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TR-1292

Microrectenna Arrays for IFF and Power/Data Transfer Applications: Summary Report on Sample Set

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1. SUMMARY

MIM diodes consisting of stacks composed of Au-NbOx-Al were fabricated and tested at MIT LL. Devices showed asymmetric IV characteristics, indicating that the film stacks are suitable for use in fabricating rectenna arrays.

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2. INTRODUCTION

Direct conversion of high-frequency electromagnetic radiation into DC electric current is of great interest for efficient generation of electric signals. Rectifying antennas, or rectennas, performing such a function have been demonstrated for some time at RF frequencies (~10 GHz), but their extension to infrared and visible frequencies is a novel concept. If successful, rectennas operating in the IR-visible part of the spectrum can enable efficient, compact devices for active identify-friend-or-foe (IFF) applications or laser-based power/data transfer applications, such as remote power beaming to an airborne asset.

Successful operation of a rectenna array for power beaming requires the formation of a metal–insulator–metal (MIM) stack in which the DC current voltage (IV) characteristic displays asymmetry, and the geometry of the rectenna produces optical resonances at the desired wavelengths. The first step in rectenna fabrication is the generation of an MIM stack with a high quality insulating layer, in this case niobium oxide (NbOx; nominally Nb_2O_5), and characterization of the current-voltage characteristics of the resulting diode. A diode with high asymmetry (ratio of forward- and reverse-biased current) will have high conversion efficiency; therefore, the goal is to create an MIM stack with highly asymmetric IV characteristics.

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3. SAMPLE PREPARATION

MIM diodes were fabricated at MIT LL using the process described in this section. The starting substrate is a 200-mm silicon wafer (resistivity = 10–20 Ω-cm) with a 500-nm thick layer of thermal oxide and 100 nm of aluminum deposited by electron beam evaporation in a Temescal 1800 system. A layer of niobium oxide (NbOx) is deposited on top of the aluminum layer via atomic layer deposition (ALD) in a Cambridge Ultratech ALD system. The NbOx is deposited at 200°C with plasma assisted deposition using (t-Butylimido)tris(diethylamino)niobium(V) (Strem Chemicals) as a precursor, with a bubbler. Sections of the wafer are masked during the deposition to provide electrical contact to the aluminum layer in the final structure. The wafer is cleaved into sections of size 1” × 2” and sections are processed individually in the final fabrication step to form the top metal contact. The top diode pad contacts are patterned in AZ1518 photoresist using a Heidelberg MLA150 direct write system. The chip with patterned resist is exposed to an oxygen plasma for 30 seconds to remove organics and improve adhesion. The top contacts consist of 100 nm of gold deposited by electron-beam evaporation. After gold deposition, the excess gold is removed via liftoff by soaking the chip in acetone overnight with no agitation and rinsing in isopropanol. The final structure is shown in Figure 1.

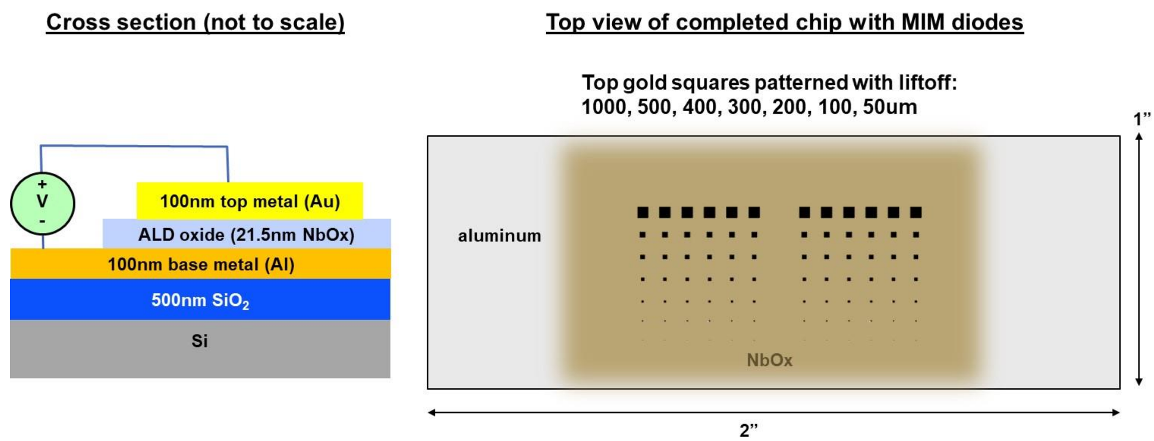

Figure 1. MIM diode test structures.

3.1 PREPARATION OF TEST SAMPLES PROVIDED IN SEPTEMBER 2022

The ALD system used to deposit the NbOx films was reconfigured in 2019 to remove the Nb source and bubbler for other users of the system. We attempted to prepare new NbOx films in the summer of 2022, after procuring new precursor material and reinstalling the bubbler. Test depositions resulted in films with very high particulate levels, leading to a complete system chamber clean and replacement of the bubbler manifold. We did not have resources to prepare additional new NbOx films, therefore the samples used to obtain the test results shown in this report were prepared using film stacks prepared in 2019, prior to the system reconfiguration. NbOx characterization data are also obtained from films prepared in 2019.

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4. NIOBIUM OXIDE CHARACTERIZATION

NbOx films deposited via ALD were characterized using ellipsometry, transmission electron microscopy (TEM), and X-ray photoelectron spectroscopy (XPS).

4.1 ELLIPSOMETRY

Ellipsometry of NbOx (nominal thickness ~100nm) deposited on an Si wafer was done by J.A. Woollam in June 2019. A Woollam RC2-DI spectroscopic ellipsometer took data from 193 nm to 1690 nm and an IR-Vase spectroscopic ellipsometer took data from 1.7 um to 30 um (fitting analysis was only performed up to 8 um). The model used was a Kramers-Kronig consistent B-spline fit with a thin (1 nm) layer of surface roughness on the NbOx film. The fitted thickness of the NbOx film was 85.93 nm. A full copy of the report and fitted optical constants are appended to this report. Figure 2 shows the modeled refractive index and loss in the 200 nm–8 μm range. The index of refraction in the region of interest (2–3 μm) ranges from 2.25 to 2.21 with negliable loss (k > 1e-4).

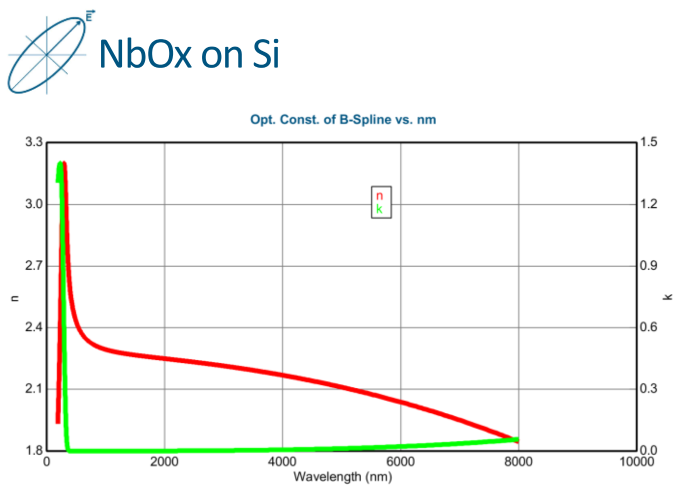


Figure 2. Optical constants of NbOx from a KK-consistent B-spline fit of spectroscopic ellipsometry of NbOx on Si. Performed at J.A. Woollam.

4.2 TRANSMISSION ELECTRON MICROSCOPY (TEM)

Representative films deposited at MIT LL were prepared and imaged using Harvard University TEM facilities. Figure 3 shows TEM images of two different film thicknesses. In each case, NbOx films were deposited on silicon substrates using plasma-assisted deposition with a bubbler. TEM measurements were used to confirm ellipsometer thickness measurements.

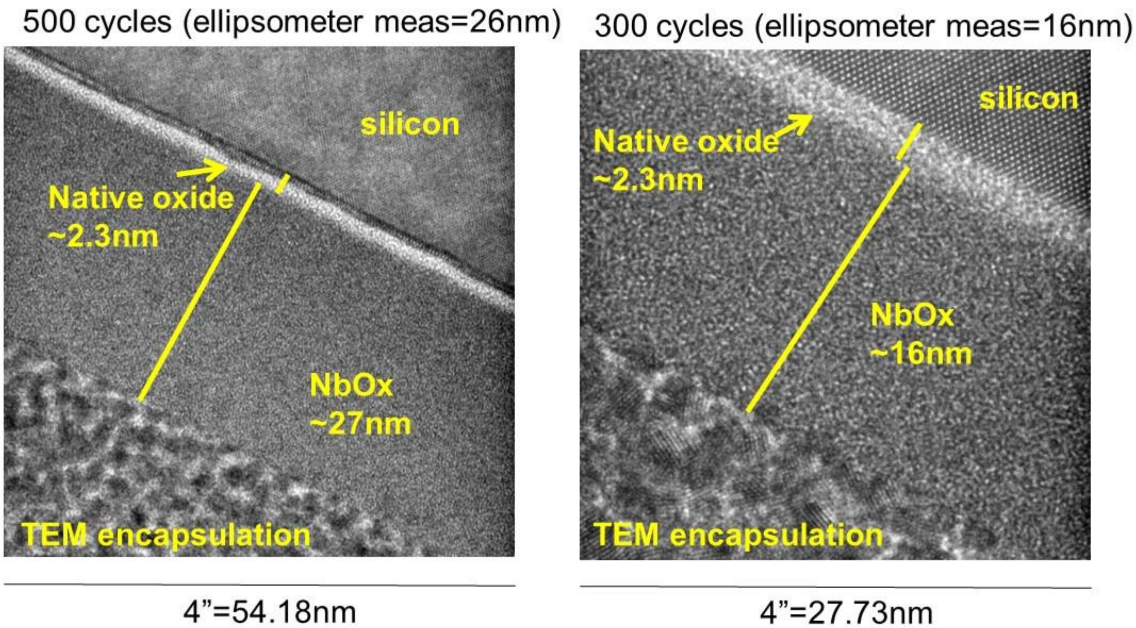


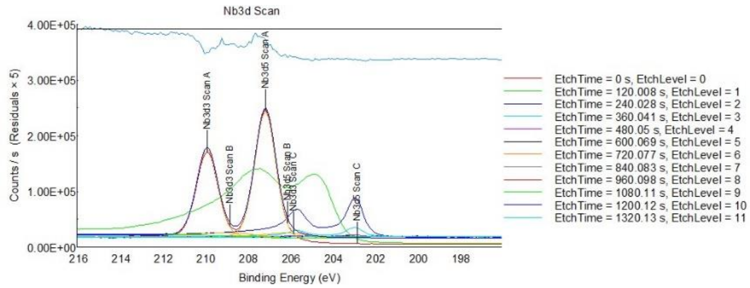
Figure 3. TEM of NbOx films deposited at MIT LL. Imaging was done at Harvard University.

4.3 X-RAY PHOTOELECTRON SPECTROSCOPY (XPS)

Stoichiometry of the NbOx films was determined by XPS analysis of representative films using a Thermo Fisher K-alpha Plus system at Harvard University. Two sets of data were taken: for films deposited in 2019 and also for films deposited in 2022.

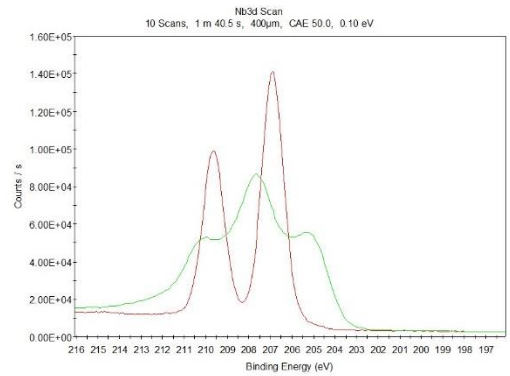
Figure 4a shows the XPS data from films deposited in 2019. These films were used to fabricate the MIM diodes prepared in May 2022 to be transferred to Natick. The peaks indicate that the film is predominantly Nb_2O_5, with atomic ratios confirming this.

	11nm	13nm	19nm
Name	Atomic %	Atomic %	Atomic %
O1s	64.54	60.51	61.07
Nb3d	25.26	23.3	23.71
C1s	10.2	16.18	15.23



A)

	23nm	50nm
Name	Atomic % (%)	Atomic % (%)
Nb 3d	30.8198	32.9389
C 1s	2.78343	1.86873
O1s	66.3968	65.1923
Nb	1	1
Ox	2.154355317	1.97918874



B)

Figure 4. XPS analysis of ALD deposited films. A) Films deposited in 2019 have a 60:25 Oxygen to Niobium ratio, which confirms the samples are predominately Nb_2O_5. B) Films deposited in 2022 have a ratio of 65:30, indicating stoichiometry closer to NbO_2. These films also had high particulate levels and were not used to make MIM diodes.

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5. TESTING

Samples were prepared as described in the previous section. Two sets of test samples were prepared, one with NbOx thickness of 15.3 nm and the second with NbOx thickness of 21.5 nm. Devices with the thinner NbOx layer were consistently shorted, but devices with the thicker layer consistently displayed diode-like behavior.

MIM diodes were tested by biasing as shown in Figure 1. Each chip was tested on a probe station, with one probe applied to the exposed aluminum layer serving as the bottom contact, and one probe contacting the top gold pad serving as the top contact to the diode. Voltage was applied with a Keithley 2440 SourceMeter instrument, and current voltage scans were done at successively increasing voltage ranges until asymmetry was observed. Voltage was scanned from zero to a positive voltage, then from zero to the equivalent negative voltage. Figure 5 shows representative IV scans for several diodes. Multiple scans were done on each device to show robust repeatable behavior. Tested devices showed asymmetries of 5 to 50.

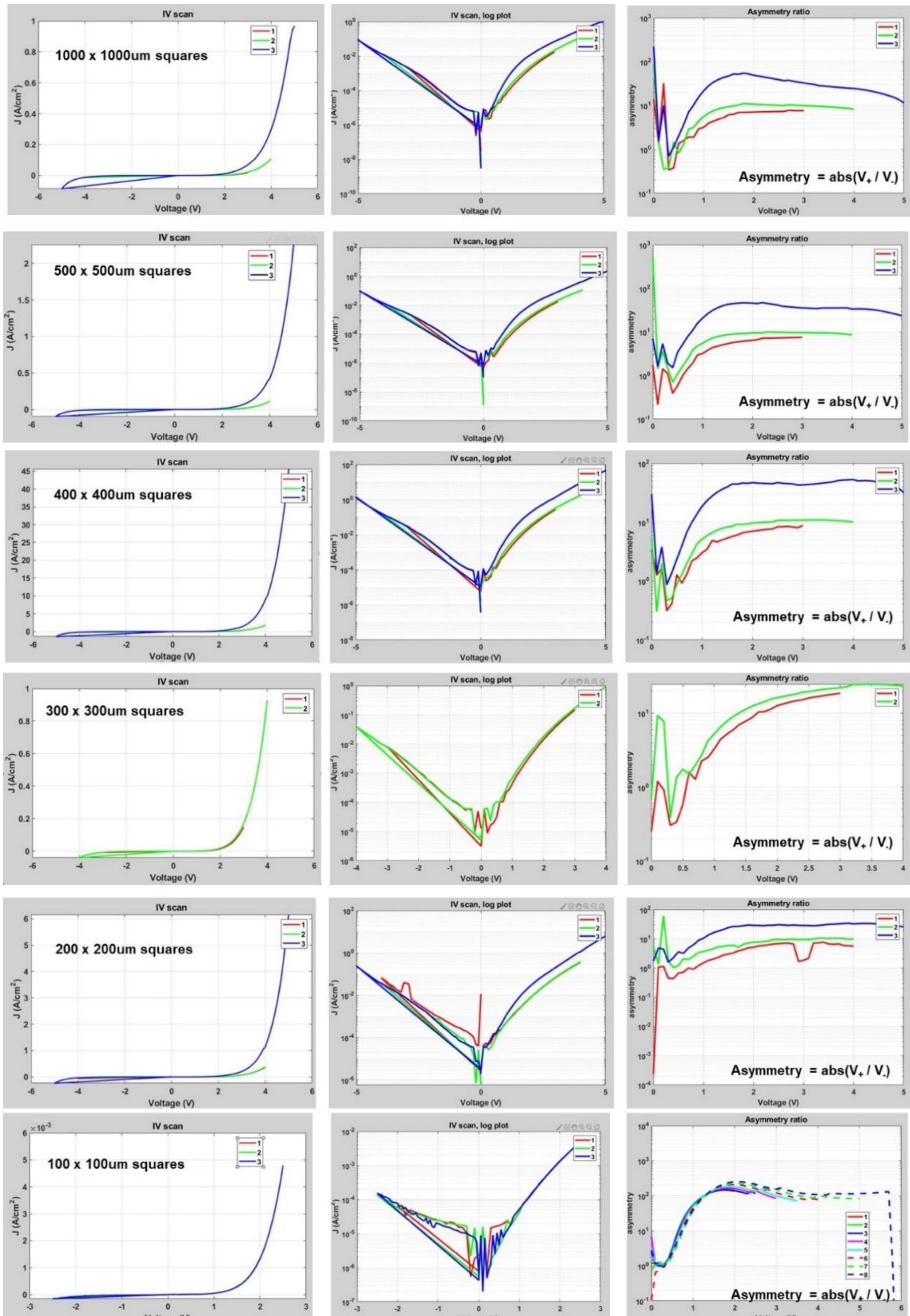

Figure 5. IV scans of MIM diodes. Each row represents multiple scans from one device, with IV scans plotted in linear and logarithmic form and asymmetry plotted as a function of voltage.