Fabrication of Nb / AL-N_x / NbTiN Junctions for SIS Mixer Applications

B. Bumble, H. G. LeDuc, J. A. Stern , and K..G.Megerian

***Abstract*— We discuss a processing technique for fabrication of superconductor-insulator-superconductor (SIS) junctions which typically exhibit a 3.5 mV sum-gap voltage. Junctions have a sub-gap to normal state resistance ratio of Rsg / R_N = 27 for resistance - area products down to R_NA = 8 $\Omega\,\mu m^2$ and high quality junctions have been produced with RnA products as low as 4 $\Omega\,\mu m^2$. The device structure incorporates Nb as a base layer, tunnel barrier formed by plasma nitridation of a thin Al proximity layer, and NbTiN as a counter-electrode material. Results for all Nb junctions with high current density aluminum-nitride barriers are also shown. Nitridation of the aluminum layer is investigated by control of the DC floating potential on a separate RF driven electrode in the vacuum process chamber. Devices are integrated to mixer antenna structures incorporating NbTiN as a ground plane. The wire circuit layer can be either normal metal or NbTiN. Annealing results show improved I-V characteristics with increased RnA products. Recent receiver measurements employing these junctions exhibit low noise performance up to 850 GHz. [1]**

***Index Terms*—AlN, Niobium, NbTiN, Superconductor device, SIS Mixers, THz detector.**

I. INTRODUCTION

HETERODYNE detectors used in radioastronomy are improving rapidly with the event of new projects such as the Stratospheric Observatory of Infraed Astronomy (SOFIA) and the Far Infrared Space Telescope (FIRST). Receivers are being developed to operate at higher frequency and larger band width. To accomplish these goals there is a recognized need for low junction capacitance and resistance which demands small area and thin tunnel barrier materials. Nb/Al-Ox/Nb tunnel junctions have produced low noise temperatures in heterodyne receivers up to 1 THz.[2] Low noise results have been achieved above the energy gap frequency of Nb ($2\Delta/h \sim 700$GHz) by using high conductivity normal metal (aluminum) tuning circuits. However, the ideal superconductor-insulator-superconductor (SIS) junction for THz heterodyne receivers should incorporate a high transition temperature (Tc), low-loss superconductor. Applications of tunnel junctions fabricated with NbN/MgO/NbN and NbN/AlN/NbN have been reported, but performance seems to be limited by either gap rounding in the current- voltage (I-V) characteristic or surface resistance in the NbN. [3,4]. Deposited barrier layers are prone to tunneling irregularities due to surface reactions and roughness resulting from thin spots or "pin-holes." Transmission electron microscope (TEM) images of Nb/Al-Ox/Nb junctions clearly show that aluminum smoothes out over the granular niobium surface. [7] Thermal oxidation of the aluminum surface produces a dense and uniform insulator. The niobium counter-electrode may degrade slightly at the interface, but not over the distance of its relatively long coherence length.

Recent measurements from mixers fabricated with NbTiN have shown that losses can be quite low. [6] However, the integration of Nb/Al-Ox/Nb junctions with NbTiN ground planes and wires suffers from gap reduction due to quasiparticle trapping at the Nb/ NbTiN interfaces on both sides of the junction. We have experimented with NbTiN as a counterelectrode on oxides and found that excess oxygen on an Al_2O_3 surface will tend to react with a deposited NbTiN layer, thus degrading the superconductor at the interface. Likewise, depositing pure Al on a NbTiN base depletes the superconductor of nitrogen at the interface. Depositing NbTiN on a layer of AlN shows less of an ill effect on the superconductor. Thermal oxidation of Al is much easier than producing the thermal nitride because the triple bond of N_2 is harder to break than the double bond in O_2. Producing a nitride requires either higher temperatures or creating a plasma to break the N_2 molecule. We did attempt using a more reactive gas (NH_3) which showed no improvement. The work presented here deals only with plasma nitridation at near room temperature.

AlN is an insulator of similar properties to Al_2O_3 with band gap energy ~4 eV and dielectric constant of 8.5. [8] Nb/Al-Nx/Nb Josephson junctions produced by plasma nitridation of aluminum have been previously investigated by Shiota, et al and shown to exhibit improved annealing stability over oxide barriers. [10] Replacing the counter-electrode with NbTiN has the advantage of moving the sum-gap voltage out by as much as 1 mV since $\Delta_{Nb} = 1.5$ mV and $\Delta_{NbTiN} = 2.5$ mV. Thus, a THz receiver would have a substantial improvement in bias range. Figure 1 shows a comparison of a Nb/Al-Nx/Nb junction with 2.7 mV gap to a Nb/Al-Nx/NbTiN junction with 3.7mV gap. Both junctions have $R_NA \sim 10$ $\Omega-\mu m^2$ and are plotted on the same scale so that the gap voltages and step features can be compared.

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All authors are with the Center for Space Microelectronics Technology, Jet Propulsion Laboratory, California Institute of Technology, Pasadena, CA 91109, USA

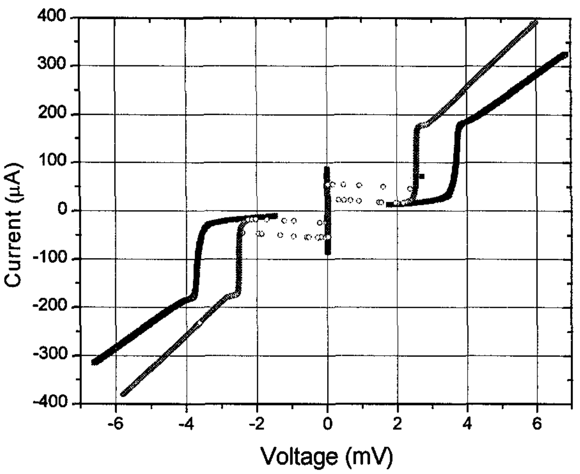

Fig. 1. Comparison of (O) Nb/Al-Nx/Nb junction RnA = 10 Ω μm^2, Rsg/Rn = 9, Vgap = 2.7mV, with (□) Nb / Al - Nx / NbTiN junction, RnA = 8 Ω μm^2, Rsg/Rn = 7, Vgap = 3.7mV.

II. EXPERIMENTAL TECHNIQUE

Junctions for this set of experiments are fabricated by a trilayer deposition and self -aligned processing technique. Details of the pattern and etch steps of this technique have been reported previously.[11] Most of the work presented here is test data on process development for trilayer deposition which involves plasma nitridation of the Al proximity layer. Film properties are investigated which may be out of the range for acceptable mixer application

Our process development is investigated for two separate UHV vacuum systems. We have made devices for receiver testing in both systems. The two cylindrical chambers are similar in many respects; system #1 is 46 cm in diameter and 36 cm high whereas system #2 is 76 cm in diameter and 48 cm high. Sputtering sources are all DC magnetrons with 7.6 cm diameter targets which are positioned to sputter upward with a target to substrate throw distance of about 6 cm. Samples are inserted through a vacuum load-lock chamber. A manipulator arm rotates about the chamber center to place the sample over the various sources located around the circumference.

Substrates used in this experiment were thermally oxidized Si wafers. They were cleaned in-situ prior to film deposit with mild Ar ion beam exposure of 150eV, 20mA for 45 seconds in system #2 and Ar plasma cleaned with comparable conditions in system #1.

Ground plane NbTiN depositions are done at substrate temperatures between 300-500 °C. Targets are 78 wt. % Nb and 22 wt. % Ti produced by vacuum arc melting. [xx] Samples are held on a stainless steel platform capable of RF bias and is surrounded by a ground shield. Reactive DC magnetron sputtering is done in an ambient of Ar and N_2. The flow ratio for optimum properties of NbTiN is integrally related to deposition rate, total gas pressure, target and substrate temperature, substrate bias, and plasma dynamics which involve fixture geometry. Furthermore, there is a compromise to be made between the properties of film stress, Tc, and resistivity. Typical values for films in this study are Tc = 15-16 K , ρ_{20K} = 55-80 μ Ω cm, and compressive stress σ = 5-15 x 10^9 dynes/cm^2. A more detailed description of the NbTiN film deposition process is given in a previous paper. [11]

Trilayers are deposited in-situ with a base pressure lower than 10^{-5} Pa. The base layer of Nb is deposited under sputter conditions which produce slight compressive stress in the film of 2-5 X 10^9 dynes/cm^2. Typical deposition rates are 50 nm/min. in 10 mTorr Ar ambient. These conditions have resulted in the best results for Nb/Al-Ox/Nb junctions and we have seen indications that it is desirable for junctions with subsequent nitride layers as well.

The aluminum layers are deposited by oscillating the sample over the target such that a 7-10 nm thick film is grown with about 75 passes for system #1 and about 100 passes for system #2. This method produces a more uniform thickness distribution than by remaining stationary over the target.

Plasma nitridation of the aluminum layer is drawn schematically in figure 2. The substrate manipulator is a grounded cylindrical assembly with capabilities for RF biasing of the bottom chuck The grounded substrate is placed above an electrode driven by a 13 MHz RF generator. Nitrogen gas of 99.999 % purity is flowed into the chamber at ~10 sccm and the pressure is controlled by throttling a turbomolecular pump. RF power of less than 5 W is applied through an impedance matching network to the substrate platform. The DC floating potential developed on the substrate is feedback controlled for the required exposure time. Bias is used rather than RF power to eliminate tuning loss fluctuations.

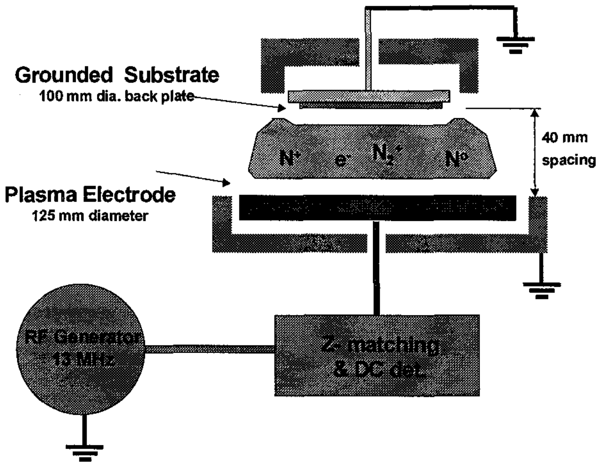

Fig. 2. . Schematic drawing of process system for plasma nitridation of aluminum proximity layer

Counter-electrode deposition of 50 nm thick NbTiN is accomplished without substrate RF bias and near room temperature to prevent excessive damage to the thin aluminum nitride barrier surface. Conditions are adjusted to come as close as possible to the properties of the ground plane described above. However, without heat and bias, the resistivity is nominally 80 µ Ω cm and Tc =14.5 K.

The wiring material is usually a low resistivity normal metal which is deposited by electron beam evaporation after an in-situ Ar ion cleaning step. We have explored the normal metals listed below in Table I. Sources are 6-9's purity and evaporated at a background pressure of 10^{-5} Pa.

TABLE I
NORMAL METAL WIRE INVESTIGATION

Material & Tempeature (°C)	Resisitivity at 4 K (µΩ cm)	Resistace Ratio R (295 K) / R (4 K)
Au @ 30	0.5	5.5
Au @ 200	0.22	11.5
Cu @ 30	0.34	6.3
Cu @ 200	0.26	7.6
Ag @ 30	0.28	7.1
Ag @ 200	0.13	16.7

Our interest in using NbTiN as a superconducting wire up to 1.2 Thz lead us to attempt sputter deposition of heated layers. We found the I-V characteristic to improve for NbTiN wire deposited at 200 °C under vacuum, however R_NA increased by a factor of two.

III. PROCESS VARIATIONS AND RESULTS

Junctions are characterized by low frequency electrical testing in liquid He at near 4.2 K in temperature. Test chips each have 12 various sized square junctions with side dimensions on the lithography mask designed from 0.8 µm up to 5 µm. We have chosen to use the parameter R_NA (product of normal state resistance and junction area) rather than current density because this value is derived by statistically fitting the measured R_N with the junction dimensions. Since the gap voltage (Vg) is typically 3.5 mV, current density (Jc) can be calculated by the Ambegaokar-Baratoff relation $JcR_NA = \pi Vg/4$. [12]

A. Bias Variation

Fig. 4 is a plot of junction R_NA product for DC floating potential values ranging from -30 to -60 V. This data exists for system #2 at the present time, but a similar trend is followed in system #1. The background nitrogen pressure is held constant at 20 mTorr and exposure time is fixed as a family of curves for 1, 3, 5, and 15 minutes. Increasing the floating potential means that both ion energy and density will be increased. The R_NA value does increase with bias and it is inferred that AlN thickness grows faster by increasing bias.

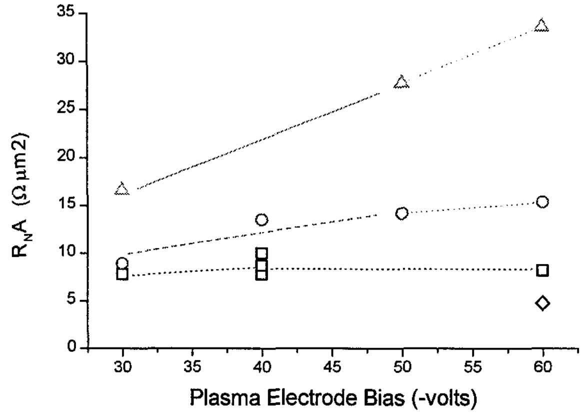

Fig. 3. Junction R_NA produced at various electrode bias voltages in System #2 for exposure times of (♦) 1, (☐) 3, (o) 5, and (Δ) 15 minutes with a background of 20 mTorr N_2.

B. Plasma Exposure Time

The data above is replotted in Fig. 4 as a function of plasma exposure for fixed bias voltages. Lines are drawn to guide the eye only. Scatter in the data for 3 minute exposures at –40 V bias demonstrates the run-to run reproducibility. Corresponding Rsg/R_N is not plotted , but it should be noted our highest quality junctions (Rsg/R_N~20) were produced with exposures between 1 to 2 minutes in system #1 with a bias of – 40 V.

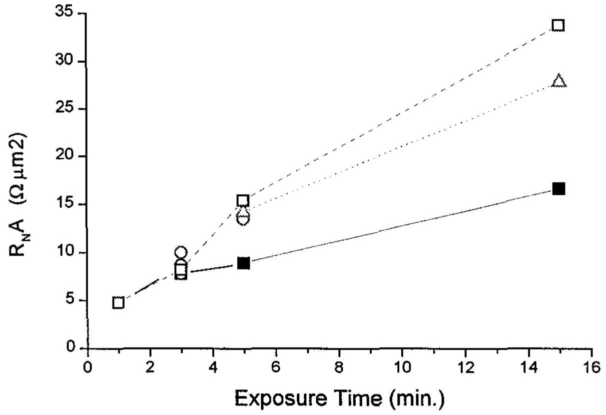

Fig. 4. Junction R_NA plotted as a function of exposure time for electrode bias voltages (♦) –30 V, (O) –40 V, (Δ) –50 V, and (☐) –60 V.

C. Junction Quality

Quality of junctions produced under many different nitridation conditions is plotted in Figure 5 as the resistance ratio Rsg/R_N against junction R_NA product. The sub-gap resistance is taken at 2 mV with the Josephson current supressed by an applied magnetic field. The normal state tunnel resistance is taken at 5 mV. Most of the data for vacuum system #2 is clustered around R_NA = 10 Ω µm^2 since that is the current design target for mixer applications. Values plotted for Rsg/R_N are obtained from statistics on 10 or more junctions of the size range given above which do not have extraneous processing flaws. System #1 produced the best junctions with the highest average ratio of 27 for $R_NA = 8\ \Omega\ \mu m^2$ as shown in Fig. 5.

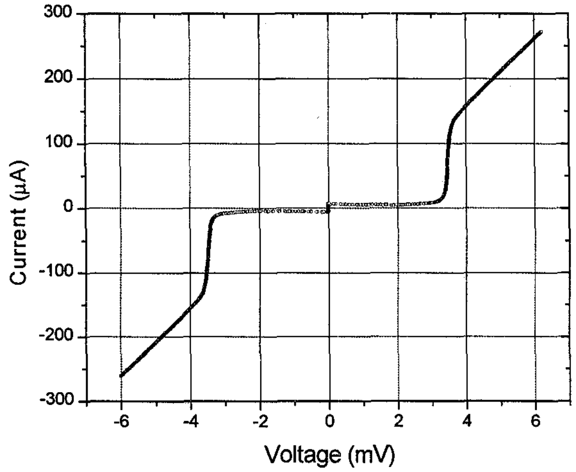

Fig. 1. Nb / Al - Nx / NbTiN junction, area = 0.58 x 0.58 μm ^{2}, RnA = 8 $\Omega\ \mu m^2$, Rsg/Rn = 27,

Junctions with larger R_NA may show higher quality, but processing in not optimized around high R_NA in this set of experiments. System #2 has never produced a junctions with comparable quality to system #1 at the same R_NA value. There is a trend exhibited in both systems to rapidly change junction quality in the range between 3 to 8 $\Omega\ \mu m^2$. Curves are drawn in the plot to highlight the envelopes of the data sets for each process system. Junctions less than 1 $\Omega\ \mu m^2$ have been made with resistance ratio of about 5.

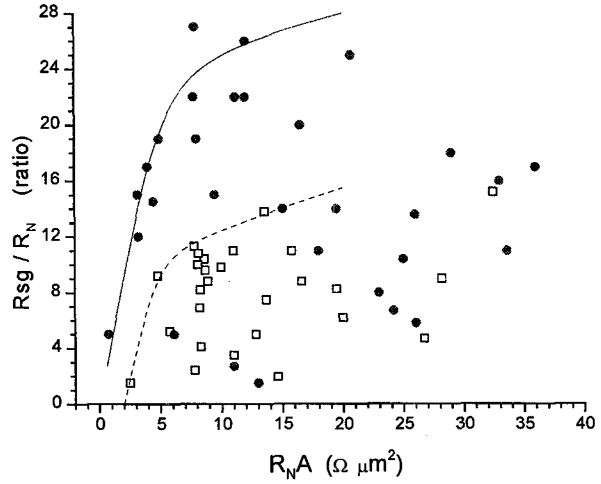

Fig. 5. Junction quality as sub-gap to normal state resistance ratio for various process conditions plotted their resistanace – area product. Results from system # 1 (O) and system #2 (□).

IV. CONCLUSIONS

We have presented our results from process development of Nb/Al-Nx/NbTiN junctions which is focused on RF plasma nitridation of the aluminum proximity layer.

System #1 is shown to produce the higher quality junctions, but system #2 has more characterized results. SIS mixers with $R_NA = 8\ \Omega\ \mu m^2$ and resistance ratios of 27 can be fabricated by this method with reasonable run-to-run variations. Heated wire layer results indicate that low RnA junctions should be made at room temperature, but that annealling in vacuum will improve their quality. Control of R_NA valves can be met to within about 30%. We think that the voltage gap of 3.5mV will bring a significant improvement in bias range for THz SIS receivers. Recent receiver measuremnets up to 900 GHz demonstrate that low noise temperatures have resulted from low-loss NbTiN tuning circuits combined with the junction's sharp I-V behavior. [1]

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