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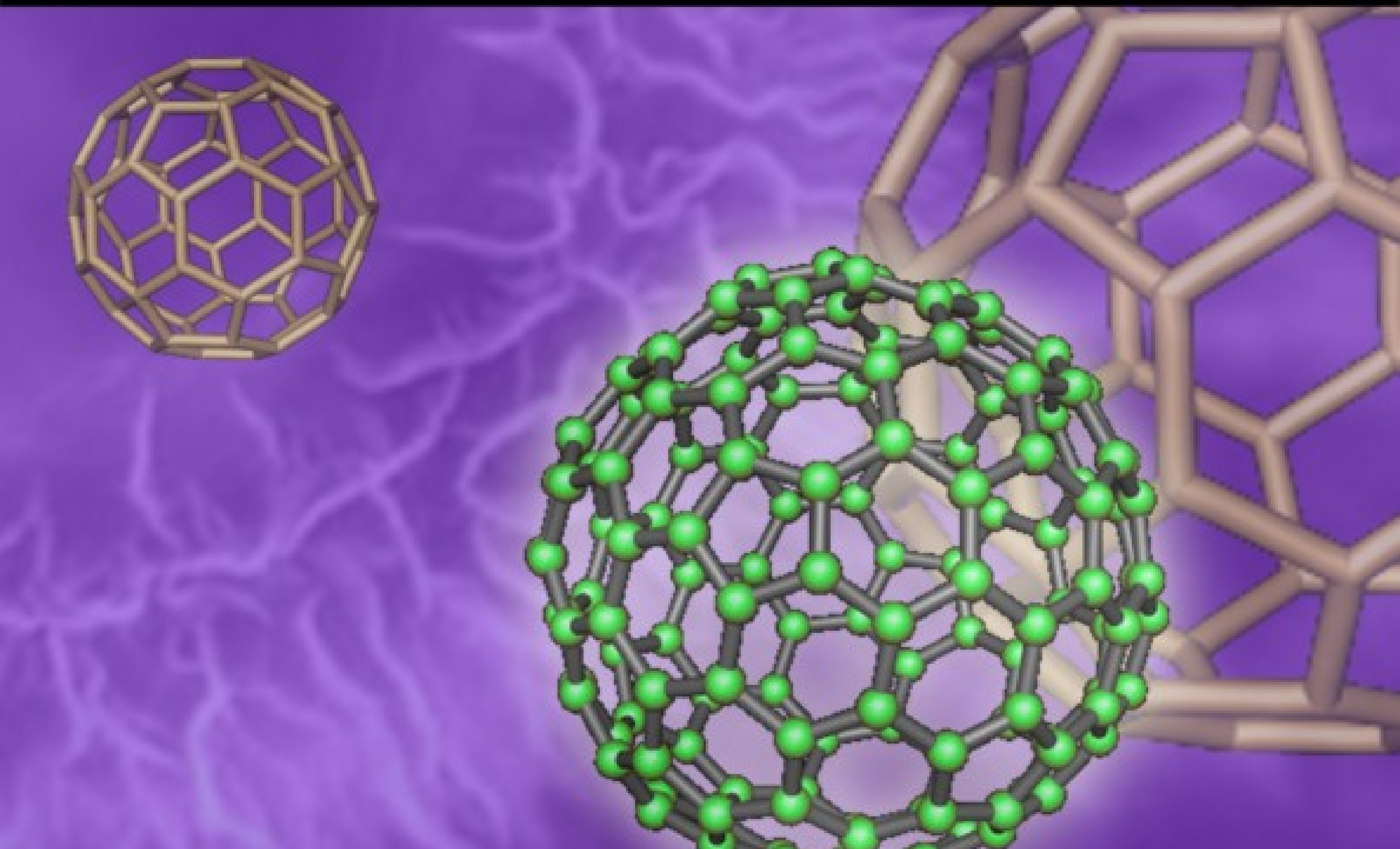

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Contents

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Resolution Enhancement of Thermal and Optical Nanolithography Using an Organic Dry Developing Resist and an Optimized Tip

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Abstract: Ultrahigh nanolithography resolution of 31 nm was achieved using thin film layers of naphthoquinones compounds. Dry nanolithography processes, negative and positive were developed, utilizing the thermal and optical properties of these compounds, by using an Atomic Force Microscope with a tapered optical tip. Negative nanolithography was achieved using a specially designed optical fiber tip transmitting 488 nm of light that functions as a near field optical source. Positive nanolithography was achieved by coating the fiber tip with a metal film to serve as a nano-heating source. Sub-wavelength gratings with a variable line width, fabricated using these processes, are demonstrated. *Copyright © 2009 IFSA.*

Keywords: Nanolithography, AFM

1. Introduction

A number of near field patterning techniques have been explored recently to extend the resolution of optical lithography beyond the conventional diffraction limits. Improved performance of the lithographic process is the key to the ongoing miniaturization of electronic and optical devices. Among the main nanolithographic methods in use, we can mention interference lithography [1], evanescent interferometric lithography [2] near field optical lithography [3] near field imprint lithography [4] and near field two photon nanolithography [5]. The lithographic methods that are based on interference are mainly used for the fabrication of periodic structures, while serial methods are, in general, unrestricted by this constraint and can be used also for non periodic structures.

The use of an AFM for direct nano-scale lithography with a normal photoresist is a well known method. The principle of this technique is the replacement of the regular pyramid tip, normally used for surface force sensing, with a "system" designed to deliver the required optical energy, in a very controlled manner to the surface, during the exposure stage of the lithographic process.

The advantage of using an AFM for direct writing, beyond considerations of simplicity and cost effectiveness, is that it's a mask-less process compared to other near field methods [3]. Different methods of utilizing this concept are mentioned in previous reports each having its own limitations. There are techniques that use a micromachined AFM cantilever with an integrated silicon probe tip that acts as a source of electrons [6], while in other methods the tip acts as optical source. Using this method we can mention the work that has been done [7] with a solid immersion lens (SIL) mounted on a cantilever and scanned while in contact with the photoresist, resulting in a line-width of 190 nm. This result was achieved because of the geometrical shape and the high index of the lens, n=2.2, that reduces the effective wavelength of the light illuminating the recording layer, thus reducing the spot size. The advantage of this method is that the scan rate can be increased up to 1cm/sec. The enhanced speed is a result of the high optical efficiency (about 10-1) of the SIL. This is several orders of magnitude faster than typical reports of near-field lithography using tapered optical fibers. In this paper we want to focus on, and utilize, the method of using the AFM to position a tapered optical fiber or a micropipette, which acts as a near-field optical source, to within a few nanometers of the surface of a photosensitive material [8-10]. In this manner the illuminating beam profile remains constant and approximately equal to the aperture of the source. This system enables direct optical writing onto the photoresist with nanometric resolution. The main drawback of this method is slow scanning speed (less than 100 μm/sec), which is due to the low level of energy which is delivered by the fiber. In order to overcome this disadvantage, resist materials with higher sensitivity are needed, as well as improved energy transfer to the resist, which can be done by the optimization of the fiber tip geometry.

From the material sensitivity perspective we want to suggest the use of the organic resist, naphthoquinone. This thin film material can function as a photosensitive material but can also be inscribed thermally by a nano-heating source. Both processes apply the method of the near field source as described above and both processes are dry processes, which do not require chemical development.

There follows a description of naphthoquinone film properties, fiber tip geometrical optimization, and the optical and thermal nanolithography results. This study is based on the methods published by us in a previous article [11]. In this paper we will present more advanced results of the thermal lithographic process via optimization of the optical tip, as well as a more thorough description of the optical tip which enabled us to obtain the new results of 31 nm line width lithography.

2. Experimental Details

The photosensitive and the heat sensitive properties of napthoquinone compounds made them a natural choice for use as a photoresist. The interaction of visible light with organic solutions of 2-arylamino-3-cycloalkylaminonaphthoquinones-1,4 causes the compounds to form ring structure derivatives of the naphthimidazoles [12]. A study of the thermal stability of the solid phase of these types of naphthoquinones, has shown that they are chemically stable below ~ 100 ^{o}C. However, in air they sublimate directly at melting point (~100 ^{o}C), and in a vacuum at lower temperatures [12]. On the other hand, the isomer created by exposure to light is stable at higher temperatures (~ 115 ^{o}C). Hence, appropriate optical treatment enables the separation of the two phases. The separation between the two phases being dependent on the napthoquinone compounds.

These two features, photosensitivity and stability up to the sublimation point, suggests the use of napthoquinones as a photoresist [13], which are developed after exposure by sublimation. Since the illuminated phase has a higher thermal stability, it will act as a negative photoresist. Alternatively, by inscribing a pattern thermally, the film will act as a positive resist without requiring further development. These two methods of inscribing a pattern allow for totally dry processes that eliminate certain steps, as compared to classical optical photolithography, and are effective in terms of total cost and time. Thermal inscription also allows real time lithography. Real time lithography may enable the development of new manufacturing nanotechnological processes.

As mentioned earlier, the efficiency and effectiveness of the lithographic process is limited by the ability to deliver sufficient energy to the photoresist. An experimental study to optimize the geometrical shape of the bend-tapered fiber was preformed in order to ensure that the maximum amount of light would emerge from the tip end. The study traces the optical power loss in the fiber. The optical fiber can be divided into five optical waveguide sections, as shown in Fig. 1:

1. A straight waveguide: In this section the optical power transmission is approximately the same as a regular straight waveguide. Apart from the coupling loss, all other losses can be ignored.

2. The bend radius R: Most of the optical power loss occurs in this area and it is due to the bend radius and the defect areas caused by the bending process. The light transmission of this section can be significantly improved by an optimized R parameter.

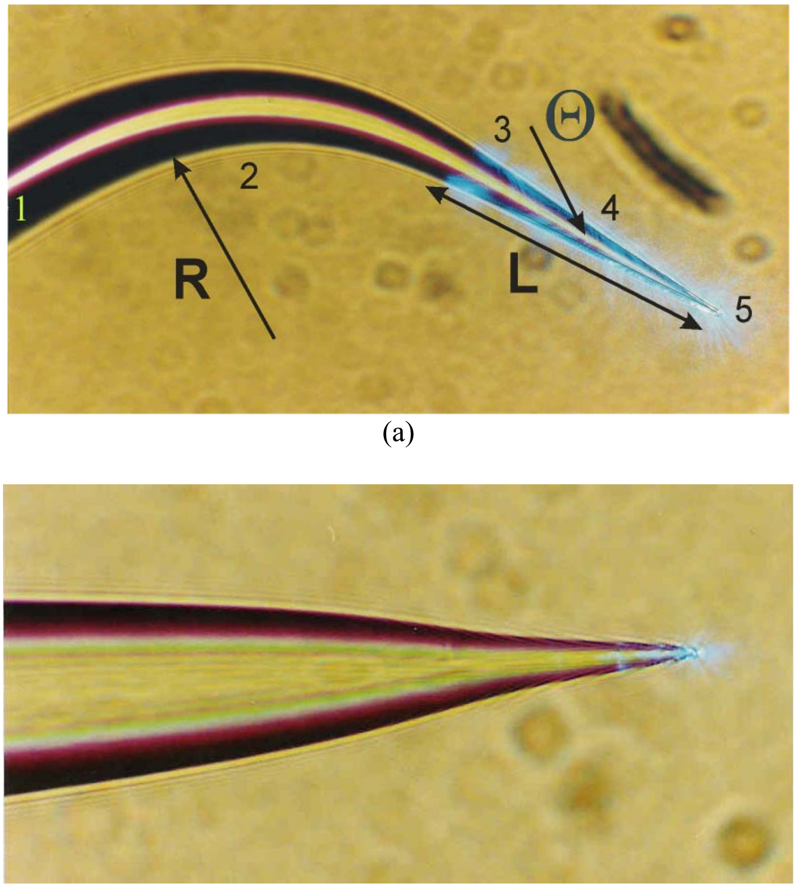
3. Cone waveguide area: In this metal covered section (angle 100-150), the border between the core and the cladding is not fully clear. The diameter of this area begins at 20 μm and its length is around 100 μm. This area melts during the bending stage so that the waveguide can be approximately described as a glass core with a metal conductive envelope. In this area only the fundamental TE optical mode exists, assuming perfect conductivity of the metal coated tip. The fundamental TE mode cutoff term is where is the wavelength (in our case = 488 nm), r is the core radius and n is the index of refraction. The optical LP01 mode in the single mode fiber is mainly coupled with the fundamental TE mode of the conical waveguide. In the region below cutoff all the modes except the fundamental TE mode, are either refracted or absorbed and considered as heat which diffuses to the coated tip area [14].

4. The cone waveguide section is divided into two parts, the first one is a tapered cone waveguide with a dimension larger than the wavelength, and the second part up to the NSOM (Near filed Scanning Optical Microscope) aperture is a tapered cone waveguide with a dimension less than ~0.5λ. In this section the optical power decreases exponentially with the distance [15]. The overall distance of this two tapered cone waveguide is ~100 μm.

5. NSOM aperture- aperture area: The aperture is smaller than the optical wavelength launch into the fiber and the final dimension of the aperture will determine the system resolution. There is a reciprocal relationship between the resolution and the power intensity that is delivered to the resist given by: where *a* is the aperture radius [16].

Optimization of the fiber performance was achieved through the control of three parameters: R the bend radius, Θ the angle and L the length of an adiabatic tip waveguide. Figs. 1a and 1b show fiber tips without the metal coating. Fig. 1a shows the light loss in the bent area of an un-optimized fiber tip. The five-waveguide sections are marked and one can track the light scattering along the fiber tip from before the waveguide cut off area until the end tip. The light guiding can be seen by controlling these geometrical parameters as shown in Fig. 1b, which shows an optimized transmission end tip. The optical and the thermal probe design follow the same guidelines. The only difference being, that the thermal tip in section five is coated with metal in order to absorb the light and convert it to heat. Maximized radiation delivery to the tip is desirable for both processes.

The first set of experiments includes a lithographic process based on near field optical exposure where negative lithography was achieved. A non-period grating was fabricated directly onto a naphthoquinone resist film with nano resolution. The fabrication involved the complete lithographic process i.e. exposure, developing and fixation. The resulting developed resist serves as the final element.



(a)

(b)

Fig. 1. An un-optimized tip with its five-waveguide sections (a); Optimized transmission end tip (b).

Commercial AFM (Topometrix, Santa Clara, USA) with an optimized optical probe, was used for the nanolithography system. CW Ar+ ion laser (488 nm) coupled to a single mode optical fiber was used as the light source. The output of this fiber is coupled to a bent and tapered Cr/Al coated quartz fiber tip (Nanonics, Jerusalem, Israel). This tip replaces the regular scanning force probe of the AFM. This tip consists of a single mode elongated quartz fiber (core diameter 3.7 μm) with a 0.13 numerical aperture. The fiber is pulled in a CO_2 laser powered micropipette puller (Sutter Instruments P-2000). Passing the fiber rapidly through the focal spot of a second CO_2 laser resulted in sharp, accurate, and very reproducible bends less then 100 microns from the tip. Varying the amount of time that the tip was in the beam controlled the bend angle. The fiber tip was glued to a small stub, leaving a free cantilever length of between 100 to 400 microns. The precise cantilever length can be controlled so as to obtain a force constant K, from 0.01-10 N/m for contact mode, and tip resonance frequency from 180 to 380 kHz. The K values can be varied from 10-100 N/m for non-contact methods. The bent glass elements were then coated with tens of microns from the sub wavelength aperture which was left uncoated, so that the fiber functioned as a near field optical source. The optimized geometry was chosen for the experiment.

The probe was held a few nanometers above the sample in order to be in the near-field mode. Calculations indicated [17, 18] that the light emanating from a sub-wavelength aperture is initially confined to the diameter of the aperture and has, in the near field, an exponentially decreasing intensity distribution as its beam spreads out from the surface of the aperture. The standard atomic force microscope feedback mechanism regulates the distance between the tip and sample, in the non-contact mode. The sample was then raster scanned to create the desired pattern.

3. Result and Discussion

Fig. 2 shows the absorption curve of the 2-cyclohexylamino-3-piperidinonaphtoquinone-1,4 used as a photoresist. The absorption peak of close to 488 nm can be observed. This material sublimates in air at a relatively low temperature (~100 °C). The melting point of the remaining solid phase occurs at 115 °C.

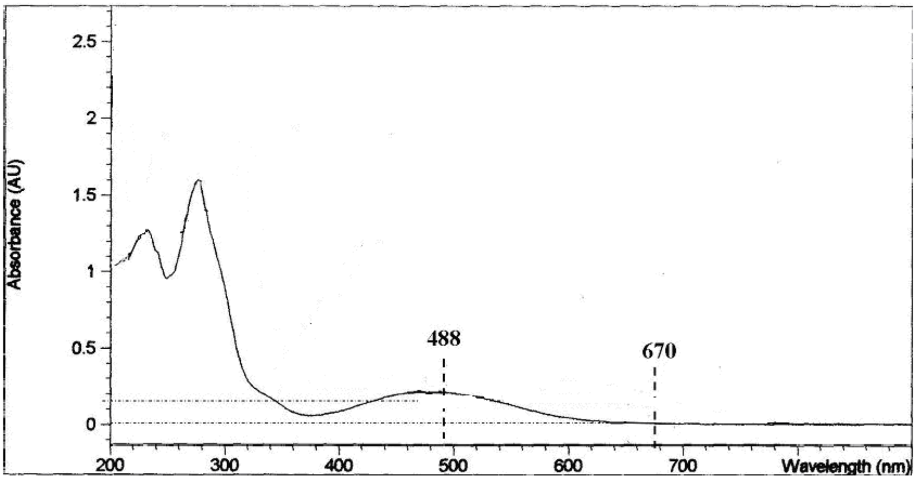
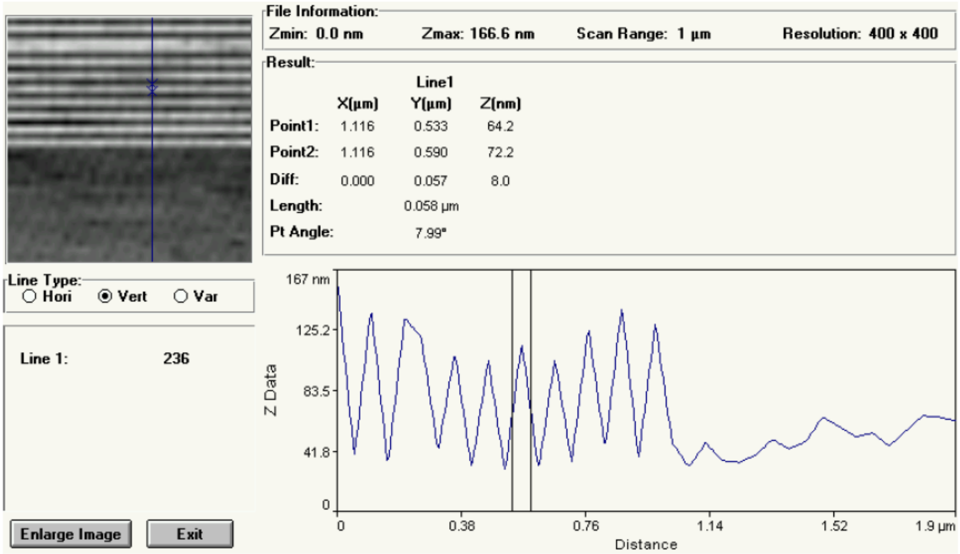


Fig. 2. Absorption spectra of naphthoquinone photoresist 2-cyclohexylamino-3-piperidinonaphthoquinone-1,4.

Resist films of this compound were used in subsequent experiments. The thickness of the resist films was approximately 0.12 μm. The photoresist films were prepared by vacuum thermal evaporation at about 120 °C and $4 \bullet 10^{-6}$ Torr. The glass roughness was 1 nm according to interferometer microscope measurement and the film roughness was about 2-5 nm according to an AFM measurements. Fiber tips with open ends of 70-100 nm outer diameters served as the near field optical source. The working parameters of the AFM system for near field lithography were: a non contact mode, a relative set point 40 %-50 % of the free range, a resonance frequency of 24 kHz and a scan rate of 3 μm/sec. The lower resonance frequencies of the tip are preferable since they give better feedback signals, and enable a more stable lithographic exposure process. The scanned areas were 4 x 4 μm^2. After exposure, a developing and fixation thermal treatment was performed and the film was brought back to the AFM for measurement and characterization with the same tip- now without launching any light into the fiber.

The lithographic results are shown in Fig. 3. The measurement of a 57 nm full width half maximum (FWHM) line width can be seen (Y diff in the figure) with an approximate etched depth of 80 nm, giving a 5:7 aspect ratio. The photoresist behaves, as expected, as a negative one, where the non-exposed photoresist disappears during the development process performed by thermal treatment. This is visualized by the fact that the bottom of the grating is at the same level as the substrate where the non-exposed photoresist has been removed by the developing process.



File Information:
Zmin: 0.0 nm
Zmax: 166.6 nm
Scan Range: 1 µm
Resolution: 400 x 400

Result:

	X(µm)	Line1 Y(µm)	Z(nm)
Point1:	1.116	0.533	64.2
Point2:	1.116	0.590	72.2
Diff:	0.000	0.057	8.0
Length:		0.058 µm	
Pt Angle:		7.99°	

Line Type: ○ Hori ● Vert ○ Var

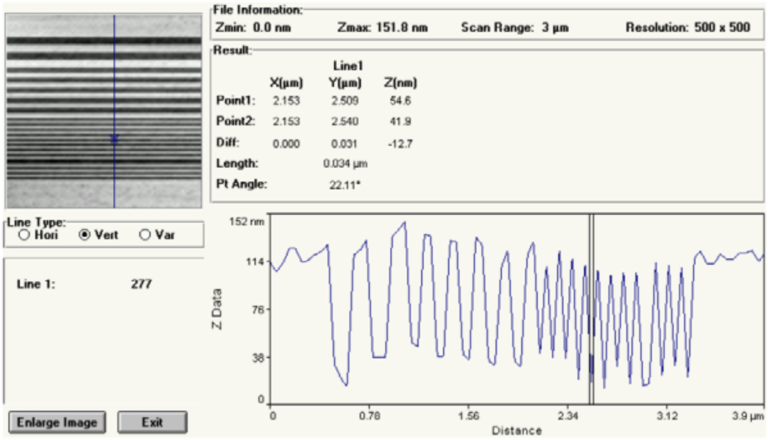
Line 1: 236

Fig. 3. Sub-wavelength grating made with optical near field exposure on 2-cyclohexylamino-3-piperidinonaphthoquinone-1,4.

Because this material sublimates in air at low temperatures (~100 °C) and the remaining solid phase melts at ~115 °C, a pattern can be inscribed by means of a localized nano heating source. By utilizing the same geometry of the fiber tip but this time with a metal-coated blocked end tip, the previous near field optical exposure source converted to a nano-heating source. The requirement that has to be fulfilled is that the localized temperature at the edge has to be above the sublimation temperature of the exposed phase (115 °C). The heat source is the light, transferred into heat, by utilizing the conductivity of the metal that covers the tip, and causes local heating and evaporation of the "exposed" material. This approach eliminates the thermal developing required when the film is optically inscribed. It also eliminates the need to remove the sample after exposure from the system in order to obtain the structure of the film. This procedure will enable the achievement of a "real time" nano lithographic process. This may open new horizons in nanotechnological manufacturing. In addition the introduction of a controlled external source to heat the fiber ends, could create a continuous lithographic process.

In the following set of experiments aimed at achieving thermal lithography, the same material that is used for optical lithography (2-cyclohexylamino-3-piperidinonaphtoquinone-1,4) with similar layer thicknesses was used. This time a blocked, metal-coated tip, with an outer diameter of 50-70 nm, was used. The assumption was that no light escapes, but only heats the edge of the tip. Heating the metal coating is not, however, solely confined to the immediate tip vicinity. The sensitivity of the thermo-chemical lithographic process therefore strongly depends on the tip induced thermal field enhancement caused by its shape and coating irregularities. The AFM system parameters were: a non contact mode, a relative set point 40 %-50 % of the free range, a resonance frequency of 32 kHz and a scan rate of 7 µm/sec. After heat exposure the film was characterized with the same tip.

Analysis of a cross section of the grating is shown in Fig. 4. Measurement of the 31 nm line width (FWHM) can be seen (Y diff), with the approximate etched depth of 90 nm, giving approximately a 3:9 aspect ratio. Although the physics governing the process still needs to be elucidated, one can clearly see that the pattern obtained is that of a positive resist. This is apparent from the fact that the top of the grating is at the same level as the original level of the photoresist and the process of exposure caused the removal of the photoresist. Therefore, using the same material under different exposure conditions, one can obtain a positive or negative pattern in the resist with a very high nano-scale resolution.



File Information:
Zmin: 0.0 nm
Zmax: 151.8 nm
Scan Range: 3 µm
Resolution: 500 x 500

Result:

	Line1 X(µm)	Y(µm)	Z(nm)
Point1:	2.153	2.509	54.6
Point2:	2.153	2.540	41.9
Diff:	0.000	0.031	-12.7
Length:		0.034 µm	
Pt Angle:		22.11°	

Line Type: ○ Hori ● Vert ○ Var

Line 1: 277

Fig. 4. Cross section of sub-wavelength grating made with a localized thermal exposure on 2-cyclohexylamino-3-piperidinonaphthoquinone-1,4.

4. Conclusion

Nanolithography has been demonstrated in naphthoquinone films, using an AFM with an optimized modified fiber optic tip. By using the same material under different exposure conditions we obtain either positive or negative nanolithography. Negative lithography is achieved using optical near field exposure, and positive lithography is achieved using a localized thermal nanoheating source. Based on thermal lithography, a "real time" dry process nanolithography was developed. These two methods can be applied for the creation of a variety of patterns, periodic or non-periodic, that demand nano-scale resolution. Using this system a record of 31 nm line width was achieved.

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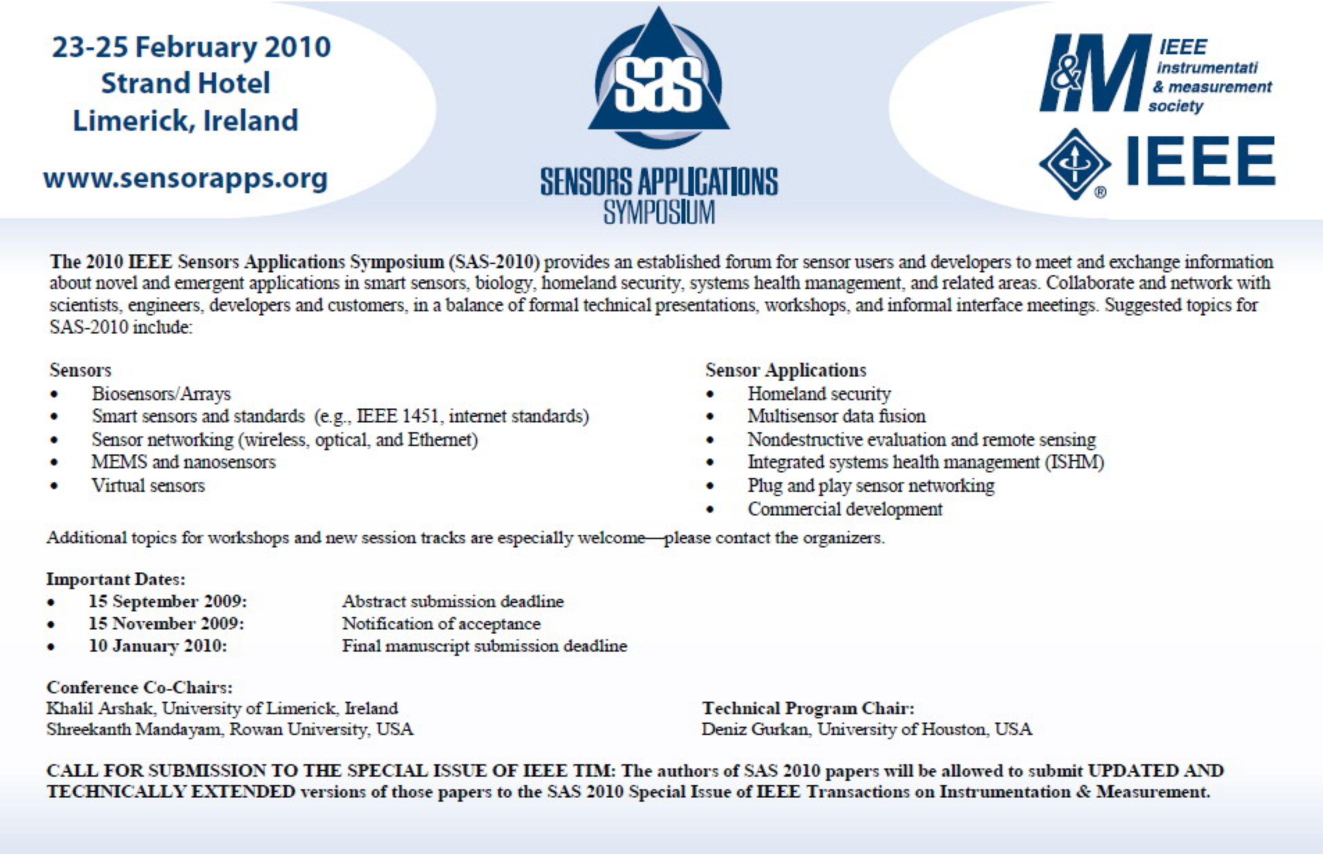

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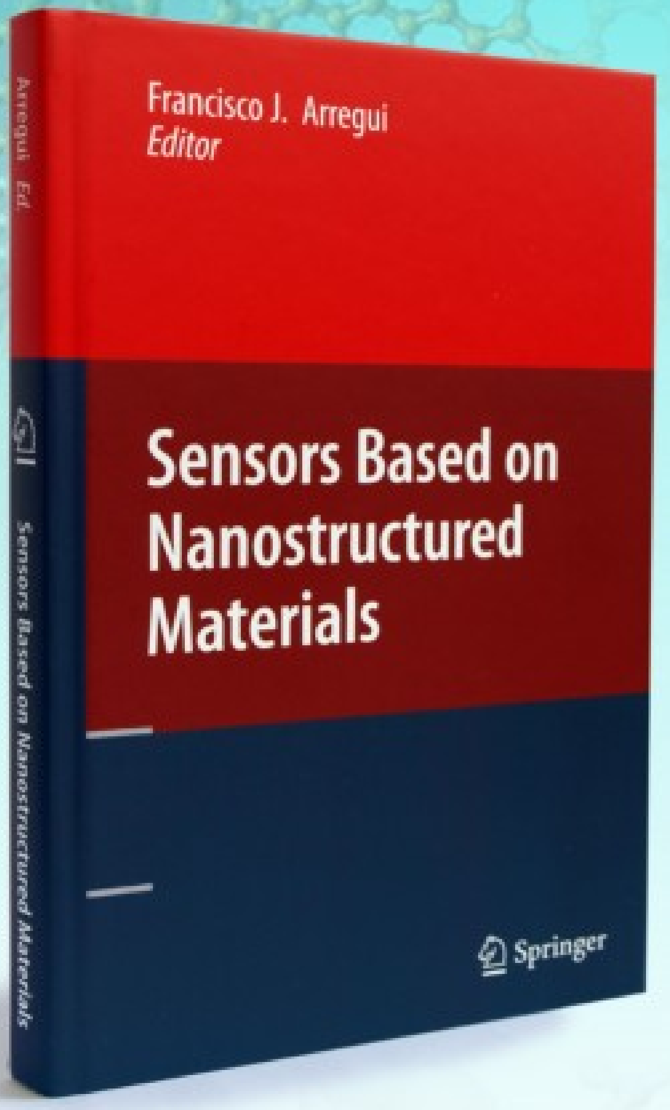
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