# **Anisotropic Wet Etching of Si as a Fabrication Tool Enabling 3-D Microphotonics Structures and Devices**

Grant W. Bidney,<sup>1,2</sup> Boya Jin,<sup>1</sup> Lou Deguzman,<sup>1</sup> Thomas C. Hutchens,<sup>1</sup> Joshua M. Duran,<sup>2</sup> Gamini Ariyawansa,<sup>2</sup> Igor Anisimov,<sup>2</sup> Nicholaos I. Limberopoulos,<sup>2</sup> Augustine M. Urbas,<sup>3</sup> Kenneth W. Allen,<sup>4</sup> Sarath D. Gunapala,<sup>5</sup> and Vasily N. Astratov<sup>1,2,\*</sup>

<sup>1</sup>Department of Physics and Optical Science, Center for Optoelectronics and Optical Communications,
University of North Carolina at Charlotte, Charlotte, NC 28223-0001, USA

<sup>2</sup>Air Force Research Laboratory, Sensors Directorate, Wright Patterson AFB, OH 45433, USA

<sup>3</sup>Air Force Research Laboratory, Materials and Manufacturing Directorate, Wright-Patterson AFB, OH 45433, USA

<sup>4</sup>Advanced Concepts Laboratory, Georgia Tech Research Institute, Georgia Institute of Technology, Atlanta, GA 30332, USA

<sup>5</sup>Jet Propulsion Laboratory, California Institute of Technology, 4800 Oak Grove Dr., Pasadena, California 91030, USA

\*Tel: 1 (704) 687 8131, Fax: 1 (704) 687 8197, E-mail: astratov@uncc.edu

Abstract— It is shown that due to high speed and large volume of production, anisotropic wet etching of Si is a unique method of manufacturing high quality micropyramidal arrays for silicon photonics. Four types of arrays were fabricated: (i) square pyramids with the 54.7° slope of the sidewall surface, (ii) microcones with the 45° slope of sidewall surface, (iii) inverted square pyramids with 54.7° slope, and (iv) inverted triangular pyramids with different angles of the sidewalls. It is shown that the first type of arrays has a capability to produce tightly focused mid-wave infrared (MWIR) beams at the tips of the pyramids. Other arrays can be also used as light concentrators and various multifunctional microphotonic components.

Keywords—silicon photonics, infrared imaging, light concentrators, focal plane arrays

## I. INTRODUCTION

In recent years, there appeared two proposals of light-concentrating structures based on using direct [1] or inverted [2] Si micropyramidal arrays where the sidewall surface was covered with a thin layer of metal. Concentration of energy in such structures is provided due to adiabatic compression of plasmons towards the apex of pyramids. These structures can be used for coupling electromagnetic energy into extremely compact photodetectors to increase the photon collection efficiency in infrared focal plane arrays (FPAs). These structures already demonstrated almost up to a hundred times enhanced photoresponse in comparison with the conventional flat designs of FPAs.

At the same time, light concentrating properties were proposed and realized using purely dielectric components such as microspheres [3-6] or microcones [7-12] placed in contact with the photodetectors. Similar to the case of plasmonic concentrators, the technology of anisotropic wet etching of Si has been found to be particularly attractive for fabricating high-index microcones [9-11]. Besides increased photon collection efficiency, these structures can resonantly trap light inside the microcones leading to strong spectral peaks in sensitivity of photodetectors integrated with such microcones. It can be used for increasing the sensitivity, reducing the size and, consequently, the thermal noise of uncooled mid-wave infrared (MWIR) imaging devices [13, 14].

Most of these structures and designs are based on using anisotropic wet etching of Si as the main fabrication tool. This technology has been studied by the microelectromechanical systems (MEMS) community for several decades [15-20]. Many properties of this technological process have been understood. These include such factors as the crystallographic orientation of the wafer ((100), (110) or (111)), use of different Si etchants (tetramethylammonium hydroxide (TMAH) or KOH) and photoresists, and use of surfactants reducing the undercutting of the photoresist caps (Triton X-100). However, the central effort was on the fabrication of structures for MEMS applications. The optical applications were relatively limited and, as a result, the capabilities of this technology for fabricating light concentrators, back reflectors, diffractive gratings and other functional photonic microstructures have not been systematically studied in the previous work.

In this work, we developed fabrication of four types of Si structures for photonic applications by using anisotropic wet etching: (i) square pyramids with the 54.7° slope of the sidewall surface illustrated in Figs. 2(a, b); (ii) microcones with the 45° slope of the sidewalls illustrated in Figs. 2(c, d); (iii) inverted square pyramids with 54.7° slope illustrated in Figs. 2(e, f); (iv) inverted triangular pyramids with different angles of the sidewalls illustrated in Figs. 2(g, h). Using MWIR laser illumination, we experimentally demonstrate that the micropyramidal arrays of the type (i) have a capability to tightly focus light at the smaller bases of the pyramids. However, the efficiency and concentration factors require further studies. The fabricated structures can be integrated with the photodetector FPAs. It can be achieved either by depositing such metals as Ni or Au to form metal/silicide Schottky barrier photodetectors for operation in SWIR or MWIR ranges, respectively [21, 22]. Alternatively, such structures can be heterogeneously integrated with the frontilluminated FPAs fabricated in various material systems with high quantum yield [13, 23]. Fabricated high-index periodic mesoscale structures can in principle possess interesting reflection/diffraction properties [26]. The microcones can potentially be used as contact superlenses similar to the case of high-index microspheres [27-29], however all these possibilities require further studies.

### II. FABRICATION

The microstructures all follow similar fabrication steps, but differ due to mask alignment, type of photoresist used, and whether or not the surfactant Triton X-100 was used, which helps reduce the undercutting of the silica mask during anisotropic wet etching with TMAH. Fig. 1 shows the photolithography pattern used to create the four different microstructures represented in the four rows of Fig. 2.

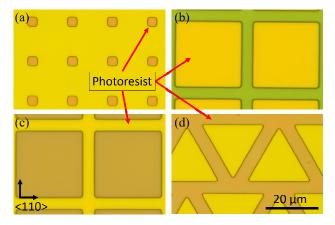


Fig. 1. Photolithography patterns of (a) 3.6  $\mu$ m squares with 15.0  $\mu$ m pitch, (b) 25.5  $\mu$ m squares with 30.0  $\mu$ m pitch, (c) 4.5  $\mu$ m square grid patterns with 30.0  $\mu$ m pitch, and (d) 4.5  $\mu$ m horizontal and diagonal stripes forming triangles with 24.0  $\mu$ m side length with the inverted triangles occurring every 30.0  $\mu$ m pitch.

The microstructures were fabricated on single-side polished (100) n-type Si wafers with 300 nm thermal silica on both sides. First, the wafers were cleaned with isopropyl alcohol and a dehydration bake on a hot plate for 75 seconds at 115° C was performed. In order to apply positive photoresist, the adhesion promoter hexamethyldisilazane (HMDS) was spin coated at 4000 RPM for 45 seconds with a ramp rate of 400 RPM/s and then baked for 75 seconds at 115° C to ensure a direct transfer of the photoresist pattern later in the fabrication. Afterwards, the same procedure was repeated to spin coat Microposit S1813 positive photoresist (Fig. 1(a, b, d)). In order to apply negative photoresist, no adhesion promoter was used but instead NR9-1500 negative photoresist was spin coated with the same specifications (Fig. 1(c)). The wafers were then patterned through photolithography, and developed with CD-26 for positive photoresist and with RD6 for negative photoresist. Next, the wafers were placed in an O2 plasma cleaner for 4 minutes to remove any photoresist that may remain after developing. Afterwards, the pattern was transferred to the silica layer by placing the wafers in a 10:1 buffered oxide etchant (BOE) bath for 7.5 minutes. These steps were completed in order to fabricate the four different 3-D Si structures shown in the four different rows of Fig. (2).

The different 3-D microstructures were then ready to be fabricated through anisotropic wet etching. The fabricated microstructures differ due to mask alignment, type of photoresist used, and whether or not Triton X-100 was used.

The pattern in Fig. 1(a) was defined with S1813 positive photoresist and etched with 200 ml of 25% TMAH plus 0.4 ml Triton X-100 at 80° C for 25 minutes to obtain Fig. 2(a-b).

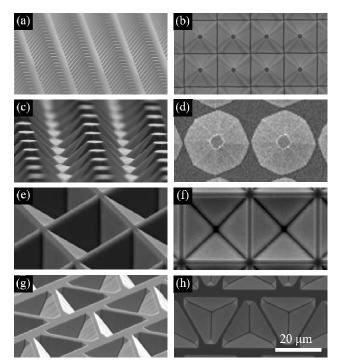


Fig. 2. SEM images of 2.4  $\mu m$  top and 15.0  $\mu m$  pitch Si micropyramids with 54.7° sidewall angles taken from (a) angled and (b) top-down views. SEM images of 7.3  $\mu m$  top and 30.0  $\mu m$  pitch Si microcones with ~45° sidewall angles taken from (c) angled and (d) top-down views. SEM images of 2.0  $\mu m$  bottom and 30.0  $\mu m$  pitch inverted Si square micropyramids with 54.7° sidewall angles taken from (e) angled and (f) top-down views. SEM images of 23.3  $\mu m$  long and 20.7  $\mu m$  wide Si triangular micropyramids with the one side aligned to the <110> direction having 54.7° sidewall angle and with the two sides not aligned to the <110> direction having ~48° sidewall angles, taken from (g) angled and (h) top-down views.

The pattern in Fig. 1(b) was defined with S1813 positive photoresist and etched with 200 ml of 25% TMAH at 80° C for 20 minutes to obtain Fig. 2(c-d).

The pattern in Fig. 1(c) was defined with NR9-1500 negative photoresist and etched with 200 ml of 25% TMAH at 80° C for 20 minutes to obtain Fig. 2(e-f).

The pattern in Fig. 1(d) was defined with S1813 positive photoresist and etched with 200 ml of 25% TMAH plus 0.4 ml Triton X-100 at 80° C for 25 minutes to obtain Fig. 2(g-h).

The microstructures in Fig. 2(a-b, e-f) all have  $54.7^{\circ}$  sidewall angles due to etching along the (111) planes, while the microstructures in Fig. 2(c-d) have  $\sim 45^{\circ}$  sidewall angles. Also, the microstructures in Fig. 2(g-h) have one side aligned to the <110> direction with  $54.7^{\circ}$  sidewall angles, and two sides not aligned to the <110> direction with  $\sim 48^{\circ}$  sidewall angles.

Thus, the performed studies showed multiple advantages of anisotropic wet etching for application in silicon photonics: a) parallel fabrication of large-scale 3-D arrays of direct or inverted micropyramidal arrays takes just few minutes, b) different sidewall slops (54.7° and 45°) can be realized for arrays fabricated on (100) wafer, and c) Very smooth sidewall surface can be obtained for 54.7° slopes that can reduce scattering losses in various photonic devices.

### III. OPTICAL CHARACTERIZATION

Optical characterization was performed for the micropyramidal arrays of the first type illustrated in Figs. 2(a, b). A typical array with  $\sim\!\!11~\mu m$  top and 15.0  $\mu m$  pitch is illustrated in Fig. 3(b). The purpose of such optical characterization was to observe the spatial patterns of light scattered by the tips of the micropyramids and to study the theoretically predicted [9] light concentrating properties of the micropyramids.

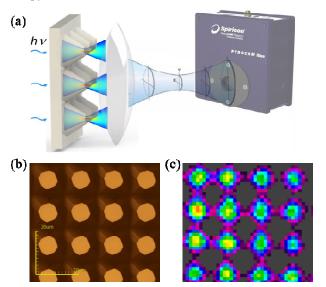


Fig. 3. (a) Diagram depicting the setup to examine the microstructures by illuminating with a 2.96  $\mu m$  wavelength Sheaumann laser, where the light is then collected by the Spiricon camera. (b) 3-D confocal microscope image of  $\sim\!\!11~\mu m$  top with 15.0  $\mu m$  pitch micropyramids. (c) Spiricon camera image of the same micropyramids showing potential for the microstructure to be used for light concentrating purposes.

The experimental setup is illustrating in Fig. 3(a). The array with Si micropyramids was illuminated through an unpolished surface of the wafer using a collimated broad beam (~2 mm aperture) produced by the Er:YAG Sheaumann laser with  $\lambda =$ 2.96 µm wavelength. Due to the small absorption of Si in MWIR range, the light traverses the Si slab without significant absorption losses and reaches the micropyramids from their wider bases, as schematically illustrated in Fig. 3(a). Propagation of the beams inside the micropyramids can be rather complicated and can have resonant properties [9]. However, these processes were outside the scope of our studies since we performed characterization at a single wavelength. The beams transmitted through the narrower bases of the micropyramids became visible due to light scattering at the tips of the micropyramids. The pattern of scattered light in this plane was observed by a Spiricon camera using a CaF2 lens, as also schematically illustrated in Fig. 3(a).

The direct visualization of the transmitted beams is illustrated in Fig. 3(c). It is seen that the transmitted beams are centered with the axis of the micropyramids and that they are approximately diffraction-limited with full width at the half maximum (FWHM)  $\sim \lambda/(2NA) \sim 4$  µm, where NA is a numerical aperture of the lens. Further studies of these effects

using an objective with stronger magnification (to reduce the image pixilation introduced by the Spiricon camera) and larger NA are required to reveal the minimal FWHMs for the transmitted beams. Along with the minimization of the spot sizes, another important parameter is represented by the "power enhancement factor" [9]. It is determined as a ratio of the power measured by the compact photodetector with and without the light-concentrating microstructure. More detailed studies of these microstructure properties will be performed in our future work.

#### IV. CONCLUSIONS

Our results show that due to high speed and large production volume, anisotropic wet etching of Si is a unique method for producing high quality micropyramidal arrays for photonics applications. We demonstrated four different types of arrays including direct and inverted micropyramids with different cross sections and slopes of the sidewalls. Our research was driven by recent proposals of light-concentrating microstructures for enhancing the performance of MWIR FPAs [1,2, 7-12]. We experimentally demonstrated that one of the fabricated arrays formed by direct square pyramids with the 54.7° slope of the sidewall surface has light concentrating properties which are found to be in qualitative agreement with the theoretical modeling results [9]. However, further studies are required to establish quantitative agreement with the theory.

Besides the applications as light-concentrating microstructures, the fabricated arrays should possess interesting reflection/diffraction properties which can have some analogies with the properties of deeply etched photonic crystal structures [26]. Due to a complex interrelationship between the focusing and imaging applications, one can also suggest that in the mesoscale regime (characteristic dimensions comparable to the wavelength of light), high-index micropyramids placed in contact with the objects can operate as contact superlens in superresolution imaging applications similar to the case of high-index microspheres [27-29]. These possibilities show that anisotropic wet etching of Si can be an extremely useful tool for the fabrication of new structures for applications as light-concentrating structures, superlenses, diffraction gratings, and other devices.

### ACKNOWLEDGMENT

This work was supported by Center for Metamaterials, an NSF I/U CRC, award number 1068050. Part of this work was carried out at the Jet Propulsion Laboratory, California Institute of Technology, under a contract with the National Aeronautics and Space Administration (80NM0018D0004).

## REFERENCES

- B. Desiatov, I. Goykhman, N. Mazurski, J. Shappir, J. Khurgin, and U. Levy, "Plasmonic enhanced silicon pyramids for internal photoemission Schottky detectors in the near-infrared regime," Optica 2, 335-338 (2015).
- [2] H.-J. Syu, H.-C. Chuang, M.-J. Lin, C.-C. Cheng, P.-J. Huang, and C.-F. Lin, "Ultra-broadband photoresponse of localized surface plasmon

- resonance from Si-based pyramid structures," Photonics Res. 7(10), 1119–1126 (2019).
- [3] V. N. Astratov, K. W. Allen Jr., N. I. Limberopoulos, A. Urbas, and J. M. Duran, "Photodetector focal plane array systems and methods," patent application14/587,068 filed on 12/31/2014. U.S. patent 9,362,324 (7 June 2016).
- [4] K. W. Allen, F. Abolmaali, J. M. Duran, G. Ariyawansa, N. I. Limberopoulos, A. M. Urbas, and V. N. Astratov, "Increasing sensitivity and angle-of-view of mid-wave infrared detectors by integration with dielectric microspheres," Appl. Phys. Lett. 108(24), 241108 (2016).
- [5] F. Abolmaali, A. Brettin, A. Green, N. I. Limberopoulos, A. M. Urbas, and V. N. Astratov, "Photonic jets for highly efficient mid-IR focal plane arrays with large angle-of-view," Opt. Express 25(25), 31174–31185 (2017).
- [6] A. Brettin, F. Abolmaali, N. I. Limberopoulos, A. Green, I. Anisimov, A. M. Urbas, and V. N. Astratov, "Towards fabrication of mid-IR FPAs with enhanced sensitivity and reduced dark current by using integration with microspherical arrays," NAECON 2018 IEEE National Aerospace and Electronics Conference, 2018, pp. 533-535, doi: 10.1109/NAECON.2018.8556727.
- [7] V. N. Astratov, A. Brettin, N. I. Limberopoulos, and A. Urbas, "Photodetector Focal Plane Array systems and methods based on microcomponents with arbitrary shapes," 06/13/2017. U.S. patent 10,585,238 (March 10, 2020).
- [8] A. Brettin, N. I. Limberopoulos, I. Anisimov, A. M. Urbas and V. N. Astratov, "Microconical arrays as novel light-concentrating structures for enhancing sensitivity, angle-of-view, and reducing dark current of mid-IR FPAs," NAECON 2018 IEEE National Aerospace and Electronics Conference, 2018, pp. 496-498, doi: 10.1109/NAECON.2018.8556800.
- [9] B. Jin, G. Bidney, A. Brettin, N. Limberopoulos, J. Duran, G. Ariyawansa, I. Anisimov, A. Urbas, S. Gunapala, H. Li, and V. Astratov, "Microconical silicon mid-IR concentrators: spectral, angular and polarization response," Opt. Express 28, 27615-27627 (2020).
- [10] B. Jin, G. W. Bidney, N. I. Limberopoulos, J. M. Duran, G. Ariyawansa, I. Anisimov, A. M. Urbas, S. D. Gunapala, and V. N. Astratov, "Design of Si Micro-Cone Light Concentrators for Heterogeneous Integration with MWIR FPAs," 2020 22nd International Conference on Transparent Optical Networks (ICTON), 2020, Tu.B7.5, doi: 10.1109/ICTON51198.2020.9203159.
- [11] B. Jin, G. W. Bidney, A. Brettin, N. I. Limberopoulos, I. Anisimov, A. M. Urbas, H. Li, and V. N. Astratov, "High-index micro-cones for focusing and concentrating light in MWIR focal plane arrays," 2019 IEEE National Aerospace and Electronics Conference (NAECON), 2019, pp. 665-668, doi: 10.1109/NAECON46414.2019.9057919.
- [12] B. Jin, A. Brettin, G. W. Bidney, N. I. Limberopoulos, J. M. Duran, G. Ariyawansa, I. Anisimov, A. M. Urbas, K. W. Allen, S. D. Gunapala, and V. N. Astratov, "Light-harvesting microconical arrays for enhancing infrared imaging devices: Proposal and demonstration", Appl. Phys. Lett. 119, 051104 (2021).
- [13] A. Rogalski, "Progress in focal plane array technologies," Prog. Quantum Electron. 36(2-3), 342–473 (2012).

- [14] S. Zhang, A. Soibel, S. A. Keo, D. Wilson, S. B. Rafol, D. Z. Ting, A. She, S. D. Gunapala, and F. Capasso, "Solid-immersion metalenses for infrared focal plane arrays," Appl. Phys. Lett. 113(11), 111104 (2018).
- [15] I. Zubel and I. Barycka, "Silicon anisotropic etching in alkaline solutions I. The geometric description of figures developed under etching Si (100) in various solutions," Sens. Actuators, A 70(3), 250– 259 (1998).
- [16] H. Lu, H. Zhang, M. Jin, T. He, G. Zhou, and L. Shui, "Two-Layer Microstructures Fabricated by One-Step AnisotropicWet Etching of Si in KOH Solution," Micromachines 7(2), 19 (2016).
- [17] P. Pal and K. Sato, "A comprehensive review on convex and concave corners in silicon bulk micromachining based on anisotropic wet chemical etching," Micro and Nano Syst. Lett. 3(1), 6 (2015).
- [18] O. Tabata, R. Asahi, H. Funabahi, K. Shimaoka, and S. Sugiyama, "Anasotropic etching of silicon in TMAH solutions," Sens. Actuators, A 34(1), 51–57 (1992).
- [19] Y. Fan, P. Han, P. Liang, Y. Xing, Z. Ye, and S. Hu, "Differences in etching characteristics of TMAH and KOH on preparing inverted pyramids for silicon solar cells," Appl. Surf. Sci. 264, 761–766 (2013).
- [20] X. Chen, R. S. Patel, J. A. Weibel, and S. V. Garimella, "Coalescence-induced jumping of multiple condensate droplets on hierarchical superhydrophobic surfaces," Sci. Rep. 6(1), 18649 (2016).
- [21] J. Duran and A. Sarangan, "Schottky-barrier photodiode internal quantum efficiency dependence on nickel silicide film thickness," IEEE Photonics J. 11(1), 1–15 (2019).
- [22] J. Duran, "Silicon-based infrared photodetectors for low-cost imaging applications," Ph.D. thesis (University of Dayton, 2019). Available at: <a href="https://etd.ohiolink.edu/pg\_10?::NO:10:P10\_ETD\_SUBID:179778">https://etd.ohiolink.edu/pg\_10?::NO:10:P10\_ETD\_SUBID:179778</a>
- [23] C. L. Tan and H. Mohseni, "Emerging technologies for high performance infrared detectors," Nanophotonics 7(1), 169–197 (2018).
- [24] T. Sherlock, A. Nasrullah, J. Litvinov, E. Cacao, J. Knoop, S. Kemper et al., "Suspended, micron-scale corner cube retroreflectors as ultra-bright optical labels," J. Vac. Sci. Technol. B Nanotechnol Microelectron. 29 (2011), doi:10.1116/1.3656801.
- [25] B. C. Park, T. B. Eom, and M. S. Chung, "Polarization properties of cube-cornerretroreflectors and their effects on signal strengthand nonlinearity in heterodyne interferometers," Appl. Optics 35(22), 4372-4380 (1996).
- [26] AD Bristow, DM Whittaker, VN Astratov, MS Skolnick, A Tahraoui, TF Krauss, M Hopkinson, MP Croucher, and GA Gehring, "Defect states and commensurability in dual-period Al x Ga 1- x As photonic crystal waveguides," Phys. Rev. B 68(3), 033303 (2003).
- [27] A.V. Maslov and V.N. Astratov, "Optical nanoscopy with contact Mieparticles: Resolution analysis," Appl. Phys. Lett. 110(26), 261107 (2017).
- [28] A.V. Maslov and V.N. Astratov, "Resolution and reciprocity in microspherical nanoscopy: point-spread function versus photonic nanojets," Phys. Rev. Appl. 11(6), 064004 (2019).
- [29] Label-free super-resolution microscopy, Ed. V. Astratov (Springer, Cham, 2019).