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A nanoscale linewidth/pitch standard for high-resolution optical microscopy and other microscopic techniques

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Abstract
We have developed a new lateral standard on the nanometre scale for use with the recently introduced high-resolution optical microscopy techniques such as deep ultraviolet microscopy (DUVM) and confocal laser scanning microscopy (CLSM). The standard provides structures in the submicron- and sub-100 nm scale, and meets the metrological requirements for accurate and traceable optical microscopy measurements. It can be used as a length measurement standard (for pitch and linewidth measurements) and for quick resolution and astigmatism testing of all these instruments. Additionally, circular gratings provide a new way for the calibration of scanning probe microscopes.

Keywords: electron beam lithography, ECR-plasma etching, linewidth standard, ultraviolet microscopy, confocal laser scanning microscopy

(Some figures in this article are in colour only in the electronic version)

1. Introduction

In the current state-of-the-art optical bright field microscopy a lateral resolution of about 120 nm is achieved by using deep UV microscopy (DUV), at a wavelength of $\lambda = 248$ nm, and water immersion. Due to its high-precision rotatable objective the asymmetrical aberrations can be reduced to less than 1 nm [1]. The lateral resolution limit of confocal laser scanning microscopes (CLSM, at $\lambda = 405$ nm) is in the range from 200 nm to 250 nm. In order to test the optical performance of these high-resolution microscopes artefacts with structure widths (in our case lines with defined linewidths) in the submicron range down to 100 nm are required. However, there is a lack of suitable and commonly available standards which would allow one to test and calibrate the lateral resolution of the microscopes. The focus of the presented standard is for lateral use. Well height standards are already available. It is not suitable to build a lateral standard on the basis of quantum dots or nanoparticles, because an accurate quantitative determination of the structure widths is not possible, since rigorous modelling tools are not currently available for the interaction of the object with the illumination of the microscope. Consequently, it is not possible to determine these structures with measurement uncertainties for $k = 2$ of less than 20 nm.

The new lateral standards have to fulfil several requirements: they need to be produced at a high quality using today's standard equipment and technology in order to have an efficient and economical manufacture, and they need to have linewidths with fine increments within the lateral resolution range of the microscopes, i.e. from 100 nm to 300 nm. Additionally, they should contain not only isolated

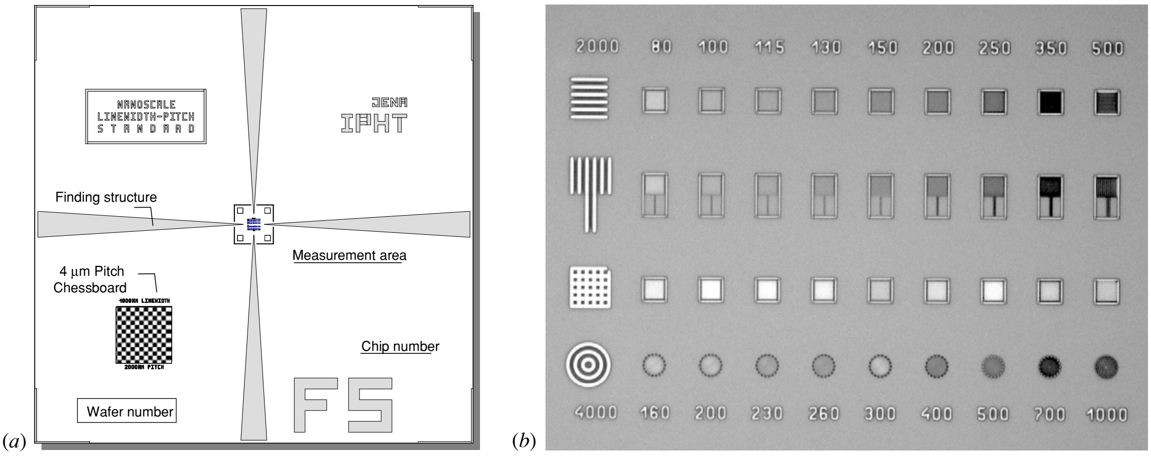


Figure 1. (*a*) Sketch of the 8 mm × 8 mm calibration chip with finding structures. (*b*) Measurement area of the nanoscale linewidth/pitch standard, the column numbers state the nominal pitch, below, and the nominal linewidth in nanometres, above.

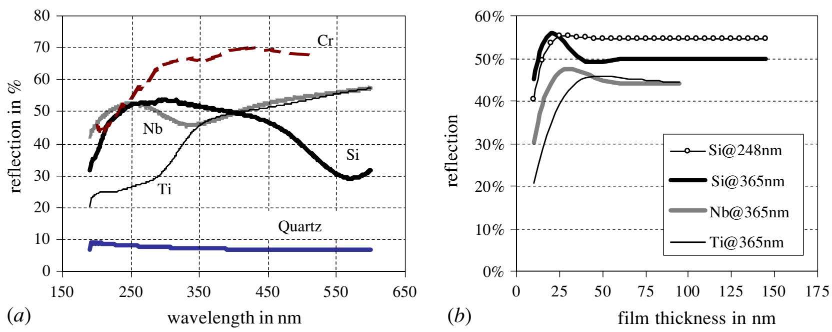


Figure 2. (*a*) Measured spectral reflectance of different thin film materials (Cr, Nb, Ti and Si), with thickness 50 nm, with quartz as reference; (*b*) dependence of reflection on film thickness (simulation).

lines but also linear, circular and cross gratings and it should be possible to model the interaction of the light with the structures using state-of-the-art rigorous diffraction simulation tools such as the rigorous coupled wave analysis (RCWA) method or the finite elements (FEM) method [2].

2. Materials and fabrication

2.1. Layout pattern

The high-resolution linewidth and pitch patterns are arranged in the centre of a quartz chip (figure 1(*a*)) which has a size of 8 mm × 8 mm and a thickness of 1 mm. The chip contains suitable finding structures which make it easy to locate the patterns. The patterns consist of different grating structures etched in a thin film layer which has been sputtered on the quartz substrate. The standard contains one-dimensional line gratings (for both x and y), two-dimensional gratings (cross gratings) and circular gratings. The duty cycle (ratio of the width of the line and the trench) of the gratings is nearly 1:1. Isolated line structures for the determination of linewidth (CD, critical dimension) are added on one side of the line gratings (figures 1(*b*) and 7). The pitch values of the gratings are 160 nm, 200 nm, 230 nm, 260 nm, 300 nm, 400 nm, 500 nm, 700 nm, 1 μm and 4 μm, i.e. the linewidths are between 80 nm and 2 μm. The different gratings are arranged in columns each of which contains gratings with the same pitch value. With the exception of the larger 4 μm structures each grating has an area of around 10 × 10 μm^2. In addition to the high-resolution pattern the chip contains the chip number, the patch number and also a large-scale 4 μm chessboard (size: 1 mm × 1 mm) in order to enable measurements at lower magnification (figure 1(*a*)).

2.2. Choice of material

In order to obtain a high contrast in UV microscopy in both the reflection and transmission modes, a range of potential materials and manufacturing processes were tested [3, 4]. For the grating material we have investigated sputtered thin films of silicon, niobium and titanium with a thickness of 30–50 nm. From lifetime experiments we know that high-dose UV exposure (4 h at $\lambda = 250$ nm and 2 mW cm^{-2}) of Nb thin films does not significantly change the reflectance.

Spectral reflectance measurements have shown that Cr, Si and Nb films have a sufficient optical contrast to quartz in the UV and DUV range (figure 2). On the other hand, titanium thin films show a weak reflectance, and thus poor contrast to quartz, in the DUV range. Amorphous silicon has, in contrast to crystalline silicon, nearly constant reflectance in the 250–450 nm wavelength range. Furthermore, calculations show that the UV reflectance is almost independent of thickness for thicknesses down to about 30 nm (figure 2(*b*)). In order to obtain a good contrast in transmission microscopy it is important that the structures absorb the UV radiation to a significant extent. If the structures would only reflect the (*a*) (*b*)

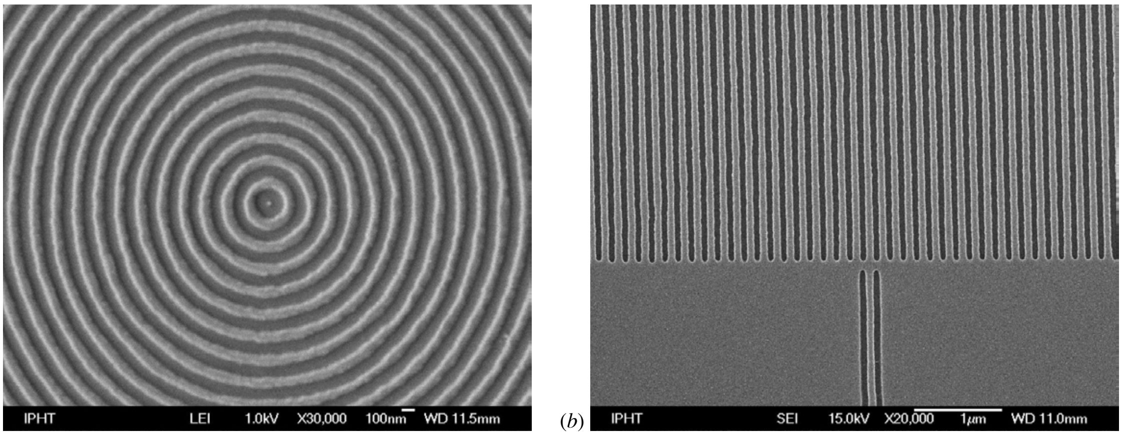


Figure 3. SEM images showing (*a*) the 160 nm circular grating etched in a 25 nm thick amorphous silicon on quartz and (*b*) the resist mask of a 160 nm line grating with isolated line structure (Linewidth (CD): 80 nm).

light this would lead to higher stray light, because this light would be backscattered on the quartz substrate. Simulations and measurements show that Si thin films of at least 30 nm thickness absorb more than 90% of the UV radiation below a wavelength of about 350 nm and are thus a promising material for transmission microscopy.

In summary, these studies have shown that amorphous silicon on quartz is a suitable candidate for a standard used in UV microscopy. Therefore, our nanoscale linewidth/pitch standard is made in 25–30 nm thick amorphous (or nanocrystalline) silicon films on quartz. Thin objects have a reduced edge transition range for a given edge angle and thus provide a better definition of the edge position.

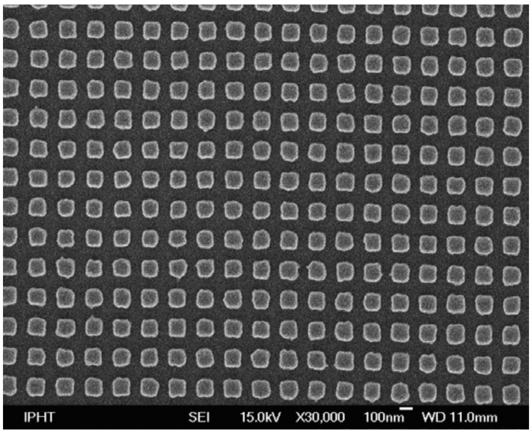
2.3. Fabrication

First, an amorphous (or nanocrystalline) silicon film is deposited by ion-beam sputtering onto a quartz wafer at room temperature. For the target an Si wafer was used and argon was the sputtering gas in a Kaufmann-type ion beam source. Before deposition the substrate was cleaned by a short exposure to the ion beam and the target underwent a pre-sputter clean. The surface roughness of the sputtered amorphous Si films was measured to be smaller than 3 nm by means of the 'total integrating scattering method' [4].

For the e-beam resist a 120 nm thick PMMA resist ARP671.04 (molecular weight: 950 000 g mol^{-1}) from 'Allresist GmbH Berlin' was used. The resist film was prepared by the spin-coating technique, baked for 1 h at 180 °C on a hotplate and was developed after the e-beam exposure in a 1:3 mixture of methylisobutylketone (MIBK) and isopropanol (IPA). In order to obtain a high-resolution resist pattern a commercial Leica LION-LV1 e-beam lithography system was used. The e-beam system provides minimal Gaussian beam diameters from 2 nm at 20 keV to 6 nm at 2.5 keV and has a high-precision *xy* stage [6]. High-resolution patterns were exposed as single pixel lines in the CPC mode (continuous path control) at a beam energy of 20 keV and a beam current of 50 pA. The CPC mode is a special feature of the LION e-beam writer and means that during an exposure the stage moves with a quasi-fixed e-beam along the pattern geometry. In contrast to the most commonly used step and repeat mode the CPC mode provides stitching free exposures, i.e. stitching errors do not occur. Moreover, the CPC mode allows the generation of very sophisticated curved structures, in our case the circular gratings. The linewidths were controlled by the number of e-beam tracks per structure (depending on the target pattern size) and by the electron dose for fixed development conditions.

For the pattern transfer of the PMMA mask into the amorphous silicon we use an electron cyclotron resonance (ECR) high-density plasma system with an RF-biased substrate. The substrates enter through a load–lock system, thereby giving a good base pressure to the etch chamber. The gas choice was CHF_3. Our etch conditions gave a ratio of about 2 between the resist mask and Si etch rate, although thermal load on the resist mask has an influence on this ratio. For example, it was seen that if the Si was continuously etched the mask would be completely etched away after about 70 s; however, if a pause was introduced this could be increased to 120 s. Contrastingly, the Si was etched with a steady etch rate, of about 40 nm per minute. Therefore, useful ratios could be achieved. After etching the films were cleaned both by acetone and isopropanol in an ultrasound bath, and then by exposure to an oxygen plasma.

Several samples of this new standard have been fabricated and evaluated using state-of-the-art optical UV, DUV and CLS microscopes, and scanning electron microscopy (SEM) and atomic force microscopy (AFM) equipment. In order to interpret the measurement results and the images made by the optical microscopy tools, knowledge about the real size and shape of the fabricated structures is important. Therefore, the patterns were characterized by means of a high-resolution SEM (JEOL JSM6700F, using an in-lens SE detector) during the whole fabrication process. In order to avoid charging effects during the SEM inspection the samples were covered with a 10 nm Au film. An important point in nanoscale manufacturing is the structure transfer from the resist mask to the Si film without a change in the size or shape. The images in figure 3 show examples of the resist mask and the etched Si structure for two different 160 nm gratings. It can be seen that the pattern transfer works very well.

Measurements by means of an AFM (*Veritekt* from Carl Zeiss AG) and SEM show that the very thin silicon structures have an edge angle of about 60° [4]. The pitch values of the gratings are very close to the design values; the uncertainty of the mean pitch was measured to be 3 nm (1σ). Inside the (*a*) 

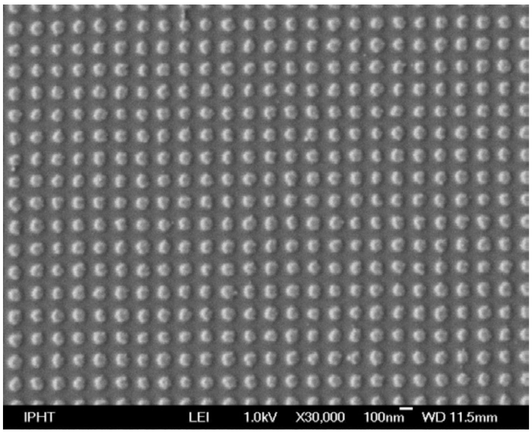
(*b*) 

Figure 4. (*a*) Details of the 230 nm pitch cross grating and (*b*) 160 nm pitch cross grating.

Table 1. Specifications of the nanoscale linewidth/pitch standard.

Parameter	Values
Pitches	160 nm, 200 nm, 230 nm, 260 nm, 300 nm, 400 nm, 500 nm, 700 nm, 1000 nm and 4 μm. Uncertainty of mean pitch: 3 nm 1σ
Linewidths of the isolated lines	Nominal: 80 nm, 100 nm, 115 nm, 130 nm, 200 nm, 250 nm, 350 nm, 500 nm and 2 μm. Linewidth variation along the lines (within a central part of 6 μm): 8 nm 1σ
Circularity of the circular gratings	$\pm 0.6\%$ deviation of mean pitch in the *x*- and *y*-directions (± 1 nm for 160 nm grating)
Edge roughness	5 nm RMS (20 nm p–p)
Sidewall angle	60°
Traceability	CD and pitch calibration by PTB Braunschweig on request

gratings a duty cycle of nearly 1:1 was achieved. In contrast to the precisely made pitch values, the linewidths of the structures can differ from the nominal values by about 20% depending on the fabrication conditions. Therefore, the CD values will be given in the specification list of the standard, see table 1. The linewidth variation of the isolated line structures is 8 nm (1σ) within a central part of 6 μm in length (measured for several samples). The circular gratings have been fabricated to a high quality. The mean grating pitch values in the *x*- and *y*-directions of the 160 nm grating agree to within ± 1 nm (0.35 nm 1σ). In principle, with our present technology it is possible to fabricate cross gratings with pitches of down to 160 nm. We have seen that the dots with dot sizes larger than 100 nm have the desired rectangular shape (figure 4(*a*)), for smaller dot sizes, however, the dots become more rounded in shape (figure 4(*b*)).

3. Measurements

We have investigated the prototypes of the nanoscale linewidth/pitch standard using state-of-the-art UV microscopes with wavelengths of down to 248 nm, CLSM, AFM and also SEM equipment.

3.1. UVM and DUVM

3.1.1. Optical resolution. In practical use, particularly the circular gratings inside the nanoscale linewidth/pitch standard allow one very quickly to obtain information about the resolution and the quality of the optical system (e.g. astigmatism). In agreement with the well-known Rayleigh resolution formula, $\Delta x = k \cdot \lambda / NA$, the best resolution was achieved by using short wavelengths and high numerical apertures (NA). The DUV microscope 'Leica INM 300 DUV' operates at an illumination wavelength of 248 nm, and two kinds of DUV objectives can be used, dry and water immersion objectives. Figure 5(*a*) shows an image of the 160 nm circular grating (having trench widths of about 55 nm and Si linewidths of about 105 nm) obtained using the DUV dry objective (150×, NA = 0.90, optical magnification: 300×). With the DUV dry objective no information about the grating is gathered. In contrast, with the DUV water immersion objective (200×, NA = 1.20, optical magnification: 400×) the 160 nm gratings could be resolved (see figures 5(*b*) and 6).

Table 2. Measured resolution limits of different microscopes, obtained for measurements on the nanoscale linewidth/pitch standard.

Microscope	Wavelength (nm)	Numerical aperture, NA	Smallest visible pitch (nm)
DUVM	$\lambda = 248$	1.2 (Immersion)	160
DUVM	$\lambda = 248$	0.8	200
UVM	$\lambda = 365$	0.9	260
CLSM	$\lambda = 405$	0.95	300

Table 2 shows the measured resolution limits of different microscopes, obtained from measurements on our nanoscale linewidth/pitch standard. For this kind of application the fine pitch steps of the gratings in our standard in the range of 160–300 nm are very helpful.

3.1.2. Linewidth measurement. An optical linewidth measurement of the 400 nm isolated line structure (200 nm nominal linewidth) was performed using an UV microscope (Leica INM, $\lambda = 365$ nm, NA = 0.9), see figure 6. The

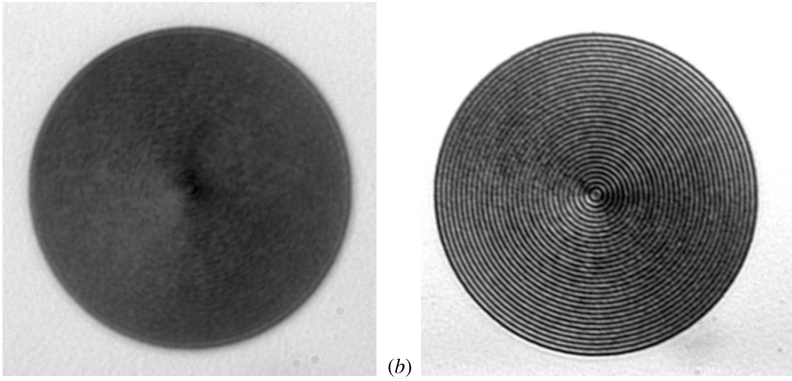


Figure 5. DUV microscope images of the 160 nm circular grating (Si on quartz) taken with (*a*) dry objective and (*b*) water immersions objective; Source: Leica Microsystems AG.

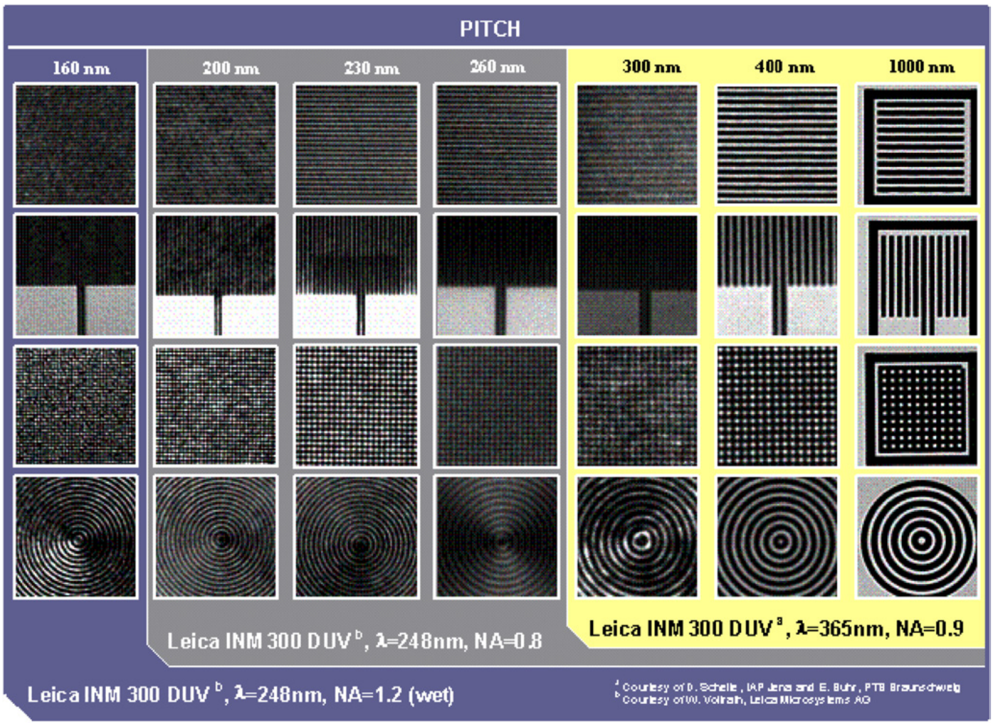


Figure 6. DUV microscope images obtained from the structures of the nanoscale linewidth/pitch standard. The column numbers show the pitch in nanometres. Only the high-resolution water immersion DUV microscope ($\lambda = 248$ nm, NA = 1.2) was able to provide clear images of the 160 nm gratings. (DUV images: courtesy of D Schelle (IAP Jena) and W Vollrath (Leica Wetzlar)).

measured line profile is shown in figure 8(*a*) together with the theoretical line profile calculated by using a rigorous coupled wave analysis method (RCWA, see [7, 8]). From the modelled line profile a linewidth of 190 nm is obtained, which is in good agreement with the SEM measurement (SEM result: 200 nm, see figure 8(*b*)).

3.2. AFM and CLSM application example

Measurements of the circularity of the circular gratings, made at the Physikalisch-Technische-Bundesanstalt, (PTB) [9], have shown that the deviations of the mean pitch in the *x*- and *y*-directions are very low (in the range of ± 1 nm for a 160 nm circular grating). Therefore, the circular gratings are suitable for a quick AFM scanner calibration. An example of such a scanner calibration made using a circular grating is presented in figure 9. This shows an AFM image of a high quality 160 nm high-resolution circular grating. From the indicated pitch data in the *x*- and *y*-directions (150 nm and 168 nm, respectively) a calibration factor for the *x*- and *y*-directions of the scanner used can be easily derived, provided the grating itself has undergone certified calibration. In this case the measurements were made by using a commercial AFM with a NanoSensor Pointprobe®-tip. The high-resolution measurements (512×512 pixels) were made over a scan area of $15 \times 15\ \mu m^2$ in the Tapping™-mode (the intermittent contact mode) with a scan rate of 1 Hz. The sample was used in its as-delivered state i.e. it was not necessary to cover the surface with an additional conducting film in order to avoid charging effects.

Prototypes of the standard have also been investigated and tested with a confocal laser scanning microscope (Carl Zeiss LSM 5 PASCAL). It was found that useable measurements in the *z*-direction need a minimum step height of 15 nm. The 30 nm thick calibration structures were easily measured with a

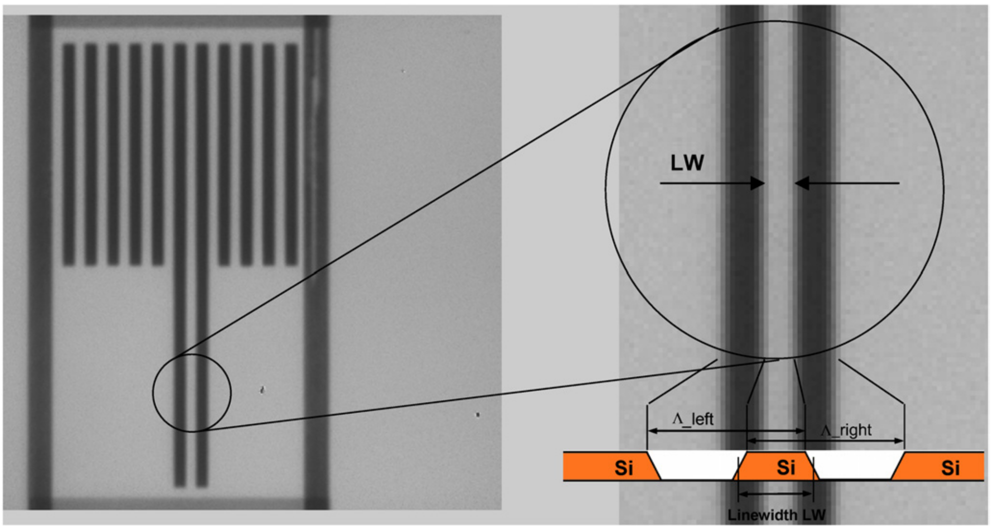


Figure 7. Left: UVM image showing the complete 400 nm linear grating with added isolated line structure, right: detail of the isolated line structure and sketch of the cross section used to define the measured parameters.

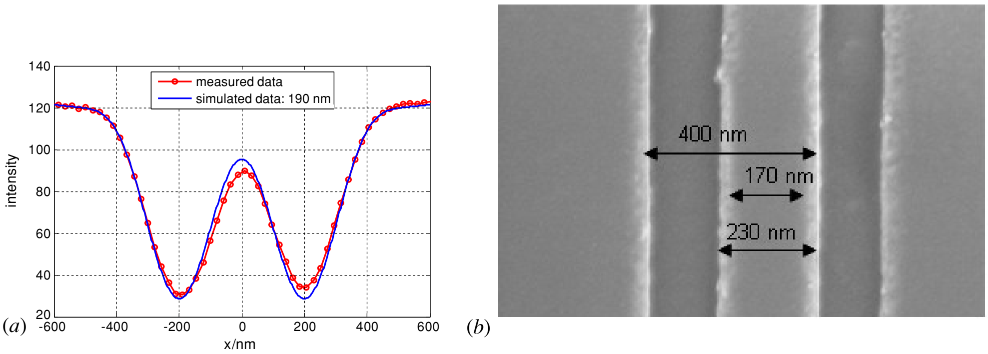


Figure 8. (*a*) Measured and simulated optical line profiles across the isolated line structure shown in figure 7. The simulation was performed using the following values for the pitch and linewidth: Λ_left = Λ_right = 400 nm, LW = 190 nm. (*b*) SEM image of this structure (made with a Zeiss LEO Supra 35VP).

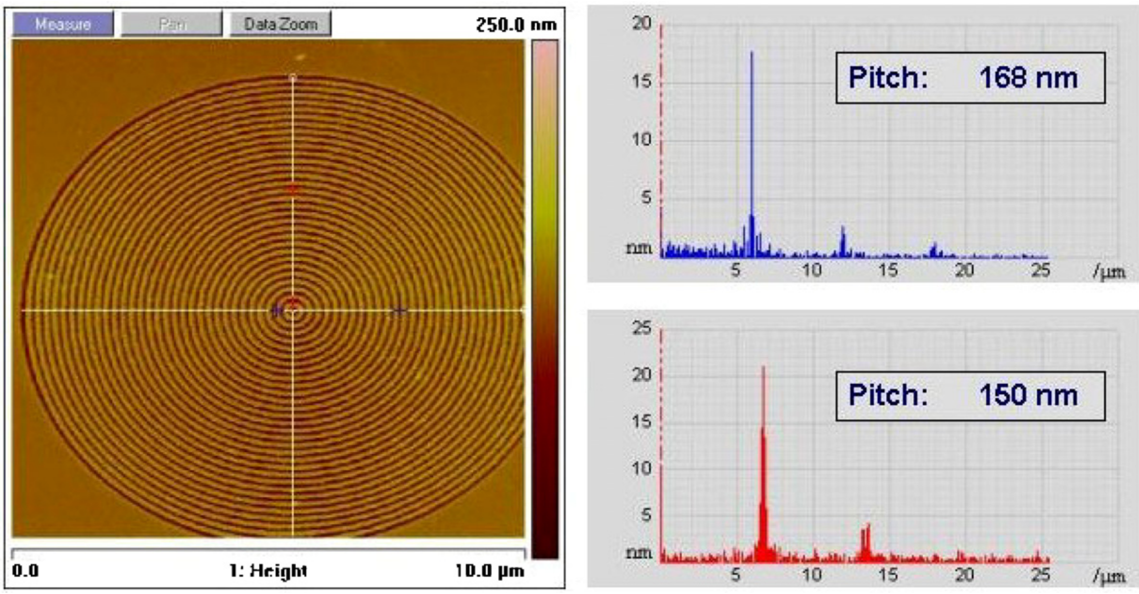


Figure 9. AFM image of a 160 nm circular grating (Si on quartz) and right: FFT-spectra and the derived pitch values from scan lines in the *x*- and *y*-directions, source: NanoWorld.

vertical resolution of about 5 nm. By using a wavelength of $\lambda = 405$ nm and a $100\times/0.95$ objective (EC Epiplan Apochromat) images of gratings with a pitch of down to 300 nm were clearly resolved. In addition, FRT GmbH has evaluated the nanoscale

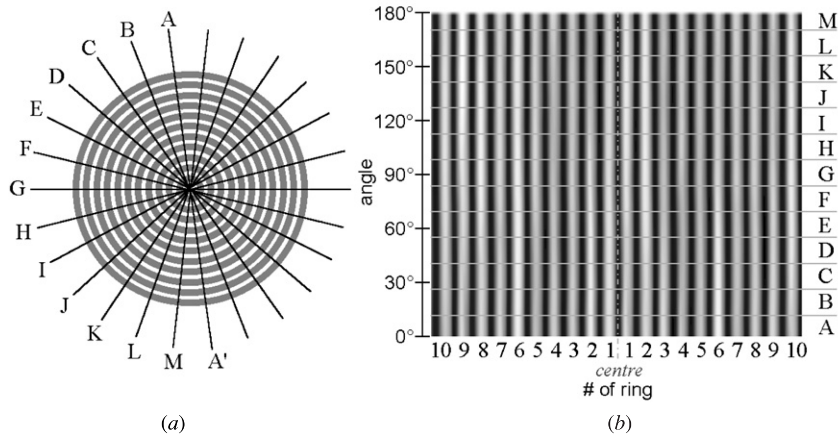


Figure 10. (*a*) Scheme of the evenly spaced radial profile scans (here: only 13 profiles A to M for clarity, typically: some hundred) measured with the Met. LR-SPM for calibration of the circular gratings. Profile A′ is recorded at the same position as profile A but with opposite scan direction (*b*) The profiles in an angle–radial axis plot so that the half rings (180°) are shown as lines. This allows a grating analysis similar to that applied to one-dimensional gratings to be used.

linewidth/pitch standard by means of its MicroGlider®, which can be used with both optical and AFM sensors. It has been shown that the high-resolution cross gratings are very interesting for use as AFM calibration objects.

3.3. Strategy for the certified calibration of the circular gratings by AFM

Prior to its application as a calibration standard, e.g. for scanning probe microscopy (SPM) as shown above, the nanoscale linewidth/pitch standard itself needs to be calibrated traceably and accurately at a National Metrology Institute (NMI) such as the PTB or a lab accredited by an NMI.

Circular gratings pose new challenges when it comes to high-quality calibrations, as the large majority of lateral standards used, e.g. for SPM calibration, consists of one-dimensional or two-dimensional gratings, and both the measurement strategy and evaluation algorithms in use focus on these kinds of gratings. However, recently various kinds of novel two-dimensional standards such as those presented here, or even three-dimensional SPM standards as suggested by Ritter *et al* [10], have been in development. These require novel calibration strategies.

With the Metrological large-range SPM (Met. LR-SPM) the PTB has developed a high-accuracy SPM, which is directly traceable with an integrated I_2-stabilized laser interferometers [11]. One advantage of this system is that the scan process can be programmed according to the best-suited measurement strategy. There is no limit e.g. on the number of pixels per scan line or the number of lines within a scan image, or on the orientation of the individual scan lines. For the circular gratings, we propose a conventional quadratic scan of the circle field first in order to determine the location of the centre of the circles. In a second step, a set of special high-resolution radial scan lines through the actual centre can be recorded at equidistant angles (see figure 10(*a*)). In this way, an angle–radial axis plot can be generated (see figure 10(*b*)). The analysis of these measurements thus becomes analogous to that of one-dimensional gratings, for which an advanced Fourier transform (FT) and a gravity centre (GC) method have been described and compared by Dai *et al* [12]. The latter also allows the determination of the deviations of the individual rings and ring segments from the ideal concentric circles. Alternatively, this goal can also be achieved by applying the fine linearity analysis of the commercial software package SPIP (Image Metrology AS, Lyngby, Denmark) which is optimized for the analysis of two-dimensional rectangular gratings. As the angle—radial axis plot shown in figure 10(*b*) is like a one-dimensional grating, it is advisable to first chop this image by adding an artificially generated and perpendicularly orientated one-dimensional grating so that each grating line (here: ring) is divided into segments of equal size [13]. The position and position deviation of each of these segments from the fitted mean circle can then be easily determined by SPIP. The analysis routine of this program first performs an FFT in order to determine the main lateral frequencies in both directions, then it determines the unit cell and cross correlates this characteristic mesh of the grating with the whole image. By calculating the gravity centres of the cross-correlation peak within each mesh, the position of each segment is determined [14]. This additional information on the local properties of the grating allows not only a better assessment of its quality and its uncertainty contribution when applied to instrument calibration, but also enables the characterization of the local behaviour of the scan system (e.g. guidance errors of the lateral axes, cross talk between them, distortions).

This calibration strategy for the circular gratings is currently being tested at the PTB. In order to compare its performance with established strategies and standards, a second instrument—another high-stability AFM with a conventional scan range of 100 μm in both lateral directions—is used for measurements on these samples. In this case, a set of conventional quadratic images of 1024 pixels × 1024 pixels is made for different scan orientations and locations. These data are analyzed by using error separation techniques in order to distinguish sample inhomogeneities from scanner irregularities. Furthermore, the radial spectrum of the images will be calculated and compared to the results obtained by the measurement and evaluation strategy chosen for the Met. LR-SPM.

4. Conclusion

Prototypes of our new nanoscale linewidth/pitch standard for high-resolution optical microscopy with patterns having a linewidth of down to 80 nm have been successfully fabricated. The structuring technology applied here ensures that the standards are free of stitching errors. Si gratings on quartz substrates with pitch structures of down to 160 nm have been evaluated by different optical, SEM and AFM methods. The results show that these structures are useful for the characterization and calibration of optical UV and DUV microscopes, confocal laser scanning microscopes and AFM tools. The standard is especially useful for the quantitative assessment of the lateral resolution of UV and confocal laser scanning microscopes. It can be used every day to check the microscope parameters, and also—after certified calibration of the standard—for a calibration of pitch and linewidth measurement tools. The nanoscale linewidth/pitch standard is now available to customers (distributor: Supracon AG [15]).

Acknowledgments

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Queries

(1) Author: Please update reference [10].
(2) Author: Please check whether reference [13] is OK as set.
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