

Investigation of Line Edge Roughness in Current-Controlled Field-Emission Scanning Probe Lithography: Image Reversal vs. Direct-Write

by

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**Investigation of Line Edge Roughness in Current-Controlled
Field-Emission Scanning Probe Lithography: Image Reversal vs.
Direct-Write**

Koç University

Graduate School of Sciences and Engineering

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ABSTRACT

Current-controlled field-emission scanning probe lithography offers unique capabilities to achieve sub-10 nm feature sizes with a high resolution in ambient conditions without any vacuum or special gas requirements. Patterned features can be scanned directly after the exposure using the same exposure setup and tip. With linewidths thus reduced from microns to nanometers, the line edge roughness has recently become even more important, since it does not scale with the decreasing feature size having a direct effect on the critical dimension of the printed features, thereupon, also on the speed and functionality of fabricated devices. For this purpose, lines patterned with a 200-nm nominal pitch through direct-write in scanning probe lithography are compared to those obtained by image reversal. Negative-tone exposure before development (latent image) is also considered thanks to the resolving power of imaging through non-contact AFM mode and used for validation purposes. Images collected are analyzed using edge detection techniques with the average linewidths for image reversal and direct-write found to be 50.01 nm and 26.81 nm, respectively. We report 16.44 nm average line edge roughness for the latent image, 16.78 nm for image reversal and 10.95 nm for direct-write. Presenting a thorough comparison of patterning through image reversal of calixarene molecular glass resist from negative-tone to positive-tone as well as direct-write, this thesis discusses various aspects of the quality of resulting patterns.

ÖZETÇE

Akım kontrollü alan-salımlı tarama probu litografi, ortam koşullarında herhangi bir vakum veya özel gaz gereksinimi olmadan yüksek çözünürlükte 10 nm altındaki şekil boyutlarını elde etmek için benzersiz yetenekler sunar. Modellenen şekiller, şekillendirmeden sonra doğrudan aynı düzenek ve uç kullanılarak taranabilir. Çizgi kalınlığının mikrondan nanometreye düşmesiyle birlikte, çizgi kenarı pürüzlülüğü, azalan şekil boyutuyla ölçeklenmediğinden modellenen şekillerin hız ve işlevselliği üzerinde çok daha fazla önem barındıran bir parametre haline gelmiştir. Bu amaçla, tarama probu litografisinde doğrudan yazma yoluyla 200 nm nominal genişlikte şekillenen çizgiler, görüntü ters çevirme ile elde edilenlerle karşılaştırıldı. Banyo etmeden önce negatif tonlu pozlama (gizli görüntü), temassız atomsal kuvvet mikroskopisi modu aracılığıyla görüntüleme gücü sayesinde deney sonuçlarının doğruluğunu kontrol etme amacıyla kullanıldı. Toplanan görüntülerden kenar algılama teknikleri kullanılarak tersine çevirme ve doğrudan yazma için sırasıyla ortalama 50.01 nm ve 26.81 nm olan çizgi kalınlığı tespit edildi. Gizli görüntü için 16.44 nm , görüntü ters çevirme için 16.78 nm ve doğrudan yazma için 10.95 nm ortalama çizgi kenarı pürüzlülüğü bildirildi. Bu tez çalışması calixarene moleküler cam resisti üzerinde negatif bir tondan pozitif bir tona görüntünün tersine çevrilmesi yoluyla oluşan şekillerin ve doğrudan yazılan şekillerin kapsamlı bir analizinin karşılaştırılması sonucu şekillerin kalitesinin çeşitli yönlerden tartışılmasını sunar.

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NOMENCLATURE

IRDS	International Roadmap For Devices and Systems
EUV	Extreme Ultraviolet
NIL	Nanoimprint Lithography
SPL	Scanning Probe Lithography
FE-SPL	Field Emission Scanning Probe Lithography
EBL	Electron Beam Lithography
CD	Critical Dimension
LER	Line Edge Roughness
LWR	Line Width Roughness
AFM	Atomic Force Microscopy
SEM	Scanning Electron Microscopy
CD-AFM	Critical Dimension Atomic Force Microscopy
CD-SEM	Critical Dimension Scanning Electron Microscopy
HHCF	Height Height Correlation Function
PSD	Power Spectral Density
MIBK	Methyl Isobutyl Ketone
FILM	Fractilia Inverse Linescan Model

Chapter 1

INTRODUCTION

For more than 50 years Moore's law was followed closely while scaling down the feature size [1]. With Moore's law having reached its physical limits, maintaining the miniaturization trends in the single-digit nanometer regime with a high yield and cost-effectiveness became a challenge [2]. According to the International Roadmap for Devices and Systems (IRDS), feature sizes are expected to decrease to less than 5 nm until 2027 [3]. New lithography methods are thus required to overcome the challenges encountered for under-10-nm feature sizes. With extreme ultraviolet (EUV) lithography and nanoimprint lithography (NIL) listed as some of the candidate technologies for further miniaturization, scanning probe lithography (SPL) represents an unconventional, alternative, cost effective and promising solution to pattern features in the single-digit regime [4].

There are a series of SPL technologies based on different working principles, including thermal/thermomechanical, bias induced, oxidation, mechanical and dip-pen SPL [5]. The foremost advantage of SPL is that it generally operates under controlled environmental conditions without any need for vacuum or any special gas decreasing the tool overhead, cost and facility needs. Unlike electron microscopy, the imaging process is nondestructive and does not cause any accidental patterning. SPL tools can be employed to pattern many types of substrates including polymers and ceramics [5].

Field emission SPL (FE-SPL) is a bias induced SPL which employs Fowler-Nordheim electron emission from a very sharp tip [6]. For FE-SPL no complex electronics or

optics are required [7]. Compared to electron-beam lithography, proximity effect is significantly reduced due to ultra-low energy electrons. Although the limited throughput of scanning probe technology can be considered as a limitation for its practical applications, mix-and-match patterning can be a solution in this aspect [8]. Furthermore, SPL can be used for fabricating molds for nanoimprint lithography (NIL) reducing further the limitation of serial processing [9].

After SPL patterning, a solubility change is observed in the exposed area resulting in either positive-tone patterning, if the exposed region becomes soluble, or in negative-tone otherwise [10]. When the resist is exposed, a latent image is observed on the resist before development in negative-tone patterning. Calixarene molecular glass resist can be used as a negative-tone resist in electron-beam lithography (EBL) [11] and as a positive-tone resist in FE-SPL [12]. Although, negative-tone patterning in FE-SPL requires a subsequent wet development step, this is not the case for positive-patterning, where the exposed area is ablated. Furthermore, the tone of the image can be altered by image reversal to benefit from advantages of a particular resist.

Such process variations within the single-digit nanometer regime also give rise to a new set of challenges. The critical dimension (CD) of a feature is the most important parameter with decreasing feature size [13]. In addition to CD, meeting the line edge roughness (LER) and linewidth roughness (LWR) requirements are the other two main challenges [13]. IRDS requires 1.2 nm metal LWR for the 5-nm node as of 2021. However, currently there is no available manufacturing solution to fulfil this roughness requirement posing a serious problem against further scaling of the feature size and improving the performance.

LER for thermal probe maskless lithography was investigated previously through SEM micrographs [14]. However, there are no previous LER reports involving imaging

using the same SPL setup. Similarly, while LER of latent images was studied before in a series of papers [15, 16, 17], there was no previous report on LER for latent images in SPL. Addressing these points missing in the literature, this work investigates LER in FE-SPL for the three cases of i) latent image of negative-tone exposure, ii) image reversal, iii) and direct-write, all imaged using the same SPL equipment in AFM mode.

In the remainder of this thesis, the working principle and advantages of current-controlled field-emission scanning probe lithography are introduced first. This is followed by LER study with fractal analysis. A validation study is then introduced, where our results are compared to those of a commercial software. A description of the experimental procedure including sample preparation and edge detection methodology is provided. The three aforementioned cases will be considered with 30 lines characterized in each case, i.e. latent image, image reversal and direct-write. The work is concluded with LER, LWR, linewidth, pitch values and spatial parameter reported for each case.

Chapter 2

THEORY AND BACKGROUND

2.1 Current Controlled Field-Emission Scanning Probe Lithography

Current-controlled FE-SPL is a tip-based nanofabrication method relying on self-actuating and self-sensing active cantilevers[18]. A scanning probe enables the user to image, align, pattern and inspect a surface at the atomic scale. This versatile nature of the active scanning probes makes them an irreplaceable tool in many nanofabrication, positioning, overlay and characterization applications. AFM images of the patterned features can be obtained directly after exposure using the same tool and cantilever. In the literature, direct fabrication of features smaller than 10 nm on molecular glass resist calixarene was reported using FE-SPL without development[19]. Scaling the feature sizes down to the single-digit region allows one to utilize quantum-based effects such as Coulomb blockade and single-electron tunneling[20].

While patterning, a strong, non-uniform electric field is applied between the tip and a biased sample leading to a current flow of low-energy electrons in between [21]. Low-energy electrons are regulated by a current feedback control in the SPL system and modulated by respective controller parameters. The resist can be either crosslinked or ablated, i.e. directly removed, depending on the applied exposure dose, a combination of speed and current, and applied bias voltage [22]. While low exposure doses result in crosslinking of the resist (negative-tone patterning), higher exposure doses result in direct-write (positive-tone patterning). Similarly, positive-tone patterning can be achieved by image reversal.

The working principle of FE-SPL with two feedback control loops is demonstrated

in Fig. 2.1a. The setup utilizes thermally actuated, piezoresistive cantilevers both for lithography and metrology purposes. Use of such active cantilevers enables higher flexibility, higher speeds and easier integration compared to existing passive cantilever methods [23]. The SPL platform consists of an approach system and a top scanner system, where the cantilever is mounted. The FE-SPL is able to pattern an area of up to $100 \times 100 \text{ mm}^2$ and scan an area of $200 \times 200 \mu\text{m}^2$ with an additional bottom stage enabling step-and-repeat operation. Both AFM and SPL are performed under room conditions keeping temperature and relative humidity constant at $22 \pm 1^\circ\text{C}$ and $35 \pm 5\%$, respectively, which are continuously monitored and recorded during patterning.

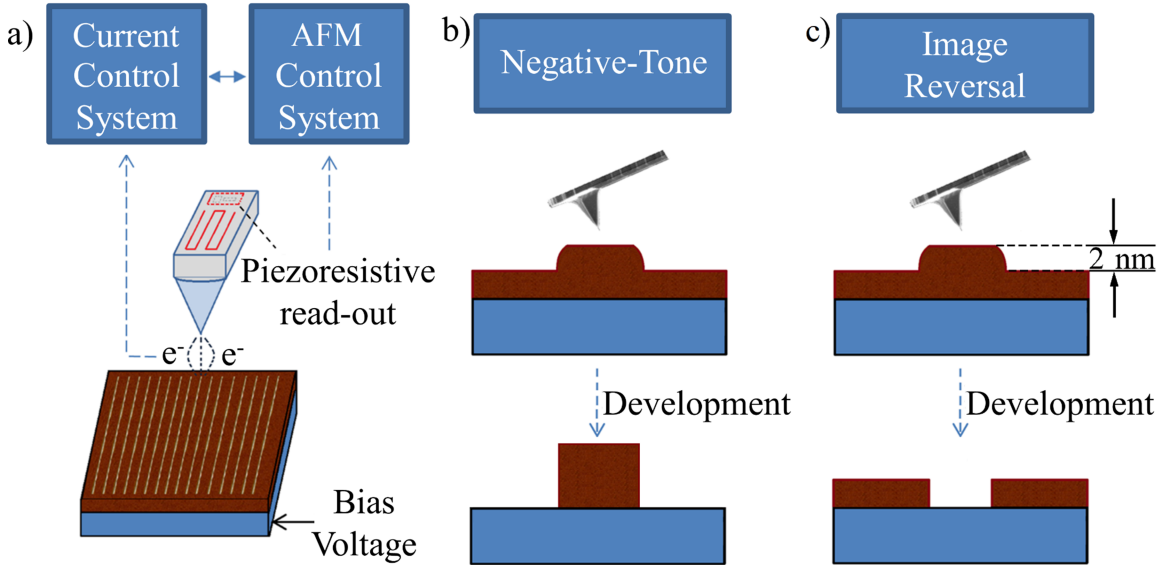


Figure 2.1: a) Working principle of current-controlled FE-SPL with two feedback loops utilizing thermally actuated piezoresistive cantilevers, b) negative-tone patterning upon development in MIBK and c) image reversal upon development in PMGI101A, both showing the latent image after exposure.

FE-SPL provides the unique capability of scanning latent images of exposed features before development among all other lithographic methods. Similarly, it allows

one to observe any other local topography changes in the resist induced by exposure. This observation facilitates any evaluation of the effect of development on the final shape of the structure. Observation of the latent image allows one to understand the contribution of process parameters to LER and linewidth. Generally, the height of the structures in latent images is on the order of 1-2 nm making it very challenging to measure using techniques other than non-contact AFM mode. When the patterned structures are exposed in negative-tone and then developed in methyl isobutyl ketone (MIBK) solution, unexposed areas are removed resulting the resist profile shown in Fig. 2.1b. If the latent features after negative-tone patterning are developed in PMGI101A developer, an image reversal from negative to positive-tone takes place as presented in Fig. 2.1c. Latent image topography can be measured through atomic force microscopy [24, 25]. In the present study, latent image is directly obtained after exposure in non-contact AFM mode of FE-SPL device.

2.2 Line Edge Roughness

LER is described as the roughness of random variations on a printed pattern. Similarly, LWR represents variations over the width of a patterned feature [26]. For LER investigation, deviations from a straight edge are examined, while for LWR, it is required to examine fluctuations in the distance between two edges.

Several sources contribute to LER depending of the specific lithography technique. Among the leading sources of LER one can mention LER of mask patterns in the use of a hard mask, variations in the exposure dose, photon flux variation (shot noise), nonuniformity in the chemical composition of the resist, acid diffusion during chemical amplification, development, dynamic and static speckle [27].

LER can be investigated using several tools such as scanning electron microscope (SEM), atomic force microscope (AFM) and their special variations such as critical dimension SEM (CD-SEM). CD-SEM and CD-AFM can quantify LER more precisely

for small feature sizes, can be automated, and provide information about line wall profiles. Each method has its tradeoffs. For example, in SEM, charging of the resist pattern causes blurring of the image, especially along the edges. Although AFM provides a higher accuracy in this respect, it is a slower method compared to SEM [28].

Using SEM, LER can be calculated using either on-line or off-line image analysis. On-line LER analysis is a real-time measurement technique that is automated, easier and faster. In on-line analysis, the number of the sampling points might be limited depending on the particular SEM setup and resist can be overexposed while imaging. Off-line analysis has the advantages of avoiding any overexposure of the resist pattern in SEM, the possibility of highest single pixel resolution and the availability of spatial and fractal analysis [29]. It was shown that such off-line analysis of SEM and AFM images is capable of providing the same level of accuracy in LER quantification as that of the on-line analysis [30].

IRDS provided guidelines for the measurement of LER and LWR based on standard the definition of Semiconductor Equipment and Materials International according to which LER and LWR are calculated as 3σ of residuals along $2\text{-}\mu\text{m}$ -long lines [3]. Defined as the standard deviation of deviations from a straight line, σ_{LER} , is given by:

$$\sigma_{LER} = \sqrt{\sum_{i=1}^N \frac{1}{N} (x_i - \bar{x})^2} \quad (2.1)$$

$$\bar{x} = \frac{1}{N} \sum_{i=1}^N x_i \quad (2.2)$$

where x_i is the local position of the edge point i on the line, $\bar{x}$ is the average of the points on the line and N is the total number of points, as defined in the sketch in Fig. 2.2.

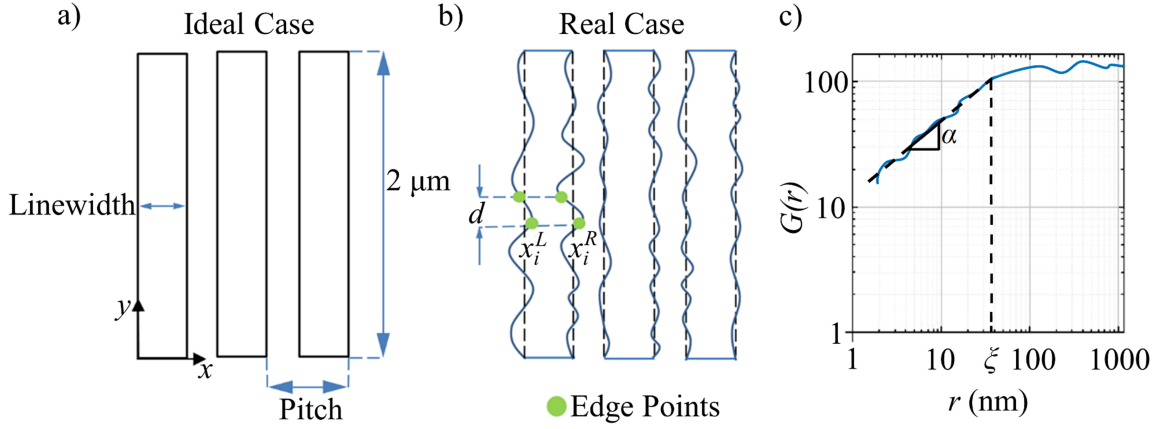


Figure 2.2: Sketch of a series of resist lines in a) ideal and b) real case with the illustration of LER definition as a function of edge points with the distance d between two consecutive edge points c) representative HHCF of a line edge with correlation length δ and roughness exponent α .

LWR can also be calculated using the aforementioned method. The width of a line at the position i , w_i , can be calculated as follows:

$$w_i = x_i^R - x_i^L \quad (2.3)$$

with x_i^R and x_i^L defined in Fig. 2.2.

The average width, $\bar{w}$, at the i^{th} interval and standard deviation of line width, σ_{LWR} , can be found using Eqn. 2.4 and Eqn. 2.5.

$$\sigma_{LWR} = \sqrt{\sum_{i=1}^N \frac{1}{N} (w_i - \bar{w})^2} \quad (2.4)$$

$$\bar{w} = \frac{1}{N} \sum_{i=1}^N w_i \quad (2.5)$$

In addition to standard deviations of line width and line edge, spatial scaling and fractal analysis are necessary for the complete description of the edge profile.

Frequency analysis provides the required correlation length, δ , and fractal dimension, D , in this respect. These parameters can be obtained by using three different methods, namely, the height-height correlation function (HHCF), the Fourier transform or the power spectral density (PSD) [31]. In this study, we utilized HHCF to find the spatial aspects of LER.

HHCF, $G(r)$, describes the spatial distribution of roughness of an edge by studying the correlation between the distances of edge points from a linear fit of all edge points [30]. $G(r)$ is defined at $r = md$ with m representing the degree of correlation and d is the distance between two edge points along y -axis (Eq. 2.6).

$$G(r) = \sqrt{\frac{1}{N-m} \sum_{i=1}^{N-m} (\delta_{i+m} - \delta_i)^2} \quad (2.6)$$

where δ_i is the distance of an edge point from the linear fit and N is the total number of equidistant points on along the y -axis of edge points. The linear portion of HHCF exhibits a power law behavior and the exponent of the power law is the roughness exponent, σ , which is related to the fractal dimension, D as shown in Fig. 2.2c. In HHCF, the power law behavior extends up to a certain point, beyond which $G(r)$ stabilizes. The distance at which HHCF assumes $\sqrt{(1-1/e)}$ of its maximum value is defined as the correlation length, δ , again depicted in Fig. 2.2c.

Chapter 3

EXPERIMENTAL PROCEDURE

In this study, we used 10-nm thick spin-coated calixarene molecular glass resist on Si wafer with resistivity of 1-15 Ω .cm. For each case of latent image, image reversal and direct-write, three exposure areas were utilized to study LER. In each exposure area, 10 lines with a nominal pitch of 200-nm were patterned with two different exposure doses of 25 nC/cm and 200 nC/cm for negative and positive-tone, respectively. Bias voltage was set to 40V for a stable lithography. It is to be emphasized that this particular set of exposure doses and bias voltage values were chosen to keep linewidths at their minimum without compromising the stability of lithography. The linewidth of each features is increased as a result of increasing exposure dose and bias voltage in positive tone as well as negative-tone patterning[12]. After the exposure, the lines were scanned using the same SPL tip in non-contact AFM mode. Later, the negative-tone sample was developed by using PMGI101A developer for 5 secs. for image reversal shown, whereas no development was necessary in the case of direct-write (Fig. 3.1). After development, the same sample was scanned one more time to observe LER after image reversal. Special attention was paid to scanning the same area for overlapping latent image and image reversal.

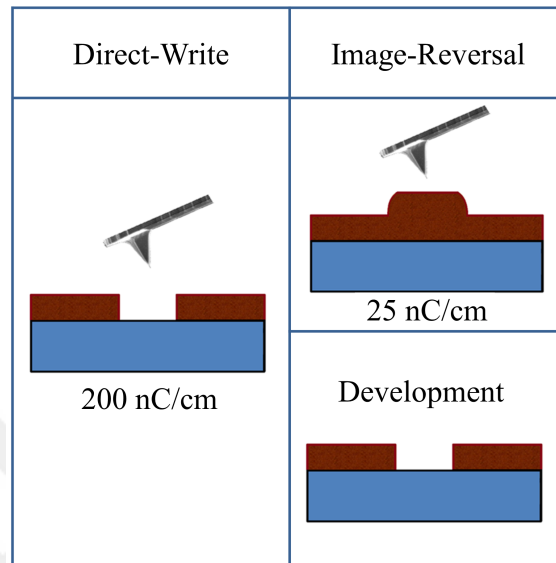


Figure 3.1: Summary of the SPL exposures used in this study. The performance of direct-write was compared to that of image reversal.

Generally, the SPL tip is subjected to wear and tear. Tip can be either broken completely or double-tip behavior can be observed after several use. Such defects can definitely affect the performance of lithography and imaging. Within this study, the SPL tip was characterized under SEM before patterning and imaging to ensure sharpness of the tip as shown in Fig. 3.2.

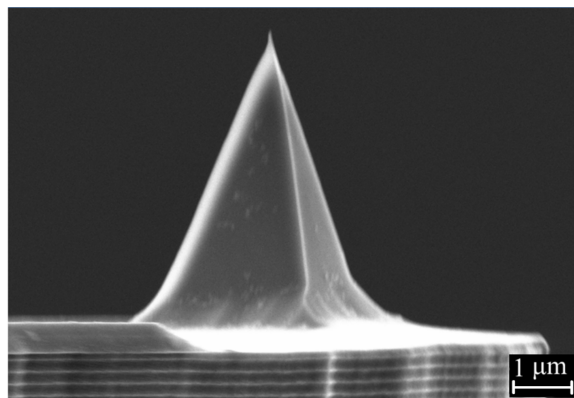


Figure 3.2: SEM image of the active cantilever tip employed in FE-SPL before use.

In line with the corresponding IRDS recommendation, imaging and image analysis were conducted by scanning lines with $2\text{-}\mu\text{m}$ length in a $2\times 2\text{ }\mu\text{m}^2$ window. Images were taken with the highest possible resolution of the SPL platform, *i.e.* 1024×1024 px, with a pixel size of 1.95 nm .

We used the Image Processing toolbox of Matlab with Canny edge detection algorithm. Canny algorithm is based on the calculation of the intensity gradients using the derivative of a Gaussian filter. The size and the standard deviation of this Gaussian filter are the two parameters that need to be carefully selected, as any improper selection might lead to inaccurate results [32]. Hence, filter parameters should be optimized by observing the dependence of the correlation length on the standard deviation of the Gaussian filter. Signal threshold with high and low threshold parameters obtained by Otsu's method was applied to remove weak edges [33]. All edges with an intensity below the lower threshold were disregarded, whereas those with an intensity above the high threshold were preserved. While detecting the edge points there were some missing pixels along the line edge which were replaced using a suitable interpolation by considering the continuity of the line. Spatial analysis was conducted by calculating HHCF of the lines on the image. Corresponding correlation length and roughness exponent were calculated from HHCF. The edge detection algorithm used in this study is presented in Appendix A.

Chapter 4

RESULTS AND DISCUSSION

AFM images were collected after exposure for latent image characterization. The step height of the latent features was about 2 nm, and resolution capability of FE-SPL tool in AFM-mode enabled in-situ imaging of the features. Imaging latent features with any other approach would be unreliable, since the height of the features was very low. Similarly, after PMGI101A development, AFM images were obtained totalling to two images for image reversal characterization. On the contrary, since direct-write is a single step process without any development step, one AFM image for each exposure was sufficient. With three areas exposed in each case a total of 9 AFM images as depicted in Fig. B.1, Fig. B.2 and Fig. B.5 were studied for latent image, image reversal and direct-write.

Collected AFM images were analyzed in Matlab using the aforementioned method. Edge point positions were obtained along x, and y axes. Corresponding LER, LWR and spatial parameters were calculated using the formulation as laid out by Eqns. (1)-(6). Fig.4.1a-c illustrates detected edge points superimposed on the actual representative AFM images in grayscale for latent image, image reversal and direct-write, respectively. A close-up view with three lines is presented below each image. Corresponding HHCF for latent image, image reversal and direct-write are depicted in Fig.4.1d-f, respectively.

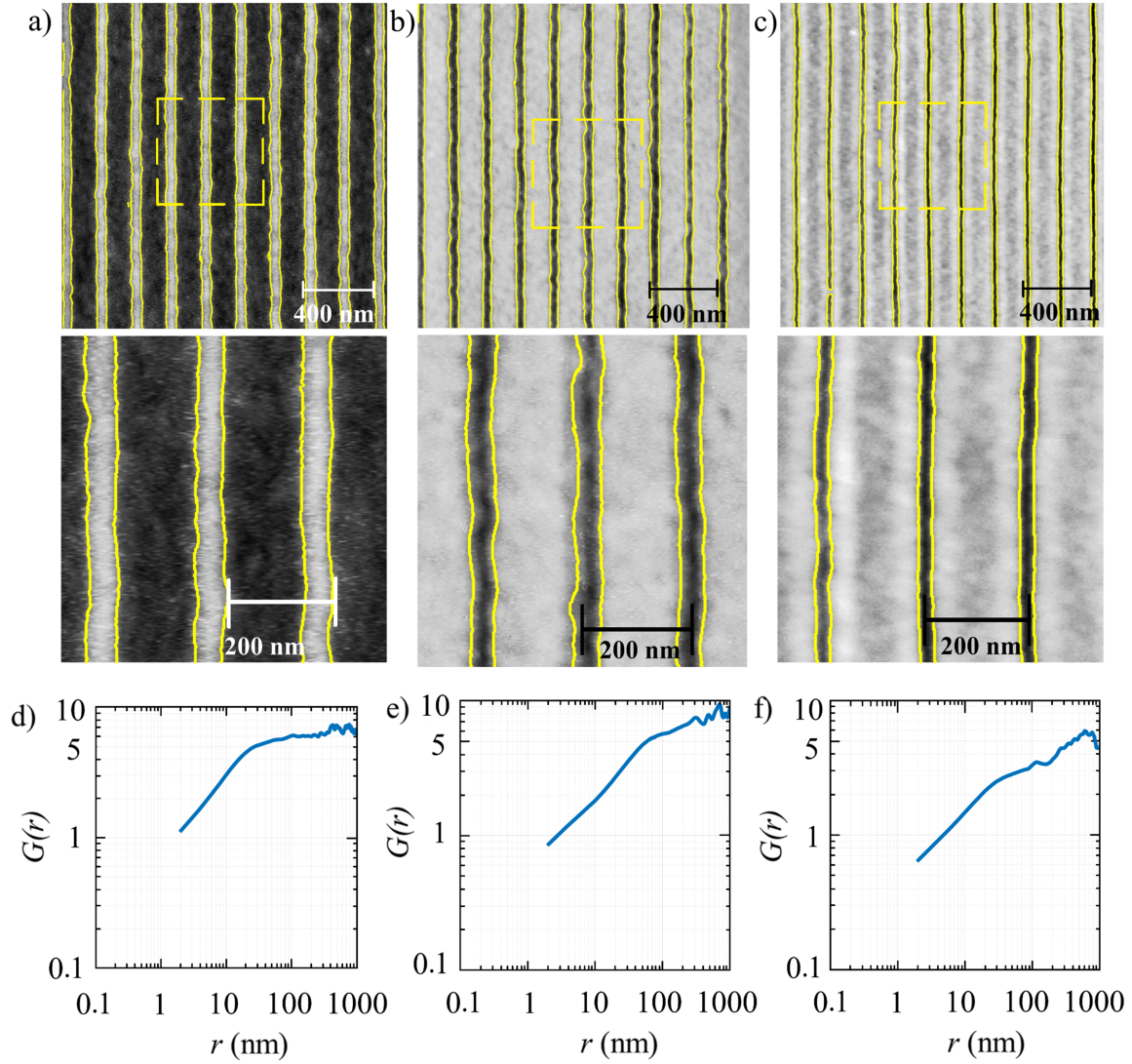


Figure 4.1: Representative edge profiles extracted from AFM images of 10 lines for a) negative-tone exposure (latent image) (b) image reversal and (c) direct-write in FE-SPL with the insets showing close-up views and corresponding HHCF for d) latent image (e) image reversal and (f) direct-write.

Roughness values and related correlation length, δ , as well as the roughness exponent, α , were calculated and averaged for each line in the representative latent image in Fig. 4.1a and reported in the first column of Table 4.1. An additional study was carried out to evaluate the reliability of the proposed methodology by comparing

these results with those of a commercial analysis. Our roughness calculation technique was compared with the unbiased roughness calculation of MetroLER software (Fractilia, LLC). MetroLER software utilized the patented Fractilia Inverse Linescan Model (FILM) edge detection algorithm to measure and remove the noise without filtering to find unbiased roughness values [34]. The results demonstrate that our analysis is capable of capturing similar LER, LWR and fractal values with MetroLER software with a maximum of 9% difference in LER. This difference can be attributed to the application of filtering to the image. Unlike our analysis utilizing Gaussian filtering, MetroLER does not rely on the use of any filtering. In addition to roughness parameters, linewidth and pitch between lines were calculated as 55.16 ± 2.08 nm and 212.1 ± 1.5 nm, respectively. It is to be emphasized that these are the results obtained on 10 lines of Fig. 4.1a.

Table 4.1: Roughness parameters for latent image: Current analysis vs MetroLER

Parameter	Analysis	MetroLER
$3\sigma_{LER}$ (nm)	16.44 ± 5.52	17.90 ± 1.72
$3\sigma_{LWR}$ (nm)	21.74 ± 5.98	21.42 ± 3.23
δ (nm)	30.33 ± 5.96	53.96 ± 3.10
α	0.69 ± 0.02	0.50 ± 0.00

To compare the outcome of image reversal and direct-write, images presented in Fig. 4.1b and Fig. 4.1c were analyzed. The results are summarized in Table 4.2. LWR and LER of lines associated with image reversal were found to be higher than those of direct-write. Ratio of LER to linewidth was 0.37 and 0.39 for image reversal and direct-write, respectively. In both studies, we found average pitch values to be close to the nominal value of 200 nm. Direct-write resulted in a smaller α compared to image reversal, as high frequency components contributed to the roughness to a larger extent in direct-write compared to image reversal.

Table 4.2: Comparison of roughness parameters obtained from image reversal and direct-write calculated on 10 lines.

Parameter	Image Reversal	Direct-Write
$3\sigma_{LER}$ (nm)	15.99 ± 7.98	9.44 ± 1.62
$3\sigma_{LWR}$ (nm)	20.18 ± 8.61	6.02 ± 2.87
δ (nm)	35.79 ± 6.82	48.34 ± 7.13
α	0.70 ± 0.02	0.66 ± 0.02
Linewidth (nm)	43.68 ± 4.06	24.48 ± 0.36
Pitch (nm)	201.4 ± 4.62	200.9 ± 1.06

It is to be noted that the above analysis related to both Table 4.1 and Table 4.2 were carried out using 10 lines that fit in a single $2 \times 2 \mu\text{m}^2$ scan. As a final point, we analyzed three $2 \times 2 \mu\text{m}^2$ images amounting to 30 lines, the results of which are summarized in Table 4.3. As clearly seen from Table 4.3, FE-SPL was able to reproduce lines with a designated nominal pitch value of 200 nm. By comparing the results of the single image presented in Table II and those in Table 3, we obtained similar LER results with 4% and 16% maximum deviation for image reversal and direct-write, respectively. Furthermore, the small LER deviation between latent image and image reversal shows that for LER generation the patterning process is more effective compared to the development. Finally, the higher standard deviation for LER in image reversal indicates the stochastic nature of the development, where the chemical composition of the resist, fluctuations in the SPL tip during patterning, instrument noise during AFM are expected to be some of the reasons.

Table 4.3: Roughness parameters for all exposure types obtained on 30 lines.

Parameter	Latent Image	Image Reversal	Direct-Write
$3\sigma_{LER}$ (nm)	16.44 ± 4.69	16.78 ± 5.81	10.95 ± 2.47
$3\sigma_{LWR}$ (nm)	20.63 ± 5.64	16.56 ± 7.24	5.57 ± 2.92
Linewidth (nm)	56.83 ± 3.50	50.01 ± 4.05	26.81 ± 3.48
Pitch (nm)	203.1 ± 10.47	201.2 ± 3.99	199.9 ± 2.38

An additional sample was prepared by coating 100-nm thick 950 PMMA C2 on Si and patterned by electron-beam lithography (EBL). Several lines with the analogous geometric shapes were patterned by Vistec electron-beam lithography system with an exposure dose of $650 \mu\text{C}/\text{cm}^2$. Later, this sample was developed in MIBK/IPA 1:3 solution for 1 min., followed by 30 sec. in IPA. After rinsing, the sample was dried by nitrogen air. EBL sample was used to compare results with SPL. The sample prepared by EBL was also scanned using the SPL tip in the same equipment. The AFM image of the EBL sample scanned using the FE-SPL setup can be seen in Fig. 4.2a with it's corresponding HHCF in Fig. 4.2b.

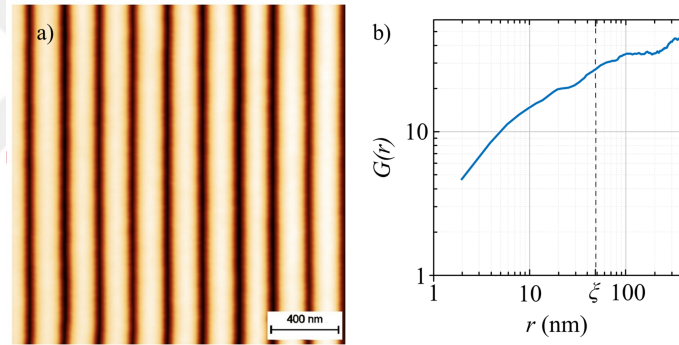


Figure 4.2: a) AFM image of sample fabricated by EBL and (b) corresponding HCCF.

Edge points of lines were detected using the same method and further roughness parameters were calculated as presented in Table 4.4. We observed smaller LER and LWR for EBL samples with 56.3 nm average linewidth.

Table 4.4: Calculated roughness parameters for E-Beam Lithography

Parameter	Matlab Analysis
$3\sigma_{LER}$	6.46 ± 0.81
$3\sigma_{LWR}$	4.53 ± 0.64
Linewidth (nm)	56.3 ± 6.17
Pitch (nm)	205.7 ± 5.96
δ (nm)	48.75 ± 15.36
α	0.44 ± 0.06

This work presents the first of its kind by patterning and analyzing lines using the same FE-SPL setup by detecting edge points on latent image, image reversal and direct-write. Image reversal of calixarene molecular glass resist from negative-tone to positive-tone is studied for the first time in FE-SPL. A methodology of LER investigation is presented to compare image reversal with direct-write, where the lines were fabricated using single exposure doses for image reversal (25 nC/cm) and direct-write (200 nC/cm) by considering the linewidth of the final structures and the stability of the patterning. In the future, this study can be extended by studying the effect of different exposure doses on LER. A further comparison with CD-SEM or CD-AFM can be provided to validate measurement capability of the current FE-SPL setup.

Chapter 5

CONCLUSION

In this thesis we report LER of lines as a result of image reversal and direct-write in current-controlled FE-SPL. Three exposure areas were patterned with 10 lines per each area using a 10-nm thick calixarene molecular glass resist on Si wafer. Image reversal and direct-write were utilized using 25 nC/cm and 200 nC/cm exposure doses, respectively. The sample prepared by negative-tone exposure (25nC/cm) was developed by PMGI101A developer for 5 seconds for image reversal from negative-tone to positive-tone. Additionally, line edge roughness of a latent image was studied for validation purposes. All AFM images were obtained in non-contact AFM mode using the same FE-SPL setup used for patterning. With the height of the latent features on the order of 2 nm, resolution capability of the setup proved to be adequate for in-situ imaging. This observation of the latent image was also crucial for understanding the effect of process parameters to LER of final structures.

A Matlab script was generated to calculate LER, LWR, linewidth and pitch values as well as spatial parameters. Canny edge detection algorithm was applied to find edge points. We utilized Otsu's method to find threshold values required for the algorithm. From HHCF, correlation length and roughness exponent for each line were found. For the verification of presented methodology, we compared our methodology with a commercially available software, MetroLER, for the latent image characterization. Both analyses were able to produce comparable results with 9% deviation showing the accuracy of the proposed technique.

The analysis showed that positive-tone generated by direct-write resulted in a lower LER and LWR. However, the ratio of LER to linewidth was higher in direct-write compared to image reversal. Although direct-write was able to produce lines

with a smaller deviation, the deviation between the lines obtained by image reversal was higher. This can be explained by the random nature of the development in image reversal. In all studies, the pitch between the lines was found to be on the order of the nominal value of 200 nm. This study can be extended by observing the effect of exposure dose on image reversal and direct-write and including a comparison with other metrology tools such as CD-SEM.



Appendix A

EDGE DETECTION ALGORITHM

- 1 Load image to Matlab
- 2 Determine pixel size in nm
- 3 Transfer image in array of pixel intensities
- 4 Determine high threshold using Otsu's method
- 5 Set low threshold to half of high threshold
- 6 Subtract background intensity
- 7 Apply noise filtering (Gaussian)
- 8 Apply edge detection algorithm with two thresholds (Canny)
- 9 Determine edge points
- 10 Calculate LER, LWR, linewidth and pitch
- 11 Calculate HHCF
- 12 Find corresponding roughness exponent and correlation length

Appendix B

AFM IMAGES OF LINES PATTERNED IN FE-SPL

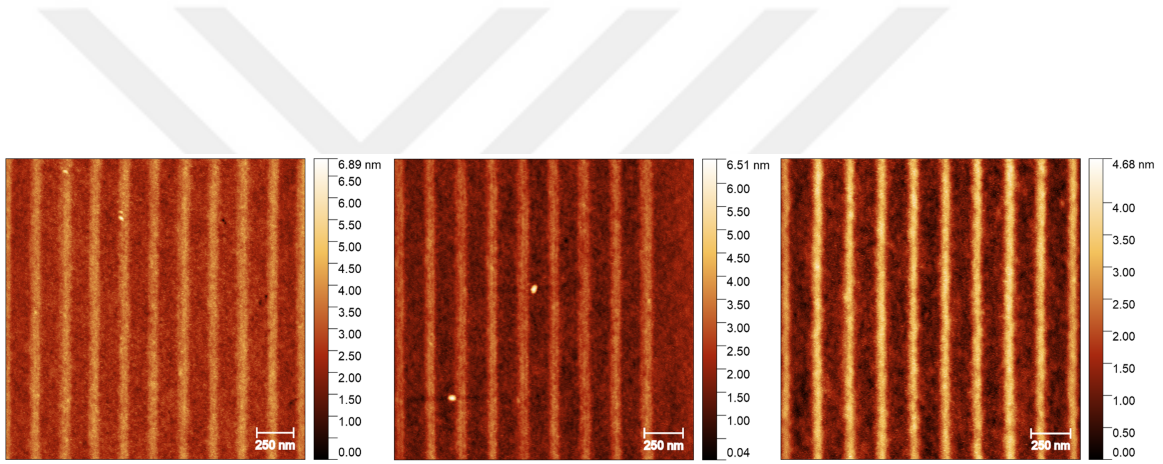


Figure B.1: $2 \times 2 \mu\text{m}$ 1024x1024px AFM latent images of the features on

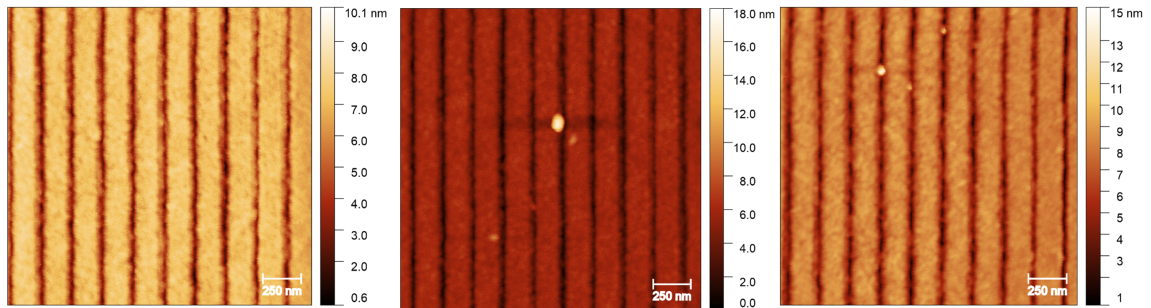


Figure B.2: $2 \times 2 \mu\text{m}$ 1024x1024px AFM images of the image reversal features after 5 secs. development

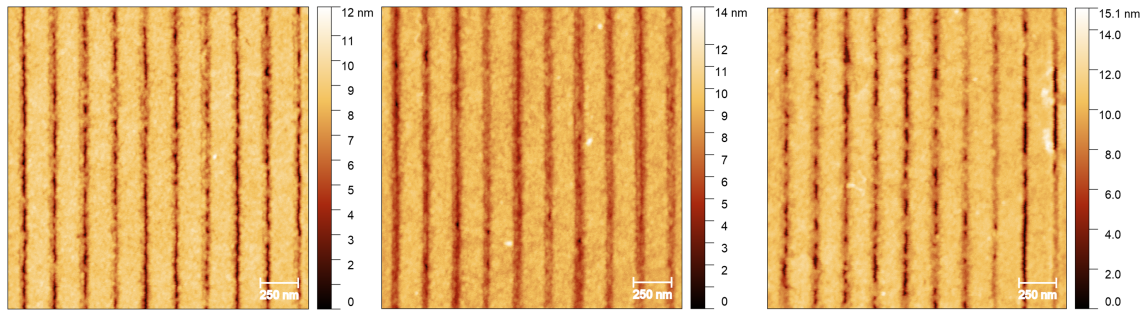


Figure B.3: $2 \times 2 \mu\text{m}$ 1024x1024px AFM images of the image reversal features after 10 secs. development

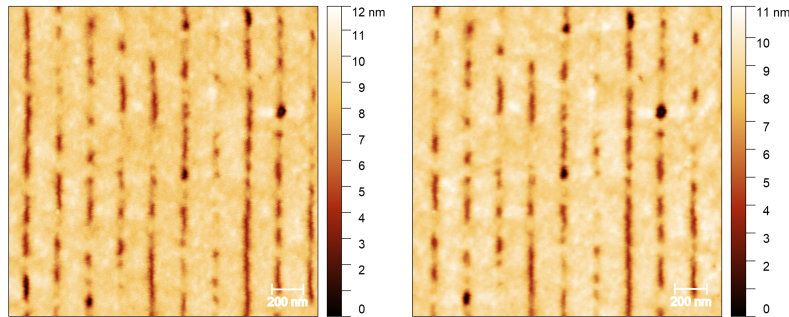


Figure B.4: $2 \times 2 \mu\text{m}$ 1024x1024px AFM images of the image reversal features after 30 secs. development

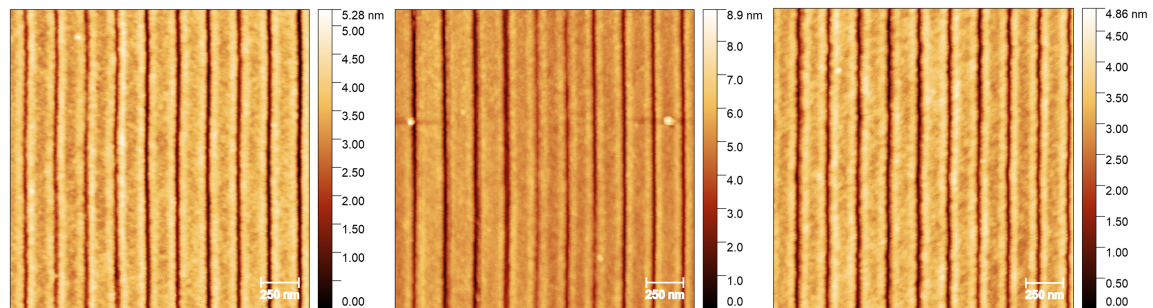


Figure B.5: $2 \times 2 \mu\text{m}$ 1024x1024px AFM images of the direct-write features

Appendix C

CONVERSION OF DIGITAL, PIXEL-BASED IMAGES TO EXTENSIBLE MARKUP LANGUAGE

Conversion is required to pattern raster images made with pixels and letters in various kind of file formats such as jpg, png and tif. Firstly, digital, pixel-based images should be converted to vector based scalable vector graphics (.svg) using a vector graphics editor such as CorelDraw. Scalable vector graphics should be converted to extensible markup language to to be compatible to LithoPatternEditor

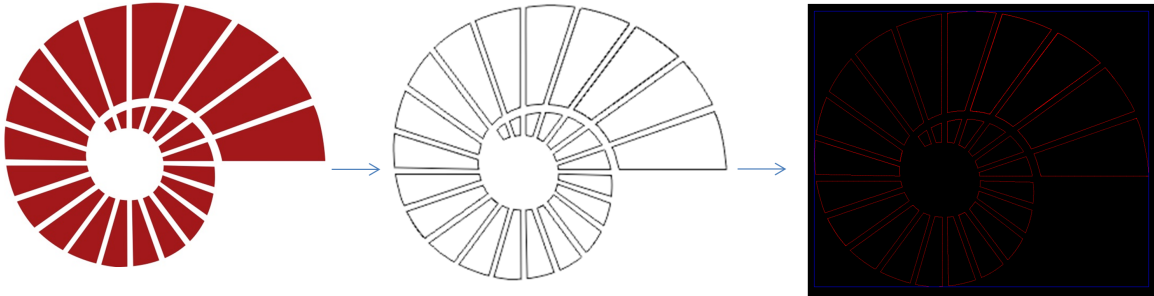


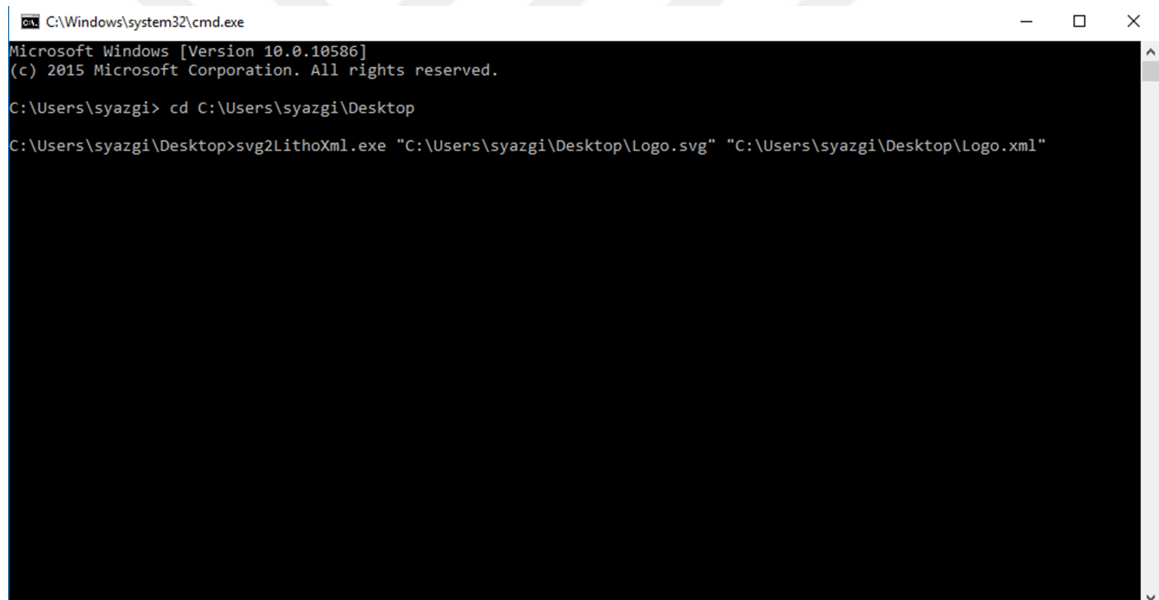
Figure C.1: Conversion of digital, pixel-based Koc University logo to vector based scalable graphics through scalable vector graphics.

First of all, the desired image should be imported to vector graphics editor. Then, the image should be vectorized using PowerTrace function of the editor. After vectorization, the vectors should be converted to curves, and additional nodes should be added to ensure smooth curves. Optionally, shapes can be ungrouped before adding nodes. After increasing the node count arcs and splines should be converted to straight lines. Finally, the processed image can be saved as scalable vector graphics. Scalable vector graphics should be further converted to extensible markup language using a

converter tool. The tool has to be run from the command line:

- Press “Windows key” + “R” to open the program execution dialog
- Enter “cmd”
- Change to the directory where the tool is stored and run the tool

You can find a screenshot of the command windows below in Fig. C.2.



```
C:\Windows\system32\cmd.exe
Microsoft Windows [Version 10.0.10586]
(c) 2015 Microsoft Corporation. All rights reserved.

C:\Users\syazgi> cd C:\Users\syazgi\Desktop
C:\Users\syazgi\Desktop>svg2LithoXml.exe "C:\Users\syazgi\Desktop\Logo.svg" "C:\Users\syazgi\Desktop\Logo.xml"
```

Figure C.2: Command window for conversion of scalable vector graphics to extensible markup language.

After creating extensible markup language file which is readable by LithoPatternEditor, Koc University logo was patterned on a Si chip with calixerene together with initials of Mechanical Characterization Laboratory (MCL). While Koc University logo was patterned in positive tone with -200nC/cm exposure dose, letters are patterned in negative tone with -25nC/cm. The patterning was conducted in a sequential manner to demonstrate imaging, alignment, inspection and patterning capability of FE-SPL. The surface was imaged before and after every pattern. In overall, the entire

structure was created by four sequential read/write cycles. The resultant structure together with intermediate steps between each sequence can be seen in Fig. C.3.

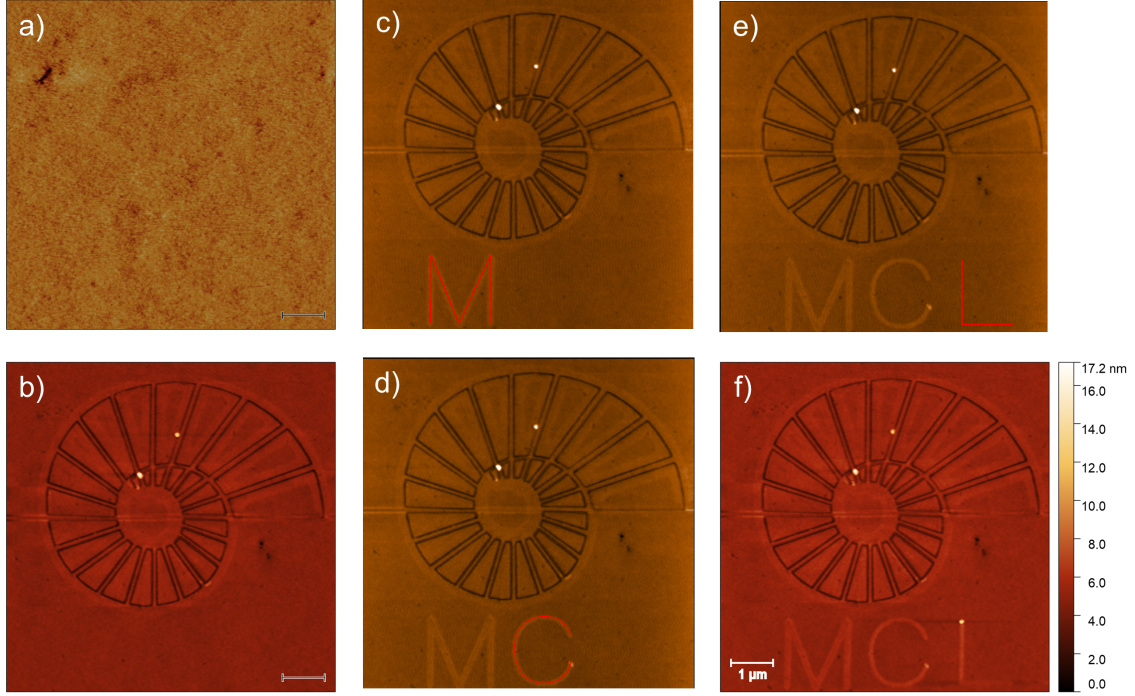


Figure C.3: Successive imaging/patterning cycle for Koç University logo with initials of Mechanical Characterization Laboratory (MCL). Images from (a) to (e) shows the generation of the patterns in a stepwise fashion with intermediate imaging, alignment and inspection sequences. From (a) to (g) MCL letters are patterned in negative tone, whereas the Koc University logo is patterned in positive tone. The resultant structure is shown in (f). The imaging in every sequence is conducted in non-contact AFM mode with the same cantilever used for patterning. The structures was not developed in any stage of the patterning between (a) and (f).

Appendix D

CONVERSION OF MATHEMATICAL FUNCTIONS TO EXTENSIBLE MARKUP LANGUAGE

Shapes with a known mathematical function can be patterned in FE-SPL, however conversion is required. There are two special Matlab codes to convert mathematical functions. First, the x and y coordinated mathematical function should be created and plotted in Matlab. Then, if the shape is as desired, it has to be converted to extensible markup language using another Matlab code.

Mathematical functions of heart and logarithmic spiral are converted to extensible markup language using defined method. The shapes are patterned on a Si chip with calixarene resist in negative tone with $-25\text{nC}/\text{cm}$ exposure dose. The resultant structures can be seen in Fig. D.1 and Fig. D.2. The resultant structures was not developed.

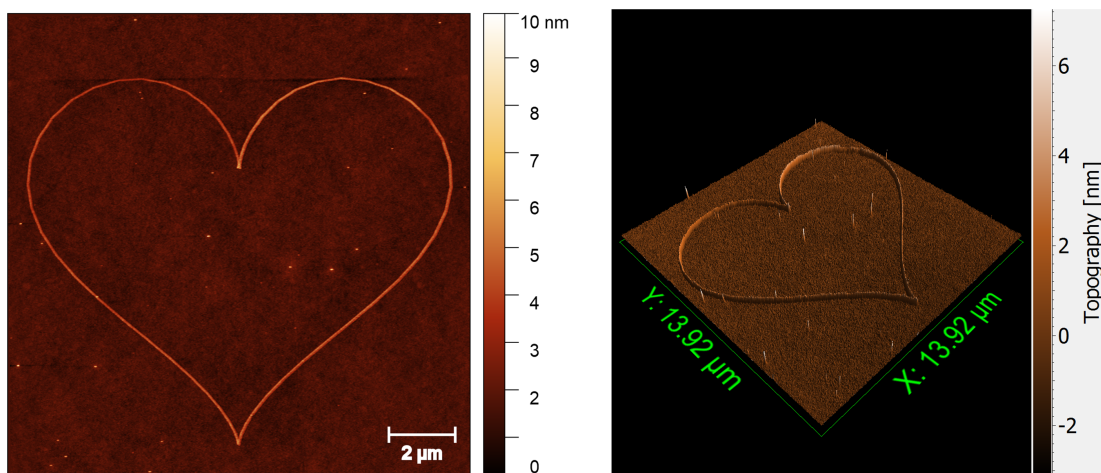


Figure D.1: 2D and 3D AFM image of hearth shape patterned on calixerene molecular glass resist in negative tone.

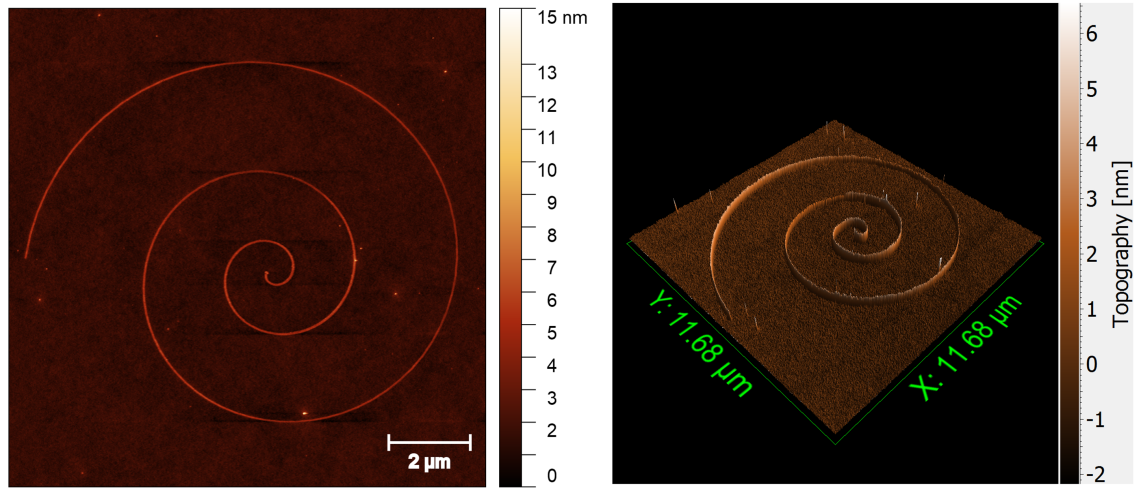


Figure D.2: 2D and 3D AFM image of logarithmic spiral patterned on calixerene molecular glass resist in negative tone.

Appendix E

LIFT-OFF PROCESS IN SCANNING PROBE LITHOGRAPHY

Bi-layer lift-off method is utilized to obtain Koc University logo and resonator structures out of chromium. Two photoresist are used to do bi-layer lift-off. 10nm thick calixerene is spin coated on top of 45-50 nm thick lift-off resist PMGI. n-type Si chip is used. After cleaning and preparing chip for coating without HF dip, the chip is pre-baked at 250 °C for 3 mins., a small amount of PMGI resist is applied by a clean pipette. The chip is rotated at 4000 rpm for 45 secs. Then, it is post-baked at 250°C for 7 mins. On top of lift-off resist, calixarene is coated using the same procedure explained in Section 3.1. After spin coating, the chip is ready for patterning. The structures were patterned in positive tone with -500nC/cm. After patterning, the chip is developed on for 30 secs. using PMGI101A developer. After 30 secs DI water is poured on top of chip and rotated at 1400 rpm for 30 secs. at spin coater.

The chip evaporated 10 nm of chrome after PMGI development. After metallization, the chrome is lifted-off in 3:1 Dimethylsulfoxide-Cyclopentanone solution at 80 °C for 30 mins. Then, the chip is placed to ultrasonic bath at 50% power for 2 mins. Following to ultrasonic bath, IPA is poured onto chip. The chip is dried out by a jet of nitrogen gas.

You can see the Koc University logo with 10 nm high chromium in Fig. E.1. Different type of split ring resonator structures can be seen in Fig. E.2 and Fig. E.3. For SRR structures gap is modulated between 50 nm and 500 nm. 5 row of structures are patterned for repeatability purposes.

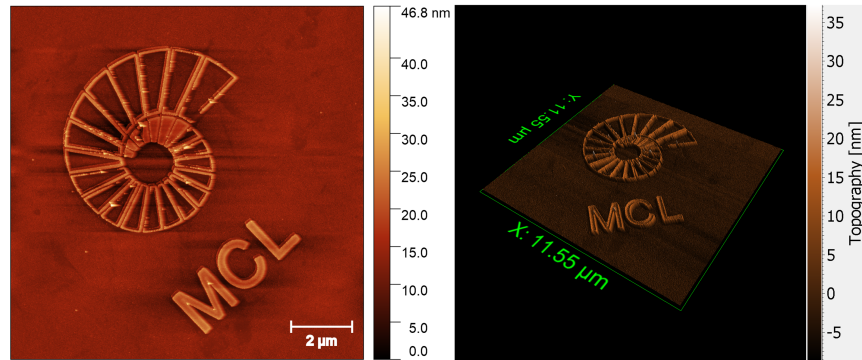


Figure E.1: 2D and 3D AFM image of logarithmic spiral patterned on calixerene molecular glass resist on negative tone.

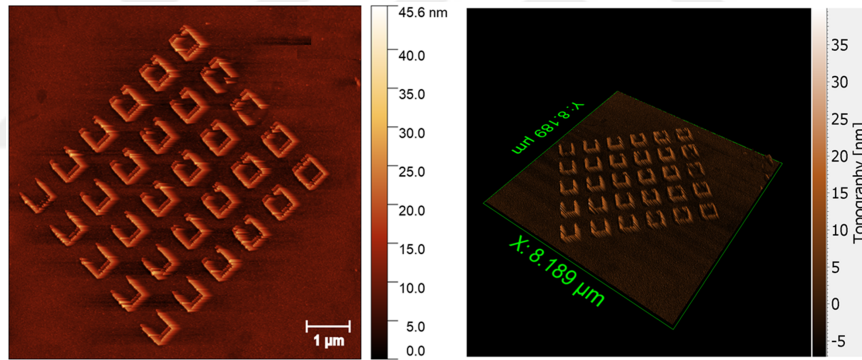


Figure E.2: 2D and 3D AFM image of logarithmic spiral patterned on calixerene molecular glass resist on negative tone.

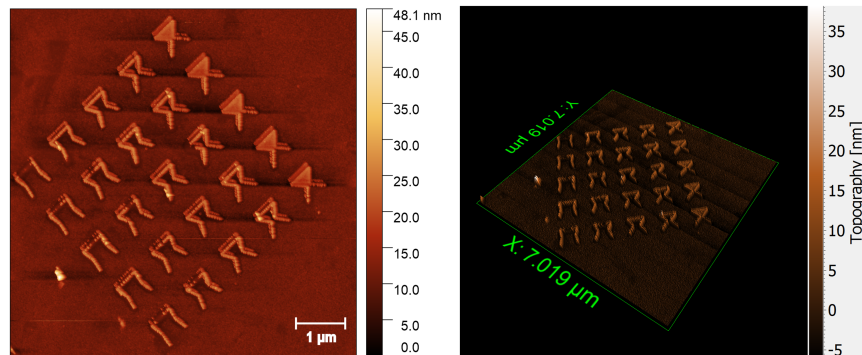


Figure E.3: 2D and 3D AFM image of logarithmic spiral patterned on calixerene molecular glass resist on negative tone.

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