

Interfacial Symmetry Controls Transparency and Thermal Stability in Nb/ZrO₂/Nb Josephson Junctions

CNF Project Number: 2091-11

Principal Investigator(s): Gregory David Fuchs

User(s): Thomas Varghese

Affiliation: Department of Applied and Engineering Physics and Department of Materials Science and Engineering, Cornell University

Primary Source(s) of Research Funding: Air Force Office of Scientific Research

Contact: gdf9@cornell.edu, tv92@cornell.edu

Research Group Website: <https://fuchs.research.engineering.cornell.edu/>

Primary CNF Tools Used: AJA sputter deposition system; GCA 6300 DSW 5X g-line wafer stepper; PT770 etcher; Oxford 81 etcher; SC4500 even-hour evaporator; DISCO dicing saw

Abstract:

Niobium-based Josephson junctions are attractive for superconducting electronics because niobium has a large superconducting energy gap and high critical temperature relative to aluminum. Their performance, however, is highly sensitive to the chemistry and structure of the ultrathin tunnel barrier. We investigate how interface termination controls transport and thermal stability in niobium/zirconium oxide/niobium junctions. Symmetric junctions containing zirconium at both barrier interfaces are compared with asymmetric junctions containing zirconium at only one interface. Measurements across multiple junction diameters, chips, and fabrication batches showed that symmetric barriers have lower resistance-area product and higher transparency. Sequential annealing further shows that oxidation completeness is an important determinant of thermal stability.

Summary of Research:

Josephson-junction properties depend exponentially on the thickness, composition, and uniformity of the tunnel barrier. Conventional aluminum oxide barriers provide excellent performance but limit the use of higher-critical-temperature superconductors and can undergo oxygen redistribution during processing. Zirconium oxide provides an alternative crystalline barrier for refractory niobium junctions [1]. Our objective is to determine whether the interface materials of the tunnel barrier can be used to control transparency without changing the nominal zirconium oxide deposition.

Fabrication:

The Josephson junctions are fabricated from Nb|Zr|O₂|Nb multilayers deposited by sputtering. Symmetric junctions are produced by adding a 1 nm zirconium

layer at the upper zirconium oxide interface, forming an Nb|Zr|O₂|Zr|Nb structure, while the remaining deposition and fabrication steps are unchanged from Ref. [1]. For this analysis, the nominal zirconium layer that is oxidized to create a barrier thickness is 3 nanometers. Circular area junctions with diameters ranging from 2 to 9 micrometers were patterned using the GCA 6300 DSW 5X g-line wafer stepper. The junction mesas were defined by chlorine-based inductively coupled plasma etching in the PT770 etcher. After mesa formation, a silicon dioxide spacer layer was deposited using the SC4500 evaporator.

The upper wiring layer was subsequently deposited by AJA 1. The completed wafers were diced using the DISCO dicing saw and electrical measurements were performed using a four-probe probestation.



(a) Symmetric junction

(b) Asymmetric junction

Figure 1: Symmetric and asymmetric niobium/zirconium oxide/niobium Josephson-junction architectures.

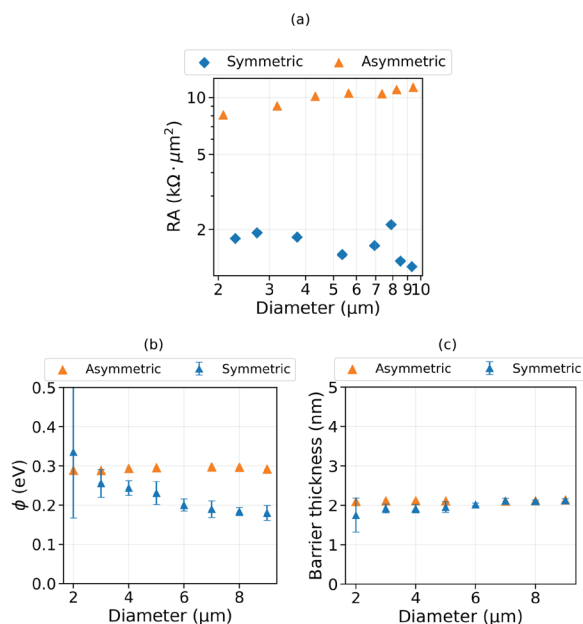


Figure 2: Comparison of room-temperature transport parameters for symmetric and asymmetric junctions: (a) resistance-area product, (b) effective barrier height, and (c) effective barrier thickness as functions of junction diameter.

Four-probe room-temperature measurements revealed a reproducible reduction in the resistance-area product of the symmetric junctions relative to the asymmetric junctions (Fig. 2a). Tunnelling parameters were extracted by fitting the low-bias current–voltage characteristics using the Simmons tunnelling model [2] and are shown in Figures 2b and 2c. The fitted effective barrier height was approximately 0.2 electronvolts for symmetric junctions and approximately 0.3 electronvolts for asymmetric junctions.

Low-temperature measurements below 20 mK confirmed that the room-temperature difference persists in the superconducting state. Symmetric junctions exhibit larger critical-current and lower normal-state resistance, together with stronger subgap conduction (Fig. 3). Reproducible subgap features in several symmetric devices are consistent with multiple Andreev reflection [3], although the limited number of resolved orders requires a conservative assignment.

Junction type	J_c (A/cm ²)	R_n (Ω)	Δ/Δ_{Nb}
Symmetric (3nm, 6μm)	57.67	55	0.62
Symmetric (5nm, 6μm)	35.4	72	0.67
Asymmetric (3nm, 6μm)	1.45	862	0.96

Figure 3: Comparison of critical-current density (J_c), normal-state resistance (R_n), and normalized superconducting energy gap (Δ/Δ_{Nb}) for symmetric and asymmetric junction architectures.

Thermal stability is evaluated by comparing 3 nm symmetric and asymmetric junctions. The asymmetric architecture showed smaller changes in low-bias resistance and fitted barrier parameters over the measured range. These observations show that interface termination and oxidation completeness must be considered together when designing a transparent and thermally robust barrier.

Conclusions and Future Steps:

Symmetric zirconium termination provides a strong means of increasing transparency in Nb|Zr|O₂|Zr|Nb Josephson junctions. This behavior could result from a modified oxygen distribution across the barrier. Annealing measurements further show that junction stability depends on the amount of residual zirconium available for continued oxidation. Future work will quantify microwave loss and determine whether the improved direct-current transport can be translated into low-dissipation superconducting circuits.

References:

- [1] J. Choi et al., “Low barrier ZrOx-based Josephson junctions,” *APL Materials* 13, 111103 (2025).
- [2] J. G. Simmons, “Generalized formula for the electric tunnel effect between similar electrodes separated by a thin insulating film,” *Journal of Applied Physics* 34, 1793–1803 (1963).
- [3] M. Octavio, M. Tinkham, G. E. Blonder, and T. M. Klapwijk, “Subharmonic energy-gap structure in superconducting constrictions,” *Physical Review B* 27, 6739–6746 (1983).

Co-Fabricated Diamond Acoustic Resonators for Nitrogen-Vacancy Spin Control and Rotation Sensing

CNF Project Number: 2126-12

Principal Investigator(s): Greg Fuchs

User(s): Chao Shang, Anuj Ashok

Affiliation(s): Applied and Engineering Physics, Cornell University, Electrical and Computer Engineering, Purdue University

Primary Source(s) of Research Funding: Defense Advanced Research Projects Agency

Contact: gdf9@cornell.edu, cs2485@cornell.edu, ashok12@purdue.edu

Primary CNF Tools Used: AJA Sputter 1; OEM M1 Aluminum Nitride Sputter System; HB16 Semi-Automatic Wire Bonder

Abstract:

This work co-fabricates high-overtone bulk acoustic resonators (HBARs) and film bulk acoustic resonators (FBARs) on a single-crystal diamond chip to provide complementary acoustic drive channels for a nitrogen-vacancy (NV) center ensemble. The HBAR mechanically drives the NV electron-spin double-quantum transition, while the FBAR is designed for future spectrally selective control of the three nitrogen-14 hyperfine sublevels in a nuclear-spin-based gyroscope. The report describes fabrication of a multi-geometry resonator die at the Cornell NanoScale Science and Technology Facility and the Birck Nanotechnology Center, together with one-port reflection measurements from 0.5 to 5.0 GHz. The HBAR exhibits a dense comb of sharp resonance dips that reach -18.3 dB, whereas the FBAR exhibits a weaker and sparser response over the same frequency range. These contrasting spectra are consistent with the different acoustic cavities formed by the two device geometries on the shared diamond substrate.

Summary of Research:

Nitrogen-vacancy centers in diamond are optically addressable solid-state spin qubits. Their ground-state electron-spin sublevels, $m_s = 0, +1$, and -1 , are coherently controlled by microwave magnetic fields, acoustic strain, or a combination of both. Acoustic driving directly addresses the $m_s = -1$ to $+1$ double-quantum transition, which is inaccessible to a conventional magnetic-dipole microwave drive [1]. Each nitrogen-vacancy center also contains a nitrogen-14 nuclear spin with $I = 1$. Hyperfine coupling splits each electron-spin transition into three lines. Because the nuclear spin can retain coherence much longer than the electron spin, it provides an attractive sensing degree of freedom for rotation measurements based on geometric phase accumulation [2,3].

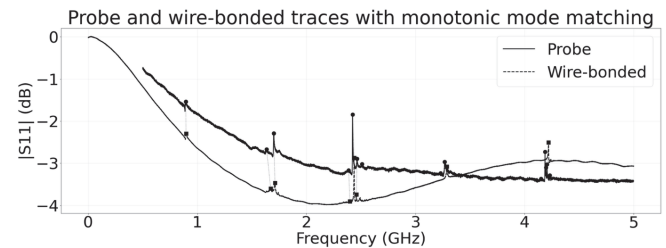


Figure 1: One-port S_{11} magnitude from 0.5 to 5.0 GHz for an FBAR device on the co-fabricated diamond chip before and after gold wire bonding.

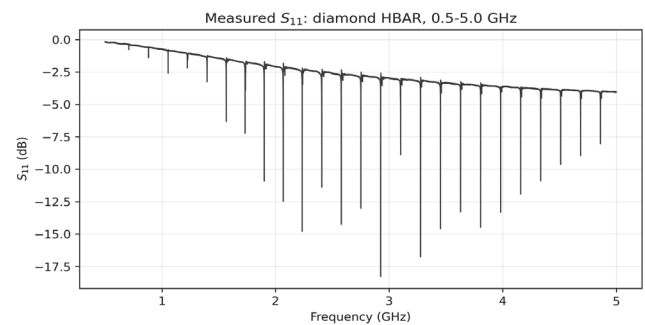


Figure 2: One-port S_{11} magnitude from 0.5 to 5.0 GHz for an HBAR device on the same diamond chip. The spectrum contains a dense comb of high-overtone resonances under a 50-ohm calibrated measurement.

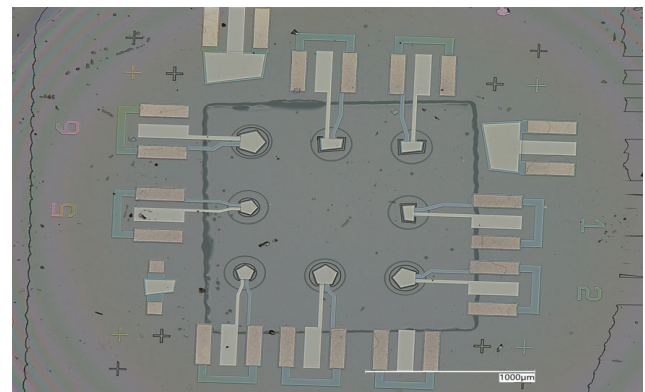


Figure 3: Optical micrograph of the fabricated diamond die. Multiple co-fabricated FBAR and HBAR electrode geometries connect through independent coplanar routing traces to bond pads at the die periphery. The scale bar is 1000 micrometers.

The diamond die integrates HBAR and FBAR structures side by side. In the HBAR geometry, a thin aluminum nitride transducer excites acoustic modes through the full diamond thickness, which acts as a Fabry-Perot cavity. In the FBAR geometry, the acoustic mode is more strongly localized near the transducer. Both device types use a 1 micrometer aluminum nitride piezoelectric film between a continuous bottom ground electrode and a patterned top signal electrode. The die includes tapered fan-shaped, pentagonal, and compact rounded top-electrode geometries. Independent coplanar traces connect each device to peripheral bond pads for individual electrical probing.

The fabrication workflow is divided between two facilities. At the Cornell NanoScale Science and Technology Facility, the AJA Sputter 1 deposits the metal electrode layers, and the OEM M1 Aluminum Nitride Sputter System deposits the 1 micrometer piezoelectric film. Photolithography, pattern transfer, and dry etching are completed at the Birck Nanotechnology Center at Purdue University. Figure 3 shows the completed diamond die, including the co-fabricated electrode geometries, coplanar feed traces, and bond pads. Device packaging uses the HB16 semi-automatic wire bonder with gold wire. Figure 3 shows the devices after the wire bounding.

One-port S11 reflection measurements are performed from 0.5 to 5.0 GHz with a vector network analyzer after a full one-port calibration. Figure 1 compares the FBAR response before and after wire bonding. The spectrum contains a small number of relatively shallow resonance features on a comparatively smooth background. Figure 2 presents the HBAR response over the same frequency range. The HBAR produces a dense sequence of sharp resonance dips that reach -18.3 dB, consistent with the high overtone density of a full-thickness diamond acoustic cavity. Measurements from the two device types use the same die and comparable calibration conditions, which supports comparison of the effects of electrode and cavity geometry.

Conclusions and Future Work:

The co-fabricated HBAR and FBAR devices show distinct one-port acoustic responses that support their complementary roles. The HBAR provides a dense set of mechanical resonances for electron-spin double-quantum control, while the FBAR provides a more localized platform for hyperfine-selective nuclear-spin control. The nitrogen-14 hyperfine lines are separated by approximately 2.16 MHz for a single-quantum transition and approximately 4.32 MHz for a double-quantum transition. Ongoing work characterizes the

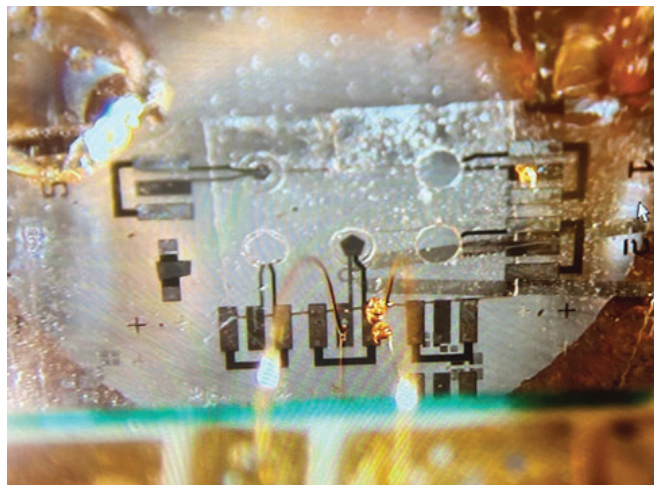


Figure 4: Optical micrograph of the wire bounded devices.

target FBAR mode, including its resonance frequency, -3 dB bandwidth, and stability, to determine whether it supports selective addressing of individual hyperfine lines or deliberate single-tone coverage of the full hyperfine manifold. Subsequent experiments integrate the selected resonator mode with optical nitrogen-vacancy initialization and readout for on-chip rotation sensing.

References:

- [1] E. R. MacQuarrie, T. A. Gosavi, N. R. Jungwirth, S. A. Bhawe, and G. D. Fuchs, "Mechanical spin control of nitrogen-vacancy centers in diamond," *Physical Review Letters* 111, 227602 (2013).
- [2] E. R. MacQuarrie, T. A. Gosavi, S. A. Bhawe, and G. D. Fuchs, "Continuous dynamical decoupling of a single diamond nitrogen-vacancy center spin with a mechanical resonator," *Optica* 2, 233 (2015).
- [3] M. P. Ledbetter et al., "Gyroscopes based on nitrogen-vacancy centers in diamond," *Physical Review A* 86, 052116 (2012).

Advancing the Sequence-defined Polypeptoid EUV Resist Platform: Multi-site Decarboxylation and Metal Sensitization

CNF Project Number: 2733-18

Principal Investigator(s): Christopher Ober

User(s): Chenyun Yuan

Affiliation: Department of Materials Science and Engineering, Cornell University

Primary Source(s) of Research Funding: Department of Energy

Contact: cko3@cornell.edu, cy479@cornell.edu

Research Group Website: <https://ober.mse.cornell.edu/>

Primary CNF Tools Used: JEOL JBX-6300FS E-beam Lithography System, JEOL JBX-9500FS E-beam Lithography System, ASML DUV Stepper, AFM - Veeco Icon, Zeiss Gemini SEM, J.A. Woollam RC2 Spectroscopic Ellipsometer

Abstract:

Our sequence-defined polypeptoids function as single-component, PAG-free negative-tone resists under EUV and electron-beam (e-beam) exposure, with patterning driven by decarboxylation of the peptoid chain by loss of carboxylic acid ($-\text{COOH}$) groups. In the original system, a single C-terminal $-\text{COOH}$ per chain, a serendipitous byproduct of solid-phase submonomer synthesis, served as the solubility-switch group, and the fully organic composition limited absorption at EUV wavelength. This year we extended the platform along two directions. First, sequence-controlled synthesis was used to install multiple $-\text{COOH}$ groups at defined positions along the chain, transforming the $-\text{COOH}$ from a synthesis byproduct into rationally placed solubility-switch sites. Residual gas analysis of five multi- $-\text{COOH}$ decamer sequences under EUV exposure revealed that the nominally "inactive" aromatic side groups (phenyl versus phenol) substantially modulate decarboxylation, and that $-\text{COOH}$ groups at the C-terminal, mid-chain, and N-terminal positions react with distinct efficiencies. Second, high-Z metal elements were incorporated by strategies including physical blending of metal salts, and a sequence-controlled chelation strategy in which 8-hydroxyquinoline submonomers coordinate Zn(II) or Sn(II) into dimeric metal-peptoid complexes. EUV reflectometry confirmed the expected enhancement in linear attenuation, with the magnitude trending with the atomic photoabsorption cross-section of the bound metal. Together, these directions yield a set of design rules for next generation of polypeptoid EUV resists.

Summary of Research:

Our previous studies indicated that the molecular homogeneity of the polypeptoid platform means that the loss of a single functional group per chain produces a detectable, uniform solubility contrast, and that every

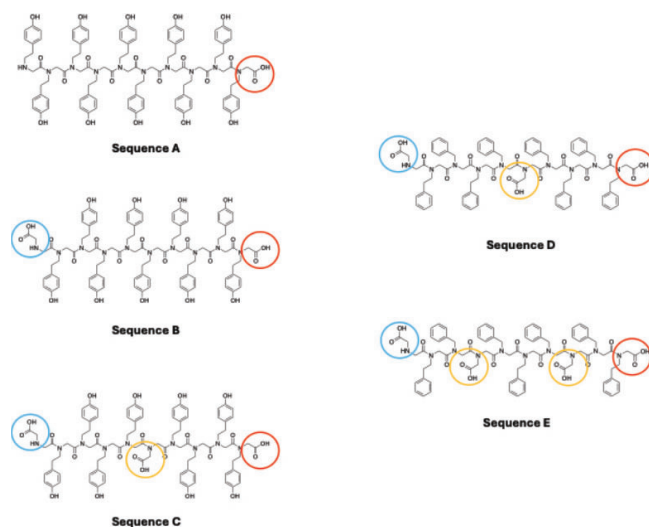


Figure 1: Molecular structures of the five multi- $-\text{COOH}$ decamer polypeptoid sequences (A-E). Sequences A-C bear phenol side groups and Sequences D-E bear phenyl side groups; each carries one to four $-\text{COOH}$ groups distributed across the C-terminal (red), N-terminal (blue), and mid-chain (yellow) positions.

chain in the film is structurally identical, making the resist not only a candidate material but also an unusually clean model system for studying resist patterning chemistry [1]. Building on this, we placed $-\text{COOH}$ groups at chosen positions along the chain (Figure 1). Five decamer sequences (A-E) were synthesized, carrying one to four $-\text{COOH}$ groups distributed across C-terminal, N-terminal, and mid-chain positions, and built with either phenol (A-C) or phenyl (D-E) aromatic side groups. Because decarboxylation releases CO_2 , residual gas analysis (RGA) during EUV exposure provides a direct probe of the reaction, monitored through the CO_2 signal at m/z 44 (Figure 2).

Two findings emerged. First, the phenyl-containing sequences outgassed nearly an order of magnitude more CO_2 than their phenol-containing counterparts. A direct comparison of Sequences C and D, which differ only by the phenol hydroxyl, isolates this effect from

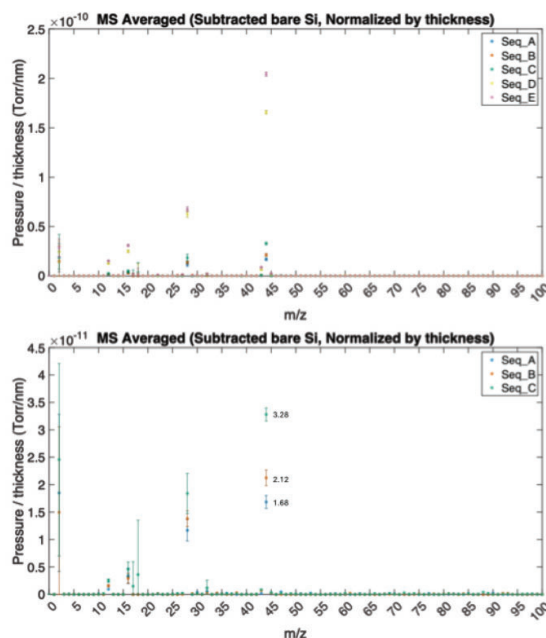


Figure 2: Thickness-normalized RGA mass spectra of outgassed species from Sequences A-E (Top) and A-C (bottom, zoomed-in) under EUV exposure.

compositional differences and points to the phenolic -OH as the likely source of suppression, plausibly through competing radical or electron/hole scavenging pathways. Second, the three types of -COOH groups decarboxylate with distinct efficiencies, in the approximate order C-terminal > mid-chain > N-terminal, as the thickness-normalized CO_2 signals across the sequences do not follow a simple stoichiometric ratio. Preliminary e-beam patterning, used here as a proxy for EUV, confirmed that these multi-COOH sequences are patternable (Figure 3), with development conditions tailored to the hydrophilicity of each sequence.

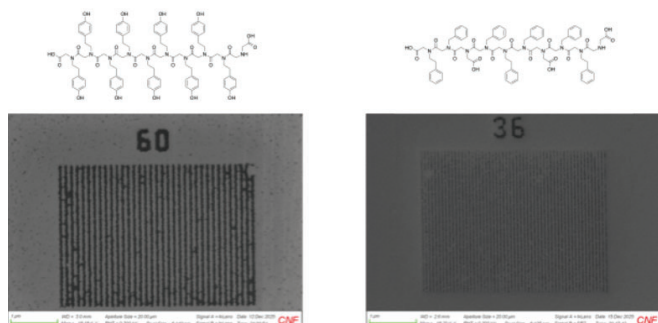


Figure 3: Preliminary e-beam patterning results from Sequences B (left) and E (right), demonstrating the patternability of multi-COOH polypeptoid sequences under e-beam exposure.

To raise EUV absorption beyond the low cross-sections of C, H, O, and N, high-Z elements were incorporated by two strategies [2]. Physical blending of ZnCl_2 with a tyramine-based hexamer at a 1 Zn : 2 -COOH ratio increased e-beam sensitivity, lowering the dose required

to pattern, as a proof of concept, though at the cost of reduced resolution and post-development surface residues, and with no control over metal placement. A sequence-controlled route was then developed using an 8-hydroxyquinoline (8-HQ) submonomer [3], a strong bidentate chelator, installed at a defined chain position. UV-Vis titration confirmed formation of $\text{M}(8\text{-HQ})_2$ complexes with $\text{Zn}(\text{II})$ or $\text{Sn}(\text{II})$, saturating at a 1:2 metal-to-peptoid ratio, which couples two chains through a single shared metal center into a dimeric architecture with the metal fixed at a sequence-defined position. EUV reflectometry at the Center for X-ray Optics (LBNL) confirmed the intended enhancement [4]: the linear attenuation coefficient at 13.5 nm rose from $4.744 \mu\text{m}^{-1}$ for the metal-free reference to $5.117 \mu\text{m}^{-1}$ for the Zn-peptoid and $5.396 \mu\text{m}^{-1}$ for the Sn-peptoid, in the order expected from the atomic photoabsorption cross-sections of the bound metals.

Conclusions and Future Steps:

These two directions extend the polypeptoid resist platform along chemistry (the number and location of reactive decarboxylation sites) and elemental composition (the placement of high-Z atoms at sequence-defined positions), while preserving the molecular precision that distinguishes it from conventional polymeric resists. Treated as model systems, they yield design rules for next-generation polypeptoid resists: which chain positions decarboxylate most efficiently, how nominally inactive side groups modulate that chemistry, and how metals can be localized at defined sites. Future work will combine sequence-defined decarboxylation sites and metal-binding sites within a single chain, and scale metal loading by installing multiple 8-HQ units to further amplify EUV absorption.

References:

- [1] Chenyun Yuan, Bernhard Lüttgenau, Honggu Im, et al. "Sequence-defined polypeptoid resists for molecularly precise stochastic control and mechanistic innovation in EUV lithography", *Proc. SPIE 13983, Advances in Patterning Materials and Processes XLIII*, 139830V (2026).
- [2] Roberto Fallica, Jarich Haitjema, Lianjia Wu, et al. "Absorption coefficient of metal-containing photoresists in the extreme ultraviolet," *Journal of Micro/Nanolithography, MEMS, and MOEMS* 17(2), 023505 (2018).
- [3] Galia Maayan, Michael D. Ward, Kent Kirshenbaum; Metallopeptoids. *Chem. Commun.* 45(1): 56–58 (2009).
- [4] Oleg Kostko, Terry R. McAfee, Jonathan Ma, et al. "Experimental characterization of model resist materials", *Proc. SPIE 11854, International Conference on Extreme Ultraviolet Lithography 2021*, 1185407 (2021).

Strain-induced Moiré Superlattice in 2D Materials Using Stressor Films

CNF Project Number: 2967-21

Principal Investigator(s): Judy Cha

User(s): Yu-Mi Wu, Sihun Lee

Affiliation: Department of Materials Science and Engineering, Cornell University

Primary Source(s) of Research Funding: The Gordon and Betty Moore Foundation's EPiQS Initiative (GBMF9062.01); Department of Energy, Basic Energy Sciences program Grant No. DE-SC0023905

Contact: jc476@cornell.edu, yw2658@cornell.edu, sl2859@cornell.edu

Research Group Website: <https://cha.mse.cornell.edu>

Primary CNF Tools Used: SC4500 Evaporator, Zeiss Supra scanning electron microscope, Nabity Nanometer Pattern Generator System, Flexus film stress measurement

Abstract:

This project developed a scalable fabrication approach for constructing strain-induced moiré superlattices in two-dimensional (2D) materials using lithographically patterned thin-film stressors. Patterned $\text{Al}_2\text{O}_3/\text{SiO}_2/\text{Al}_2\text{O}_3$ stressor films were fabricated by electron beam lithography and electron beam evaporation to introduce localized heterostrain into exfoliated 2H- MoS_2 flakes. Raman spectroscopy confirmed the transferred strain, while scanning transmission electron microscopy (STEM) and four-dimensional STEM (4D-STEM) revealed the resulting lattice reconstruction and moiré superlattice formation. The induced strain generated distinct stripe and distorted hexagonal moiré patterns depending on the local strain symmetry. This work demonstrates a reproducible fabrication process for producing and characterizing strain-engineered 2D materials [1].

Summary of Research:

Moiré superlattices in 2D materials have become an important platform for studying emergent electronic phenomena. Conventionally, moiré superlattices are created by manually twisting and stacking atomically thin layers or by using intrinsic lattice mismatch between different materials. These approaches typically require repeated fabrication attempts to achieve the desired twist angle or stacking configuration and offer limited control over the resulting moiré geometry. Developing a scalable and deterministic method for constructing moiré superlattices therefore remains an important technical challenge. In this work, we developed a fabrication approach based on lithographically patterned thin-film stressors that locally introduce heterostrain into MoS_2 , enabling formation of strain-induced moiré superlattices using standard microfabrication processes.

We fabricated samples on both SiO_2/Si substrates

and SiN_x TEM grids to investigate stressor-induced heterostrain in MoS_2 . 2H- MoS_2 bulk crystals were mechanically exfoliated using Scotch tape onto pre-cleaned SiO_2/Si substrates. Target flakes were then identified by optical microscopy and transferred onto SiN_x membrane TEM grids and SiO_2/Si substrates using a polypropylene carbonate (PPC) stamp. The TEM grids were mounted onto a custom holder and spin-coated with poly(methyl methacrylate) as the electron-beam resist. Electron-beam lithography (Nabity NPGS, Zeiss Supra SEM) was used to write $500 \times 10 \mu\text{m}$ stripe patterns with $10 \mu\text{m}$ spacing. After pattern development, a stressor film of Al_2O_3 (10 nm)/ SiO_2 (50 nm)/ Al_2O_3 (10 nm) was deposited using e-beam evaporation (SC4500 Even-hour evaporator). The residual film stress was characterized using Flexus Film Stress Measurement System, which measured a compressive stress of -105.7 MPa .

Figure 1a shows a schematic of the sample geometry, consisting of a MoS_2 flake capped with a patterned stressor film. Figure 1b shows an optical image of a representative fabricated sample. The fabricated samples were characterized using Raman spectroscopy, atomic-resolution STEM, and 4D-STEM to evaluate the strain distribution and resulting structural reconstruction. In Figure 1c and 1d, Raman measurements showed shifts in the E_{2g} and A_{1g} vibrational modes, confirming successful strain transfer from the stressor films into the underlying MoS_2 without introducing significant sample damage (Fig. 1e). To understand how deeply the applied strain penetrates into the sample, cross-sectional high-angle annular dark-field (HAADF)-STEM imaging was performed on the top few MoS_2 layers near the edge of the patterned stressor film. Figure 2a shows local stacking changes induced by the stressor, while measured Mo-Mo interatomic distances in Figure 2b reveals that the applied strain was concentrated primarily within the top two MoS_2 layers.

We next investigated the resulting structural

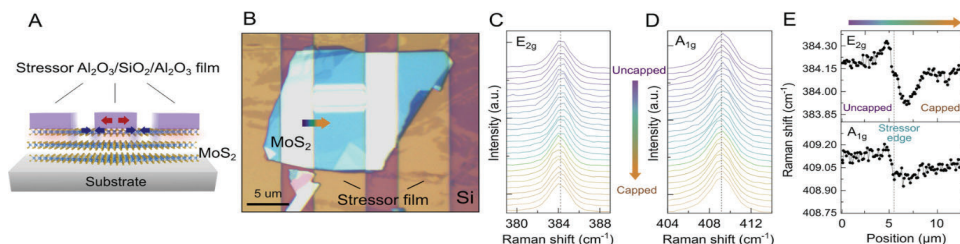


Figure 1: (A) Schematic of patterned $\text{Al}_2\text{O}_3/\text{SiO}_2/\text{Al}_2\text{O}_3$ stressor film transferring heterostrain onto MoS_2 locally. Red and blue arrows indicate the strain direction. (B) Optical image of a MoS_2 flake under the patterned film on the Si substrate. The Raman spectra shown in (C,D) come from regions indicated by the arrow. (C,D) Raman spectra of E_{2g} and A_{1g} modes of MoS_2 across the uncapped to capped regions, respectively. The spectra are plotted with a vertical offset for visual clarity, with the spectra from the uncapped region at the top and from the capped region at the bottom. (E) Corresponding E_{2g} and A_{1g} peak position profiles obtained by Lorentzian fitting.

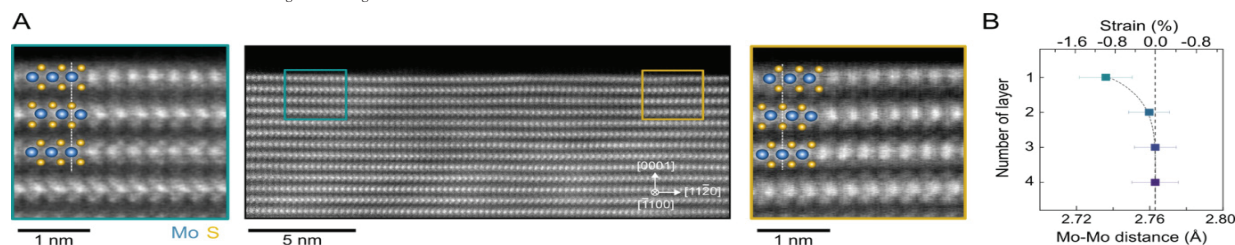


Figure 2: (A) Cross-sectional atomic-resolution HAADF-STEM images of MoS_2 under the region close to the stressor edge. The panels on the left and right show magnified views of the regions highlighted by the green and yellow boxes in the center image. (B) Layer-dependent Mo-Mo interatomic distances from the experiment obtained by quantitative atom tracking from (A).

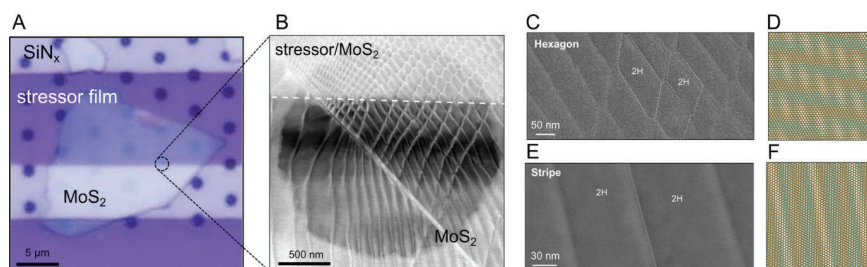


Figure 3: (A) Optical microscopy image of MoS_2 under the patterned stressor film. (B) Reconstructed virtual annular dark-field image from the 4D-STEM dataset. The image corresponds to a zoomed-in region near the stressor film edge marked in (A). (C) Plan-view low magnification HAADF-STEM image of distorted hexagonal moiré with domain boundaries highlighted by white dashed lines. (D) Schematic of rigid moiré pattern generated by biaxial and shear heterostrain. (E) Low-magnification HAADF-STEM image of stripe-type moiré with domain boundaries highlighted by white dashed lines. (F) Schematic of rigid moiré pattern generated by uniaxial and shear heterostrain.

reconstruction in plan view. Figure 3a shows an optical image of the sample on a TEM grid. Figure 3b presents a reconstructed virtual annular dark-field (ADF) STEM image from the 4D-STEM dataset, focusing on the region near the edge of the patterned stressor film where the strain variations are most pronounced. Two distinct types of strain-induced moiré patterns are observed. Near the stressor edge, where the applied strain is relatively biaxial, a distorted hexagonal moiré pattern forms (Figures 3c and 3d). Farther away from the stressor, the strain becomes predominantly uniaxial, and the moiré structure gradually evolves into a stripe pattern (Figures 3e and 3f). This spatial transition demonstrates that the local symmetry of the applied strain determines the resulting moiré geometry.

Conclusions and Future Steps:

We demonstrated a scalable and deterministic approach for constructing heterostrain-induced moiré superlattices in MoS_2 using lithographically patterned stressor films. By combining Raman spectroscopy, atomic-resolution STEM, and 4D-STEM, we resolved the induced strain, lattice reconstruction, and resulting moiré geometries. This scalable fabrication strategy provides a practical route for engineering strain-controlled moiré superlattices in 2D materials. Future work will extend this approach to other layered materials and stressor designs to achieve greater control over moiré geometries and their associated electronic properties.

References:

- [1] Y.-M. Wu et al. Proceedings of the National Academy of Sciences 123, e2602234123 (2026).

Area Selective Deposition

CNF Project Number: 2990-21

Principal Investigator(s): James R. Engstrom

User(s): James T. Jensen

Affiliation: Chemical Engineering, Cornell University

Primary Source(s) of Research Funding: Department of Energy

Contact: jre7@cornell.edu, jtj33@cornell.edu

Primary CNF Tools Used: Woollam RC2 Spectroscopic Ellipsometer, VCA Contact Angle, Oxford FlexAL

Abstract:

In this work, we aim to further develop the fundamental framework behind area selective deposition (ASD), extending results for blocking Al_2O_3 atomic layer deposition (ALD) on SiO_2 to a new system blocking SiO_2 deposition on Al_2O_3 . We compare results for two Al precursors and two blocking molecules selective to SiO_2 with results for a new Si precursor and a blocking molecule selective to Al_2O_3 .

Summary of Research:

We are extending our previous efforts in ASD by investigating a new Si precursor and a blocking molecule selective to Al_2O_3 . Our earlier work involved depositing Al_2O_3 on metal surfaces while blocking growth on SiO_2 , and we have now moved to depositing SiO_2 on metal surfaces while blocking growth on Al_2O_3 .

We operate a custom-built deposition reactor employing a quartz crystal microbalance (QCM) as a deposition monitor in situ and in real time [1–3]. For the Al_2O_3 deposition results, we prepared 20 nm of SiO_2 on Au QCM crystals in the CNF to create a starting surface for blocking molecules to selectively cover. The two blocking molecules in use in the blocked Al_2O_3 deposition system were (N,N-dimethylamino)trimethylsilane (DMATMS) in the vapor phase and octadecyltrichlorosilane (ODTS) in a liquid solution phase. We confirmed the changes in surface chemistry by taking water contact angle images of SiO_2 surfaces before and after formation of these blocking layers. After forming the layers, we conducted ALD cycles to compare with untreated SiO_2 . For Al_2O_3 deposition, we employed trimethylaluminum (TMA) and bisdimethylamino-diamino-Aluminum (BDMADA-Al) as thin film precursors and H_2O as the co-reactant. Most deposition studies were completed at $T = 120^\circ\text{C}$, with one at $T = 285^\circ\text{C}$. Figure 1 shows QCM data collected during deposition of the Al_2O_3 films from TMA on both unblocked and blocked SiO_2 , while Figure 2 shows similar experiments with the sterically bulkier BDMADA-Al as the thin film precursor. We

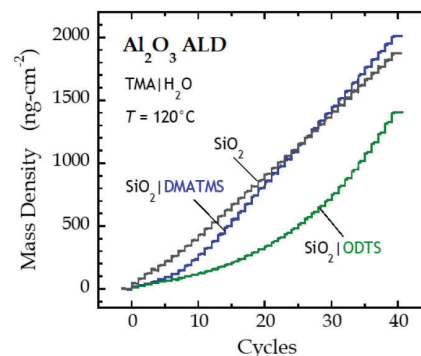


Figure 1: QCM data showing mass densities vs number of cycles for Al_2O_3 using TMA-Al/ H_2O process chemistry at $T = 120^\circ\text{C}$ on SiO_2 crystals passivated with either DMATMS or ODTS.

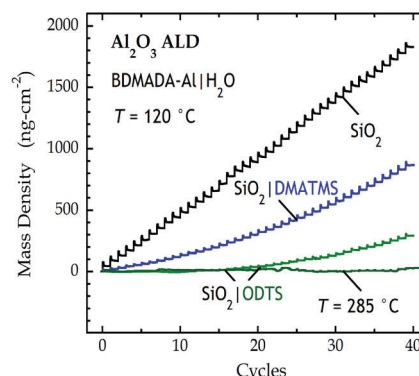


Figure 2: QCM data showing mass densities vs number of cycles for Al_2O_3 using BDMADA-Al/ H_2O process chemistry at $T = 120^\circ\text{C}$ on SiO_2 crystals passivated with either DMATMS or ODTS. One experiment on ODTS was conducted at $T = 285^\circ\text{C}$.

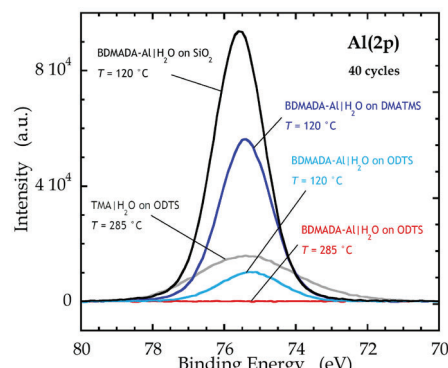


Figure 3: X-ray photoelectron detailed spectra of the Al(2p) region on SiO_2 coupons for a variety of ALD process chemistries and blocking molecules.

have also included data for one experiment at $T = 285^\circ\text{C}$ for BDAMDA-Al deposition on ODTs in Figure 2. We show for both precursors that ODTs blocks deposition more effectively than DMATMS due to enhanced steric hindrance between the larger blocking molecule and the precursor. Similarly, we show that BDMADA-Al provides a deposition system which is easier to block than TMA for Al_2O_3 deposition. We show X-ray photoelectron spectra of the Al(2p) region of samples after different ASD strategies for Al_2O_3 in Figure 3, showing agreement with the QCM traces. Notably, there is no deposition detected with BDMADA-Al on ODTs at $T = 285^\circ\text{C}$.

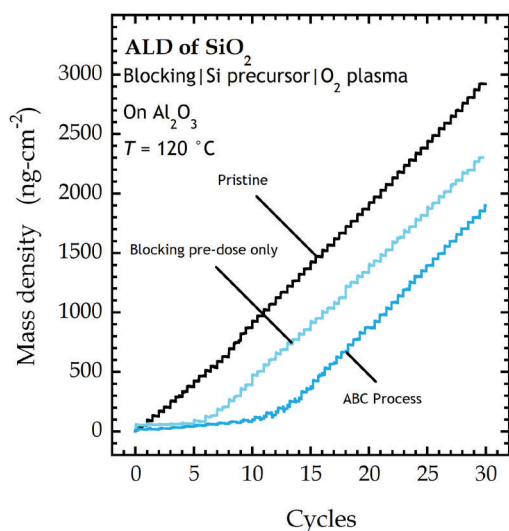


Figure 4: QCM data showing mass densities vs number of cycles for SiO_2 using a new Si precursor and O_2 plasma process chemistry at $T = 120^\circ\text{C}$ on Al_2O_3 crystals passivated with an Al_2O_3 blocking molecule. The ABC process involves a re-dose of the Al_2O_3 blocking molecule for every cycle.

For the SiO_2 deposition system, we started with a CNF-prepared 20 nm thick Al_2O_3 surface and a selective blocking molecule referred to here as the Al_2O_3 blocking molecule. We used a new Si precursor containing only Si-N and Si-H bonds, with no Si-C or Si-Si bonds. The co-reactant was O_2 plasma. Figure 4 shows initial results for SiO_2 deposition blocked on an Al_2O_3 surface. The Al_2O_3 blocking molecule is removed or the layer is damaged by the fifth cycle, after which deposition begins. To extend blocking performance, we show an ABC dosing sequence including a re-dose of the Al_2O_3 blocking molecule after each O_2 plasma dose. As can be seen, blocking is extended to ten cycles. The plasma-activated SiO_2 deposition process exhibits a different type of blocking breakdown than is seen with the thermally activated Al_2O_3 deposition process due to the more aggressive and high-energy nature of plasma processes.

Conclusions and Future Steps:

We show here that steric hindrance serves as a good predictor for blocking performance on non-growth surfaces in ASD systems, considering both Al_2O_3 deposition blocked on SiO_2 surfaces and SiO_2 deposition blocked on Al_2O_3 surfaces. We have exhibited initial results with a new Si precursor, showing blocked growth of the first ten cycles of ALD even in a plasma process. Future work involves tuning reactor conditions, examining new blocking molecules for use with the same Si precursor and O_2 plasma system, and further *ex situ* characterization of the resulting films on both growth and non-growth surfaces.

References:

- [1] J. V. Swarup, H.-R. Chuang, J. T. Jensen, J. Gao, A. L. You, and J. R. Engstrom, "Nonpyrophoric alternative to trimethylaluminum for the atomic layer deposition of Al_2O_3 ," *J. Vac. Sci. Technol. A*, 43(2), 022404 (2025), doi: 10.1116/6.0004171.
- [2] J. V. Swarup, H.-R. Chuang, A. L. You, and J. R. Engstrom, "Effect of Co-Reactants on Interfacial Oxidation in Atomic Layer Deposition of Oxides on Metal Surfaces," *ACS Appl. Mater. Interfaces* 16(13), 16983–16995 (2024), <https://doi.org/10.1021/acsami.3c19033>.
- [3] J. V. Swarup, R. T. Mercogliano, J. T. Jensen, G. Chheda, R. A. Jr. Distasio, and J. R. Engstrom, "ALD with Alternative Coreactants: Which Work, Which Do Not, and Why," *J. Phys. Chem. C* 130(33), 11570–11584 (2026), doi: <https://doi.org/10.1021/acs.jpcc.6c01772>&ref=pdf.

Initiated CVD (iCVD) Polymerization in the LC to Synthesize Polymer Particles

CNF Project Number: 3177-24

Principal Investigator(s): Rong Yang

User(s): Shiqi Li; Soumyamouli Pal

Affiliation: Smith School of Chemical and Biomolecular Engineering, Cornell University

Primary Source(s) of Research Funding: National Science Foundation

Contact: ryang@cornell.edu, sl2869@cornell.edu; sp2476@cornell.edu

Primary CNF Tools Used: Leica 300 CPD

Abstract:

In our prior work, we have demonstrated that the morphology of shape-controlled polymer particles of DVB is governed by the LC-polymer interfacial energies and the LC elastic energy. However, the full spectrum of morphologies that can be accessed by iCVD-in-LC remains to be fully understood. Here, we address the objective of synthesizing polymeric morphologies with tailored chemistries by using a new monomer, glycidyl methacrylate (GMA). One of the technical bottlenecks of characterizing the products is removing the LC before using SEM. To keep the morphology of polymer products and remove LC at the same time, we use Critical point drying to obtain the polymers after deposition.

Summary of Research:

GMA monomer: We performed iCVD polymerization reactions into the E7 LC template in its nematic phase using GMA as the monomer and TBPO as the initiator. Two deposition conditions were explored in detail, with varying monomer and initiator partial pressures. After crosslinking the linear polymer materials in LC, we used critical point drying (in Figure 1) to remove the LC and maintained most of the polymers deposited in LC. Then we used SEM to observe material structures. Templated by elasticity of LC, the polymer formed nanoparticle assemblies (Figure 2) with nanoscale diameter and microscale length.

Conclusions and Future Steps:

Anisotropic nanoparticle assemblies of pGMA were obtained through the iCVD-in-LC process. This morphology is different from the pDVB microparticles in our previous work. This gave us more information about polymeric morphologies with tailored chemistries that could be obtained in LC.

References:

[1] Jain et al., Sci. Adv. 10, eadp5573 (2024).

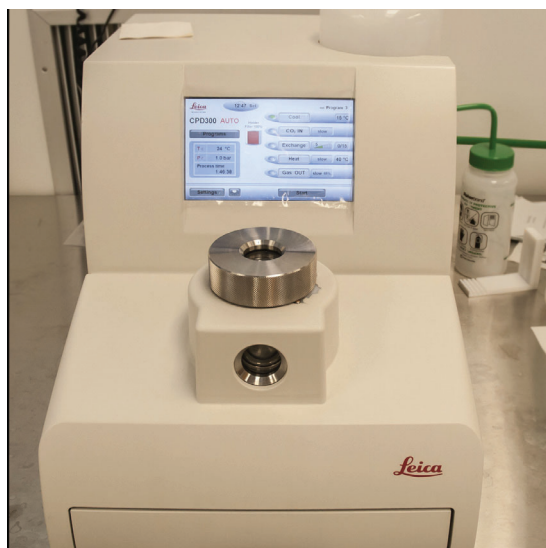


Figure 1: Photo of critical point drying.

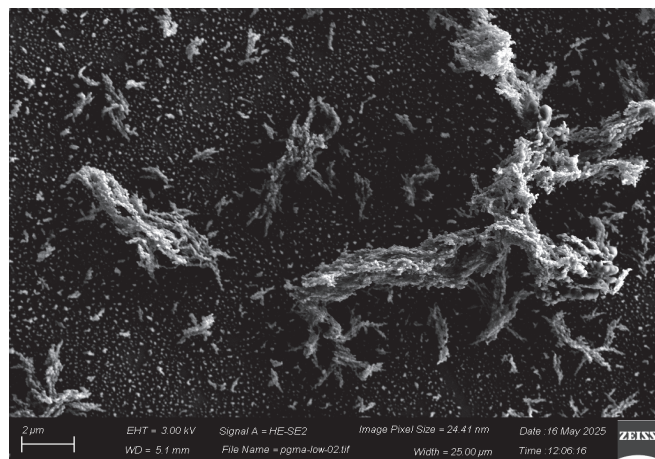


Figure 2: SEM image of pGMA material obtained in LC.

Magnon-Exciton Coupling in the Van der Waals Antiferromagnet CrSBr

CNF Project Number: 3184-24

Principal Investigator(s): Youn Jue Bae

User(s): Jiacheng Tang, Silin Guo

Affiliation: Department of Chemistry and Chemical Biology

Primary Source(s) of Research Funding: Department of Energy

Contact: yb293@cornell.edu, jt925@cornell.edu, sg2628@cornell.edu

Research Group Website: <https://bae-lab-at-cornell.org/>

Primary CNF Tools Used: AJA Sputter 1; YES HMDS Oven; Heidelberg Instruments DWL 2000; ABM aligner; PT770 Etcher; DISCO Dicing Saw

Abstract:

Hybrid devices combining magnetic materials with superconducting microwave resonators provide a platform for quantum transduction, sensing, and nonlinear magnonics[1–3]. In this project, we developed niobium coplanar waveguide resonators for microwave excitation of magnons in the layered antiferromagnetic semiconductor CrSBr. The ultimate design target is a high-quality-factor resonator operating near 30 GHz, corresponding to the zero-field magnon frequency range of the CrSBr samples studied. Resonator performance was optimized by varying the niobium-film thickness and device geometry, including the signal-line width, dielectric-gap width, and signal-line pattern.

The fabricated devices currently exhibit resonances near 20 GHz. A dielectric-gap-to-signal-width ratio of approximately 2:1 produced suitable microwave impedance and strong magnetic-field concentration near the resonator gaps. Field-dependent microwave measurements revealed anticrossings between the resonator and CrSBr magnon modes, demonstrating magnon–photon coupling. The resonator platform also enables optical measurements of the driven magnetic response through the excitonic properties of CrSBr.

Summary of Research:

Hybrid magnetic devices enable coupling between spin dynamic and photon in microwave resonators[1–3]. Such systems are promising for microwave-to-spin transduction, studies of coherent light–matter interaction, magnetic sensing, and the development of hybrid quantum technologies. CrSBr is particularly useful for this purpose because it is a layered 2D magnetic semiconductor with both strong magnetic excitations and an optical response that is sensitive to its magnetic state. This combination allows the same magnon dynamics to be investigated through microwave transmission and optical measurements.

The objective of this project was to fabricate superconducting coplanar waveguide resonators that efficiently excite CrSBr magnons while remaining compatible with optical access to the CrSBr flake. The resonator frequency must overlap with a magnon branch to produce measurable coupling. Because the zero-field magnon response of the studied CrSBr samples occurs in the tens-of-gigahertz range, the long-term design goal is a resonator frequency near 30 GHz. The present devices were designed near 20 GHz so that the magnon frequency could be tuned through the resonator frequency using an external magnetic field.

Niobium coplanar waveguide resonators with different film thicknesses and geometries were fabricated using CNF facilities. The fabrication process included niobium-film preparation, lithographic patterning of the microwave circuit, plasma etching of the niobium, and characterization of the resulting film and device dimensions. Several geometric parameters were varied, including resonator length, signal-line width, dielectric-gap width, and the shape of the signal-line pattern.

The resonator length was selected to set the fundamental microwave mode near 30 GHz. A dielectric-gap-to-signal-width ratio of approximately 2:1 was used to maintain impedance matching near 50 ohms while producing a strong microwave magnetic field in the dielectric-gap regions. Electromagnetic simulations were used to evaluate the resonant frequency, microwave-field distribution, and expected overlap between the resonator field and the CrSBr sample. These simulations showed that the strongest microwave magnetic field is concentrated near the dielectric gaps. CrSBr flakes were therefore positioned to maximize their overlap with these regions.

The fabricated resonators were characterized using a vector network analyzer. Magnetic-field-dependent microwave transmission measurements were performed in a Physical Property Measurement System. When a

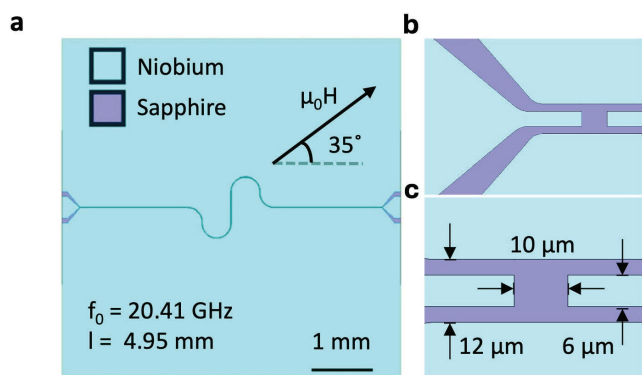


Figure 1: Design and HFSS simulation of the on-chip microwave resonator. Geometry of the on-chip microwave resonator[4].

field-tunable CrSBr magnon mode approached the resonator frequency, the microwave transmission spectrum showed an anticrossing rather than a simple crossing. This spectral splitting indicates hybridization between the magnon and microwave-photon modes and demonstrates that the on-chip niobium resonator can effectively drive magnons in relatively thick CrSBr flakes.

The devices were also designed to support optical access to the CrSBr sample. Optical measurements provide information that is not available from microwave transmission alone. A vector network analyzer measures the spatially averaged response of the entire hybrid device, whereas the excitonic optical response of CrSBr can probe the locally driven magnetic dynamics. Differential-reflectance measurements can therefore be used to determine where the resonator most strongly excites the material and how the optical response changes when the microwave frequency overlaps with a magnon resonance.

CNF fabrication was essential for producing reproducible superconducting structures with controlled film thicknesses, micron-scale gaps, and accurately defined resonator lengths. These capabilities increased the microwave magnetic-field amplitude at the CrSBr position and improved the measurable magnon response. The fabricated platform connects superconducting microwave-circuit engineering with spatially resolved optical detection of spin dynamics in a van der Waals magnetic semiconductor.

Conclusions and Future Steps:

The fabricated niobium coplanar resonators successfully excited magnons in thick CrSBr samples, and field-dependent microwave transmission measurements revealed anticrossings consistent with magnon–photon coupling. The results show that resonator geometry and sample placement can be engineered to enhance the microwave magnetic field interacting with CrSBr.

Future work will optimize the geometry and configuration to increase sensitivity to thinner CrSBr flakes and lower spin densities. Combining these improved resonators with spatially resolved optical measurements will enable quantitative imaging of microwave-driven magnon dynamics.

References:

- [1] A. Osada, Phys. Rev. Lett. 116, 223601 (2016), DOI 10.1103/PhysRevLett.116.223601.
- [2] D. Lachance-Quirion, Y. Tabuchi, A. Gloppe, K. Usami, Y. Nakamura, Appl. Phys. Express 12, 070101 (2019).
- [3] Y. J. Bae, J. Wang, A. Scheie, J. Xu, D. G. Chica, G. M. Diederich, J. Cenker, M. E. Ziebel, Y. Bai, H. Ren, C. R. Dean, M. Delor, X. Xu, X. Roy, A. D. Kent, X. Zhu, Nature 609, 282 (2022).
- [4] J. Tang, A. Singh, N. J. Brennan, D. G. Chica, Y. Li, X. Roy, F. Rana, Y. J. Bae, Nano Lett. 25, 10912 (2025).

Silicon Interposer for Millimeter-Wave Heterogeneous Integration: Doped vs High Resistivity Substrates

CNF Summer Student: Aditya Chembravattom

Student Affiliation: Department of Chemical Engineering, University of Florida

Summer Program(s): 2026 Cornell NanoScale Facility Research Experience for Undergraduates (CNF REU) Program

Principal Investigator(s): James C. M. Hwang; Department of Materials Science and Engineering, Cornell University

Mentor(s): Jin Hong Joo

Primary Source(s) of Research Funding: SUPREME, Grant No. NNCI-2025233

Contact: jch263@cornell.edu, jj593@cornell.edu, ad.chembravattom@ufl.edu

Summer Program Website(s): <https://www.cnf.cornell.edu/education/reu/2026>

Primary CNF Tools Used: SUSS MA6-BA6 Contact Aligner, AJA Sputter Deposition, UNAXIS 770 Deep Silicon Etcher, Veeco Savannah ALD, ReynoldsTech Cu ECD Hood, DC Probe Station, Microwave Small-Signal Probe Station

Abstract:

As the demand for heterogeneous-integrated RF chips increases, interposers for millimeter-wave circuits using through-silicon vias (TSVs) have become increasingly important due to their low loss and high-power capacity even at extremely high frequencies. For frequencies above 110 GHz, substrate-integrated waveguides (SIWs) are small enough to be integrated in Si interposers for high-power interconnects.

They can also be used to form high-quality passive devices such as filters and antennas, which have been difficult to integrate on-chip. This enables system-on-chip. In this study, we investigate the fabrication of SIWs and grounded coplanar waveguides (GCPWs) in Si interposers with a thickness of around 200 μm with resistivities ranging from 10-10000 $\Omega\cdot\text{cm}$. Thin Si wafers were patterned and etched using the Bosch deep reactive ion etching (DRIE) process to create TSVs. The TSVs were then metallized with platinum (Pt) using atomic layer deposition (ALD) and filled with copper (Cu) using electroplating deposition, before patterning the frontside.

DC measurements confirm a TSV series resistance of less than 1 Ω . Small-signal millimeter-wave-on-wafer measurements show that the coplanar interconnects fabricated on high-resistivity (HR, $\rho > 1000 \Omega\cdot\text{cm}$) Si have an insertion loss of 0.7 dB/mm at 40 GHz, an order of magnitude better than the same coplanar interconnects fabricated on doped Si ($\rho > 10 \Omega\cdot\text{cm}$).

Summary of Research:

Si is the most extensively used material in semiconductor devices due to its exceptional electrical and mechanical properties, including a high dielectric constant, electrical resistivity, breakdown strength, and low loss tangent. These characteristics, and its cost effectiveness,

make it an attractive candidate for SIWs. However, its relatively low mechanical toughness and high thermal conductivity compared to materials like silicon carbide (SiC) pose challenges during processing, particularly in etching processes. Our group has previously demonstrated SiC as a viable substrate material for SIW processing, and we adapted the same methodology as a proof of principle for Si SIW fabrication.

To develop a processing recipe for Si-based SIWs, we used a thinned HR Si wafer with a thickness of approximately 200 μm and resistivity greater than 1000 $\Omega\cdot\text{cm}$. Building on a similar methodology used for SiC SIWs while taking advantage of the more advanced etching processes available for Si, we began by depositing a 50 nm layer of aluminum oxide (Al_2O_3) on the wafer's backside using AJA Sputter deposition. Al_2O_3 was selected as the etch mask due to its excellent masking performance in the Bosch DRIE process for Si, providing a high selectivity greater than 1:1000.

The DRIE was performed using the UNAXIS 770 Deep Silicon Etcher. The etching chemistry employed C_4F_8 and SF_6 , which react with Si to anisotropically form vias. As shown in Figure 1, this process yielded 200 μm -deep TSVs with a diameter of 50 μm and

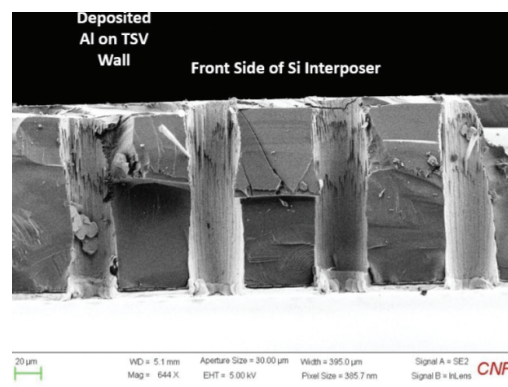


Figure 1: Cross-sectional SEM Images of TSVs.

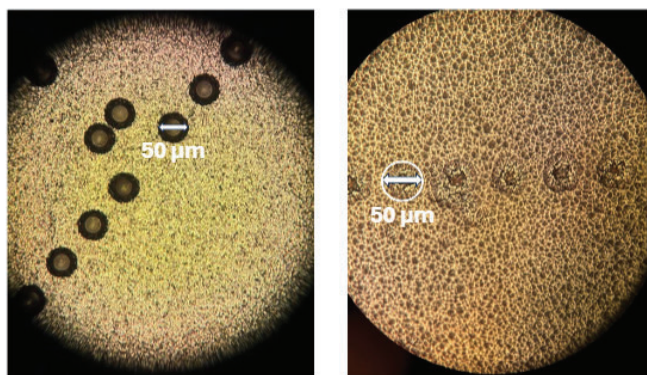


Figure 2: Top-View Optical Image of TSVs After 3 and 12 Hours of Cu Electroplating.

sidewall angles ranging from 80 to 90 degrees. After the TSVs were fully etched through, ALD was used to coat the TSV sidewalls with a Pt seed layer, functioning also as an effective diffusion barrier and adhesion layer. Cu was then electroplated to fill the TSVs, initiating from the Pt seed layer, as illustrated in Figure 2.

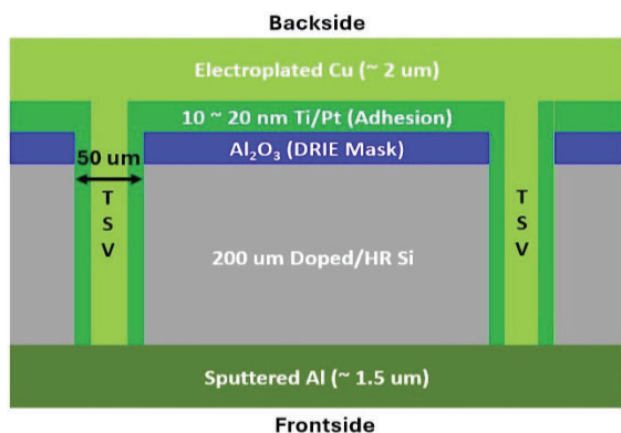


Figure 3: Schematic of Si Interposer Layout.

Once the backside processing was completed, the front side was patterned with SIW and GCPW lines. A platinum/aluminum (Pt/Al) layer was subsequently deposited using AJA Sputter deposition to complete the vertical interposer pathway. The structural details and final device configuration is depicted in Figure 3.

Both HR and doped Si devices were tested at the High Frequency Test Lab (HFTL) where TSV series resistance and GCPW line performance were evaluated. Using the DC probe station, I-V measurements showed that the TSV series resistance was typically below 1 Ω for both HR and doped Si substrates. I-V curves also confirmed a resistivity ranging from 4.7 to 17.4 $\text{k}\Omega\cdot\text{cm}$,

confirming that high resistivity of the HR Si substrate. RF measurements were then conducted over the 1-40 GHz range using the small-signal probe station, as shown in Figure 4.

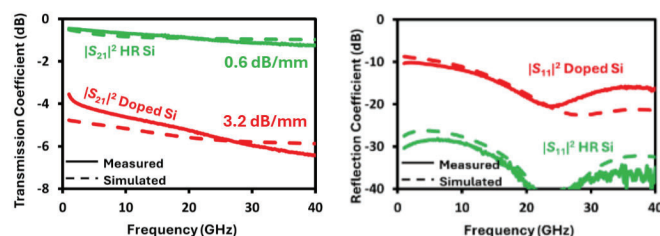


Figure 4: Measured Insertion Loss (S_{21}) and Return Loss (S_{11}) of Doped and HR Si Substrates Across 1–40 GHz.

For HR Si, the insertion loss (S_{21}) value was approximately 0.7 dB/mm, with return loss (S_{11}) exceeding 20 dB. In comparison, doped Si devices exhibited a much higher insertion loss of approximately 3.2 dB/mm and a return loss greater than 10 dB.

Conclusions and Future Steps:

The fabrication of Si interposers focused on optimizing Bosch DRIE of thinned Si wafers and refining metallization processes to achieve uniform anisotropic etching and consistent metal filling across the wafer. Measurements comparing HR and doped Si substrates revealed that HR Si shows insertion losses an order of magnitude lower than the doped Si, highlighting its potential as a promising substrate material for millimeter-wave applications. Looking ahead, with updated tools and improved techniques for etching and metallization, fully metal-filled TSVs are expected to achieve insertion losses below 0.5 dB/mm at frequencies up to 220 GHz, utilizing the 220 GHz single-sweep probe station at HFTL; once these targets are achieved, our group hopes to achieve heterogeneous integration in the cleanroom with flip chip bonding.

References:

- [1] M. J. Asadi, L. Li, K. Nomoto, Y. Tang, W. Zhao, P. Fay, D. Jena, H. G. Xing, and J. C. M. Hwang, "SiC Substrate Integrated Waveguides for High-Power Monolithic Integrated Circuits Above 110 GHz," 2021 IEEE MTT-S Int. Microw. Symp. (IMS), Atlanta, GA, USA, 2021, pp. 669–672.
- [2] D. Malta, E. Vick, S. Goodwin, C. Gregory, M. Lueck, A. Huffman, and D. Temple, "Fabrication of TSV-based silicon interposers," 2010 IEEE International 3D Systems Integration Conference (3DIC), Munich, Germany, 2010, pp. 1–6.
- [3] M. Leskela and M. Ritala, Thin Solid Films 409, 138 (2002).
- [4] F. Laermer et al., IEEE Microelectromech. Syst. 211 (1999).

Characterization of Deep Reactive Ion Etching for Silicon Plasma Singulation

CNF Intern: Caleb Pelland

Student Affiliation: Department of Materials Science and Engineering, Cornell University

Summer Program(s): 2026 Cornell NanoScale Facility Summer Internship

Mentor(s): Jeremy Clark, CNF

Primary Source(s) of Research Funding: NORDTECH

Contact: cap363@cornell.edu, clark@cnf.cornell.edu

Summer Program Website(s): <https://www.cnf.cornell.edu/education/reu/2026>

Abstract:

Bosch deep reactive ion etching (DRIE) was characterized for silicon plasma singulation using SPR 220-4.5 positive photoresist [1]. A 250 cycle study quantified aspect-ratio-dependent etching (ARDE) from 5–100 μm dicing streets, or trenches. Long runs established local 30 μm through-wafer capability and the photoresist limit. Depth decreased from 460.59 μm at 100 μm width to 222.64 μm at 5 μm . A cycle-dependent passivation run showed 0.80–1.00 μm widening, compared with provisional fixed-passivation values of 4–7 μm at matched widths. Optical monitoring identified mask failure but did not qualify a silicon endpoint.

Summary of Research:

1. Campaign from Screening to Final Monitor

Each Bosch cycle [2] used 2.2 s of C4F8-rich polymer passivation, a 2.0 s ion-assisted SF6 trench-bottom clear at 275 W platen bias, a 3.0 s zero-bias high-flow SF6 silicon etch, and a 1.0 s low-flow SF6 transition. Fixed passivation means that the 2.2 s step did not change with cycle number. Reflectometry measured resist loss, and scanning electron microscopy (SEM) measured depth. Aspect ratio was depth divided by nominal width, D/w . Normalized depth was D_w/D_{100} .

2. Selectivity and Photoresist Survival

A five-run platen-bias/passivation-time screen was completed. Both streaked 225 W conditions were rejected. Accepted conditions spanned 53.45–57.37 nm/cycle and 6.38:1–10.03:1 silicon-depth/resist-loss selectivity. The non-flat 29.27:1 result was rejected for anomalously small resist loss. Clearing trials selected the 275 W/2.2 s fixed-passivation process.

The 350 cycle photoresist-survival run reduced center resist from 3789.592 nm to 542.434 nm, corresponding to 9.278 nm/cycle consumption. Site loss was 2.896–

3.247 μm . With nominal 530 μm silicon, the conditional thickness-to-loss ratio was 163:1–183:1. It represents selectivity only where clearing is independently confirmed, not full-wafer mask uniformity. 500 cycle testing cleared 30 μm streets but consumed the photoresist, motivating a more durable oxide or metal hard mask for narrower features [3].

Dense repeated streets with 33.8% open area lost resist, while normally spaced 30 μm die streets with 2.7% open area retained it. This is not a universal density threshold. Silicon depth was not mapped, so accelerated local silicon etching is unproven.

3. Photoresist Loss Decreased with Radius

Matched thin-film reflectometry measurements show radial photoresist consumption. Photoresist-survival sites decreased from 9.23 nm/cycle in the inner radial bin to 8.37 nm/cycle near the edge. The ramped-passivation run decreased from 9.03 nm/cycle to 7.57 nm/cycle. Linear slopes were -0.029 and -0.046 nm/cycle/mm. These are resist-loss, not silicon etch-rate, measurements.

4. ARDE is the Dominant Small-Street Limit

The 250-cycle measurements in Table 1 decrease monotonically with narrower streets. Image scaling was calibrated, but mounting and projection uncertainty were not quantified. The 5 μm trench had a 44.53:1 nominal-width ratio but only 48.3% of the 100 μm -street depth. Through 530 μm silicon would require a geometric ratio near 106:1.

Width (μm)	Depth (μm)	D/w	D_w/D_{100}
5	222.64	44.53	0.483
15	256.04	17.07	0.556
30	306.13	10.20	0.665
50	395.19	7.90	0.858
100	460.59	4.61	1.000

Table 1: SEM-measured depth, nominal-width aspect ratio, and depth normalized to the 100 μm street after 250 fixed-passivation cycles. Each width has one cross-section.

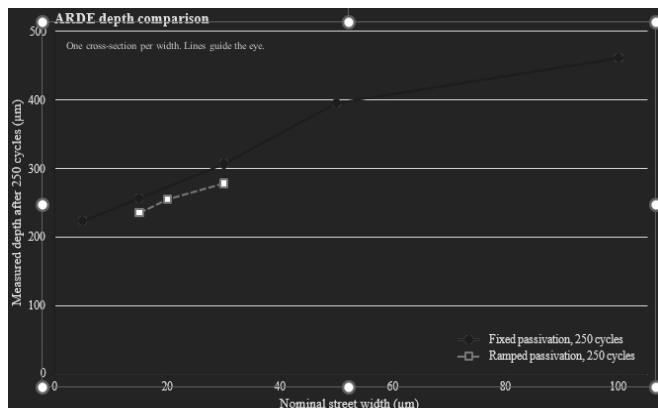


Figure 1: SEM-measured silicon trench depth after 250 Bosch cycles versus nominal opening width. A fixed 2.2 s polymer-passivation step is compared with a linear increase from 2.2–2.5 s. Each point is one cross-section. Lines guide the eye.

5. The Ramped-Passivation Run Showed Less Widening

The ramped-passivation run increased the polymer-deposition step from 2.2 s to 2.5 s over 250 cycles, as a depth-compensation strategy [4], [5]. Confirmed 15, 20, and 30 μm trenches reached 234.95, 254.87, and 277.86 μm, with 0.801, 0.591, and 1.000 μm top-to-bottom widening. Relative to the fixed-passivation depth study's provisional 15 μm and 30 μm widening of 4 and 7 μm, the new values were 80% and 86% smaller. Depth was 8.2% and 9.2% lower at those widths. Nominal-width aspect ratios were 15.66:1, 12.74:1, and 9.26:1. Each profile has one cross-section and no replicate uncertainty.

Ramped-passivation run: confirmed 250-cycle cross sections

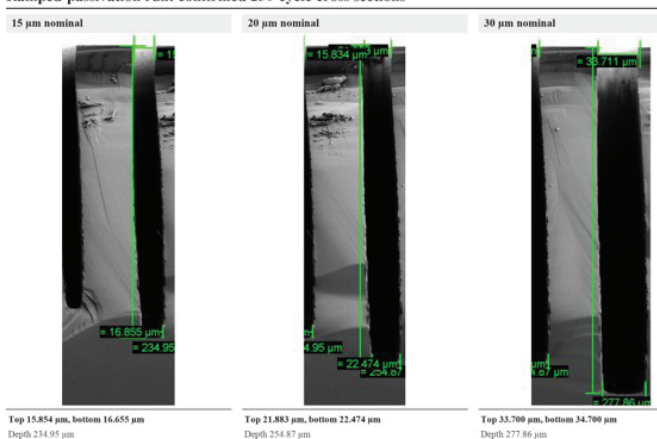


Figure 2: Cross-sectional SEMs after the 250-cycle ramped-passivation process show top width, bottom width, and depth for nominal 15, 20, and 30 μm streets. Green annotations are instrument measurements. No geometric correction was applied.

6. Optical Monitoring Detected Mask Failure, Not Silicon Breakthrough

Optical emission spectroscopy (OES) monitoring was evaluated as a possible method for stopping the etch

when silicon streets clear [6], [7]. The trial established a compact data-collection method and identified a late optical transition associated with local photoresist loss. It did not establish a silicon-breakthrough endpoint because the transition was not correlated with physical clearing of 30 μm streets. Automatic termination therefore remains outside the characterized process scope until the optical transition is reproduced and verified against physical breakthrough.

Conclusions and Future Steps:

Fixed passivation enabled normally spaced 30 μm streets with bottom widening. Ramped passivation showed less widening but lower depth. ARDE, pattern density, and mask survival constrain smaller streets. Hard masks are the smaller-street path. Optical termination remains disabled pending repeatable physical clearing.

Acknowledgements:

This work was performed at the Cornell NanoScale Facility (CNF), part of the National Nanotechnology Coordinated Infrastructure supported by National Science Foundation Grant NNCI-2025233. Jeremy Clark and CNF staff are acknowledged. This internship was sponsored by NORDTECH.

References:

- [1] Kayaku Advanced Materials, MEGAPOSIT SPR220 Series Photoresists product data sheet, Kayaku Advanced Materials. [Online]. Available: <https://kayakuam.com/wp-content/uploads/2019/09/SPR220-Data-Sheet.pdf>.
- [2] F. Laermer and A. Schilp, "Method of anisotropically etching silicon," US 5,501,893, Mar. 26, 1996. [Online]. Available: <https://patents.google.com/patent/US5501893A/en>.
- [3] M. S. Gerlt, N. F. Läubli, M. Manser, B. J. Nelson, and J. Dual, "Reduced etch lag and high aspect ratios by deep reactive ion etching (DRIE)," *Micromachines*, vol. 12, no. 5, p. 542, 2021. doi: 10.3390/mi12050542.
- [4] B. Chang, P. Leussink, F. Jensen, J. Hübner, and H. Jansen, "DREM: Infinite etch selectivity and optimized scallop size distribution with conventional photoresists in an adapted multiplexed bosch DRIE process," *Microelectronic Engineering*, vol. 191, pp. 77–83, 2018. doi: 10.1016/j.mee.2018.01.034.
- [5] Y. Tang, A. Sandoughsaz, K. J. Owen, and K. Najafi, "Ultra deep reactive ion etching of high aspect-ratio and thick silicon using a ramped-parameter process," *Journal of Microelectromechanical Systems*, vol. 27, no. 4, pp. 686–697, 2018. doi: 10.1109/JMEMS.2018.2843722.
- [6] Plasma-Therm LLC, Cortex mds documentation, Offline Help supplied with the MDS system; copyright 2013–2015, Plasma-Therm LLC, 2015.
- [7] Plasma-Therm LLC, Endpointworks 4 user's guide and integrated help, Offline Help supplied with EndpointWorks 4, Plasma-Therm LLC, 2022.

Atomic Layer Deposition Dielectric Film Quality

CNF Intern: Nadiyah Rahaman

Student Affiliation: Department of Engineering, Cornell University

Summer Program(s): 2026 Cornell NanoScale Facility Summer Internship

Mentor(s): Jeremy Clark, CNF

Primary Source(s) of Research Funding: NORDTECH

Contact: nsr45@cornell.edu, clark@cnf.cornell.edu

Summer Program Website(s): <https://www.cnf.cornell.edu/education/reu/2026>

Primary CNF Tools Used: Oxford FlexAL, Oxford ASP, Woolam RC-2 Ellipsometer, Kiethy Probe Station

Abstract:

As semiconductor devices continue to scale, dielectric films must become thinner while maintaining reliable electrical isolation. Atomic Layer Deposition (ALD) can be used to create thin oxide layers. These oxide layers are used to passivate electrical signals from one electrode to another with a very thin layer of oxide material. The Oxford FlexAL and Oxford ASP tools allow for thin oxide Atomic Layer Deposition (ALD) films. Deposition parameters including temperature, oxide chemistry, and ALD tool can influence film growth, density, and electrical properties. The Oxford ASP is a new ALD tool in the Cornell Nanoscale Facility (CNF) Cleanroom. The purpose of this experiment is to explore baselining the ASP. Due to tool availability and the duration of the study, the results presented represent an initial baseline rather than a complete process optimization. SiO₂ and Al₂O₃ films were deposited under varying process conditions and characterized using deposition rate, wet etch rate, and electrical measurements. Deposition rate was used to evaluate film growth behavior, while wet etch rate provided a relative indication of film density. Metal-oxide-semiconductor capacitors (MOSCAPs) were fabricated to evaluate electrical behavior using capacitance-voltage (C-V) and current-voltage (I-V) measurements. Together, these measurements establish preliminary relationships between ALD process conditions and resulting dielectric film properties and provide a foundation for continued optimization of the Oxford ASP.

Summary of Research:

This research investigated the quality of dielectric films deposited by atomic layer deposition (ALD), with emphasis on establishing baseline processes for the Oxford ASP and comparing its performance with the established Oxford FlexAL system. The study focused primarily on silicon dioxide (SiO₂) and aluminum oxide (Al₂O₃) films. Process variables included deposition

temperature, oxide chemistry, deposition method, and ALD tool. Film quality was evaluated through deposition behavior, wet chemical etch behavior, and electrical characterization of fabricated metal-oxide-semiconductor capacitors (MOSCAPs).

ALD Film Deposition and Characterization

Overall, the deposition-rate measurements demonstrate measurable differences between the two ALD systems as well as temperature-dependent growth behavior.

Wet Etch Characterization

Wet chemical etching was used as an indirect method of comparing film density and chemical resistance. This data is drawn from the Oxford FlexAL.

For FlexAL Al₂O₃, the measured etch rate decreased with increasing deposition temperature as expected.

The FlexAL SiO₂ results displayed a different trend. The measured etch rate reached a maximum at the intermediate 200 °C deposition condition. Because this trend differed from the expected decrease in etch rate with increasing deposition temperature, additional measurements are required to determine its reproducibility.

MOSCAP Fabrication

MOSCAP devices were fabricated to characterize the electrical properties of the deposited dielectric films. The fabrication sequence consisted of wafer cleaning, ALD dielectric deposition, dielectric thickness measurement, patterned top-metal deposition, backside cleaning, backside-metal deposition, and electrical characterization. SiO₂ and Al₂O₃ dielectric layers were deposited using either the Oxford FlexAL. SiO₂ films were deposited for device fabrication from the ASP while Al₂O₃ films were not available for deposition on the ASP. Top-metal electrodes were deposited through a shadow mask using the AJA Sputter, producing an array of circular MOSCAP structures with different electrode areas. Completed devices were electrically characterized using the Keithley Probe Station.

Electrical Characterization

Capacitance vs. voltage (C-V) measurements were performed to characterize the electrical response of the fabricated MOSCAP structures during a voltage sweep. The measured curves demonstrate transitions between accumulation, depletion, and inversion behavior. Both p-type and n-type silicon substrates were used during device fabrication, causing the direction and voltage of the C-V transition depending on substrate type. FlexAl film devices were constructed on p-type wafers while ASP devices were constructed on n-type wafers. This difference should be considered when comparing samples.

For the FlexAL Al_2O_3 samples, the 200 °C wafer exhibited a C-V transition voltage of approximately +0.88 V with a median hysteresis of 2.29 V, while the 300 °C wafer exhibited a median transition voltage of approximately +0.58 V and a lower median hysteresis of 1.01 V. This reduction in hysteresis suggests improved electrical stability at the higher deposition temperature.

The FlexAL SiO_2 sample deposited at 300 °C exhibited a transition voltage of approximately -3.05 V and a median hysteresis of 0.18 V. For ASP SiO_2 , the 200 °C sample exhibited a transition voltage of approximately -4.79 V and median hysteresis of 1.07 V, while the 300 °C sample exhibited a transition voltage of approximately -4.05 V and median hysteresis of 0.62 V. The lower hysteresis measured at 300 °C suggests a possible improvement in electrical stability with increasing ASP deposition temperature.

Current vs. voltage (I-V) measurements were used to evaluate dielectric leakage.

The ASP results show a decrease in median leakage at the higher deposition temperature, although additional measurements are necessary for conclusivity.

Significant variation was observed in several I-V measurements when measuring the same size electrode, with leakage spanning multiple orders of magnitude. This variability may result from local dielectric defects, wafer nonuniformity, probe contact, electrode-area uncertainty, contamination, or fabrication effects such as shadow-mask bowing and unintended metal deposition beneath the mask. Based on acquired data, samples should be sorted to determine accuracy of each measurement. The electrical measurements should be considered preliminary indicators of process performance rather than definitive material properties. Additional device measurements and improved fabrication repeatability are recommended before establishing electrical baselines for the ASP.

Figure 1.

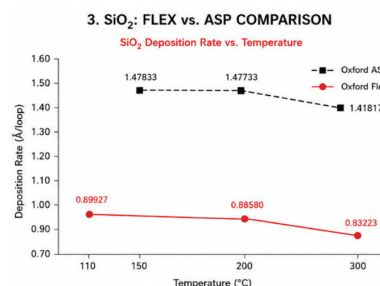


Figure 2.

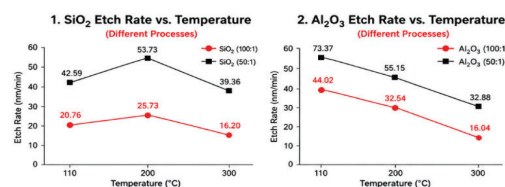


Figure 3.

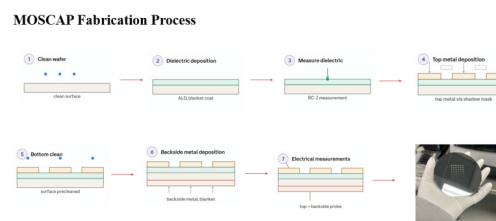
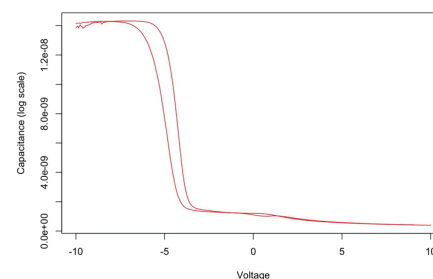


Figure 4.



Conclusions and Future Steps:

This study established repeatable experimental procedures for ALD deposition characterization, wet etch testing, MOSCAP fabrication, and electrical measurement. The deposition results demonstrate that film growth rate depends on both deposition temperature and ALD system. Wet etch measurements further indicate that deposition conditions affect the chemical resistance of the resulting films, although the unexpected intermediate temperature behavior observed for FlexAL SiO_2 requires additional investigation. MOSCAP fabrication successfully enabled C-V and I-V characterization of selected dielectric films. These measurements provide an electrical complement to the deposition and etch-rate studies by evaluating the behavior of the dielectric within a functional device structure. MOSCAP construction and electrical measurements should be expanded to test a wider variety of oxide films. Upon further research, results will provide a complete comparison between the Oxford ASP and Oxford FlexAL and contribute to establishing baseline dielectric processes for the ASP system.

Niobium Seeded Tantalum Film Stress Characterization on AJA-Q

CNF Intern: Gavin Simoneau

**Student Affiliation: Department of Mechanical and Mechatronics Engineering
Technology, Rochester Institute of Technology (RIT)**

Summer Program(s): 2026 Cornell NanoScale Facility Summer Internship

Mentor(s): Shilling Du, CNF

Primary Source(s) of Research Funding: NORDTECH

Contact: gavinsimoneau24@gmail.com, xd255@cornell.edu

Summer Program Website(s): <https://www.cnf.cornell.edu/education/reu/2026>

Primary CNF Tools Used: AJA-Q2, FleXus Film Stress Measurement, Filmetrics R50, and the P7 Profilometer

Abstract:

This research investigated how niobium (Nb) and tantalum (Ta) sputtering pressures affect the stress and electrical properties of Nb-seeded Ta thin films intended for superconducting devices. The tested film structure consisted of approximately 5 nm of Nb beneath 100 nm of Ta on a silicon substrate. Nb deposition pressures of 3, 7, 15, and 20 mTorr were combined with Ta pressures of 3, 7, and 20 mTorr, creating 12 deposition conditions. Before deposition, each wafer received a MOS clean within four hours of being loaded into the deposition system. Film stress, thickness, deposition rate, and resistivity were evaluated for each condition. The results showed that both Nb and Ta deposition pressures influenced film stress. The 7 mTorr Nb and 7 mTorr Ta combination produced the lowest stress magnitude among the tested Nb-seeded film.

Summary of Research:

Tantalum is a promising material for superconducting devices because of its electrical and superconducting properties. However, its performance depends strongly on its crystalline phase. Alpha-phase tantalum is preferred because it has a resistivity of approximately 15–20 $\mu\Omega\cdot\text{cm}$ and a superconducting transition temperature near 4.3 K. Beta-phase tantalum has a much higher resistivity of approximately 150–200 $\mu\Omega\cdot\text{cm}$ and a transition temperature below 1 K. A thin Nb seed layer can promote the formation of the preferred alpha-Ta phase.

In addition to having low resistivity, a deposited film must have acceptable stress properties. Excessive tensile or compressive stress can contribute to wafer bow, film cracking, delamination, and variations during later fabrication processes. These problems can reduce device uniformity and process repeatability. The objective of this research was therefore to determine

how Nb and Ta sputtering pressures affected film stress and to identify conditions that produced a low-stress film while maintaining desirable electrical properties.

Wafer preparation was an important part of the experimental procedure. Before deposition, each silicon wafer underwent a MOS clean. The clean was performed within four hours of deposition to limit the accumulation of airborne particles, organic residue, and other surface contaminants. Maintaining a clean wafer surface supported consistent film adhesion and uniform growth of the Nb seed layer and Ta film. Applying the same cleaning requirement to each wafer also helped reduce sample-preparation differences between deposition conditions.

The film stack consisted of approximately 5 nm of Nb followed by 100 nm of Ta. Four argon pressures were tested during Nb deposition: 3, 7, 15, and 20 mTorr. Each Nb pressure was paired with Ta pressures of 3, 7, and 20 mTorr. This created a matrix of 12 Nb-Ta pressure combinations. Ta-only films deposited at 3, 7, and 20 mTorr were also evaluated as baseline samples. Film stress was determined by measuring wafer curvature before and after deposition using the FleXus stress-measurement system. The change in curvature was used to calculate the stress produced by the deposited film. Stress values closer to zero represented films with lower overall stress magnitude. Film thickness was measured to confirm the deposited thickness and calculate deposition rates. Electrical measurements were used to calculate resistivity and determine whether the films had properties consistent with alpha-Ta. Heat maps were then created to compare stress and resistivity across the tested pressure combinations.

The results demonstrated that deposition pressure had a substantial effect on film stress. For the Ta-only baseline samples, the 3 mTorr Ta condition produced the greatest stress magnitude. Increasing the Ta pressure

generally reduced the magnitude of the measured stress. A similar overall relationship was observed for the Nb-seeded films, although the Nb deposition pressure also influenced the final stress.

Among the Nb-seeded samples, the combination of Nb deposited at 7 mTorr and Ta deposited at 7 mTorr

Nb-Seeded Ta Film Resistivity Non-Uniformity

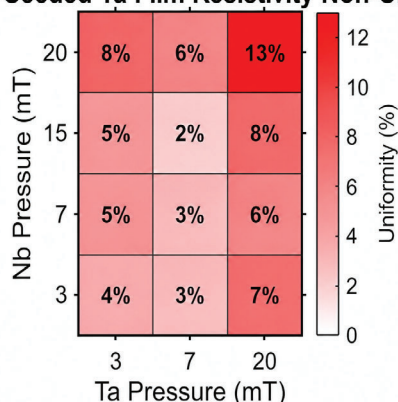


Figure 1.

Nb Seeded Ta Film Stress

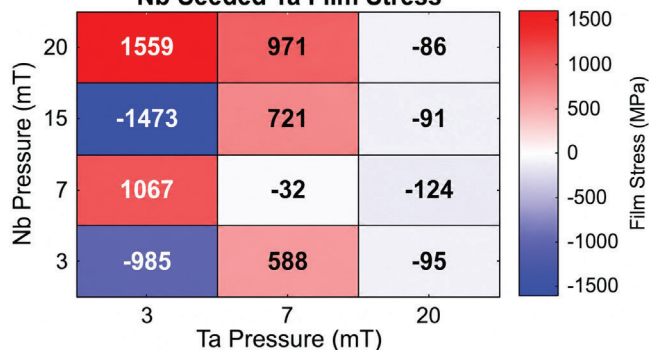


Figure 2.

Nb Seeded Ta Film Resistivity

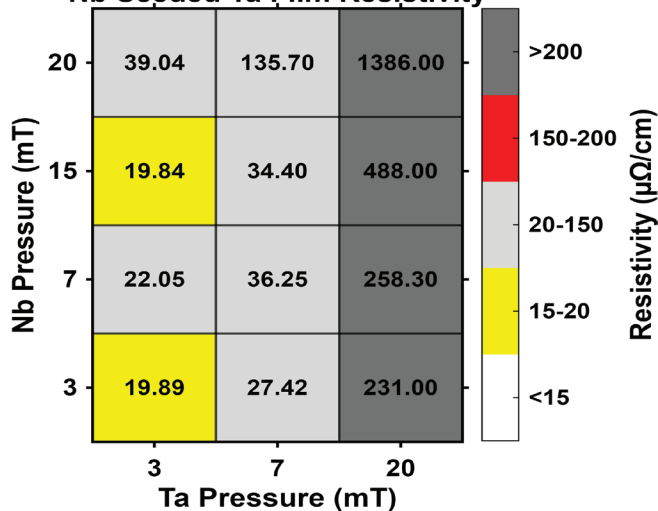


Figure 3.

produced a stress value closest to zero. It was therefore identified as the lowest-stress Nb-seeded condition tested. Several films deposited at a Ta pressure of 20 mTorr also demonstrated relatively low stress. However, changing the Ta pressure did not produce the same response for every Nb pressure. This indicates that the conditions used to deposit the seed layer affected how the Ta film grew and how stress developed within the completed stack.

Stress could not be used as the only factor when selecting an optimized process. A film with low stress would still be unsuitable for superconducting-device fabrication if it had the high resistivity associated with beta-Ta. The preferred process must balance low stress, low resistivity, wafer-scale uniformity, and repeatability. The pressure matrix narrowed the range of promising deposition conditions and identified the region near 7 mTorr Nb and 7 mTorr Ta as a candidate for further study.

Conclusions and Future Steps:

This research showed that both Nb and Ta sputtering pressures influenced the stress of Nb-seeded Ta thin films. Increasing the Ta pressure generally reduced the magnitude of film stress, but the Nb seed-layer pressure also affected the completed film. Of the 12 combinations evaluated, the 7 mTorr Nb and 7 mTorr Ta condition produced the lowest measured stress magnitude.

Consistent wafer preparation was also necessary for comparing the samples. Performing a MOS clean within four hours of deposition helped maintain a clean surface and supported uniform film adhesion and growth. This cleaning requirement should be maintained in future experiments so that differences between samples are primarily caused by deposition conditions rather than surface contamination.

Future work should repeat the most promising conditions to determine whether the results are reproducible. Additional pressures near 7 mTorr Nb and 7 mTorr Ta should be tested to define the optimum process more precisely. Stress, thickness, and resistivity uniformity should also be measured across the wafer. Structural characterization can be used to confirm alpha-Ta formation, while low-temperature measurements can determine the superconducting transition temperature. Finally, superconducting test devices should be fabricated using the optimized film to determine whether lower stress improves device uniformity, performance, and reliability.