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'2020



«EUROPEAN SCIENCE»
BOOK 3. PART 3

SWorld
Germany



Bezusov A.T., Choporov O.N., Danylina H.V., Guido M., Kukhar V.V. et al.

**INTELLEKTUELLES KAPITAL - DIE GRUNDLAGE FÜR
INNOVATIVE ENTWICKLUNG**
INNOVATIVE TECHNIK UND TECHNOLOGIE, INFORMATIK
*INTELLECTUAL CAPITAL IS THE FOUNDATION OF
INNOVATIVE DEVELOPMENT*
INNOVATIVE ENGINEERING AND TECHNOLOGY, INFORMATICS

*Monographic series «European Science»
Book 3. Part 3.*

*In internationalen wissenschaftlich-geometrischen Datenbanken enthalten
Included in International scientometric databases*

MONOGRAPHIE
MONOGRAPH

*ScientificWorld-Net Akhat AV
Karlsruhe 2020*

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Intellektuelles Kapital - die grundlage für innovative entwicklung: innovative technik und technologie, informatik. Monografische Reihe «Europäische Wissenschaft». Buch 3. Teil 3. 2020.

Intellectual capital is the foundation of innovative development: innovative engineering and technology, informatics. Monographic series «European Science». Book 3. Part 3. 2020.

ISBN 978-3-949059-04-9

DOI: 10.21893/2709-2313.2020-03-03

Published by:

ScientificWorld-NetAkhatAV

Lußstr. 13

76227 Karlsruhe, Germany

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**KAPITEL 4 / CHAPTER 4.****OPTIMIZATION OF TWO-LAYER RESISTS FOR LASER LITHOGRAPHY ON SUBSTRATES REQUIRED FOR WIDE APPLICATION IN MICROWAVE SENSOR TECHNOLOGY**

**ОПТИМИЗАЦИЯ ДВУХСЛОЙНЫХ РЕЗИСТОВ ДЛЯ ЛАЗЕРНОЙ ЛИТОГРАФИИ НА ПОДЛОЖКАХ, НЕОБХОДИМЫХ ДЛЯ ШИРОКОГО ПРИМЕНЕНИЯ В СЕНСОРНОЙ СВЧ ТЕХНИКЕ
ОПТИМІЗАЦІЯ ДВОШАРОВИХ РЕЗИСТІВ ДЛЯ ЛАЗЕРНОЇ ЛІТОГРАФІЇ НА ПІДКЛАДКАХ, НЕОБХІДНИХ ДЛЯ ШИРОКОГО ЗАСТОСУВАННЯ В СЕНСОРНІЙ СВЧ ТЕХНІЦІ**

DOI: 10.21893/2709-2313.2020-03-03-012**Introduction**

We report on the optimization of laser lithography on various substrates relevant for ultra-high frequency on-chip experiments. We're aiming at fabricating microstructured photoconductive switches for transport experiments with sub-picosecond time resolution. Double layer resist systems for optimal lift-off processing of metal and semiconductor thin films are used. The double layer consist of LOR 3B as under layer and ma-P 1205 as top layer. Substrates are sapphire, i-GaAs, i-Si, SiO₂ on i-Si, and SiO₂ on p-doped Si. The target parameters for the optimization are the exposure dose and the edge roughness of microstructured elements. The latter is important to obtain high bandwidth striplines, microstrips, or coplanar waveguides.

The study of electronic transport properties of nanoscale systems on ultrashort time scales is a hot topic in solid state physics [1, 2]. There are several reasons for interest in such systems. First they comprise a variety of new physical phenomena [3-5]. Another reason is the possibility to manufacture a new generation of ultra-fast optoelectronic devices based on nanoscale systems [6, 7]. These devices include high-bandwidth striplines, microstrips, or coplanar waveguides [9, 10], as well as optical switches, such as photoconductive switches, also called Auston switches [8]. Also an interesting brunch of research in the field of terahertz are nano-devices with the properties of negative differential resistance [11, 12]. For the realization of ultra-fast electronic micro- and nanostructures and for nano-devices with negative differential resistance, the optimization of technological manufacturing methods as deposition, etching, and lift-off processes is very important. The purpose of this work is to improve the quality of resist masks used for lift-off processing under laboratory conditions to produce experimental nanostructures with dimensions down to 500 to 1000 nm, with controlled geometry and controlled boundary quality between different elements. In order to get a clean and reproducible lift-off, double-layer resists are utilized and adapted for our purposes. By using two different types of resist, one can precisely pattern the top photoresist and form an undercut in the photo insensitive bottom resist layer, resulting in a reliable lift-off profile. One should use resists with different solvents to avoid mixing of chemicals so that consistent results



across all sample batches are realized. Microchem PMGI/LOR and LOL [13] series resists work well as underlayer for i-line and broadband lithography, where the undercut rate is well controlled with standard developers [14, 15]. The uppermost layer can be a positive or negative photoresist and should typically not exceed one third of the total thickness. Especially positive photoresists as top layers are well suited for clean and repeatable lift-off processing [15]. In the present work resist of the ma-P 1200 series from microresist technology is used [16]. Optimizing the process requires an exposure series to determine the optimal exposure dose. This depends on the photoresist and the optical properties of the substrate and is described *inter alia* by the contrast curve [17]. The contrast of a photoresist is a measure of the sharpness of the transition from exposed to unexposed areas after the development process. Because the light spot is not perfectly binary, there will be grey areas where the resist is exposed by a fraction of the maximum dose. The higher the contrast of a photoresist, the less these grey areas are affected by the development process and the better the resolution of the resist system is. In this paper another parameter for the optimization of the resist process is considered, namely the edge roughness R . We find that this roughness critically depends on the exposure dose. The manufacturing process and the results of the optimization are described in detail in the following.

4.1. Experimental

The fabrication process of resist masks discussed here shall be used to manufacture photoconductive switches on various substrate materials that are relevant for ultrafast transport experiments. We study the properties of the double layer resist system for the following substrates: (a) sapphire from Pi-KEM Ltd., (b) intrinsic (low doped) gallium arsenide from Freiberger Compound Materials (i-GaAs), (c) intrinsic (low doped) silicon from Si-MAT GmbH (i-Si), (d) low-doped silicon with a thermally grown layer of silicon dioxide with a thickness 250 nm from Si-MAT GmbH (SiO_2 on Si), and (e) highly p-doped silicon with a thermally grown layer of silicon dioxide with thickness 285 nm from Nova Technology Inc. (SiO_2 on p-doped Si). Schematic images of all substrates and the double layer resist system with detailed parameters and geometries are shown in Fig. 1.

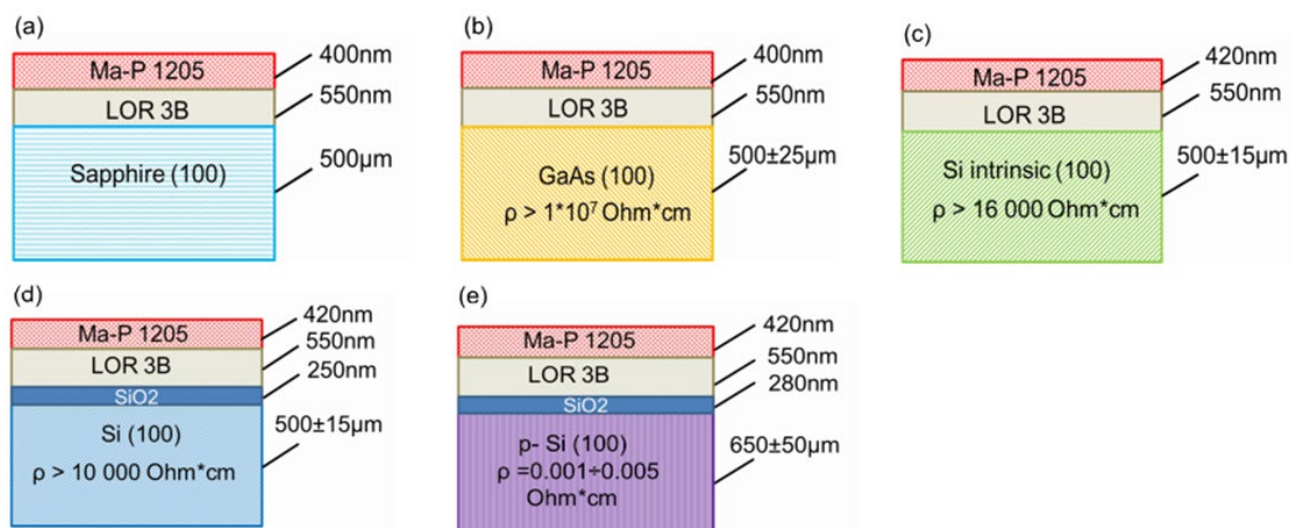


Figure1. Schematic representation of the substrates and double layer resist systems used in this study: (a) Sapphire, (b) i-GaAs, (c) i-Si, (d) SiO₂ on i-Si, and (e) SiO₂ on p-doped Si. Layer thicknesses and specific resistivity are denoted in this figure

4.2. Technology

4.2.1. Wet chemistry process and photoresist coating

The resists LOR 3B [13] and ma-P 1205 [16] are used, as this combination provides the required thickness, the undercut that is important for the later lift-off processing, as well as the sensitivity for the near UV (375 nm) laser beam of the lithography machine. All substrate types are cut into square pieces of 10 x 10 mm² using a laser prototyper LPKF ProtoLaser U3 [18], with the exception of the gallium arsenide wafers, that are cut with a diamond embedded in a custom build mechanical system for cutting wafers. After cutting, the substrates are cleaned with a standard method using acetone and isopropanol in an ultrasonic bath and washed in ultra-pure de-ionized water with a specific resistivity of 18.2 MΩ*cm [19] and dried with dry nitrogen with a purity of 99.999 %. The samples are heated to a temperature of 100 °C for 10 minutes on a hot plate to dehydrate the surface. Photoresists are applied by spin coating with a SPN150 Spin Coater [20]. First, an LOR 3B layer is spin coated on the substrate with 2000 rpm for 45 seconds. Then, the photoresist is baked on a hot plate at a temperature of 180°C for 4 minutes at an air temperature in the cleanroom at +22 °C ambient temperature and a humidity of 35 %. After slow cooling of the samples, a second photoresist of type ma-P 1205 is added by spincoating with 2000 rpm for 30 seconds. Then, the photoresist is baked on a hotplate at a temperature of 100 °C for 30 seconds in air at the abovementioned



temperature of air and humidity in the cleanroom.

4.2.2. Laser lithography

The double-layer resist is exposed in a laser lithography system μ PG 101 from Heidelberg Instruments GmbH [23]. The μ PG 101 uses a direct writing mode in near UV range, namely with a wavelength of 375 nm. To determine the contrast curves we expose arrays of rectangular elements with varying exposure dose. The initial values of the laser power and its intensity are 10 mW and 10 % while using a filter with 16 % transmission. The exposure intensity is increased in steps of 0.5 %. For the gallium arsenide substrate we increased the intensity in steps of 5%. This procedure leads to exposure doses ranging from 3 to 300 mJ/cm². For each intensity five 100 x 20 μ m² rectangles are exposed on the various substrates. All samples are developed in the developer ma-D 331/S [13] for 60 s. Deionized water is used to stop the process. The samples are rinsed in propan-2-ol [24] and dried with dry nitrogen. The developing time is kept constant at 60 s.

4.2.3. Optical inspection and AFM measurements

The lateral quality of the double-layer resist on the substrate is checked with optical microscopy (Leica DM 3000) [21]. Then the roughness of the photoresist and the substrate as well as the thickness of each layer of the photoresist are measured by atomic-force microscopy (AFM) (Bruker, Icon Dimension) [22]. AFM measurements are performed with high resolution, low power, and asymmetric shape FESPA probes. For this type of probes we use the air tapping method in the soft tapping mode, which is most adequate for soft materials. Scanning is carried out both on the photoresist surface and on the surface of the substrate. The scanning region is chosen to be 50 \times 50 μ m². The AFM measurements show that the thickness of LOR 3B ranges from 500 to 550 nm, while the thickness of ma-P 1205 ranges from 450 to 490 nm. Depending on the properties of the various substrates, the total thickness of the double layer is found to range from 960 to 980 nm. To determine the contrast curves exposed with laser lithography we measured the height of the photoresist at the edge of a microstructure with a length of 50 μ m or more. After characterization, a gold layer with a thickness of 200 nm is vapor deposited. In order to ensure better adhesion to the substrates, a titanium interlayer with a thickness between 6 and 10 nm is inserted. All deposition processes are performed in a vapor deposition system HVB 100 [25]. Finally, the lift-off process is carried out with remover mr-REM 700 [26]. Lift-off times varied depending on the substrate and ranged from 2 to 12 hours.



4.3. Results and discussion

4.3.1. Contrast curves

Figure 2 shows the contrast curve for each substrate for the double layer resist system LOR3B/ma-P 1205. In the contrast curve we plot the ratio of the resist layer remaining after development normalized to the original layer thickness as a function of the logarithmically scaled exposure dose as it is done conventionally [17]. The contrast γ is defined as

$$\gamma = \left(\log \left(\frac{D_c}{D_0} \right) \right)^{-1}, \quad (1)$$

where the so called dose-to-clear D_c is the dose at which after the development no photoresist remains on the substrate, while D_0 is the dose where the photoresist starts to develop.

Figure 2 shows the contrast curves and the concomittant fit functions versus exposure dose D for the five substrates. We obtain normalized thickness curves that follow a linear decay for all substrates. The dose-to-clear D_c has values between 70 and 160 mJ/cm² depending on the substrat material.

The calculated values for the contrast γ , the dose-to-clear D_c , and the dose where the photoresist starts to develop D_0 are listed in Table 1.

Table 1.

Exposure doses and contrast values for the five substrate materials.

Substrate	D_c (mJ/cm ²)	D_0 (mJ/cm ²)	Contrast γ
Al ₂ O ₃	110	41	2.3
i-GaAs	159	48	1.9
i-Si	102	37	2.3
SiO ₂ on i-Si	103	46	2.9
SiO ₂ on p-doped Si	68	35	3.5

The contrast values range from 1.9 to 3.5, which is the expected range for optical resists that typically provide contrast values from 0.4 to 4 [25].

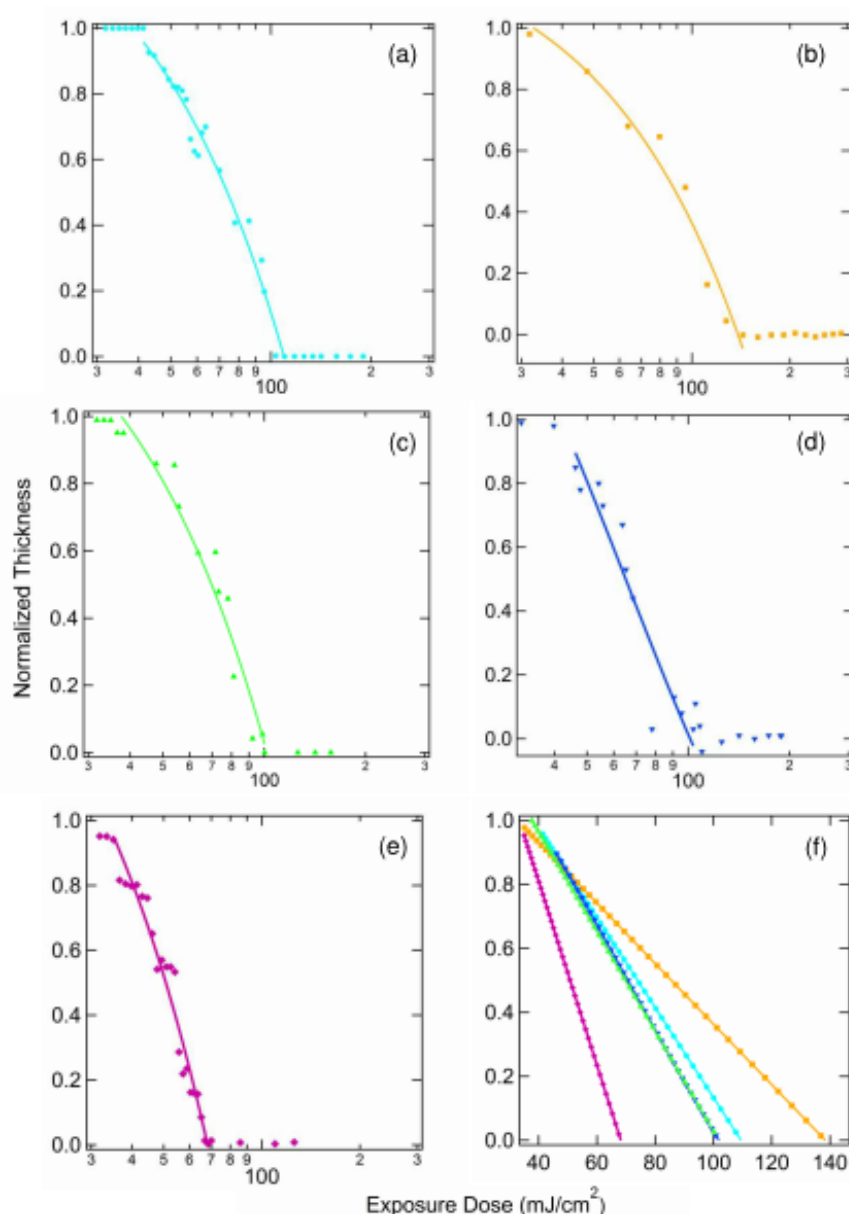


Figure 2. Contrast curves and concomitant fit curves for various substrates. (a) Sapphire, (b) i-GaAs, (c) i-Si, (d) SiO₂ on i-Si, and (e) SiO₂ on p-doped Si, (f) comparison of fit curves

4.3.2. Roughness of double layer resist

The roughness of the double layer resist system as well as of the substrate surfaces are measured with a Bruker Icon Dimension AFM using the roughness statistics software routine from Bruker. Statistics are derived following the standard B46.12 of the American Society of Mechanical Engineers (“Surface Texture: Surface Roughness, Waviness and Lay”) [22]. After plane fitting the data we evaluate the root mean square and the arithmetic mean roughness values R_q and R_a . R_q is here defined as the standard deviation of the measured z_i values within the region of interest



$$R_q = \sqrt{\frac{\sum_{i=1}^N z_i^2}{N}}, \tag{2}$$

where N is the number of points within the region of interest. In our case the region of interest of $50 \times 10 \mu\text{m}^2$ yielded $N \approx 50.000$. R_a is the arithmetic average of the absolute values of the measured z_i values within region interest

$$R_a = \frac{1}{N} \sum_{i=1}^N |z_i|. \tag{3}$$

Calculated roughnesses for the double-layer resist on all substrates for optimal dose are listed in Table 2.

Table 1.

Geometric and arithmetic roughnesses of photoresist and substrate.

Name of substrate	Roughness of photoresist (nm)		Roughness of substrate (nm)	
	R_q	R_a	R_q	R_a
Al ₂ O ₃	7.8	3.9	11.3	6.3
i-GaAs	2.6	2.0	3.2	1.7
i-Si	2.7	1.5	6.4	4.1
SiO ₂ on i-Si	3.9	3.0	7.9	6.4
SiO ₂ on p-doped Si	5.4	2.3	8.6	3.0

The roughness values for all investigated substrates are in the range of 2 to 11 nm. The roughness of the substrates at the exposed sites is 2 to 4 nm larger than the roughness on top of the double-layer resist. After spin coating, soft baking and exposure the best roughness is found for the i-GaAs substrate. During the measurement of the contrast curves, it has been observed that the edge roughness changes strongly. It is reduced for increasing dose and then saturates. To elucidate this more we investigated the edge roughness systematically. The results are presented in the following.

4.3.3. Edge roughness of microstructured double-layer photoresist

Based on the software Igor from WaveMetrics [25] we determine the edge roughness of the microstructured double-layer resist on all five substrates. First, the position x_e of the edge is determined by fitting an error function to each line scanned vertical to the resist edge. After subtracting a linear fit to the edge positions the roughness R_e is calculated by the root mean square of the edge position values. For all samples the edge roughness is calculated for an edge length of at least $50 \mu\text{m}$. All five microstructured elements of varying exposure dose are included in the calculation of

the edge roughness. Edge roughness values R_e , optimal exposure dose values from contrast curves D_c , and optimal exposure dose values from the study of the edge roughness D_e are listed in Table 3 and are shown in Figs. 3 and 4.

Table 3.

Comparison of edge roughness of microstructures, optimal dose from contrast curves, and optimal dose from edge roughness studies on various substrates.

Name of substrate	Edge roughness R_e (nm)	Optimal dose from contrast curve D_c (mJ/cm ²)	Optimal dose from edge roughness D_e (mJ/cm ²)	Contrast γ
Al ₂ O ₃	35.8	110	110	2.3
i-GaAs	10.0	159	159	1.9
i-Si	15.4	102	102	2.3
SiO ₂ on i-Si	20.2	103	110	2.9
SiO ₂ on p-doped Si	24.2	68	78	3.5

The edge roughness is in the range of 10 to 36 nm for the optimum dose D_e depending on the substrate. Figure 3 shows the dependence of the optimal exposure dose D_c on the contrast for the five substrates. From the plot it can be inferred that a high contrast requires a low exposure dose [17].

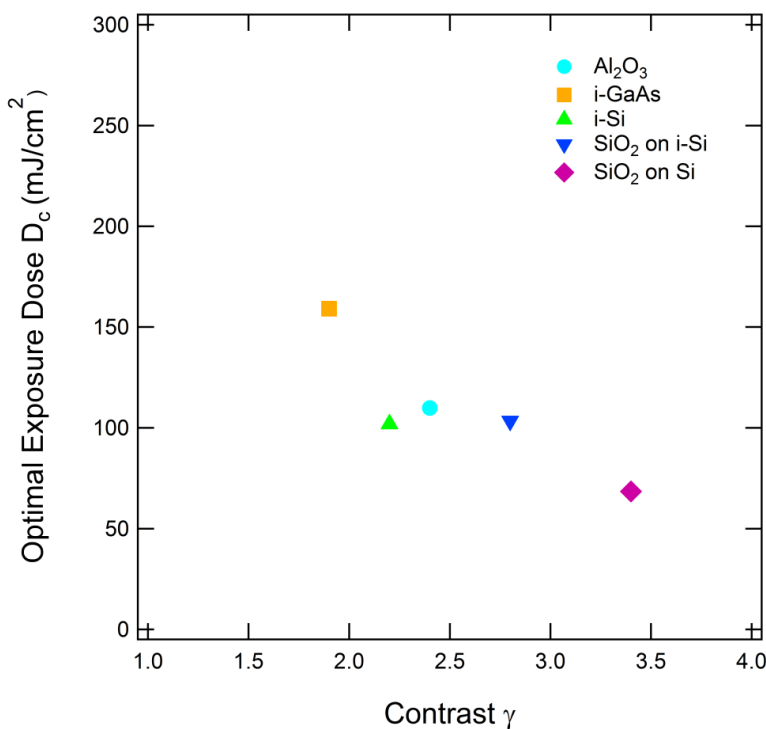


Figure 3. Dependence of the optimal exposure dose on the contrast for the five substrates

Figures 4 (a) and (b) show details of the AFM measurements in two different cases with either non-optimal and optimal dose, respectively. While Fig. 4 (a) shows the AFM image section in the case of low exposure dose yielding a rough edge, Fig. 4 (b) shows the AFM measurement for optimal exposure dose resulting in an optimal edge roughness.

Figure 4 (c) plots the edge roughness for each substrate as a function of exposure dose. Each point corresponds to an AFM measurement of the edge roughness at exposure doses between 60 mJ/cm² and 300 mJ/cm². The graph is plotted on a logarithmic scale to make the large span of edge roughnesses visible. All substrates show a similar behavior: with increasing exposure dose the edge roughness decreases and stabilizes at a characteristic optimal value. Interestingly the determination of the optimal dose from the analysis of the contrast curves and the investigation of the edge roughness lead to almost exactly the same dose values as can be seen in Table 3.

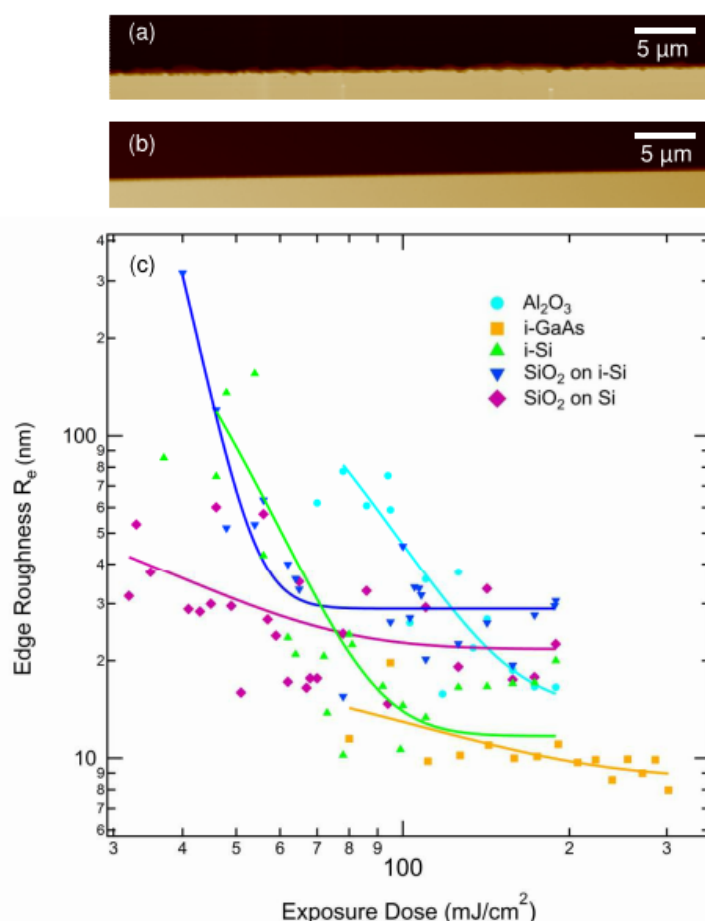


Figure 4. (a) AFM image section in the case of low exposure dose. (b) AFM image section for optimal exposure dose. (c) Measured edge roughness versus exposure dose for all five substrates



Conclusion

We optimized a double layer resist system for lift-off processing. The process is applicable for various substrates such as sapphire, i-GaAs, i-Si, SiO₂ on i-Si and SiO₂ on p-doped Si, which are relevant for ultra-high frequency on-chip experiments. The optimal exposure doses were determined and the contrast curves were generated for five different substrates. We obtain normalized contrast curves that follow an exponential course for all substrates. Depending on the substrate, the optimal exposure dose has a value between 70 and 160 mJ/cm². The contrast values are between 1.9 and 3.4, which corresponds to the expected range for positive optical resists. The edge roughness is an important parameter for optimizing the process. The edge roughness values have been determined at the optimum exposure doses and have been found to range from 10 to 36 nm. The edge roughness versus exposure dose follow an exponential course for all substrates before they stabilize at a characteristic optimal value. The edge roughness values are excellent when considering the wavelength of the UV light used in the laser lithography process.



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