700,4	1,2	49	2	2,4	0,15
MIKES	FI	June 2008	700,712	0,034	18	2	0,068	0,65
NMC/A*STAR	Singapo-re	Aug. 2008	700,8	0,4	∞	2	0,8	0,05



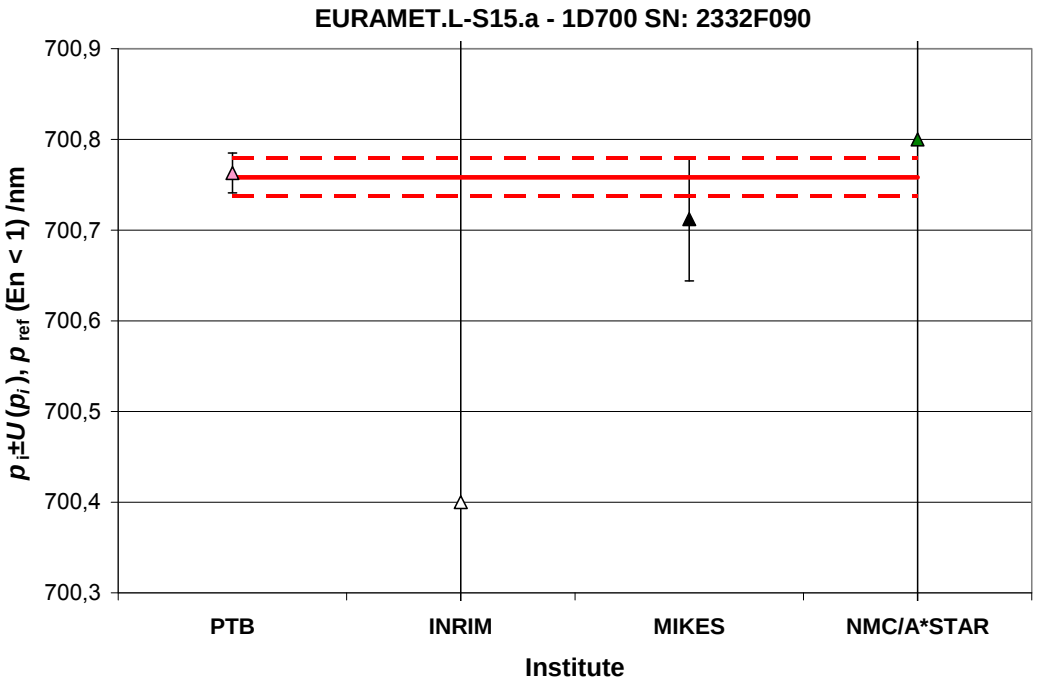


Fig. 17: Measured pitch values p_i of the institutes and reference values p_{ref} (red line) and expanded uncertainties of the reference values (red dotted line) calculated from all values, since for all of them En $\leq$ 1.

8 UNCERTAINTY BUDGET

The uncertainty of the measurement was estimated according to the *Guide to the Expression of Uncertainty in Measurement (GUM)*. The participating laboratories were encouraged to use all known influence parameters for the method applied by them. The values for step height *h* and pitch *p* of the standards are expressed as a function of the input quantities x_i (for simplicity all values are referred to as *h* instead of *h* and *p*)

$$h = f(x_i). \qquad (1)$$

The combined standard uncertainty $u_c(h)$ is the square sum of the standard uncertainties of the input quantities $u(x_i)$, each weighted by a sensitivity coefficient c_i

$$u_c^2(h) = \sum_i c_i^2 u^2(x_i) \text{ with } \qquad c_i = \frac{\partial h}{\partial x_i}. \qquad (2)$$

The uncertainty components should be divided into components associated with the realisation of the object compared, and those associated with the comparison method.

Contributions to the uncertainty budgets depend on the method and the instrument used, for instance:

1. calibration
- vacuum wavelengths of lasers
- refraction index of the air
- interferometer alignment
- uncertainty of calibrated standards used
- non-linearity of the instrument
- angular motion of translation stages
- Abbe offset

2. measurement
- sample alignment
- noise of instrument
- repeatability

3. evaluation
- profile evaluation and filtering
- roughness of the standard
- non-planarity/out of plane motion
- temperature of the standard

9 ANALYSIS OF DATA

9.1 REFERENCE VALUE AND ITS UNCERTAINTY

The reference value (h_{ref}) for this comparison is calculated as the weighted mean of all measurements (h_i). The weights are $u^{-2}(h_i)$. For each standard a reference value was calculated. To set up the |En| ≤ 1 criterion, the expanded uncertainty U with a coverage factor of k = 2 was used. After the correction of 2 values (see chapter 7) all results passed the |En| ≤ 1 criterion. Therefore, all values contributed to the reference value.

Reference value $$h_{ref} = \frac{\sum_{i=1}^{n} u^{-2}(h_i) \cdot h_i}{\sum_{i=1}^{n} u^{-2}(h_i)} \quad (3)$$

Combined standard uncertainty $$u_c(h_{ref}) = \left(\sum_{i=1}^{n} u^{-2}(h_i) \right)^{-\frac{1}{2}} \quad (4)$$

Degree of freedom $$\nu_{eff}(h_{ref}) = \frac{u_c^4(h_{ref})}{\sum_{i=1}^{n} \frac{u_i^4(h_{ref})}{\nu_{eff}(h_i)}} \text{ with } u_i(h_{ref}) = |c_i| \cdot u(h_i) = \frac{u^{-1}(h_i)}{\sum_{i=1}^{n} u^{-2}(h_i)} \quad (5)$$

Expanded uncertainty using k=2 $$U(h_{ref}, k=2) = 2 \cdot u_c(h_{ref}) \quad (6)$$

En-criteria $$En(h_i) = \left| \frac{h_i - h_{ref}}{\sqrt{U^2(h_i) + U^2(h_{ref})}} \right| \quad (7)$$

The plus sign in the denominator of (7) is used although there is some correlation between a single measurement result and the reference value. With the plus sign the En values could be slightly too small.

Performing the calculation using the above formulas we obtained the En values listed in the tables after first and if necessary after second calculation.

The final reference values are listed in Table 11. As mentioned before, in this comparison the reference values were calculated from all results, since all results passed the |En| ≤ 1 criterion.

Table 11: Reference values, uncertainties, expanded uncertainties, degrees of freedom

Standard	h_{ref} / nm	u(h_{ref}) /nm	U(k=2) /nm	v_{eff}
SH0007	6,31	0,23	0,46	75,6
SH0040	42,26	0,33	0,66	156,7
SH1000	1014,12	0,36	0,72	85,1
SH2000	2096,81	0,44	0,88	82,8

Standard	p_{ref} / nm	u(p_{ref}) /nm	U(k=2) /nm	v_{eff}
1D300	287,600	0,008	0,016	63,4
1D700	700,758	0,010	0,020	59,3

10 CONCLUSIONS AND REMARKS

The following conclusions are drawn from this comparison:

1. The comparison was performed between a small number of participants and in a short time. All participants performed their measurements very carefully and with best detailed knowledge of their instruments.

2. Compared to EUROMET707 the step height range measured was extended down to 7 nm step heights. The results show that such step heights on samples can be measured with very small uncertainties using sophisticated instruments.

3. Nevertheless, the comparison reveals, that an instrument has to be very carefully examined before it should be used for daily calibration, specially for very high steps.

4. In the case of large uncertainties this has to be checked carefully by the user. What are the reasons and what could be done to improve this? Furthermore, in some cases the uncertainty could have been estimated too large. In these cases the budgets should be checked in future.

11 APPENDIX A

INRIM

Description of the measurement method and instrument

The standards have been imaged using a Scanning Probe Microscope (SPM) operating with interferometer and capacitance-based controls of *xyz* displacements. The instrument uses a sample-moving system; for this exercise it has been equipped with a scanning device having a working volume of 100x100x15 μm^3. The *xy* parallelogram stage uses plane-mirror linear interferometers and fast phase-meters to monitor and control the lateral displacements, whereas a piezo-capacitive stage is used for the *z*-axis. The SPM microscope makes use of the electronics and data acquisition system of a commercial SPM. The z-stage has been calibrated off-line by means of a double-pass plane-mirror interferometer setup.

STEP HEIGHTS

A number of AFM images (20x20 μm^2, 512 or 1024 pixels) have been taken at the given sampling areas (MP1-MP5) of each sample. The raw images have been processed using the SPIP software (SPIP- The Scanning Probe Image Processor, Versions 3.0.1 and 4.4.1, by Image Metrology A/S). Images have been filtered (LMS 1st order planefit, low pass). The step-height has been calculated using the "ISO 5436 step-height" routine of SPIP, applied to the full image (20x20 μm^2). At present, the images have not been zoomed down to 20x12μm^2 area as given in the TP.

Track of changes

The individual MP1 to MP5 values, the step-heights and uncertainties of the results of the samples SH1000 and SH2000 have been corrected. However, only the value for SH2000 was asked to be reviewed by the pilot. After checking this also the value obtained for SH1000 was changed on INRIM's own initiative.
The changes are due to an error occurred in INRIM's previous evaluation, namely in the calculation of the correction due to the deviation from linearity of the z-stage.

1D GRATINGS

The 1D-700 has been imaged along the central part of the patterned area (~3x4 mm^2). The sample was oriented with the patterned lines orthogonal to the x-axis. A number of images of different sizes, sampling time around 1 line/s, have been taken in subsequent runs after withdrawal and re-approach of the tip. Due to some deviations to be further investigated, the images/results taken in one of these runs (sampling point shifted along the y-axis) have not been included.
The 1D-300 has been imaged as well along the central part of the patterned area (~4x3 mm^2). The sample was oriented with the patterned lines orthogonal to the x-axis. A number of images of different sizes, sampling time around 1 line/s, have been taken in subsequent runs after withdrawal and re-approach of the tip to sampling points around the central part of the area. No significant differences have

been observed between the results obtained at the various sampling points.

The raw images have been processed using the SPIP software (SPIP- The Scanning Probe Image Processor, Versions 3.0.1 and 4.4.1, by Image Metrology A/S). Images have been filtered (LMS 1st order planefit, low pass). The pitch has been calculated using the “Accurate unit cell detection/xycalibration” tool of SPIP applied to topography and Z-error signal from both direct and reverse tip scanning for each image.

MIKES

Description of the measurement method and instrument

All measurements were made using the MIKES interferometrically traceable metrology AFM (IT-MAFM) (see Fig. A1), which has 3D online detection of the sample position by laser interferometers. The interferometers are heterodyne interferometers of type Zygo ZMI 2000. The interferometers in *x* and *y* directions are differential, in *z* direction the dead path error is corrected by software. The measurement ranges of the AFM in *x* and *y* directions are 100 µm, and in *z* direction 16 µm. A commercial AFM head (PSIA XE100) is used in the instrument. Non-linearities of the laser interferometers are measured and corrected by a self-calibration method [1]. The instrument is located in a temperature controlled laboratory and it is acoustically shielded by an enclosure. In order to improve temperature stability, there is small constant airflow inside the measurement chamber. The measurement table is supported on a vibration isolated concrete block. Temperature, pressure and humidity are monitored during the measurements and refractive index of air is corrected by updated Edlén equations. All measurements are traceable to the national standards of Finland.

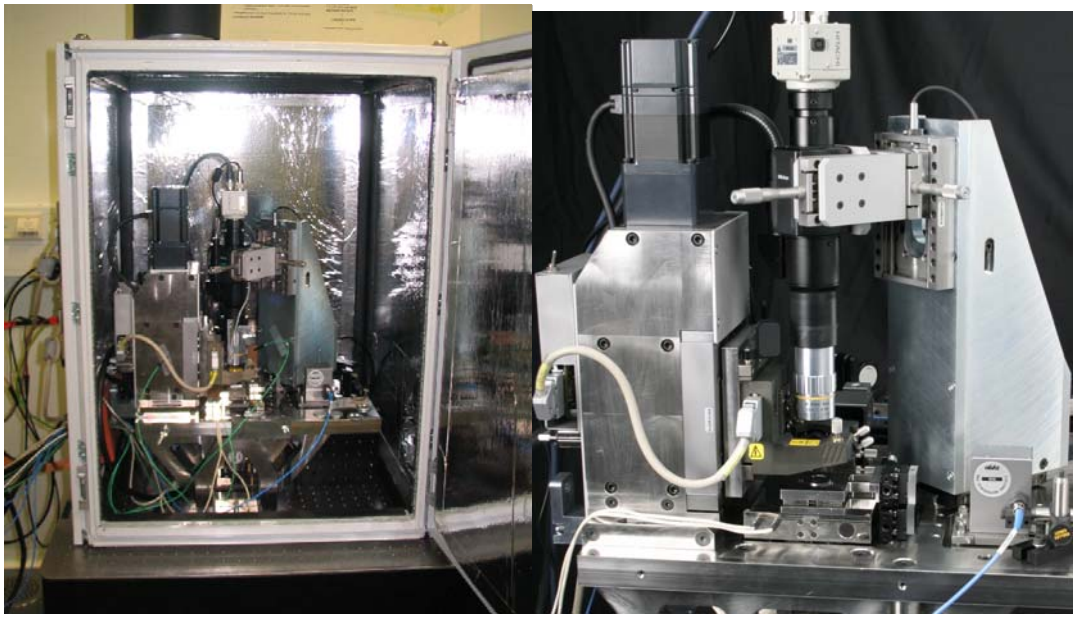


Fig. A1: Images from MIKES IT-MAFM.

STEP HEIGHTS

During the step height calibration an optical microscope was used to locate the measurement area. The measurement line was not visible in sample SH7, thus the measurement area was estimated using the alignment marks in each sides of the sample. Each single measurement contained 64 evenly spaced measurement lines for each MP. Data analysis was done by SPIP software (version 4.7.2) using ISO 5436 method. Measurement lines of poor quality were excluded from the analysis. Average of the step heights from the rest of the lines gave a single result for the MP. Measurements were repeated several times in sequence to see effects of drift and to reach equilibrium. The measurements were repeated using both non-contact and contact modes. The given results for individual MPs are averages

of repeated measurements with both modes.

1D GRATINGS

During the pitch calibration an optical microscope was used to locate the measurement area. Clean areas were selected for the measurements. The measurements were done over areas of 85 µm × 100 µm (1D700) or 70 µm × 100 µm (1D300), so that only 2.5 µm × 100 µm (1D700) or 1.25 µm × 100 µm (1D300) areas at both sizes of the measurement area were measured and the area in the middle was skipped. This reduced measurement time significantly. There were in total 512 × 128 pixels (x × y) in a single measurement. The pitch was calculated using a self made Matlab code.

A normalised 2D cross correlation function was used to calculate how much Δl in the right side of the image should be shifted in order to have the best correlation i.e. phase match between the left side and right side patterns. Sub-pixel resolution was obtained by fitting a second order polynomial around the correlation maximum. Analysis for a single line is illustrated in Fig. A2. Corresponding number of full periods was calculated using preliminary pitch value measured using a smaller measurement range. The pitch is calculated as a ratio of Δl and the number of periods. Cosine errors caused by sample tilt and sample rotation were corrected. Final results are averages of analysed pitches of several repeated measurements in different measurement positions.

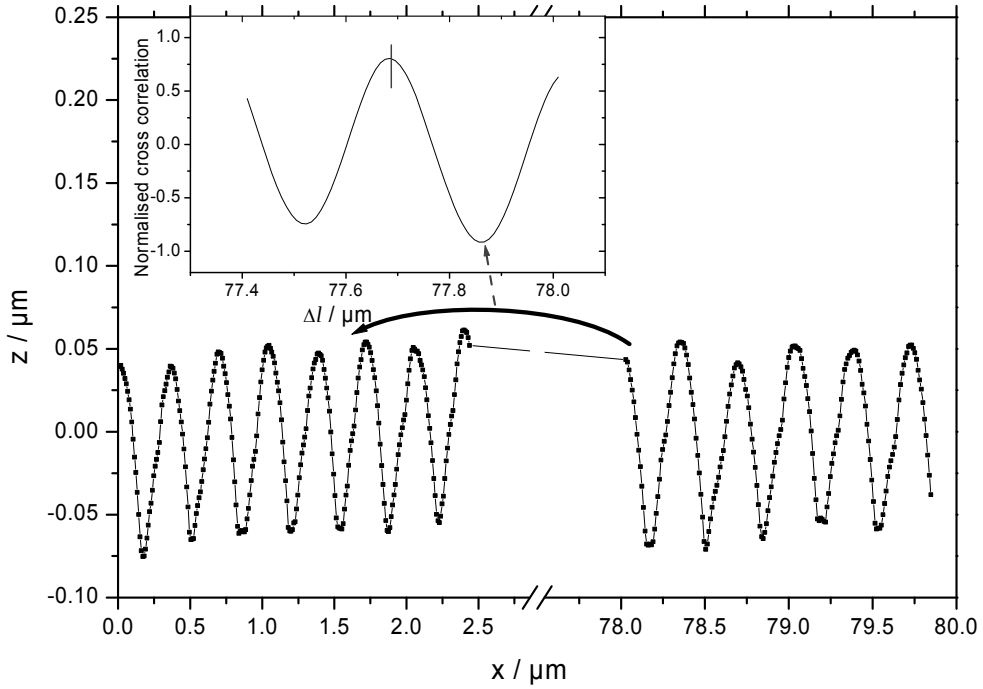


Fig. A2: Illustration of cross correlation analysis for single AFM measurement line of pitch measurement.

References:

[1] V. Korpelainen and A. Lassila, Self-calibration of non-linearities of laser interferometer and capacitive sensor combination for an interferometrically traceable AFM device, Proceedings of 4th international conference of the European society for precision engineering and nanotechnology, euspen, Glasgow, Scotland, May 31 – June 2, 2004, p 256.

NMC/A*STAR

Description of the measurement method and instrument

All measurements were carried out using a large range metrological atomic force microscope (LRM-AFM) at NMC/A*STAR, Singapore (see Fig. A3). The LRM-AFM consists of an AFM probe, a Nano Measuring Machine (NMM), control electronics and software for coordinating servo motion control, signal detection, data acquisition and analysis. An isolation table and an acoustical enclosure are also furnished to minimize the influence of external vibration and noise on the system's performance. The AFM capable of working in non-contact mode produced by Danish Micro Engineering was integrated into the NMM manufactured by SIOS Meβtechnik GmbH, Germany. The motions along the three co-ordinate axes of the NMM were measured by three He-Ne laser interferometers. The laser frequencies were calibrated by an iodine frequency stabilized laser. Hence, the measurements are directly traceable to the SI unit for length.

The NMM is capable of providing a large scanning range up to 25 mm × 25 mm × 5 mm (*x, y and z*) and moving the sample around while the AFM probe is fixed onto the NMM through a metrological frame made of thermal stable material (zerodur). The AFM tip is located at the intersection of the three measuring beams of the three laser interferometers to minimize the Abbe offset. In combination of a video microscope of the AFM probe, the high accurate large scanning stage allows an easy positioning of the surface under test to be placed near to the AFM tip.

Fig. A3: Large range metrological atomic force microscope (LRM-AFM) consisting of a Nano Measuring Machine (NMM) and a commercial AFM head.

STEP HEIGHTS

The step height measurements are done in accordance with ISO 5436 as required by the comparison technical protocol. The final measured step height is the grand average of the average step height values obtained from the 5 measurements (MP1 to MP5) on each step height standard.

1D GRATINGS

The measurement of the pitch of the 1D grating was carried out using the same measurement instrument as described above. The measurement of the 1D grating is also directly traceable to the SI unit for length.

The measurement data were evaluated using Fast Fourier Transform method for determining the mean pitch in an effective scanning range to have the number of grating pitch in N_{gp}. The final measurement result is the grand average of all the average values obtained from the 5 different measurement positions on each standard. The scanning areas of each measurement position on 1D300 and 1D700 were 25 µm × 10 µm and 30 µm × 10 µm respectively, in which the lengths of 25 µm and 30 µm were located along the direction orthogonal to the ribs of the gratings.

PTB – LR-SFM

Description of the measurement method and instrument

The samples are measured by the Metrological Large Range Scanning Force Microscope (Met.LR-SFM) [1, 2] developed based on the Nano Measuring Machine (NMM) [3] and a home-built SFM head, as shown in the Fig. A4. The position of the scanning stage is measured by three optical interferometers with frequency stabilised lasers along the x, y and z axes. The laser frequencies are calibrated to an iodine frequency-stabilised laser for achieving traceability.

The measurements were performed in intermittent-contact mode using commercially available non-contact silicon cantilever probes (type: PPP-NCHR by Nanosensors®).

The instrument is installed in a acoustic chamber inside PTB's cleanroom centre. During measurements, the temperature of the sample and surrounding air is (20.5 ± 0.5)°C; the relative humidity of the air is (46 ± 2) %.

Fig. A4: Photo of the metrological large range SFM consisting of a Nano-Measuring-Maschine (NMM) and a home-built SFM head, described in [2]

STEP HEIGHTS

SFM images are obtained at five different measurement positions as shown in Fig. 2. The measured SFM images are evaluated in compliance with ISO 5436-1. The data are processed linewise. After 1st order levelling, two edge positions and the width of the line structure *w* are determined. The step height is then calculated as the vertical distance between the top line located at the centre of the structure with a width of *w*/3 and two bottom lines with the same width. The gaps between the bottom lines to the structure's edge are also *w*/3. The step height value of each SFM image is calculated as the mean value of all recorded scan lines. The reported calibration value *h* is the average of step height values calculated from SFM images recorded at the 5 measurement positions.

1D GRATINGS

The measurements of the 1D gratings are carried out using the same instrument as described above and the results are directly traceable to the metre definition as well.

5 different areas with sizes of 1 mm x 10 µm are measured each located close to the centre of the grating. The numbers of pixels recorded in each SFM image are 50000 pixels x 16 rows for the 1D300 and 30000 pixels x 16 rows for the 1D700, respectively. The scan speeds are 10 µm/s for the D300 and 5 µm/s for the D700, respectively. The y axis is selected as the fast scan axis for both samples.

The mean pitch value of the grating is analysed using the FFT-FT method [4] from each SFM image. The FFT-FT method uses the fast Fourier transform algorithm (FFT) for a fast search of the spatial frequency component of the grating structures, which has the largest amplitude. Then it uses the Fourier transform (FT) algorithm for refining the search. Finally, phase information of each individual grating structure is calculated using the FT algorithm, and the homogeneity of the grating structures can be determined. The reported pitch value is calculated by averaging pitch values determined at the 5 measurement positions. Cosinus errors caused by the scan direction deviations are corrected.

References

[1] G. Dai, F. Pohlenz, H.-U. Danzebrink, M. Xu, K. Hasche, G. Wilkening: Metrological Large Range Scanning Probe Microscope, Rev. Sci. Instr., Vol. 75, No. 4, pp. 962–969 (2004)

[2] Dai, G.; Wolff, H.; Pohlenz, F.; Danzebrink, H.-U.: A metrological large range atomic force microscope with improved performance, Rev. Sci. Instrum. 80, 043702 (2009)

[3] Jäger, G.; Manske, E.; Hausotte, T.; Büchner, H.-J.; Grünwald, R.; Schott: W.: Nanomeasuring technology - nanomeasuring machine. In: The Sixteenth Annual Meeting, pages 23-27, Crystal City, Arlington, Virginia, 10.-15. November 2001. American Society for Precision Engineering

[4] Dai, G., Koenders, L., Pohlenz, F., Dziomba, T., Danzebrink, H.-U., Accurate and traceable calibration of one dimensional gratings. Meas. Sci. Technol. 16 (2005) 1241 – 1249

12 APPENDIX B – RESULTS FROM CMI

CMI

In the following CMI's results are presented. The measurements were performed in June 2009 on the same set of samples as for the other participants.

All results of CMI are compared to the reference values from Table 11.

Description of the measurement method and instrument

Metrology SPM based on Physik Instrumente table and six-axis 3D interferometer using Nd:Yag laser, contact mode, standard contact tip (Nanosensors PPP-CONTR).

STEP HEIGHTS

Measurement areas were chosen according to project technical guide, measurements were repeated several times. Z signal was obtained as an average value from three z interferometers, table rotation information obtained from the signals was incorporated into uncertainty of interferometer (no correction now). Final uncertainty was also influenced by continuous problems with laser stability and intensity.

SH0007 C19 R11 N197								
Institute	Country	Meas.	h / nm	u_c / nm	$\nu_{eff}(h)$	k	U(k=2) /nm	
CMI	CZ	June 2009	5,8	2,9	100	2	5,8	
Reference value			6,31	0,23	75,6	2	0,46	

SH0040 C05 R14 N261								
Institute	Country	Meas.	h / nm	u_c / nm	$\nu_{eff}(h)$	k	$U(k=2)$ /nm	
CMI	CZ	June 2009	41,8	5,8	60	2	11,6	
Reference value			42,26	0,33	156,7	2	0,66	

SH1000 C04 R06 N63								
Institute	Country	Meas.	h / nm	u_c / nm	$\nu_{eff}(h)$	k	$U(k=2)$ /nm	
CMI	CZ	June 2009	1006,0	8,2	62	2	16,4	
Reference value			1014,12	0,36	85,1	2	0,72	

SH2000 C06 R06 N65								
Institute	Country	Meas.	*h* / nm	u_c / nm	$\nu_{eff}(h)$	k	*U(k=2) /nm*	
CMI	CZ	June 2009	2064,0	13,2	50	2	26,4	
Reference value			2096,81	0,44	82,8	2	0,88	

1D GRATINGS

Measurement areas were chosen according to project technical guide, measurements were repeated several times. X signal was obtained from x interferometer, table rotation information obtained from the two Y interferometer signals was incorporated into uncertainty as a single uncertainty source. Final uncertainty was also influenced by continuous problems with laser stability and intensity.

1D300 SN: 2330D084								
Institute	Country	Meas.	*p* / nm	u_c / nm	$\nu_{eff}(p)$	k	*U*(k=2) /nm	
CMI	CZ	June 2009	287,6	0,9	44	2	1,8	
Reference value			287,600	0,008	63,4	2	0,016	

1D700 SN: 2332F090								
Institute	Country	Meas.	*p* / nm	u_c / nm	$\nu_{eff}(p)$	k	*U(k=2) /nm*	
CMI	CZ	June 2009	701,0	1,0	44	2	2,0	
Reference value			700,758	0,010	59,3	2	0,020	

Resumee

All results obtained are in full agreement with the reference values of the comparison.