Self-assembled Ge/Si islands as novel nano-roughness standards for Scanning Force Microscopy

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Abstract: The quantitative determination of nanometric surface features is increasingly important for many applications. While Scanning Force Microscopy is often used for the determination of nano-roughness, these measurements lack generally accepted standards for comparability, as neither suitable certified areal nano-roughness standards nor guidelines exist. In a joint project with IPM-RAS, candidates for novel roughness standards based on Ge/Si nano-islands are fabricated and investigated. It is shown that the well-controlled growth of these structures allows to fabricate durable nano-roughness reference samples of high homogeneity. Furthermore, the surface features are sufficiently stable to stand replication techniques such as multiple hot embossing so that negative copies can be fabricated rather easily – a prerequisite for inexpensive production.

1. INTRODUCTION

Scanning Force Microscopy (SFM) is used today not only in research and development but increasingly also in many fields of industrial fabrication and inspection. High-technologies such as semiconductor fabrication, nanotechnology and ultra-precise surface figuring attach great importance to the quantitative information these instruments provide.

1.1 SFM standards and SFM calibration guidelines: current status

A number of transfer standards suited for SFMs have already been developed and are commercially available. Most of them are either lateral standards based on homogeneous 1D or 2D gratings, step height standards with one or several steps of a discrete height, or flatness standards. A recently developed landmark-based 3D standard (so-called "Ritter pyramids") allows, apart from axes calibration, the determination of the coupling between all three axis [Ritter 2007]. For an overview on SFM standards, see e. g. www.nanoscale.de

In order to ensure a uniform calibration procedure, much attention has been devoted to a guideline for SFM calibration based on such standards in the past few years. While this guideline [VDI/VDE 2656 – Part 1] is now established with the whiteprint, its content is currently being discussed internationally at ISO TC 201 SC 9, the sub-committee in charge of Scanning Probe Microscopy.

1.2 The challenges to nano-roughness measurements by SFM

Roughness measurements are one of the crucial applications of SFM. Similar to shape measurements, they require, in principle, the individual SFM tip shape to be known and to be accounted for – this the more so, the smaller the relevant features are. Furthermore, material related effects are not negligible in SFM, and the tip can often not be regarded constant during a measurement series, as tip wear, elastic deformation and tip contamination may occur.

Compared to the long-established and standardized roughness measurement techniques typically recording profiles with many thousand measurement values, SFM allows to acquire a rectangular (usually quadratic) image of the surface topography. However, the number of pixels per image is usually limited to 1 Mpix or less in most commercial instruments currently available, and recording of single profiles at a much higher sampling rate is usually not provided either. Consequently, any attempt towards standardization of SFM roughness measurements will focus on 3D roughness parameters.

Recent activities in the field of roughness standardization e. g. in ISO TC 213 envisage both 3D roughness parameters and a wide range of very different measurement techniques [Krüger-Sehm 2007], among them many optical techniques and Scanning Probe Microscopy (SPM), especially SFM. This is expected to require significant changes: Tight rules such as the number of measurement values, requirements for the multiple subsequent repetition of profiles, the fixed settings for profile lengths to be recorded and – depending on them – fixed lower and upper spatial filter wavelengths are to be replaced or partly weakened to facilitate the application of many more different techniques.

Bearing these new developments in mind, a projected further part of the SPM calibration guideline VDI/VDE 2656 is currently being developed for roughness measurements within VDI/VDE-GMA 3.41/3.43.

A prerequisite for comparable SFM roughness measurements is the development of novel standards, as the standards available for the established roughness measurement techniques are not ideally suited for SFM. An attempt to extend the characteristics of the existing roughness standards into the nanometric regime was made by a nano-grinding process [Krüger-Sehm 2005].

As SFMs usually record images instead of single profiles, it is furthermore necessary to provide a type of standard whose roughness characteristics are isotropic, i. e. (nearly) the same irrespective of the direction and orientation of profiles extracted from the recorded images. Such isotropic samples would allow to assess to what extend the tip shape or the scanner performance is non-isotropic, as it would reveal direction-dependent characteristics of the system as anisotropy in its images and thus direction-dependent roughness parameters.

In the following, a set of such prototype standards will be introduced.

2. FABRICATION OF THE Ge/Si SAMPLES BY SELF-ASSEMBLY

2.1 Growth by molecular beam epitaxy

Self-assembly is an efficient process when it comes to the creation of nanostructures on a surface once the process is understood. As the structures organize themselves reproducibly based on inter-atomic or inter-molecular forces as long as the growth conditions are properly set, no lithographic or mechanic steps (such as polishing or grinding) are necessary to enforce the structure formation.

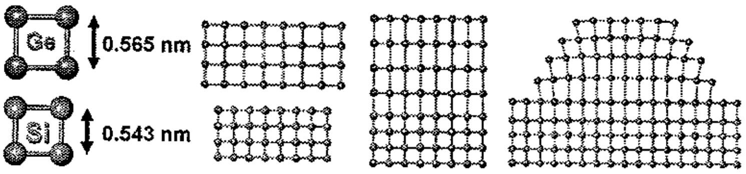

Figure 1: Mismatch of the lattice constants leading to island formation

We apply the self-assembly method to create nanoscopic Ge/Si-islands on a Si substrate [Schmidt 1999] at IPM Nizhniy Novgorod. In our case, the structure formation is caused by the slight mismatch of the lattice constants of Ge and Si, which differ by about 4 % (Figure 1). The structures are grown on Si(001) substrates from solid source through molecular beam epitaxy (MBE) [Shaleev 2005]. The entire process (Figure 2) is performed in ultra-high vacuum without any interruption of the vacuum during fabrication.

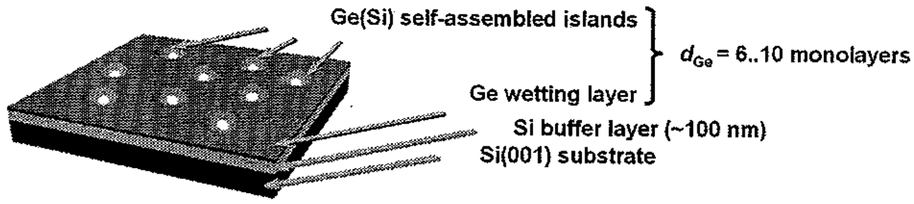

Figure 2: MBE growth of the Ge/Si samples to create nanoscopic islands

In a first step, a Si buffer layer of ~100 nm is grown at 700℃ on the wafer to create a virgin clean surface. Next a Ge wetting layer is deposited; for a few monolayers, this layer is smooth as the Ge atoms arrange on the pattern given by the Si atoms. With continuing deposition, however, the stress induced by the lattice mismatch leads to the formation of islands. Intensive investigations by IPM and its partners show that the islands do not consist of pure Ge, but contain a certain percentage of Si as well. The Si content in the islands depends on the growth conditions and is attributed to inter-diffusion between the freshly deposited Ge and the top Si layers of the substrate.

2.2 Tuneable size of the Ge islands

The type, size and coverage density of the Ge islands can be controlled by the growth parameters, above all by the growth temperature (Figure 3) and secondly, by the deposition rate and the total amount of deposited Ge.

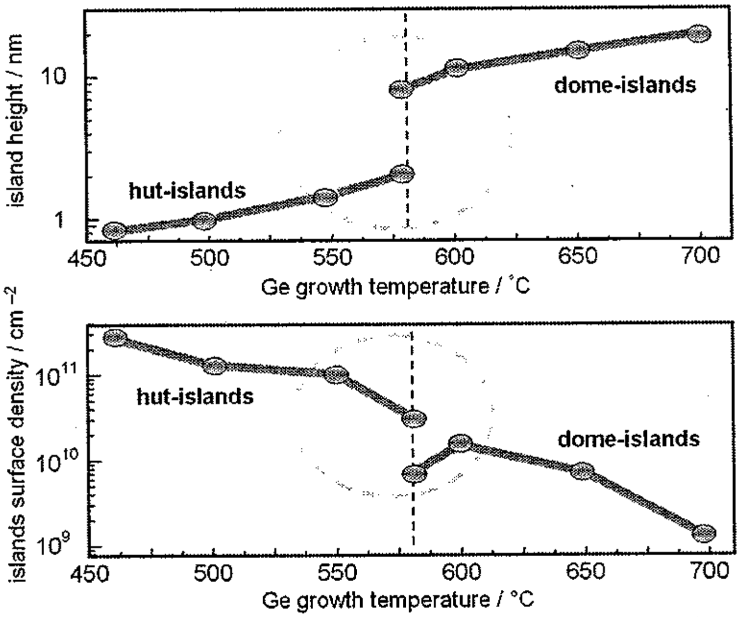

Figure 3: Influence of the growth temperature on island height and density

At temperatures below 580℃, tiny hut-shaped island s with rectangular base (baselines of some ten nanometres) and a height of 0.8 to 2 nm are formed (Figure 4 left). While the size of the islands varies across an image, the orientation of their baselines remains (nearly) the same over larger sample areas. Due to their small size, their imaging by SFM is strongly influenced by the shape of the tip used; therefore they have not yet been envisaged as standards.

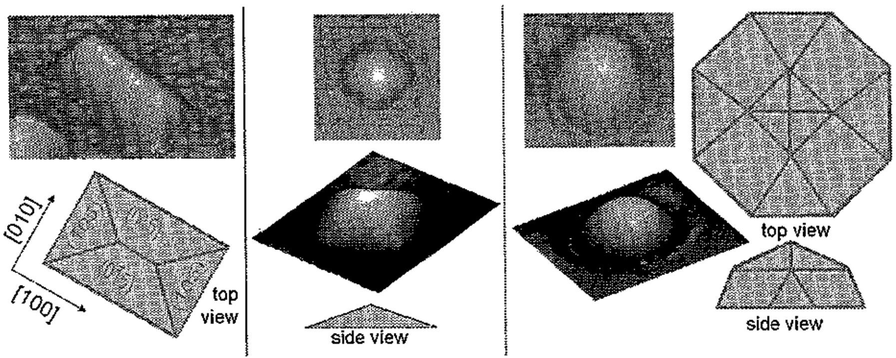

Figure 4: Types of islands: hut (left), pyramid (centre), dome (right)

Growth temperature above 580℃ result in a differen t type of islands. Investigations at IPM suggest that such dome-shaped islands have an octagonal base and are characterized by a four-sided pyramid at their top (Figure 4 right). Although these islands are much larger with a diameter of typically 60 nm to 300 nm and heights ranging from 8 nm to several 10 nm (Figure 3), the details of their shape usually do not show up in SFM investigations due to the radius of curvature of standard SFM tips being in the range of 5 to 20 nm. Consequently, they are imaged as circular islands. On the other hand, the overall apparent size of the islands is only slightly affected by the actual tip shape at this scale. Furthermore, the surface coverage is sufficiently low leading to rather long distances to the next neighbour islands, resulting in a very low percentage of measurements points affected by tip shape influences. These characteristics, and the possibility to vary their size over a larger range reproducibly, turns them into promising nanoroughness reference samples.

It should be noted that in the intermediate temperature region around 580°C both types of islands are observed, accompanied by a third type of pyramidal shape with a square base (Figure 4 centre). The latter are also found in the temperature range where dome-shaped islands prevail. However, these pyramids are rather rare and very small compared to the domes so that they are not expected to affect the roughness values significantly.

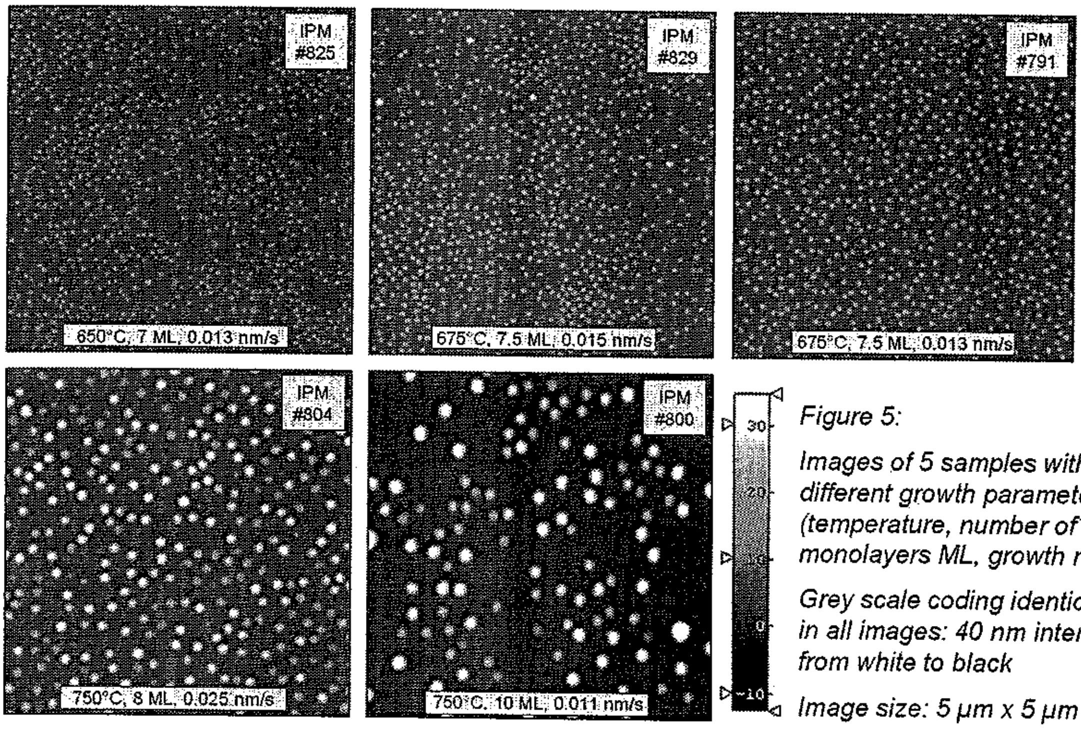

Figure 5:

Images of 5 samples with different growth parameters (temperature, number of monolayers ML, growth rate)

Grey scale coding identical in all images: 40 nm interval from white to black

Image size: 5 µm x 5 µm

3. CHARACTERIZATION OF A SET OF SAMPLES

3.1 Instrumental setup and data processing

Several sets of samples with dome islands were investigated by SFM at IPM and in the PTB cleanroom centre to assess their properties as candidates for a novel type of nanoroughness standards. The instruments used were a Solver PRO (NT-MDT, Zelenograd, Russia) at IPM and a modified Nanostation II (SIS GmbH, Herzogenrath, Germany) at PTB. While the first instrument has a very low noise floor, the latter is characterized by active position control of the lateral axes via capacitive sensors and a strain gauge as senor in z. The SIS instrument allows very accurate positioning by optical microscopy, with the axis of the optical microscope being aligned to the SFM probe tip to better than 3 µm.

All images were levelled 1st order plane and usually also linewise in advanced mode by SPIP™ (Image Metrology AS, Hørsholm, Denmark), i. e.

only those pixels outside the islands have been taken into account for the calculation of the subtraction function, followed by the application of this function to all pixels. No filtering was performed.

3.2 Roughness values

The wide range of islands sizes and surface coverage densities as a result of variation of growth temperature and Ge amount deposited is demonstrated by SFM images at one set of such standards comprising 5 samples (Figure 5).

A set of randomly selected profiles extracted from these 5 images is shown in Figure 6. While this plot helps to inform about the islands' heights and diameters, it needs to be stressed that the roughness values stated next to the profiles are for coarse orientation only; a single profile of only 5 µm length is not sufficiently representative of the whole image.

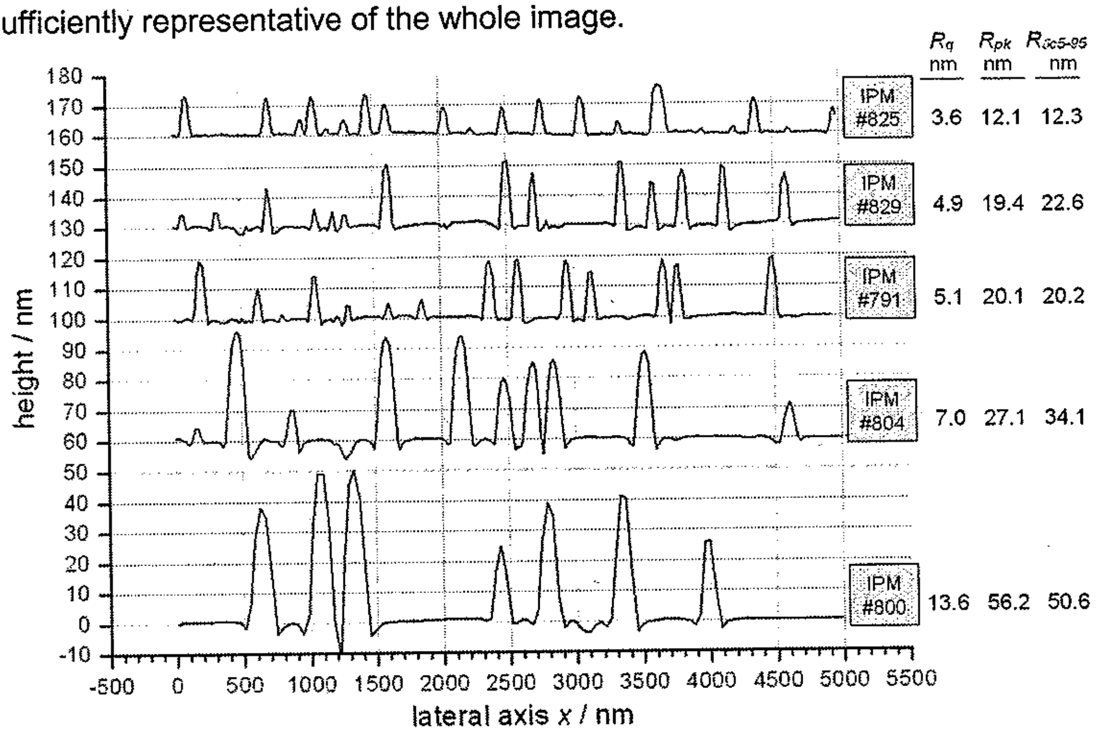

Figure 6: Profiles randomly extracted from the images in Figure 5

Surface roughness was analyzed by SPIP™. In an attempt to expand the international standards for the evaluation of profile roughness to surface roughness and adapt them for Scanning Probe Microscopy, this commercial software package follows the suggestions by K. J. Stout et al. [Stout 1994] and G. Barbato et al. [Barbato 1995]. Table I shows that the Ge deposition technology allows to vary the root mean square roughness *Sq*, the ten-point-height *Sz*, the reduced summit height *Spk* and the 5 % to 95 % interval of the bearing curve $S_{\delta c5\text{-}95}$ by a factor of about three.

The surface skewness *Ssk*, a parameter that characterizes the dominance of holes (<0) or of peaks (>0), ranges from 2.3 to 3.4, indicating that the main features are extreme peaks (i. e. *Ssk* >1). The fractal dimension *Sfd* varies around 2.5.

sample	*Sq*	*Sz*	*Ssk*	*Spk*	*Sk*	*Svk*	*Stdi*	*Sfd*	$S_{\&5\text{-}95}$
#825	3.1	22.3	2.4	10.5	2.0	0.6	92 %	2.68	10.6
#829	4.9	39.3	2.5	18.6	1.7	1.4	95 %	2.40	16.8
#791	4.8	30.7	2.3	17.6	1.3	1.0	93 %	2.34	16.0
#804	7.6	49.4	2.4	25.4	2.2	2.2	95 %	2.68	25.0
#800	9.7	72.6	3.4	42.3	3.0	3.4	88 %	2.50	30.4

Table I: Roughness values for images in Fig. 5. Values in nm except for Stdi and Sfd

4. HOMOGENEITY OF THE SAMPLES – MEASUREMENT STRATEGY

The results presented in figure 5 and table I are – strictly speaking – only valid for the selected 5 µm x 5 µm scan fields and for the particular SFM scan settings. For the use as reference objects, a sufficient uniformity over a larger area must by proved unless specific reference areas are explicitly defined. Furthermore, it needs to be discussed how tight an SFM measurement strategy needs to be in order to ensure sufficient comparability.

4.1 Uniformity across the samples

In a first step, the scan range was varied from 3 µm x 3 µm via 10 µm x 10 µm to 30 µm x 30 µm to check for the scalability of the roughness parameters. All measurements were performed at the highest possible pixel number of 1024x1024. As the images of samples #800 and #825 show, the surface coverage density of the Ge islands varies locally within a few micrometres. Consequently, the scan range needs to be sufficiently large to level out such local effects. While in the case of #825, the 5 µm x 5 µm is just large enough, #800 demands a minimum scan range of 10 µm x 10 µm. In the case of the other 3 samples shown here, even scan ranges as small as 1 µm x 1 µm will do. On the other hand, the scan range must not be chosen too large in order to still ensure sufficient sampling. In the case of the first three images in Figure 5, a scan range of 30 µm x 30 µm at 1024x1024 pixels is the maximum. In conclusion, scan range variation over a bit more than 1 decade is possible on these samples with the typical state-of-the-art pixel number of 1024x1024.

In the second step, the samples of total size of approx. 20 mm x 30 mm were measured both regionally (i. e. shift of the measurement field by a few to some ten micrometers) and globally (centre versus positions 2 mm from the rim of the sample). Individual measurements in a region have shown that the main parameters *Sq*, *Sz*, *Ssk*, *Spk*, *Sfd* and $S_{\&5\text{-}95}$ vary by typically a few percent to 10 %, with some extreme exceptions. The latter are to be attributed to some rather rare irregularities in the surface texture like a few much larger islands and holes. On a global scale across the sample, the deviations of the values were not necessarily larger than within a region – provided some minimal care is taken to make sure that only those regions are measured that look roughly the same in the optical microscope. In conclusion, the samples are quite uniform, but it is mandatory to avoid locations clearly recognizable as irregular and to exclude such measurements from the analysis.

In summary, it needs to be recommended to measure a set of suitable scan fields at slightly different locations in a region and to repeat this procedure in one or two further regions of the sample surface unless a reference area is specified.

4.2 Tip shape influences

As tip wear can clearly be identified as soon as the islands are imaged uncircular (usually triangular), a worn tip can be exchanged in time. At this stage, tip wear just starts to influence the roughness values. At the samples studied, super sharp Si tips yielded only slightly different results.

4.3 Isotropy of the samples

The routine roughness analysis tool in SPIP™ provides, among others, a 2D FFT plot and a polar plot of the angular spectrum, see Figure 7. Throughout the measurement series, distinguished directions have not been identified – unless an apparently worn shows up in the plots. Apart from blunt pyramidal tips, imperfections of the scanstage, such as line-to-line jumps, typically turned out the sole identifiable origin of apparent non-isotropy. Consequently, if artefacts related to the probe or the stage can be eliminated, a very high degree of angular isotropy similar to the one shown in figure 7 is measured. The texture direction index *Stdi* (see also Table I) is usually larger than 90 %. If needs to be added, however, that specially sharpened tips operated under optimized settings have not yet been used; maybe they would give a hint on the crystallographic facets sketched in Figure 4.

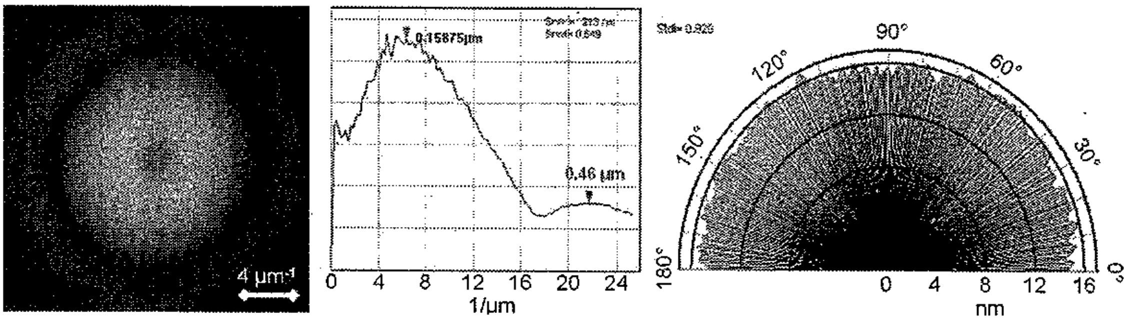

Figure 7: FFT, radial spectrum and angular spectrum of the image of IPM #791 shown in Figure 5. The local maximum around 158 nm corresponds to the measured diameter of the islands, the maximum around 460 nm to the mean distance between islands

4.4 Stability with time

A number of samples was measured by SFM at IPM immediately after their growth, several weeks or months later and then again after one more year at PTB. In the meantime, the samples were stored in the PTB cleanroom centre at 20℃ and a relative humidity of ~45 %. A significant change of the roughness values could not be observed. This confirms the overall IPM experience that such samples are stable.

5. REPLICATION BY HOT EMBOSSING

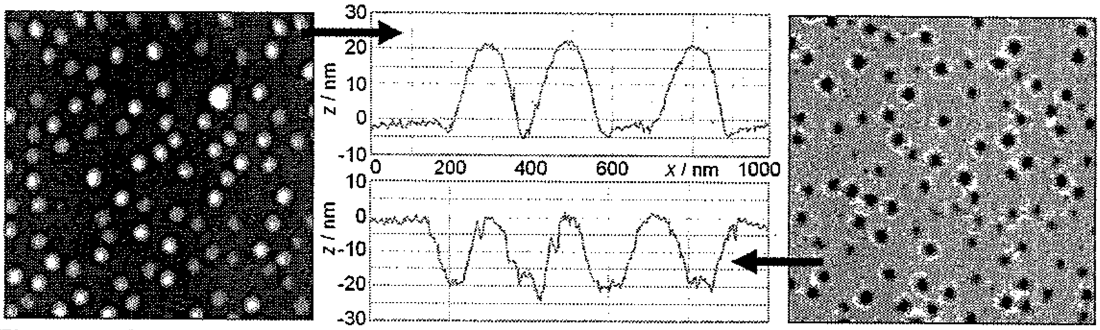

Figure 8: SFM images of 3 µm x 3 µm of master (left) and copy (right), 1 µm profiles

A great advantage compared e. g. to Au nanospheres adsorbed on a substrate is that these Ge/Si islands are firmly connected with the crystallographic lattice of the substrate. This allows to fabricate the negative of the their surface features by hot embossing into plastics, as they stand the high pressure and temperature exerted on the master.

First tests with a sample similar to #804 were performed in the hot embossing apparatus "HEX 03" at IMR Hanover. The process parameters force and temperature were varied to assess their influence: Only slight variations in the quality of the copies could be observed in the subsequent SFM investigations at identical positions on all four copies made. However, a slight gradual deterioration of the master occurs due to fragments of the plastics sticking to the master. This leads to an increase of unwanted indents in the copies if the master is not cleaned between subsequent embossing steps. For this reason, the images of the forth copy had to be excluded from analysis. Furthermore, the rim of the indents left by the islands are rigid (Figure 8). We hope that this minor drawback can be eliminated by optimization of the process parameters.

sample	*Sq*	*Sz*	*Ssk*	*Spk*	*Sk*	*Svk*	*Stdi*	*Sfd*	$S_{\delta c5\text{-}95}$
Master	6.2	61.7	2.4	**19.9**	3.2	*1.8*	94 %	2.68	20.1
Copy 1	4.4	65.6	-2.3	*2.8*	4.0	**11.7**	96 %	2.56	14.0
Copy 2	5.3	71.8	-2.2	*3.3*	5.1	**12.4**	90 %	2.54	16.1
Copy 3	3.8	54.2	-2.1	*2.8*	5.3	**8.4**	85 %	2.58	11.6

Table II: Values for a 10 µm x 10 µm field of master and three subsequent copies (images at copies at identical position). All values in nm (except Stdi, Sfd)

Table II gives an example for the similarity of master and copies. While the same positions could be localized on all three copies studies, the master had to be investigated at a different position. Table II shows that the indents on all copies are about half as deep as the islands' heights on the master (**bold** *Spk* and *Svk* values, $S_{\delta c5\text{-}95}$), resulting also in *Sq* values being about a third smaller on the copies then on the master. This points to a plastic deformation or relaxation at the end of the embossing process. Additionally, tip shape influences may have contributed to this discrepancy: Due to the finite size of the tip, the islands are imaged slightly larger and their indents smaller; this, however, needs to be analysed and simulated in detail to assess the respective share of both effects.

6. CONCLUSIONS AND OUTLOOK

Preliminary measurements at MBE-grown Ge-on-Si islands show that such systems are promising candidates for SFM nano-roughness standards. Complementary surfaces with holes can be fabricated by hot embossing. Except for some few local defects, masters and copies are very homogeneous and isotropic, but to ensure their safe application, marked reference fields might be considered. This, however, might disturb other applications such as scatterometry. The establishment of general measurement strategies and rules remains decisive for comparative SFM roughness measurements. The experience gained here and the recent trends towards surface roughness standardization will be taken into account for future work both at these reference samples and upon drafting the SFM roughness guideline as another part of VDI/VDE 2656.

7. REFERENCES

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