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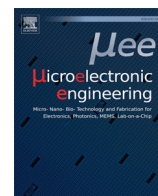
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# Nanoimprint stamps with ultra-high resolution: Optimal fabrication techniques

Mariusz Graczyk<sup>a</sup>, Andrea Cattoni<sup>b</sup>, Benedikt Rösner<sup>c</sup>, Gediminas Seniutinas<sup>c</sup>, Anette Löfstrand<sup>a</sup>, Anders Kvennefors<sup>a</sup>, Dominique Mailly<sup>b</sup>, Christian David<sup>c</sup>, Ivan Maximov<sup>a,\*</sup>

<sup>a</sup> Lund Nano Lab, Division of Solid State Physics and NanoLund, Lund University, Box 118, SE-221 00 Lund, Sweden

<sup>b</sup> Centre de Nanosciences et de Nanotechnologies (C2N) CNRS, Université Paris-Sud, Université Paris-Saclay, route de Nozay, 91460 Marcoussis, France

<sup>c</sup> Paul Scherrer Institut, 5232 Villigen-PSI, Switzerland

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## ABSTRACT

Single-digit nanometer patterning by nanoimprint lithography is a challenging task, which requires optimum stamp fabrication technique. In the current work, we present different strategies for technology of hard master stamps to make intermediate working stamps with sub-10 nm features. Methods of both negative and positive master stamps fabrication, based on EBL, RIE and ALD are described and compared. A single-step copying of negative master stamps using a polymer material is a preferred strategy to reach the ultra high-resolution. Lines as small as 5.6 nm are demonstrated in a resist using a combined thermal and UV-imprint with OrmoStamp material as a working stamp.

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## 1. Introduction

Methods of single-digit nanofabrication, i.e. the technology for fabricating nanostructures with typical feature sizes below 10 nm, represent a very rapidly developing field of nanotechnology, both for fundamental research and for industrial applications. They include different high-resolution lithographic methods and can be classified in two inherently different strategies: top-down and bottom-up techniques. The first, top-down approach, is well-developed, and represented by such methods as electron beam lithography (EBL) [1], focussed ion beams (FIB) [2], scanning probe writing [3] and recently nanoimprint lithography (NIL) [4]. The second, bottom-up approach, is based on self-organisation, and includes phenomena such as formation of regular structures in Block-Copolymer (BCP) films after vapour annealing [5], or epitaxial growth of III-V nanowires [6]. In many cases, the bottom-up methods allow formation of highly regular, sub-10 nm structures over sufficiently large areas, but their fabrication technology has to be improved to achieve sufficient reliability for use in modern technologic applications.

The top-down lithographic methods are well-established and relatively inexpensive and are typically used in academic research laboratories with a limited budget. All of them are capable of providing writing resolution of below 10 nm in resist, although with different throughput

and flexibility. EBL is undoubtedly the most popular nanolithographic method in many laboratories due to its flexibility and high resolution. However, it is generally not suitable for mass-fabrication due to its slow serial writing speed. Focused ion beams can be used for both for resist exposure as EBL, but also for ion beam milling and for ion-beam induced deposition by a controlled dissociation of suitable precursors, e.g. metalorganics or metal carbonyls. The FIB methods suffer from the same low throughput as electron beam lithography, but have the advantage of using resist-free processes to make nanostructures by a high-resolution deposition even on 3D-surfaces. Electrons can also be used for the beam-induced deposition with sub-10 nm resolution [7], although with less efficiency. Scanning probes, both Atomic Force Microscopes (AFM) and Scanning Tunnelling Microscopes (STM), being the tools with atomic resolution, can also do the single-digit nanofabrication [3], although at the expense of tremendous amount of time, required for making any reasonable pattern.

Nanoimprint lithography, proposed by S. Chou in 1995 [4] and developed by many groups world-wide since then [8,9,10,11], is a technique with sub-nm resolution and very high throughput due to a parallel printing process. A suitable resist is patterned in nanoimprint by a mechanical contact with a working stamp thus eliminating the diffraction limitations of UV-lithography. This replication process results in an exact, inverted copy of the stamp and can be realised by a thermal-(T-NIL), UV-imprint process (UV-NIL) or their combination. It is clear that the ultimate resolution of the NIL is determined by the stamp features and its quality, thus the technology regarding the fabrication,

\* Corresponding author.

E-mail address: [ivan.maximov@ftf.lth.se](mailto:ivan.maximov@ftf.lth.se) (I. Maximov).

handling and application of the nanoimprint stamp is a critical issue in NIL. Fabrication of nanoimprint stamps originally realised by EBL and reactive ion etching [4,8] using Si or SiO<sub>2</sub> as stamp material. However, soft stamps, made of PDMS, OrmoStamp or other polymeric materials, become very popular due to their low cost, reliability, mechanical flexibility and high resolution [11,12].

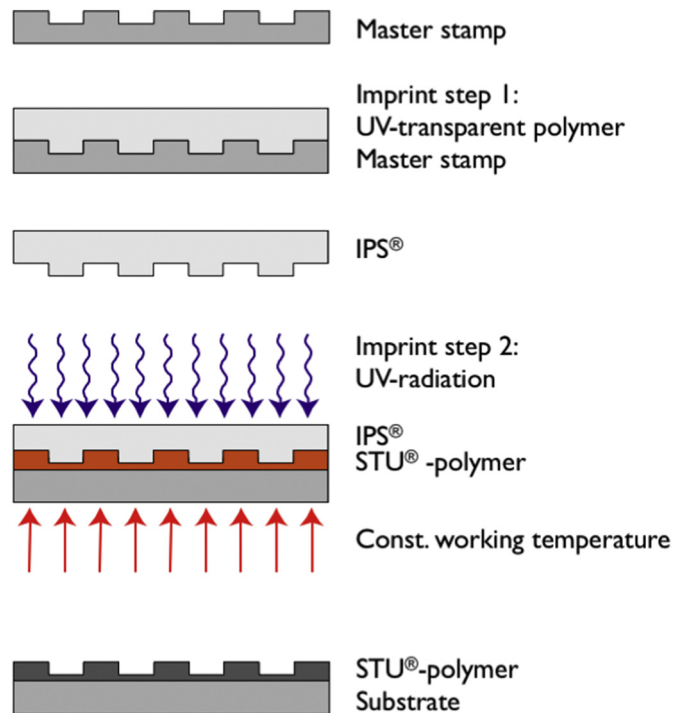
In the present work, which is performed within the framework of the EU-project NFFA-Europe (Nanoscience Foundries and Fine Analysis – Europe), we evaluated different methods of NIL stamp fabrication to realise sub-10 nm structures by nanoimprint lithography. The results of this investigation will be used in Transnational Access program within the NFFA-Europe project to provide access to ultra high-resolution nanoimprint lithography.

## 2. Experimental methods

The experiments, described in the present work, are the result of joint collaboration efforts between four research laboratories in Europe: PSI (Switzerland), C2N (France) and Lund University (Sweden). As part of the Joint Research Activity (JRA) of the NFFA-Europe project, the partners worked together to investigate the most efficient methods of fabrication of nanoimprint stamps with ultra-high resolution. All nanoimprint experiments were performed at Lund University, while the other laboratories contributed to development of NIL stamp fabrication technology and evaluation of the results.

### 2.1. Nanoimprint experiments

All nanoimprint tests have been performed using a modified version of the nanoimprint process based on a standard IPS-STU (Intermediate Polymer Stamp – Simultaneous Thermal and UV-imprint) technology proposed by Obducat AB [13], Fig. 1. The basis of the IPS-STU technology is a single-use UV-transparent polymer stamp (IPS) replicated from a Ni or Si master stamp. The IPS is used later to transfer the stamp pattern into a suitable resist on substrate surface. As a result, the expensive master stamp is never exposed to the processed samples, which extends its



**Fig. 1.** The basic principle of Obducat's IPS-STU technology [13]. In the present work we used the Si master stamps made by EBL and RIE instead of electroplated Ni masters. Reprinted with permission from Obducat AB.

life-time. The nanoimprint is typically performed at elevated temperature and UV-illumination (STU-process).

This optimised IPS-STU process is used by the Lund University group for formation of arrays of Au dots with sizes 100–200 nm for metalorganic vapour phase epitaxy (MOVPE) growth of III–V nanowires (NW) on 2" substrates [14]. The nanoimprint is used to pattern a double-layer resist system, TU7/LOR (Obducat AB, Sweden and MicroChem Corp, USA), to create holes with a negative slope for Au lift-off. One of the advantages of this process is low defect density of the single-use IPS and good mechanical flexibility of the IPS material, which can compensate some imperfections on the substrate, for example, waviness and presence of particles. At the same time, the replication of the IPS from the electroplated Ni master stamp, which take place at 160 °C pressure of 50 bar, may result in bending of the IPS features during the separation stage, especially at the edges of the stamp. Also, for the stamp features (pillars)  $\leq 100$  nm with a large separation  $> 1$   $\mu\text{m}$ , mechanical stability of the pillars becomes insufficient, leading to elongated holes in the imprinted resist due to bending of the pillars during the imprint process. Although some elongation of the resulting structures may not be critical for the NW growth, as the Au dots melt during the annealing step in MOVPE, bending of the IPS features is not acceptable for sub-10 nm features.

The above-mentioned limitations of the conventional IPS-STU process lead us to development of an alternative NIL process based on OrmoStamp (Micro Resist Technology GmbH, Germany), instead of a standard IPS polymer (Obducat AB, Sweden). The OrmoStamp organic-inorganic hybrid polymer has demonstrated sub-50 nm resolution in thermal and UV-nanoimprint lithography [15], so in the current work we used EBL-defined Si stamp as a master and OrmoStamp polymer to make the IPS. Replication of the OrmoStamp was performed in a conventional mask aligner Karl Süss MJB4 (SUSS MicroTec, Germany) by disposing a drop of the polymer on top of the Si master stamp (see part 2.2. for fabrication details), squeezing it and exposing by 365 nm UV-light with an intensity of 20 mW/cm<sup>2</sup> for 120 s. The minimum UV-exposure dose required for the crosslinking of resist is 1000 mJ/cm<sup>2</sup>, as stated by the manufacturer, but we used 2400 mJ/cm<sup>2</sup> to make sure the resist is cross-linked completely. After the cross-linking procedure, the OrmoStamp film has been heated in a convection oven for 30 min at 130 °C. Finally, the surface of the OrmoStamp IPS has been activated for 15 s with a remote oxygen plasma prior to deposition of a perfluorodecyltrichlorosilane (FDTS) monolayer to provide the required anti-sticking treatment. These process steps have been performed in an atomic layer deposition system (ALD) Fiji F200 (Cambridge NanoTech, USA). The contact angle on the substrate surface, measured after this treatment was in the range of 109–110°.

The imprint experiments using the OrmoStamp IPS have been performed in nanoimprinter Eitre 6" (Obducat AB, Sweden). First, spin-coating process of resist TU7 was optimised to minimize thickness of residue layer after imprint. The nanoimprint process was performed in temperature range of 65–75 °C under pressure of 10–20 bar. During the imprint, the TU7 resist was crosslinked by exposure to UV light (365 nm wavelength with intensity of 40 mW/cm<sup>2</sup>) for 20 s.

Resist residues were removed in an oxygen plasma (descum process) using a table-top reactive ion etching (RIE) system (Sirius T2, Trion, USA) at operating pressure of 150 mTorr and RF power of 50 W. The descum time depends on residue thickness and can vary from 5 to 10 s. The resist mask patterned by the nanoimprint and descum process can be used for pattern transfer into the substrate using e.g. reactive ion etching.

### 2.2. Technology of Si-master stamp fabrication

EBL with high resolution was chosen as the main method to define features on the master stamp with typical sizes of the order of 10 nm or below. As an alternative, structures made using BCP and reactive

ion etching, were also considered. To achieve high resolution in the nanoimprint process, the intermediate polymer stamp must be of positive polarity, i.e. to have pillars or other features on the stamp surface. In this case the amount of resist displaced during the imprint will be minimized, making it easier to copy <10 nm structures. This implies that the Si master stamp must be made with a negative polarity, having holes or other structures inside the Si substrates. Technology of the negative stamps with sub-10 nm features is a challenging task, because it includes high-precision reactive ion etching or other material removal techniques. Positive master stamps can be used, too, using a double replication of IPS. Experimental details of both approaches are described below.

### 2.2.1. Fabrication of negative master stamps

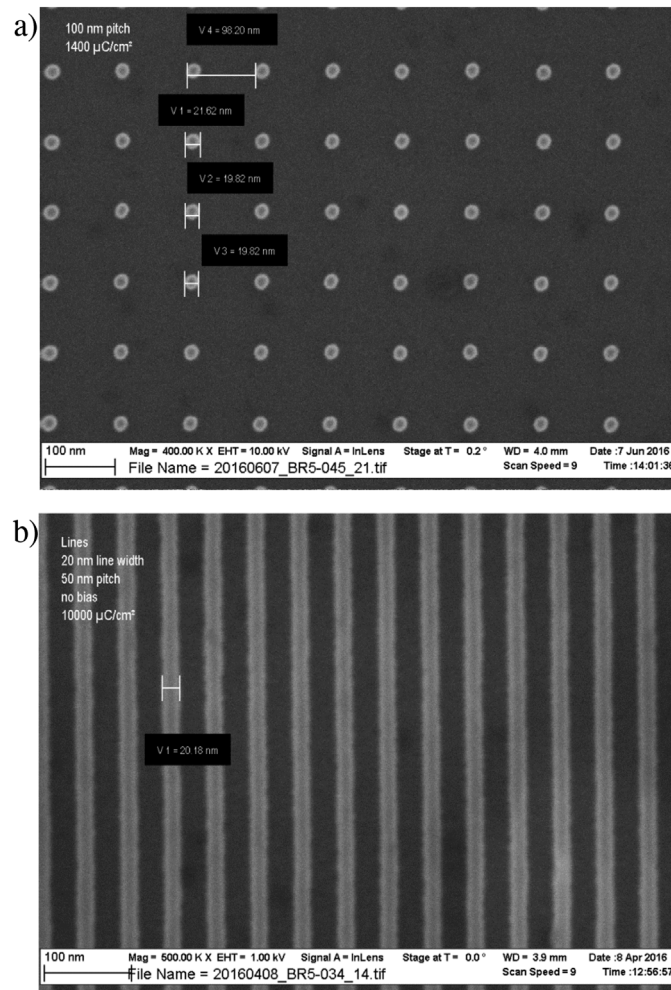
The main purpose to fabricate the negative stamp was to test OrmoStamp as the alternative IPS material in the STU-imprint process. To make negative Si master stamps a Voyager EBL system (Raith GmbH, Germany), was used with the electron beam energy of 50 keV and with a beam current of 2.2 nA. A layer of 250 nm thick positive tone e-beam resist AR-P 6200 (9% in anisole) was spin-coated onto a two-inch Si wafer and baked at 180 °C for 2 min. The exposed pattern included a number of test elements, e.g. 1000, 200, 100 and 50 nm wide lines and hexagonal array of holes with diameter of 80–90 nm and pitch of 1 µm. After the exposure, the resist was developed for 60 s in n-amylacetate with subsequent rinsing in IPA for 30 s and blowing dry with nitrogen. The reactive ion etching of Si substrate was

performed using a parallel plate RIE system (NEXTAL NE100, France) at C2N laboratory, using a mixture of SF<sub>6</sub> and CHF<sub>3</sub> with a 2:5 ratio at a working pressure of 5 mTorr and RF power of 15 W with AR-P 6200 resist as the etch mask. After the RIE process, the samples were sent to Lund University, where resist residues were stripped in NMP (Remover S-1165) and in oxygen plasma. Afterwards, the Si-master stamp was treated in the ALD tool Fiji F200 to deposit 0.4 nm thick Al<sub>2</sub>O<sub>3</sub> layer followed by deposition of FDTS monolayer for the anti-sticking purposes. This procedure results in contact angles between 113 and 114°.

### 2.2.2. Fabrication of positive master stamps

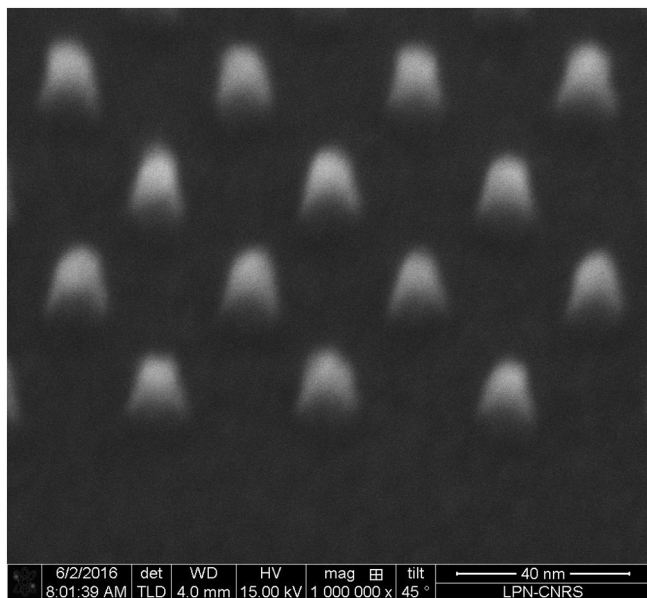
Two-inch Si master stamps of positive polarity have been patterned using 100 keV EBL tools at PSI, Switzerland and C2N, France. In both approaches, negative tone resist HSQ was used, which converts to SiO<sub>x</sub> after the EBL exposure and development. The PSI group applied the HSQ resist to make SiO<sub>x</sub> features on the Si surface (Method A), while the C2N group used the SiO<sub>x</sub> masks to etch Si substrate (Method B). In Method A, a 30 nm-thick HSQ resist was spin-coated on a 2" Si wafer and exposed with 100 keV electron beam (Vistec EBPG 5000 Plus, Raith GmbH, Germany) with a current of 500 pA to produce arrays of 20 nm wide lines and dots with different pitch. After exposure, the resist was developed in a 1:3 mixture of Microposit 351 developer and water for 5 min, followed by rinsing with water, IPA, and drying with nitrogen. Fig. 2 shows a top-view SEM images of the developed HSQ-structures.

In Method B, the HSQ-exposure was used to make silicon oxide masks for subsequent RIE of Si, Fig. 3 illustrates the final results after



**Fig. 2.** SEM images of silicon oxide lines and dots made by EBL-exposure and development of HSQ resist made at PSI, Switzerland. The 20 nm wide lines were exposed with a pitch of 50 or 100 nm using a dose of 1000 µC/cm². The dots with 20 nm diameter and pitch of 100 nm were exposed with a dose of 1400 µC/cm².





**Fig. 3.** SEM images of Si pillars with diameter of 8 nm and pitch 40 nm made by 100 kV EBL and RIE at C2N, France (Method B). Pillars with diameter up to 20 nm and pitches of 30 and 50 nm were also produced. Resulting pillars had aspect ratio of  $\approx 2$  and a conical shape.

the EBL exposure, development and RIE. The HSQ resist was spin-coated at 2500 rpm and exposed by 100 keV electrons with beam current of 2.5 nA. After the EBL exposure, the resist was developed for 20 s in 1:3 AZ400K/water developer and baked for 15 min at 430 °C to convert it to a stable  $\text{SiO}_x$  etch mask. The Si substrate was etched in the reactive ion etching system NEXTAL NE100 under the conditions described in 2.2.1. This process resulted in pillars between 8 and 20 nm in diameter, pitch of 40 nm with an aspect ratio of  $\approx 2$  and somewhat conical shape.

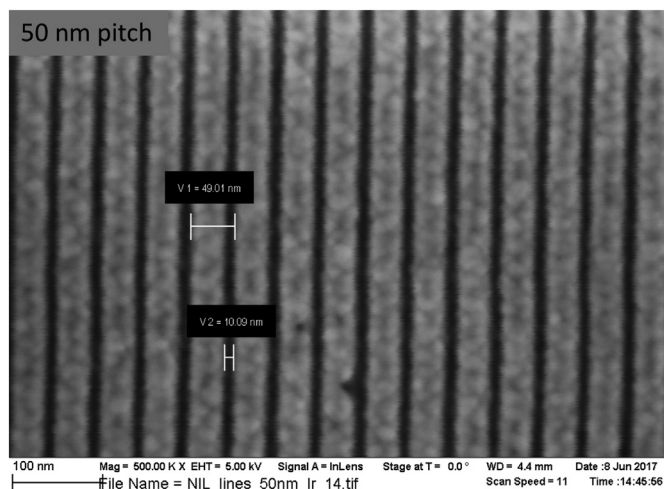
#### 2.2.3. Master stamps with a gap reduction technique

To reduce the master stamp features further, the HSQ-based structures, denoted as Method A were used in combination with ALD of Ir layer, which allowed a controlled reduction of gaps between the HSQ lines. This stamp was designed as a set of lines with different pitch of 50, 100 and 200 nm and a varied EBL exposure dose, in the range of 4000–13,750  $\mu\text{C}/\text{cm}^2$ , which varied the gap between the lines. The HSQ film (Dow Corning, XR 1541-004) was spin-coated to a thickness of 60 nm and exposed in Vistec EBPG 5000 Plus EBL. After the exposure, the resist was developed in a mixture Microposit 351 developer: deionised water (1:3) for 5 min. ALD of a 10 nm Ir layer (plasma-enhanced ALD in a Picosun ALD system at 370 °C, 275 cycles) allowed the reduction of the gaps between the lines to about 10 nm, Fig. 4.

### 3. Results and discussion

#### 3.1. Polymer stamps from the negative masters

As mentioned earlier in 2.1, stamp features below 100 nm in a standard IPS material are not stable enough for a reliable nanoimprint replication into the resist layer due to insufficient mechanical stability, which often leads to bending of the features. In the current experiments, we used the OrmoStamp material to make replicas of the Si negative master stamps with features as small as 50 nm. SEM inspection of the produced OrmoStamp copies indicated no bending of the stamp features after separation of the IPS and the stamp. The OrmoStamp IPS has been used for a RIE pattern transfer into  $\text{SiO}_2/\text{Si}$  substrate using a modified STU-process in single-layer 150 nm thick TU7, instead of a double-layer TU7/LOR resist system used for a lift-off application. The residual resist layer was removed by an oxygen plasma ashing followed by a RIE in  $\text{CHF}_3/\text{CF}_4$  gas mixture to a depth of 66 nm. The SEM images of



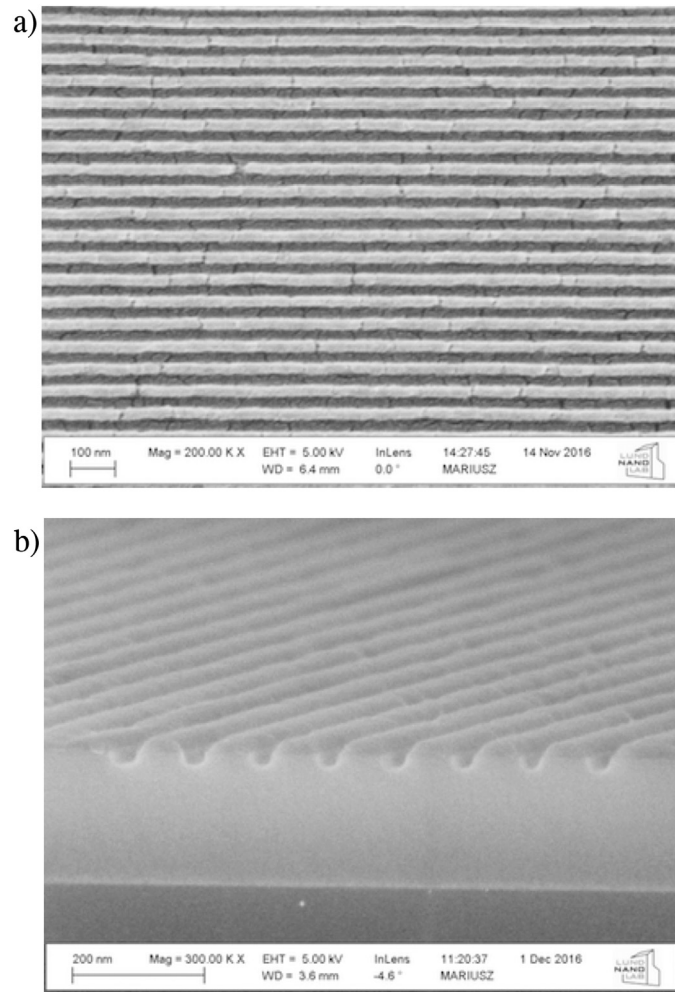
**Fig. 4.** SEM image of the area with 50 nm pitch. The HSQ-lines were exposed with different dose (4250–7500  $\mu\text{C}/\text{cm}^2$ ) to control the line width and thus the distance between the lines. The gaps between the HSQ-lines (dark lines) can be made smaller than 10 nm after ALD of Ir, at the actual image is it about 10 nm wide.

the imprinted and etched structures showed very good pattern transfer even of the smallest features of 50 nm. A single-step replication process to make the OrmoStamp IPS was found to be an advantage compared to a two-step process from the positive master stamps, see 3.2.

#### 3.2. Polymer stamps from the positive masters

The master stamps were made with features as small as 8 nm, but due to their design, replication of the IPS requires a two-step process: in the present work, we explored a OrmoStamp to OrmoStamp replication technique to fabricate a working positive IPS. Positive master stamps made using Methods A and B were used in the current experiments. As described earlier in 2.2.2, the Method A results in positive stamps based on Si substrates with  $\text{SiO}_x$  lines and pillars made by EBL exposure of the HSQ-resist. During the replication of OrmoStamp from such masters, some of the sub-20 nm pillars were found to peel-off during the separation step. The line structures were more stable, compared to pillars, which may be explained by the insufficient adhesion of the  $\text{SiO}_x$  to the Si substrate. SEM inspection of the Si masters after the replication process showed broken  $\text{SiO}_x$  pillars with length equal to the height of the original structures, confirming that the pillars were cut at the bottom. Fig. 5 shows an example of the first copy of OrmoStamp with  $\approx 20$  nm wide line arrays. Some defects, such as discontinued lines, are visible, but they are mainly as a result of thin Pd-Pt metal cracking in the 2 nm thick Pd-Pt layer which was deposited to avoid charging effects in the SEM.

The second copy of the OrmoStamp was made using a procedure described in 2.1, but instead of Si-master stamp a first copy of OrmoStamp was used. Inspection of the second copy of the stamp indicated somewhat higher level of defects as compared to the first copy, however, the second copy was used for pattern transfer into a  $\text{SiO}_2$  layer with  $\approx 20$  nm resolution. The nanoimprint has been performed in a 52 nm-thick TU7 resist spin-coated onto the thermally oxidised Si wafer using imprint temperature of 65 °C and pressure of 20 Bar. During imprint the resist was exposed to UV-light for 20 s. After separation of the IPS stamp from the substrate, the TU7 resist residues were ashed in an oxygen plasma for 5 s and afterwards the  $\text{SiO}_2$  substrate was etched to a depth of 20 nm in  $\text{CHF}_3/\text{CF}_4$  plasma at pressure of 50 mTorr for 60 s. The cross-section of the etched sample, shows a successful pattern transfer from a second copy of the OrmoStamp into the silicon oxide layer with a 20 nm resolution, Fig. 5. The demonstrated aspect ratio of the etched structures is about 1, but it can be improved by optimizing the etching process.



**Fig. 5.** Above: the SEM top view of the first copy of IPS OrmoStamp stamp after replication of a master stamp made by Method A. The  $\approx 20$  nm wide lines are successfully copied into the polymer, although some defects, such as line discontinuities, are visible. The second OrmoStamp copy was used for NIL pattern transfer. Below: a cross-section of etched silicon dioxide using imprinted TU7 resist as an etch mask (right). The width and depth of the etched lines is about 20 nm.

The positive Si master stamps have been made using Method B, see Section 2.2.2. for details, but the second copies of the OrmoStamp resulted in too shallow structures, which were difficult to use for nanoimprint experiments. There were no problems with the broken structures after the stamp replication, as they were made by RIE, but the slightly conical shape of the pillars, compare Fig. 3, was not optimal for the nanoimprint.

### 3.3. Polymer stamps from masters with reduced gaps

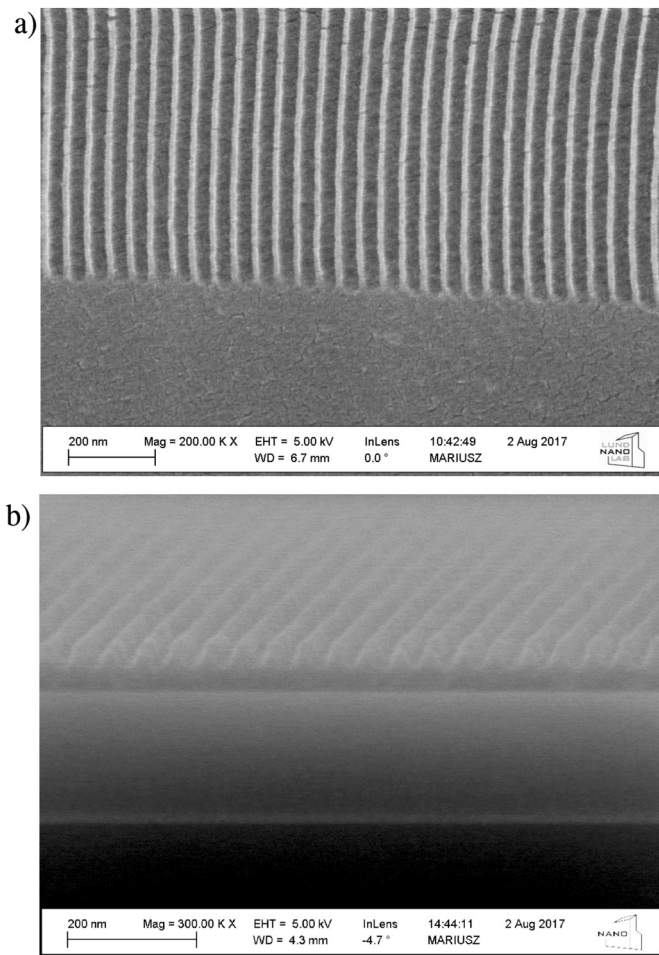
Combination of EBL-patterning using the HSQ-resist and ALD of Ir layer, described in Section 2.2.3, gives a suitable method to fabricate NIL stamps with sub-10 nm features, as shown in Fig. 4. Apart from reducing the gap by a controlled Ir deposition, the uniform coverage of the Si/SiO<sub>x</sub> surface by the Ir layer is expected to make the master stamp more stable against mechanical effects during the replication of IPS. Here we demonstrated a successful replication of the OrmoStamp IPS from the master and nanoimprint of sub-10 nm features in the TU7 resist. It should be noted that a single-step replication of IPS was used, which results in a negative IPS, however, sub-10 nm gaps between the SiO<sub>x</sub> lines were replicated, not the lines themselves, so effectively, positive stamp structures were used in this experiment. At the same time, the gaps were converted into ridges in a recessed area of the polymer, which is not optimal for the resist flow during nanoimprint. Fig. 6 illustrates this by showing the OrmoStamp copy of the Si master. The

linewidth of the ridges in the polymer is below 10 nm, as measured by SEM.

The OrmoStamp IPS shown in Fig. 6 was used to imprint sub-10 nm lines into a TU7 resist, using the imprint temperature of 75 °C, pressure of 10 bar and alternating UV-illumination. The lower part of Fig. 6 illustrates the successful imprint of sub-10 nm lines into a 60 nm thick resist by showing a cross-section of the imprinted sample. Top-view SEM inspections (not shown here) indicate imprints with line widths as small as 5.6 nm. The image on the cross-section indicates a relatively thick imprint residual layer, about 10 nm. In order to realise a pattern transfer into the underlying substrate, this layer must be removed, preferably without widening of the sub-10 nm trenches, which is a challenging task.

## 4. Conclusions

To achieve sub-10 nm resolution in nanoimprint lithography, the NIL stamp fabrication technique must be optimised. In the present work, we investigated different EBL-based methods of Si master stamps to produce working intermediate polymer stamps (IPS) with high resolution for simultaneous thermal and UV-nanoimprint process. The preferred working stamp fabrication technology include Si negative master stamp made by EBL and RIE and its replication into IPS using the OrmoStamp hybrid polymer. The master stamp feature may be further reduced by a controllable deposition of Ir layer by ALD. We have demonstrated successful pattern transfer of 20 nm features in SiO<sub>2</sub> layer by



**Fig. 6.** Above: SEM image of the OrmoStamp replica of the Si master stamp at the area with 50 nm pitch. The polymer copy has been coated with 2 nm thick Pd-Pt film to prevent charging effects during the SEM inspection. The measured line width is 11.3 nm, indicating the real width of the ridges < 10 nm. Below: a cross-section of the imprinted TU7 resist showing imprinted sub-10 nm lines. Relatively thick ( $\approx 10$  nm) residual layer must be removed prior to pattern transfer into the  $\text{SiO}_2$ .

imprint with the OrmoStamp, followed by RIE and replication of the IPS features as small as 5.6 nm in a TU7 resist.

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