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High-Aspect-Ratio Nanoscale Patterning in a Negative Tone Photoresist

Kwangki Ryoo[1*] and Jeong Bong Lee[1,2] , ***Member, KIICE***

[1]Department of Information & Communication Engineering, Hanbat National University, Daejeon 305-729, Korea
[2]Department of Electrical Engineering, University of Texas at Dallas, Richardson, TX 75083, USA

Abstract

The demand for high-aspect-ratio structures has been increasing in the field of semiconductors and other applications. Here, we present the commercially available negative-tone SU-8 as a potential resist that can be used for direct patterning of high-aspect-ratio structures at the submicron scale and the nanoscale. Such resist patterns can be used as polymeric molds to create high-aspect-ratio metallic submicron and nanoscale structures by using electroplating. Compared with poly (methyl methacrylate) (PMMA), we found that the negative tone resist required an exposure dose that was less than that of PMMA of equal thickness by a factor of 100–150. Patterning of up to 4:1 aspect ratio SU-8 structures with a minimum feature size of 500 nm was demonstrated. In addition, nanoimprint lithography was studied to further extend the aspect ratio to realize a minimum feature size of less than 10 nm with an extremely high aspect ratio in the negative resist.

Index Terms: Electron beam lithography, High aspect ratio, Nanoimprint lithography, Negative photo resist

I. INTRODUCTION

There is a growing interest in the development of high-aspect-ratio, three-dimensional (3D) structures in a variety of fields, such as 3D Fin FETs [1] and electromechanical resonator devices in the deep nanoscale range [2]. Over the years, various lithography techniques that are capable of patterning in the submicron scale and nanoscale have been developed. Electron beam lithography (EBL) [3], proton beam lithography [4, 5], extreme ultraviolet (EUV) lithography [6], and nano imprint lithography (NIL) [7] are a few among them. However, most of these lithographic techniques have been developed for the integrated circuit (IC) industry, which essentially deals with two-dimensional (2D) lithography.

One of the more common resists for EBL, poly (methyl methacrylate) (PMMA), can be used for the development of high-aspect-ratio 3D structures in the submicron/nanoscale. For example, a 3:1 aspect ratio (thickness=1 μm; minimum feature size=350 nm) submicron structure was formed in PMMA by EBL and was used as a resist mold for a metallic nanoscale electro thermal actuator [8]. However, the requirement of a large exposure dose, and thus, a long exposure time, to expose the thick PMMA film renders this material less attractive.

The development of an SU-8 epoxy-resin negative photoresist has facilitated the fabrication of high-aspect-ratio 3D structures by using conventional ultraviolet (UV) lithography in the micron scale. Good chemical and mechanical properties of cross-linked epoxy-based SU-8 have opened up a considerable number of new applications in the field of microelectromechanical systems (MEMS) [9]. With the use of this chemically amplified negative tone

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***Corresponding Author** Kwangki Ryoo (E-mail: kkryoo@hanbat.ac.kr, Tel: +82-42-821-1710)
Department of Information & Communication Engineering, Hanbat National University, 125 Dongseo-daero, Yoseong-gu, Daejeon 305-729, Korea.

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photoresist, devices with aspect ratios of approximately 20:1, features sizes in the range of tens of microns, and near-vertical sidewalls were developed [10].

Compared to a positive photoresist, such as PMMA, the negative tone photoresist SU-8 has many advantages; further, since it is a chemically amplified photoresist, it requires substantially low exposure doses. In addition, the added feature of direct write onto the photoresist for EBL or NIL eliminates the need for a mask. In this paper, we report the nanoscale patterning characterization of both EBL and NIL on SU-8.

II. ELECTRON BEAM LITHOGRAPHY ON SU-8

EBL was performed on a Texas Instruments EBL station setup custom-designed to work at an acceleration voltage of 20 kV. Three parameters (namely, density, exposure, and spot size) were available to the end-user for the characterization of any pattern/material. Spot size determines the width of the electron beam. Density defines the number of lines that are drawn to fill a unit length (1 mil=25.4 μm), while exposure defines the exposure rate in microseconds per unit length of the electron beam. A combination of the parameters exposure and density determine the total exposure dose (Q) that the photoresist is exposed to. The pattern is designed using computer-aided design (CAD) software and is then converted into the pattern description language (PDL) format that the EBL system reads. Additional lines that specify the values of exposure, density, and pattern repetition can be added to the PDL file. Spot size is varied by controlling the magnetic lenses that the electron beam passes through.

For the EBL experiment, a 3-inch silicon wafer was cleaned using a typical RCA cleaning procedure. A seed layer consisting of 100-Å chromium (Cr) and 1000-Å gold (Au) was sputter-deposited onto the wafer. Gold was chosen as the seed layer because of its excellent adhesion property to SU-8. In this study, commercially available Nano SU-8 2002 was selected to be used as the photoresist for the characterization. Spin-coating of SU-8 was carried out at 2,000 rpm for 30 seconds to obtain a photoresist thickness of approximately 1.65 μm. A two-step pre-exposure bake consisting of 60 seconds at 65°C and 60 seconds at 95°C on a hotplate was performed. After cooling to room temperature, the sample was exposed to the electron beam. A 2D array grid, shown in Fig. 1, with the exposure and density varying along each dimension was generated for each submicron pattern to be characterized. As shown in Fig. 1, the density values varied from 50 to 400 lines per unit length and the exposure values varied such that the exposure dose varied from 0.5 μC/cm^2 to 2.5 μC/cm^2. Three different submicron spot sizes of 0.07, 0.13, and 0.25 micron were tried. Each of the spot sizes was used to expose the 2D grid successively.

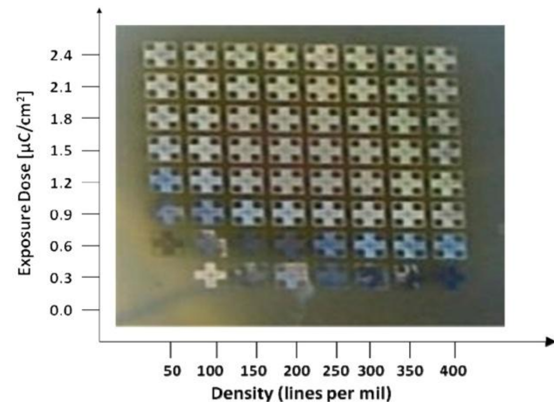

Fig. 1. Optical image of the 2D grid array used for electron beam lithography patterning characterization on SU-8 shown with the density and exposure doses used for the electron beam lithography.

After exposure, a two-step post-exposure bake consisting of baking on a hotplate at 65°C for 1 minute and at 95°C for 1 minute was carried out. The sample was then developed using the Nano SU-8 developer. The typical development time was about 60 seconds. Optimum development was verified by dipping the sample in isopropyl alcohol (IPA), which forms a white residue on the sample when underdeveloped. The developed sample was then rinsed in deionized water and dried. The developed sample was further cleaned by a short treatment of low-power oxygen plasma. During this process, about 0.15 μm of the surface SU-8 photoresist was etched back. This reactive ion etch (RIE) cleaning rendered a clean surface of the seed layer, thus promoting adhesion between the electrodeposited metallic layer and the metallic seed layer. After the RIE cleaning of the photoresist mold, the sample was dipped in an electroplating solution container and the container was placed in a low-vacuum chamber (approximately 500 mTorr) for about 30 minutes for removing any air bubbles trapped between the patterned nanoscale photoresist patterns. A plating current density of approximately 2 mA/cm^2 was identified as the optimum rate for electroplating nickel for the fabrication of high-aspect-ratio submicron structures. The growth rate for this condition was about 2 μm/hr.

SU-8 2002 with two different process conditions was studied. The preliminary test patterns used for this characterization are shown in Fig. 2. Typical features sizes varied from 0.25 μm to 5 μm. These patterns were exposed onto 1.5- and 2-μm-thick SU-8 resists by using the exposure/density grids as described above. Among all the spot sizes used, the spot sizes of 0.07 μm and 0.13 μm rendered good patterns. The beam current for the 0.07-μm spot size was about 0.9 nA and about 2.9 nA for the 0.13-μm spot. As the exposure rate is a function of the beam current, the 0.13-μm spot beam was preferred over the 0.07-

Fig. 2. Test patterns used for the electron beam lithography of high-aspect-ratio nanoscale patterning of SU-8.

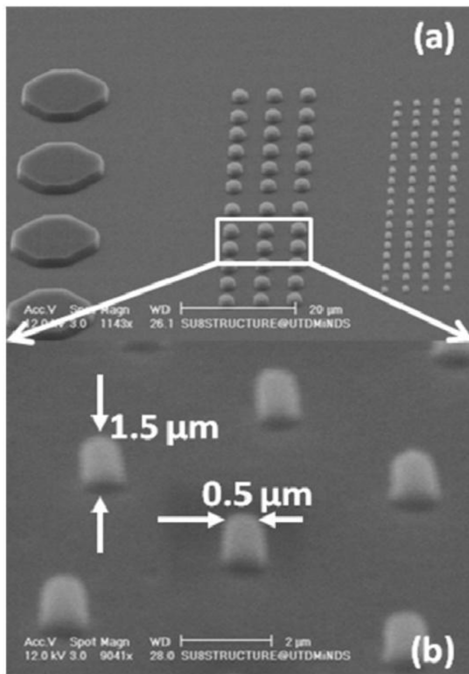

Fig. 3. Scanning electron microscopy (SEM) images of octagonal test patterns: (a) overall view of the various sizes of octagonal patterns, (b) a few octagonal pillars in high magnification showing the 3:1 aspect ratio nanoscale structures with a feature size of 500 nm.

μm spot beam, thus achieving an exposure rate that is approximately three times faster.

Various test structures obtained from the optimized EBL for SU-8 are shown in Figs. 3 and 4. The required values of density and exposure are different not only for different patterns but also for different sections of the same pattern. Fine features need a low density value with a high exposure dose, while coarse structures, such as contact pads and anchors, need a high density value with a low exposure dose. These variations are incorporated into the PDL file. The cumbersomeness of this process can be avoided if the fine and the coarse features are separated and exposed sequentially with the same global parameters of exposure and density. The fine feature layer and the coarse feature layer can be aligned and exposed as two separate layers. This also enables the use of a relatively large spot size such as 0.75 μm with a current of 75 nA for the coarse features, thus further enhancing the write speed for the pattern.

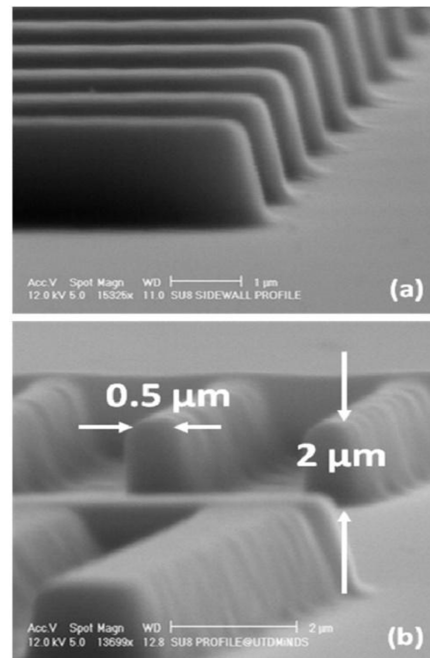

Fig. 4. Scanning electron microscopy (SEM) images of the arrayed comb pattern showing the 4:1 aspect ratio patterns defined on SU-8 with a minimum feature size of 500 nm.

As a preliminary work to build structures, a comb-drive structure was fabricated using a well-established positive photoresist PMMA for the sake of comparison. A fully fabricated 1:1 aspect ratio submicron metallic comb-drive structure is shown in Fig. 5(a). A similar comb drive but with a relatively high-aspect-ratio (3:1) mold created in SU-8 is shown in Fig. 5(b). The exposure time for the PMMA mold was about 10 min, whereas that for SU-8 was about 30 seconds. The metallic structure in the case of PMMA was obtained by lift-off. The same process is not possible in SU-8 because of two distinct reasons. Firstly, the cross-linked SU-8 is difficult to remove in a solvent and the sidewall profile has an inherently positive slope in the case of a negative photoresist and an inherently negative slope for a positive photoresist for EBL. Solubility in a solvent and the negative sidewall profile of the photoresist are essential for obtaining good lift-off structures. This inherently positive sidewall profile for negative resists and negative sidewall profile for positive resists can be attributed to the backscattering of incident electrons. Fig. 6 shows a schematic representation of the behavior of the backscattered electrons in a negative photoresist.

A closer look at the sidewall pattern of SU-8 shown in Fig. 7(a) clearly indicates the slightly positive profile due to the backscattered electrons from the substrate. This effect is not problematic when the separation between patterns is not in the deep submicron range. When the patterns are placed very close to each other, the backscattered electrons tend to

combine and the distortion in the pattern is more pronounced. This effect is known as the proximity effect in EBL. The distortion of structure due to this effect is shown in Fig. 7(b).

The electroplating of nickel was done initially in the form of growing mold structures around SU-8 structures. Fig. 8 shows nickel molds electroplated onto SU-8 structures. Submicron SU-8 molds experience a considerably severe proximity effect when compared to the submicron SU-8 structure as the exposure area for the mold structure is relatively large. This makes the plating of nickel molds easier than the growth of nickel structures.

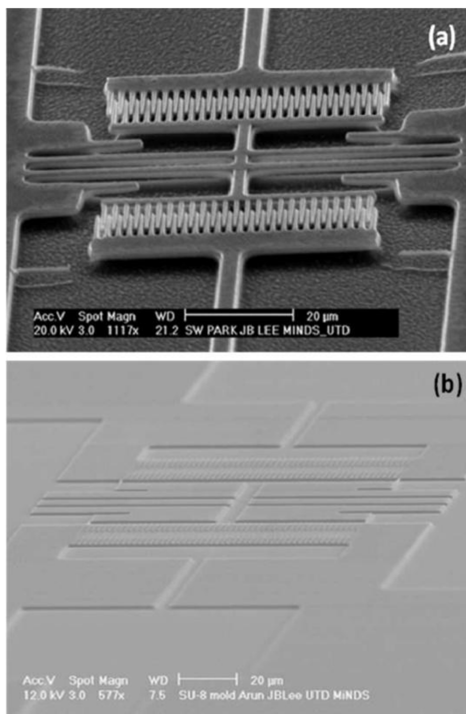

Fig. 5. Scanning electron microscopy (SEM) images of a tested comb-drive structure: (a) 1:1 aspect ratio metallic comb-drive using a PMMA mold, (b) 3:1 aspect ratio SU-8 mold.

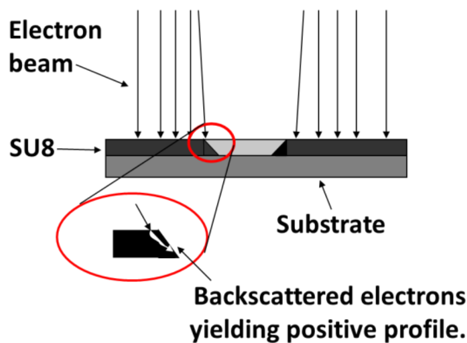

Fig. 6. A schematic representation of an inherent positive sidewall profile in a negative photoresist due to backscattered electrons.

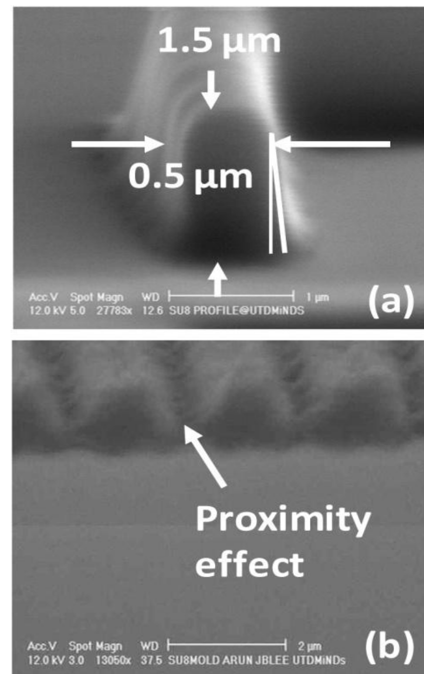

Fig. 7. Scanning electron microscopy (SEM) images showing the angled positive sidewall profile due to backscattered electrons resulting in the proximity effect.

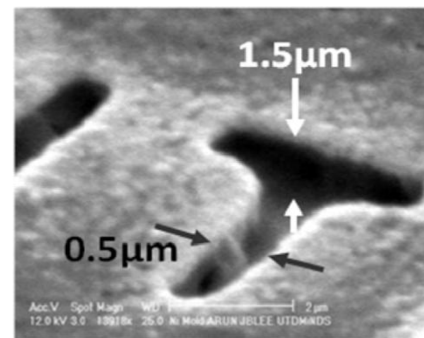

Fig. 8. Scanning electron microscopy (SEM) image of electroplated nickel using a native resist as a mold.

III. NANOIMPRINT LITHOGRAPHY ON SU-8

Although the processing time for the formation of nanoscale structures by using EBL is not prohibitively long in practice, it is desirable to have in place a fabrication process that can pattern nanoscale structures within a relatively short process time. To reduce the process time significantly, we studied an alternate method of the formation of nanoscale patterns using NIL. In this study, we used two different nano grating silicon molds, one with 10-nm-wide grating and 100-nm pitch distance and the other with 20-nm-wide grating and 200-nm pitch distance. Note that the minimum feature size studied for the NIL is

approximately 50 times smaller (from 500 nm in EBL to 10 nm in NIL), but the NIL allows a limited density range as it requires a good gap between the features (100-nm pitch distance in our case).

The fabrication was started by spin-coating a layer of 450-nm-thick SU-8 on an oxidized silicon wafer with 2-μm oxide, and the SU-8 layer was imprinted with a 20-nm-wide, 200-nm-pitch nano grating mold. 200-Å-thick Cr was evaporated on the imprinted SU-8 gratings at an oblique angle of 35°. Plasma etching in oxygen was carried out at 5 mTorr with an inductively coupled plasma (ICP) power of 300 W and a bias power of 100 W for 60 seconds to etch the exposed SU-8. The Cr layer was etched using a Cr etchant (a solution of 20 g:20 g:100 mL of $NaOH:K_3Fe(CN)_6:H_2O$). Fig. 9 shows a scanning electron microscopy (SEM) image of the cross-section of the imprinted and etched SU-8 structure, showing 30:1 aspect ratio 15- to 20-nm-wide openings at the top. During plasma etching, there was a slight widening of the nanoscale patterns beyond the dimension of the top opening in the imprinted SU-8. However, due to the highly directional nature of ICP etching, the transferred dimension at the bottom of the trench was not significantly different from the topmost dimension.

Although 30:1 aspect ratio nanoscale patterning with a minimum feature size of 15–20 nm was successful, we investigated further to find a recipe for achieving sub-10-nm patterns. In order to make sub-10-nm-wide opening patterns, a method involving the evaporation of a metal at oblique angles of more than 35° or the use of a relatively thick metal mask could be used. In our study, another alternative method was investigated. An additive 45-nm-thick SiO_2 layer was evaporated on the imprinted and etched SU-8 membrane at an oblique angle of 35°. Fig. 10 shows an SEM image of the cross section of the imprinted and etched SU-8 membrane with 45-nm-thick SiO_2 at the top, showing the smallest dimension down to 7-nm-wide openings. However, the dimension varies from one region to another and a reproduction of the nano slots with uniform openings at the top of the etched SU-8 membrane was practically impossible because the metal or dielectric material evaporation rate in the electron-beam evaporator was not uniform. Nonetheless, this study demonstrated nanoscale patterns with aspect ratios of up to 64:1 (thickness=450 nm; minimum feature size=7 nm).

Since the additive layer of the oblique-angle deposited SiO_2 was not uniform, a further study was carried out to find a method of realizing uniform sub-10-nm patterns in SU-8. To make uniform sub-10-nm-wide openings, the 10-nm-wide, 100-nm-pitch nano grating silicon mold was fabricated and tested. First, SU-8 2000.5 and SU-8 2000 thinners were mixed in a 4:1 ratio. A layer of 300-nm-thick diluted SU-8 was spun on an oxidized silicon wafer with 2-μm buried oxide and imprinted with the 10-nm-wide, 100-nm-pitch nano grating mold. Cr was evaporated on the imprinted SU-8 gratings at an oblique angle of 35°. Then, oxygen plasma etching was carried out at 5 mTorr with an ICP power of 300 W and a bias power of 100 W for 55 seconds, and a Cr layer was etched using a Cr etchant. Fig. 11 shows an SEM image of the cross section of the imprinted and etched SU-8 membrane showing 8- to 12-nm-wide openings at the top resulting in aspect ratios of up to 37.5:1. Like the 20/200-nm mold NIL, this 10/100-nm mold NIL was reproducible and consistently led to the sub-10-nm patterning result with a minimum variation of sizes.

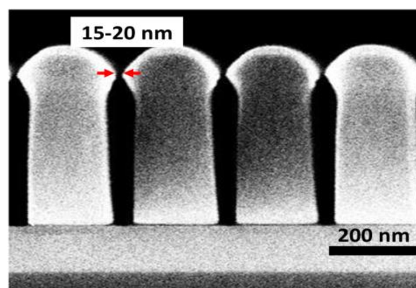

Fig. 9. Scanning electron microscopy (SEM) image of the cross section of nanoimprinted, ICP- patterned 450-nm-thick 30:1 aspect ratio SU-8 with an oblique metallic mask. ICP: inductively coupled plasma.

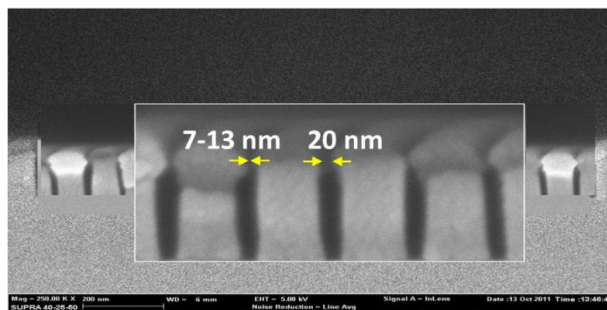

Fig. 10. Scanning electron microscopy (SEM) image of the cross section of nanoimprinted, ICP-patterned 450-nm-thick SU-8 coated with a 45-nm SiO_2 layer by evaporation at an oblique angle of 35°. ICP: inductively coupled plasma.

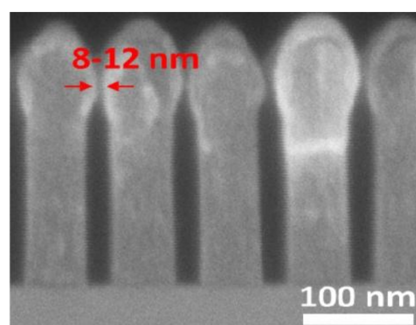

Fig. 11. Scanning electron microscopy (SEM) image of cross section of nanoimprinted, ICP-patterned 300-nm-thick 37.5:1 aspect ratio SU-8 with an oblique metallic mask. ICP: inductively coupled plasma.

IV. CONCLUSIONS

Deep submicron to sub-10-nm feature patterning was studied on a negative tone chemically amplified photoresist using both EBL and NIL. Submicron structures with an aspect ratio of 4:1, a minimum feature size of 500 nm, and near-vertical walls were obtained using EBL. Various process condition variations for different types of patterns were discussed. In the NIL, reproducible nanoscale patterning down to 8 nm with an aspect ratio of up to 37.5:1 was demonstrated. These two methodologies show considerable potential of the nanoscale patterning ability in a negative photoresist. We believe that such methods can be widely applicable to the development of 3D FinFETs and nanoscale multifunctional devices.

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Kwangki Ryoo
was born in Kongju, Chungnam Province, Korea. He received his B.S., M.S., and Ph.D. in Electronics Engineering from Hanyang University, Seoul, Korea, in 1986, 1988, and 2000, respectively. He is currently a full professor with the Department of Information and Communication Engineering, Hanbat National University, Daejeon, Korea. From 1991 to 1994, he was a full-time lecturer in the Department of Electronics Engineering at the Korea Military Academy (KMA) and worked in the System IC Design team at Electronics and Telecommunications Research Institute (ETRI) from 2000 to 2002. From 2010 to 2011, he was a visiting scholar at the University of Texas at Dallas, Richardson, Texas, USA. His research interests include engineering education, SoC platform design and verification, hardware/software co-design, and multimedia codec design.

Jeong Bong Lee
was born in Seoul, Korea. He received his B.S. in Electronics Engineering from Hanyang University, Seoul, Korea, in 1986, and his M.S. and Ph.D. in Electrical Engineering from Georgia Institute of Technology, Atlanta, Georgia, USA, in 1993 and 1997, respectively. He is currently a full professor with the Department of Electrical Engineering, The University of Texas at Dallas, Richardson, Texas, USA. His current research interests include MEMS and nanophotonics. Dr. Lee received the U.S. National Science Foundation CAREER Award in 2001. He served as an executive program sub-committee member for the Transducers 2011 Conference and a program committee member for the Transducers 2013 and IEEE Sensors Conferences. Dr. Lee also serves as an editorial board member for Micromachines Journal, The Scientific World Journal, and Journal of Nanotechnology and Smart Materials.