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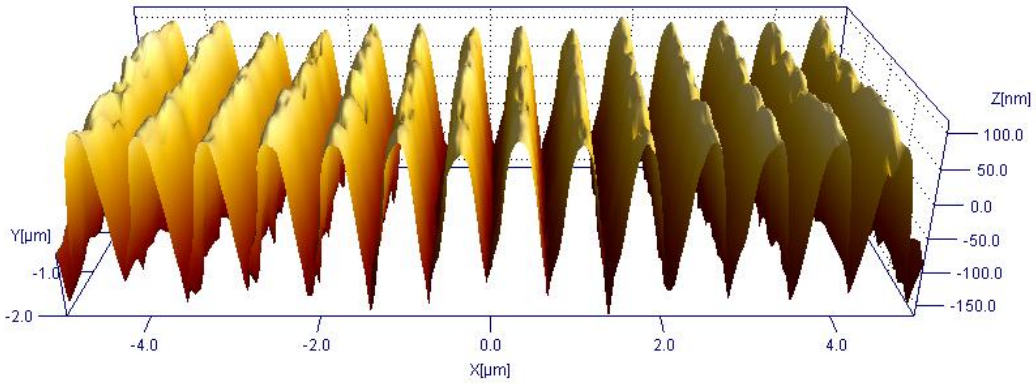
J1/2012



Intercomparison of lateral scales of SEM and AFM microscopes in research institutes in Northern Europe

S. Jeremias Seppä, Virpi Korpelainen, Sten Bergstrand,
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Centre for Metrology and Accreditation
Espoo 2012

MIKES
Mittatekniikan keskus
Centre for metrology and accreditation

Publication J1/2012

Intercomparison of lateral scales of SEM and AFM microscopes in research institutes in Northern Europe

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Mittatekniikan keskus

Espoo 2012

Abstract

An intercomparison (Nordic-nano1) of lateral scales of SEM and AFM microscopes in research institutes in Northern Europe. Grating samples (1D) were circulated among the participating laboratories. The laboratories were also asked about the calibration of their instruments. The results for both nominally 300 nm and 700 nm gratings show that a simple scale factor calibration would have corrected a large part of the deviations from the reference values. The accuracy of the uncertainty estimates varied between the laboratories, and for some laboratories the appropriateness of the calibration procedures could be considered.

Contents

1 Introduction

Traceable length scale and realistic uncertainty estimates are needed in all dimensional measurements. Without traceability, measurement results are not repeatable or comparable with other results measured in other laboratories, or even with those from the same laboratory performed at different times or with different instruments. High resolution and good repeatability may give an illusion of high accuracy. Frequent calibration, instrument stability and high resolution are all needed for reliable and accurate measurements.

In general, intercomparisons are an important part of the quality systems of national metrology institutes (NMIs) and accredited laboratories to ensure traceable units in measurements. There have been several comparisons between NMIs in nanometre scale measurements [e.g. 1,2] with good agreement between most of the institutes. The national metrology institutes – the Centre for Metrology and Accreditation (MIKES,FI), Technical Research Institute of Sweden (SP, SE), Justervesenet (JV, NO), and Metrosert (EE) –organized this comparison measurement (Nordic-nano1) for scanning electron microscopes (SEM) and atomic force microscopes (AFM) in 2010 - 2011. The purpose of the comparison was to study measurement capabilities at universities and research institutes. It started as a national comparison in Finland and was expanded to the other Nordic and Baltic countries. Most of the laboratories that measure nanometre scale structures are research laboratories without accreditation. None of the participating laboratories in this comparison are accredited.

The purpose of the intercomparison is to get information about the measurement capabilities and calibration of AFMs and SEMs. The participants obtained information about the accuracy of their instruments and their measurement capability. The comparison samples were 1-D gratings with nominally 300 nm and 700 nm pitches. The reference value was measured with the MIKES laser diffractometer [3]. Deviations from the reference value are reported in this paper. The participants were asked to estimate their measurement uncertainty. An example of an uncertainty budget for AFM measurement is also given here.

In addition, questions were asked about the calibration of the participating instruments.

2 Participants

The participants are listed in Table 1. Twenty-five laboratories participated in the comparison; 20 were from Finland, two from Sweden, two from Norway and one from Estonia. Additionally, two laboratories did not report their results. Each participant is informed about their ID numbers used in this report, and each laboratory has been given their own preliminary results soon after the measurements ended. The measurements began on 7.6.2010 and the last participant carried out their

measurements in October 2011. Some laboratories participated in the comparison with more than one instrument. In total we have results from 40 instruments: 25 SEM, 14 AFM and one profilometer. Some laboratories reported more than one result for one instrument, e.g. separate results for X and Y axes. Those results are analysed as different instruments.

Table 1. Participants in the comparison

Aalto university - Materials technology, Finland
Aalto university - Mechanical Process Technology and Recycling, Finland
Aalto university - Department of Micro- and Nanosciences, Finland
Aalto university - Forest Products Technology, Finland
Aalto university - Nanofab, Finland
Finnish institute of occupational health - Aerosol laboratory Finland
Glafo, Sweden
Institute for Energy Technology, Norway
Jyväskylän yliopisto - NSC, Finland
KTH - Nanostructure Physics, Sweden
Lappeenranta University of Technology - ASTRaL, Finland
Mikpolis Oy - Materiaalitekniikka, Finland
Optoelectronics Research Centre/TUT, Finland
Savonia, Finland
SINTEF ICT - Department of Microsystems and Nanotechnology, Norway
Tallinn University of Technology – Centre for materials research, Estonia
Tampere university of technology - Materials Science, Finland
Top Analytica Ltd., Finland
University of Eastern Finland - physics and chemistry, Finland
University of Helsinki - Accelerator laboratory, Finland
University of Helsinki - Inorganic chemistry, Finland
VTT - micro- and nanotechnology, Finland
VTT - Micronova, Finland
VTT - Microsystems and Nanoelectronics, Finland
Åbo akademi - Inorganic chemistry, Finland

3 Samples

The comparison samples were 1-D gratings with nominal pitch of 300 nm or 700 nm. The gratings are made of silicon substrate with photoresist and tungsten (W) coating. Manufactured using a holographic method, they should not have discontinuities in the sinusoidal profile. The whole top surface of the samples is patterned. The height amplitude of the grating profiles is approximately 100 nm - 200 nm. The comparison was carried out using two sets of samples. During the comparison the samples were scratched, but the measurements could be done in unscratched areas. The samples are listed in Table 2. Photographs of the samples are shown in Figure 1.

Each participant sent the samples either to the next participant or back to MIKES after their own measurement period. The measurement period was 1-2 weeks from receiving the samples.

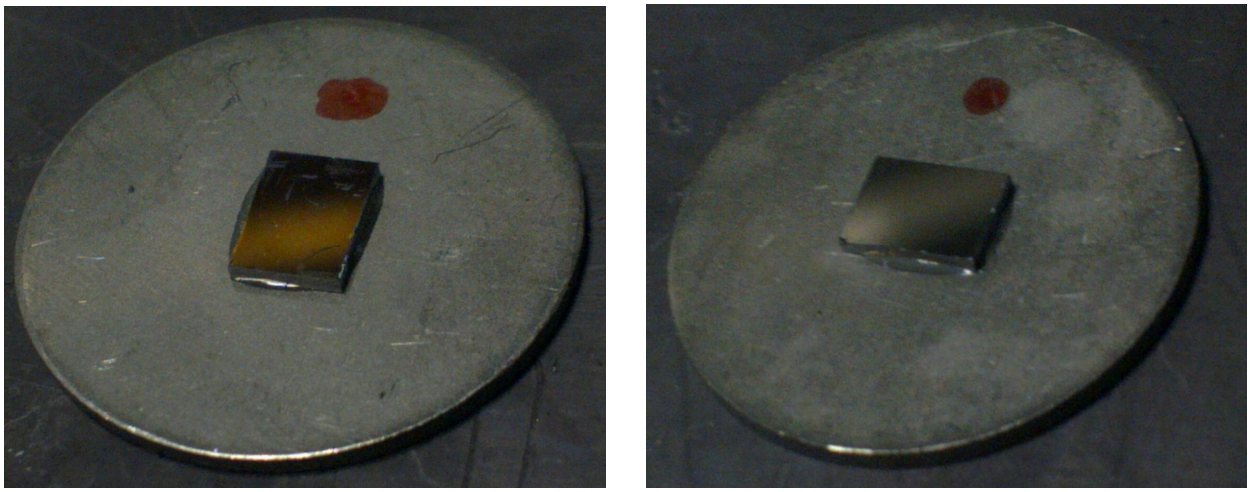


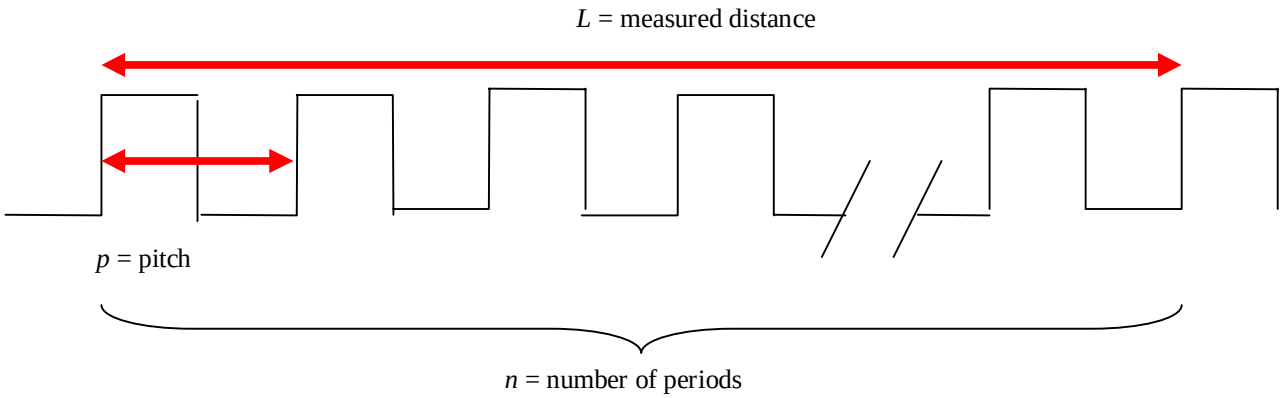
Figure 1. Photographs of both grating types with disc mount and alignment dot.

Table 2. Samples for comparison

MIKES number	Type of grating	Nominal pitch	Reference value	Expanded (k=2) uncertainty of the reference value
MIKES005950	300-1D	300 nm	287.581 nm	53 pm
MIKES005951	300-1D	300 nm	287.585 nm	53 pm
MIKES005952	700-1D	700 nm	700.756 nm	53 pm
MIKES004723	700-1D	700 nm	700.755 nm	53 pm

3.1 Measurand

The measurand used in this comparison was the average grating pitch in the centre of the standard at 20°C. The direction of the pitch is defined to be orthogonal to the grooves of the grating and parallel with the surface. An example of pitch determination from the surface profile is shown in Figure 2.



$$p = \frac{L}{n}$$

Figure 2. Example of pitch measurement from the surface profile.

3.2 Reference value

Reference values for the comparison are the pitch values measured with the MIKES laser diffractometer [3]. Laser diffractometry is the most accurate way to measure grating pitch, with an achievable uncertainty level down to 10 pm. MIKES has participated in an international comparison for 1-D gratings [4], and the accuracy of MIKES instruments was found to be excellent. The nominal pitches and reference values are given in Table 2.The expanded standard uncertainty (k=2) of the reference values for all samples was 53 pm. This is set by our internationally accepted calibration measurement capability (CMC) for grating pitch by our laser diffractometer [5].

All the samples were measured at MIKES before and after the comparison. The reference value was an average of the values measured at MIKES and was used to normalize the results for the different sample sets. The grating pitches measured by the laser diffractometer, before and after the comparison, differed by less than 20 pm for each sample.

The quality of the samples was assessed after the comparison by measuring possible linearity errors, especially “stitching errors”, or sudden discontinuities of the phase of the sinusoidal profile of the gratings. Approximately 300 µm of the grating surface profile was covered with 80 µm-long scan lines by MIKES IT-MAFM [6,7].

The linearity of the phase of the sinusoidal pattern advancing along the measurement direction was measured by taking the products of the measured profile and sine and cosine signal of the same frequency, and using a suitable moving average to extract the phase information. , see Figure 3. Possible stitching error would have been visible as step change in the curve.

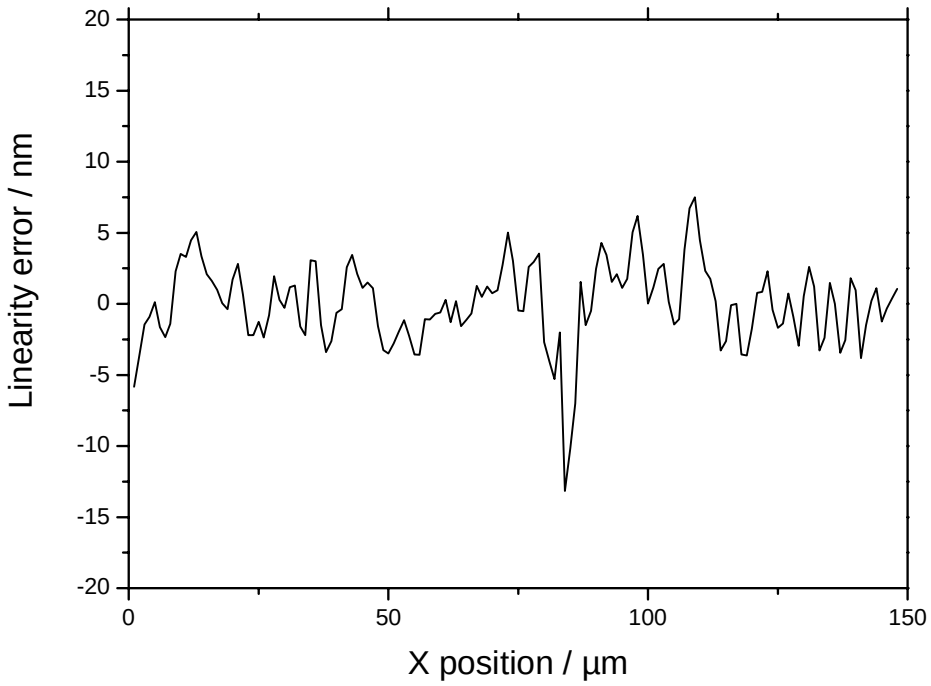


Figure 3. Analysed phase (linearity) along the scan line for one sample.

The results of this grating quality/homogeneity measurement suggest that, using e.g. 100 grating periods in a scan line for pitch measurement, the relative error in pitch due to grating homogeneity should be less than 1/1000. No evidence of severe phase jumps or other linearity errors that would have been noticed in the analysis were found. The original diffractometer measurements were repeated at three different locations on the sample surface. The results did not indicate severe inhomogeneity of the average pitch between different parts of the pattern.

4 Measurements and instruments used

Measurements could be done with any instrument suitable for measurement of this type of grating. Each measurement was reported separately. If more than one result was reported for one instrument (e.g. separate results for x and y scale) they were analysed separately. Normal measurement procedures were performed in the laboratory for this kind of measurement.

- The measurements were done in the middle of the sample. If there were scratches e.g. caused by earlier measurements, an adjacent area could be used for the measurements.
- The measurement results were reported at 20°C. The thermal expansion coefficient for the sample is 0.5×10^{-6}/K.

5 Results

The results are shown in Table 3 and Figures 4-7 as differences from the reference value, with the reported uncertainties. The laboratories are in arbitrary order. SEM and AFM results are separated.

Table 3. Differences from the reference values.

	300 nm		700 nm		
ID	Difference	Uncertainty	Difference	Uncertainty	Instrument
1	-3.59	2.84	-2.76	6.98	SEM
2	-1.59	0	3.24	0	SEM
3	0.41	2	1.74	2	SEM
4	2.11	1.2	2.44	1.1	SEM
5	-3.68	1.03	-7.66	3.85	SEM
6	-4.58	0	-13.76	0	SEM
7			-25.76	0	SEM
8	-0.58	0.6	-1.76	1	SEM
9	-1.93	0.61	-9.39	1.02	SEM
10	3.57	0.38	14.77	0.92	SEM
11	0.41	0	1.64	0	SEM
12	-3.63	0.66	-3.49	1.38	SEM
13	-8.59	14	-23.76	34	SEM
14			3.24	35	SEM
15	32.41	5	4.24	10	SEM
16	1.41	4	4.24	7	SEM
17	-0.59	5	3.25	13	SEM
18	60.41	11	103.24	19	SEM
19	12.12	1.5	19.24	2	SEM
20	-0.59	1	-3.76	2	SEM
21	-0.59	0.6	2.24	2	SEM
22	6.41	5	14.24	17	SEM
23	12.41	5	31.24	14	SEM
24	1.10	1.02	14.18	2.76	SEM
25	3.41	10	7.24	20	SEM
26					
27			-1.76	9	AFM
28	9.04	10	14.71	22	AFM
29	4.73	10	9.94	22	AFM
30	1.14	7.1	0.96	6.1	AFM
31	38.92	3.9	60.64	4	AFM
32	3.42	2	1.24	11	AFM
33	7.42	5	-6.76	15	AFM
34	82.42	2	186.24	3	AFM
35	-7.68	0.2			AFM
36	4.27	6.15	4.24	14.35	AFM
37	0.41	4	-0.76	40	AFM
38	32.42	25	89.24	90	AFM
39	6.41	5	14.24	17	AFM
40	-1.58	4	-2.76	5	AFM
41			-3.66	1.2	Profilometer

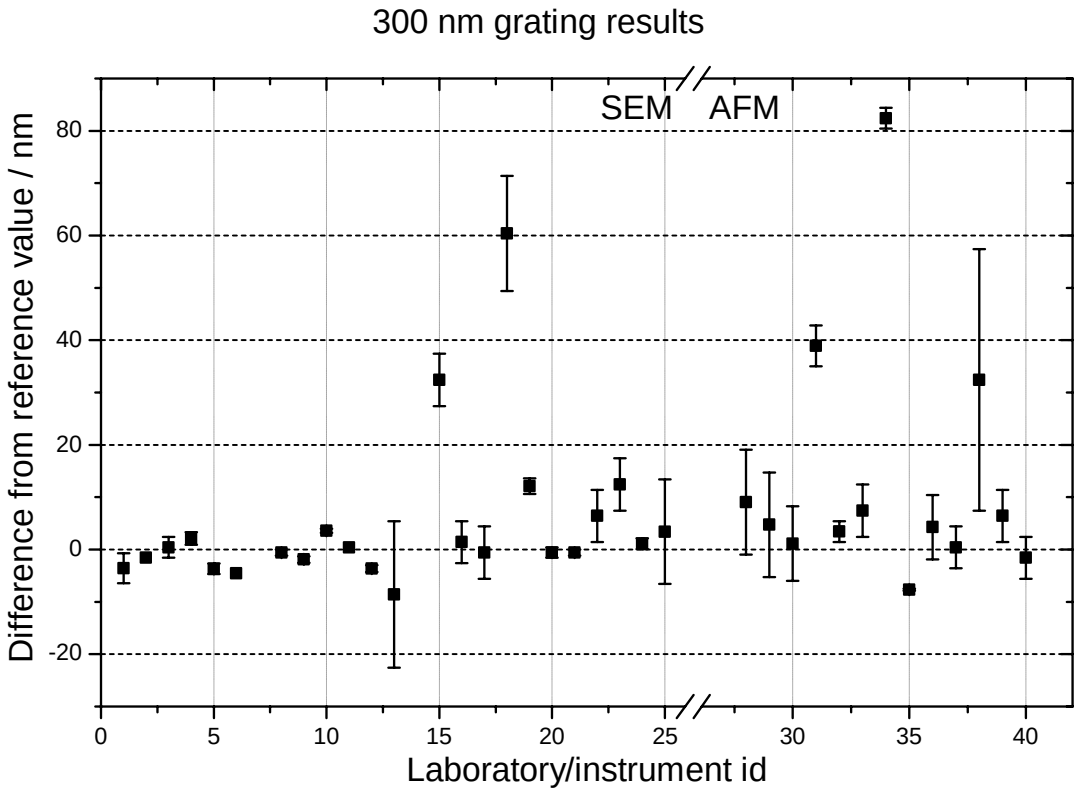


Figure 4. Deviations from the reference value for nominally 300 nm grating.

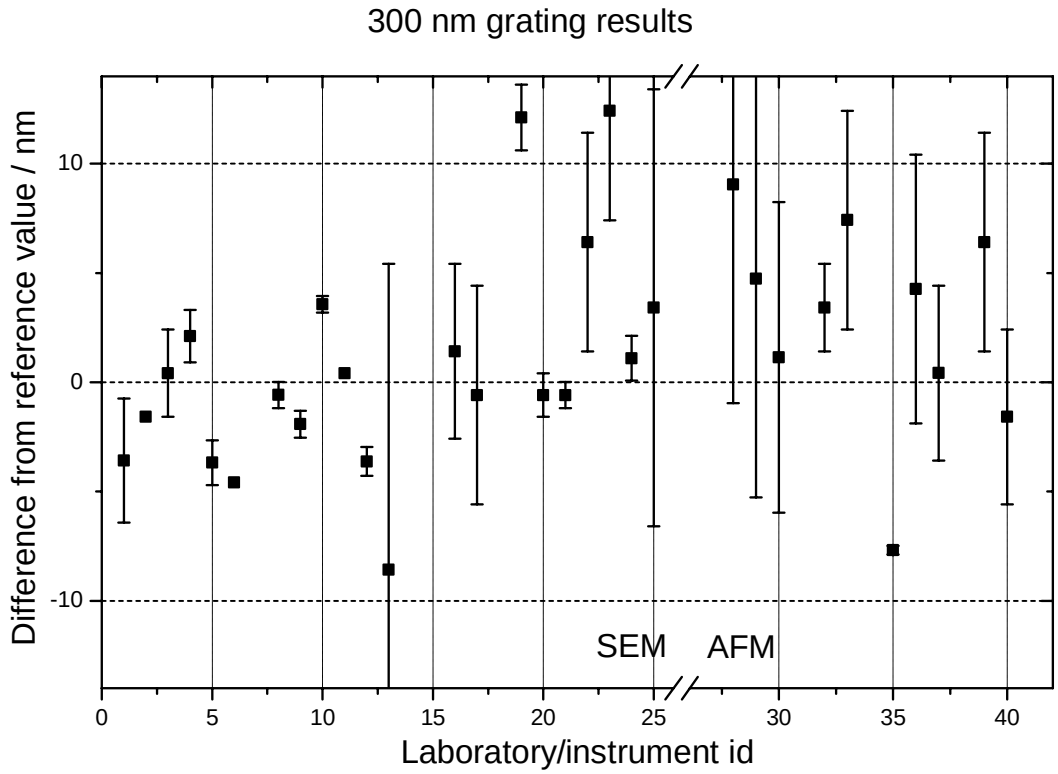


Figure 5. Deviations from the reference value for nominally 300 nm grating with zoomed axis.

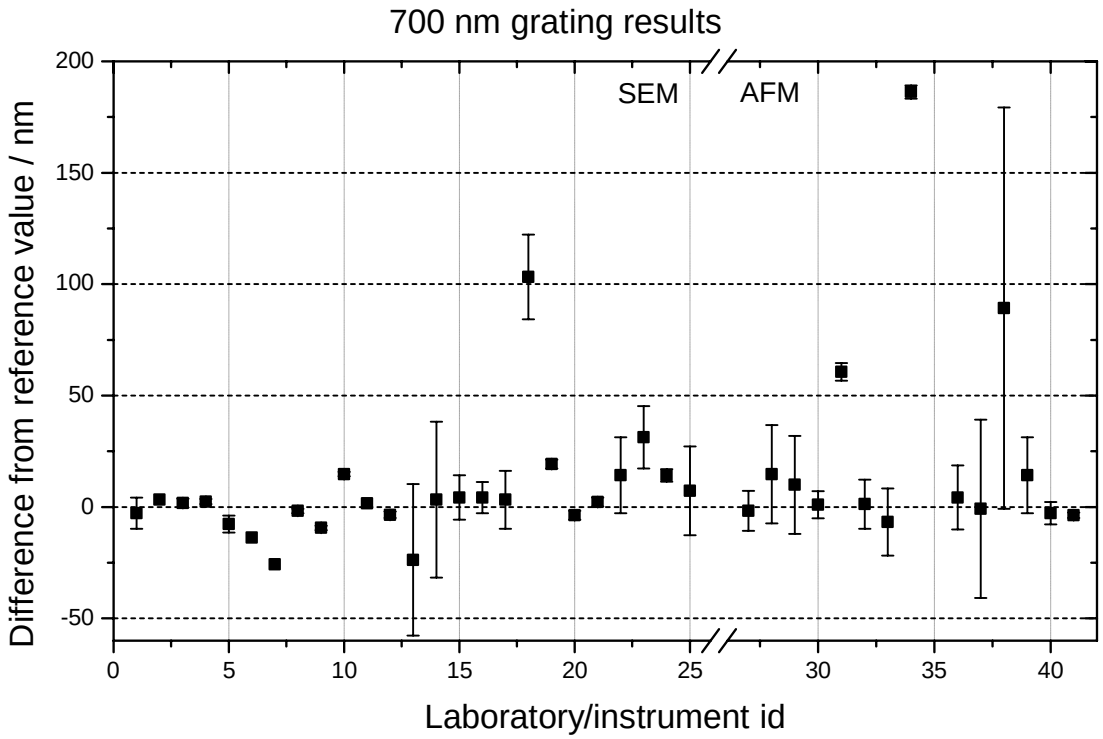


Figure 6. Deviations from the reference value for nominally 700 nm grating.

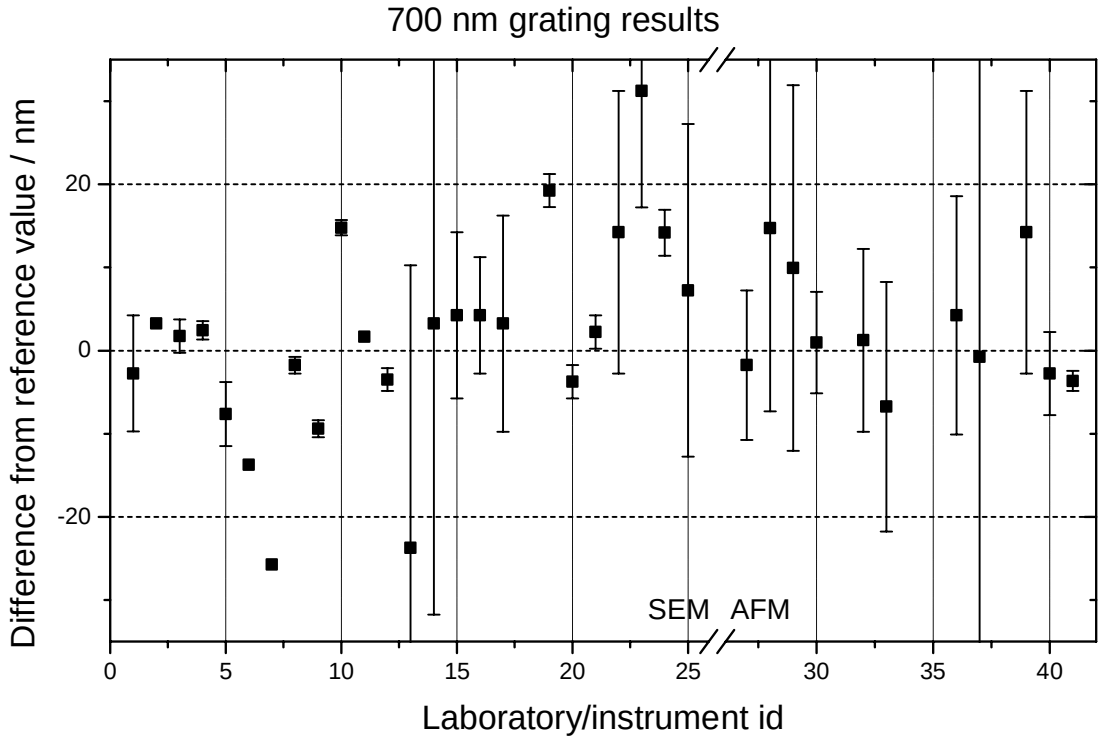


Figure 7. Deviations from the reference value for nominally 700 nm grating with zoomed axis.

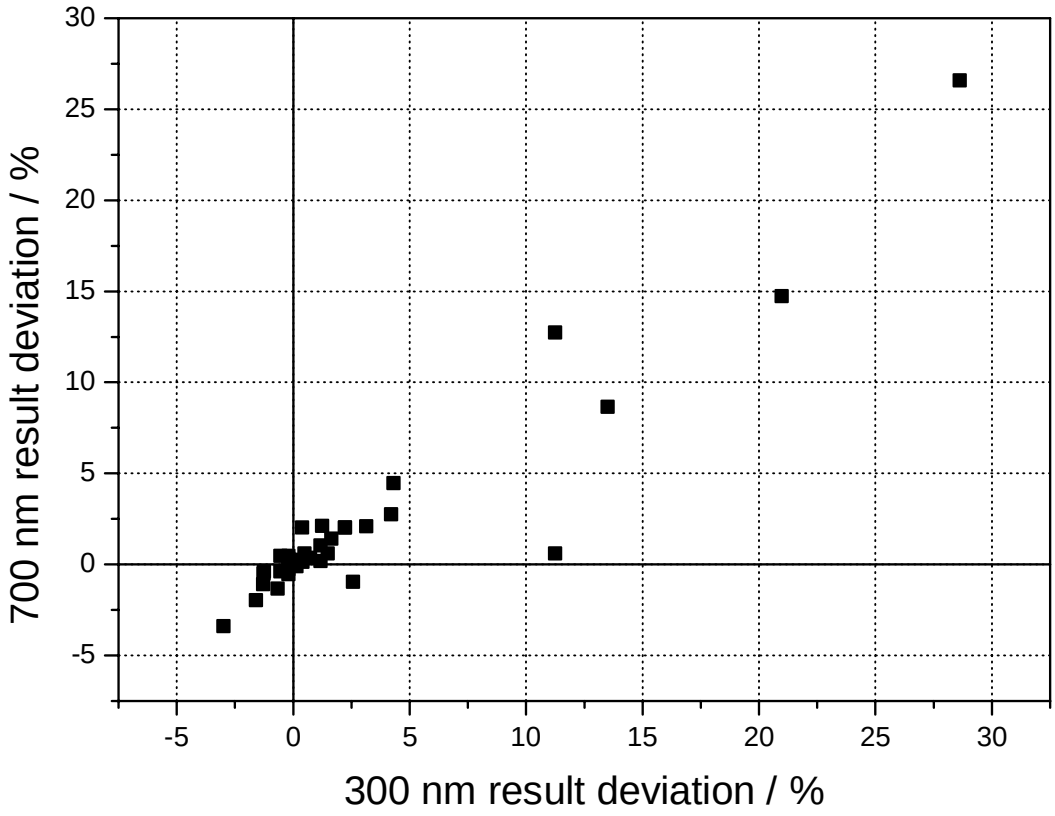


Figure 8. Deviations from the reference value for 300 nm and 700 nm in percentages as an XY plot.

From Figure 8 it is clear that the relative errors of an instrument for 300 nm and 700 nm samples are strongly correlated. This means that a simple calibration with one reference, yielding one scale correction coefficient, would have eliminated most of the error in both 300 nm and 700 nm grating pitch measurements.

6 Uncertainty estimates

Most of the participants estimated the uncertainty of the measurement. Usually the estimated uncertainty was not the standard uncertainty as usually used in calibration certificates and comparisons. Typical uncertainty estimates were only based on standard deviation of the measurement. Some participants used specifications of the instrument as an uncertainty estimate. Some uncertainty estimates were “educated guesses” based on measurement experience but the uncertainty components were not analysed. None of those were realistic uncertainty estimates that can be used to estimate the accuracy of the results and compare the results with other measurements. Standard deviation of the measurement underestimates the measurement uncertainty, because it does not include any information about systematic errors. “Educated guess” is a subjective estimate that can either under- or overestimate the uncertainty, but it often includes implicitly at least some systematic errors, and it is better than no uncertainty estimate at all.

Standard uncertainty [8] is a well-defined way to give uncertainty estimates. Proper uncertainty analysis includes a mathematical model for the measurement and estimated systematic and random errors.

Below is an example of an uncertainty budget for an AFM measurement. The measurement model is developed for the case of measuring a 1-D grating after calibrating the instrument by measuring a calibrated grating. The calibration and measurement can be done in the same environmental conditions and same part of the scanning area for better accuracy. The calibration factor uncertainty can be assessed using a similar analysis. The model is written as follows;

$$p = \frac{1}{n}\sum_{i=1}^{n}\left[c_x \frac{l_i}{N}\right]\frac{\cos\gamma}{\cos(\beta)(1+\alpha(t-20°\mathrm{C}))}$$

simplified =>

$$p \approx \frac{1}{n}\sum_{i=1}^{n}\left[c_x \frac{l_i}{N}\right] - \frac{\gamma^2}{2}P + \frac{\beta^2}{2}P + \Delta t_{20}\,\alpha\,P \qquad (1)$$

$$c_x = \frac{p_{ref\_cert}}{p_{ref\_meas}},$$

where p is the pitch result for the sample, P is the nominal pitch of the sample P=300 nm, c_X is the calibration factor for the x-scale, l is the measured length over N=10 grating periods (later l without subscript is the mean over the individual analysed lines), n is the number of analysed lines in the AFM image, Δt_{20} is the temperature difference from 20°C, α is the thermal expansion coefficient of the sample, P_{ref_cert} is the pitch value of reference from the certificate and P_{ref_meas} is the measured pitch value for the reference. cos β is the cosine error due to sample tilt in the XZ plane and cos γ is the cosine error due to the analysis line not being orthogonal to the grating grooves (in the sample surface plane). The sensitivity coefficients are shown in the following equations and in the example uncertainty budget.

$$\frac{\partial p}{\partial c_x} = P$$

$$\frac{\partial p}{\partial l} = \frac{c_x}{N} \approx 1/N$$

$$\frac{\partial p}{\partial \beta^2} = \frac{1}{2}P$$

$$\frac{\partial p}{\partial \gamma^2} = (-)\frac{1}{2}P \qquad (2)$$

$$\frac{\partial p}{\partial \Delta t_{20}} = \alpha P$$

$$\frac{\partial p}{\partial \alpha} = \Delta t_{20} P$$

$$u_c^2(p) = \sum_i \left(\frac{\partial p}{\partial i}u_i\right)^2 \qquad (3)$$

$$u_c = \sqrt{u_1(y)^2 + u_2(y)^2 + \ldots + u_i(y)^2}$$

Table 4. Example uncertainty budget for pitch measurement

input parameter	symbol	estimate, x_i	prob. distr.	standard unc. $u(x_i)$	sensitity coefficient c_i	uncertainty u_i(y) /nm
measured length, repeatability	l	3001.2 nm	normal	4.1 nm	0.1	0.41
cosine error	γ^2	0.0004 rad^2	exponential	0.0004 rad^2	150 nm/rad^2	0.06
cosine error	β^2	0.0009 rad^2	exponential	0.0009 rad^2	150 nm/rad^2	0.14
temperature diff. from 20°	Δt_{20}	5 °C	rectangular	0.9 °C	0.000771 nm/°C	0.001
thermal exp. coeff. of sample	α	2.57 ppm/°C	normal	0.02 ppm/°C	0.0015 nm °C	0.00003
calibration factor for x-scale	c_x	1.0015 relative	normal	0.004	300 nm	1.20
Measured pitch	p	300.49 nm			standard uncertainty	1.3
					expanded uncertainty (k=2)	2.6

7 Calibration of the instruments

In addition to the measurement results, the participants were asked about the calibration of the instruments. According to the replies received, most commonly the instrument is calibrated once a year when the instrument manufacturer performs its yearly maintenance. Some of the instruments have a dedicated person responsible for the calibration, twice a year, or once in a while. A few participants calibrated the instrument just before the measurements. Of those only one lab always calibrates before precision measurements. Some of the instruments have been calibrated only when first taken into use. The remaining few instruments have never been calibrated. For one instrument the information was not available (it was not known whether the instrument was calibrated or not). Some of the uncalibrated instruments were reported not to be used for quantitative measurements, part of the results being checked with other methods.

In most laboratories the users themselves do not calibrate the instruments and are not instructed about calibration in any way. In some laboratories there is mainly one user and therefore no need to instruct others. In others the user is responsible for calibration but there are no instructions.

Checking the scales of a microscope is a simple measurement that can be done regularly even if the proper calibration and adjustment are only done during maintenance. Regular calibration is an easy way to ensure proper operation and detect possible errors.

8 Conclusions

The comparison shows that the measurement capabilities of the laboratories vary significantly. For a nominally 300 nm grating, 19 results out of 36 differed from the reference value by more than the estimated uncertainty, and three results did not have an uncertainty estimate. For nominally 700 nm samples, 15 results out of 39 differed more than the estimated uncertainty and four did not have an uncertainty estimate. The largest deviations were approximately 20 % for both grating types. For most laboratories the relative error was similar for both samples, caused by a scale factor error that could be easily corrected with calibration. For some participants it may be appropriate to reconsider the uncertainty estimation. For others it may be useful to consider the target/desired accuracy level and the current calibration practices and their possible updating. Several participating laboratories also had a good comprehension of their measurement capability, matching the comparison results.

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Orders: e-mail tilaukset@mikes.fi.

ISBN 978-952-5610-96-3 (nid.)
ISBN 978-952-5610-97-0 (pdf)
ISSN 1235-2704

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