FABRICATION AND DC-CHARACTERIZATION OF NbTiN BASED SIS MIXERS FOR USE BETWEEN 600 AND 1200 GHZ*

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SIS mixers incorporating two-junction, NbTiN tuning-circuits have been designed and fabricated using two different types of tunnel-junctions. The first type of tunnel junction--NbTiN/MgO/NbTiN--has the advantage of a large gap voltage (5 mV), but has a relatively soft I-V characteristic and high specific capacitance as compared to Nb/Al-O_x/Nb tunnel-junctions. The second type of junction--Nb/Al-AlN_x/NbTiN, is a hybrid structure, which has many of the advantages of Nb junctions, with a slightly larger energy gap voltage (3.5 mV) and more robust thermal properties. A detailed description of the deposition techniques used in making the AlN_x devices will be given in a separate paper[1]. In this paper, we discuss the deposition of high-quality NbTiN films, and the trade-off between stress and quality in these films. We also discuss the deposition details for the NbTiN/MgO/NbTiN junctions. We measured the magnetic-penetration-depth of our NbTiN films with SQUID circuits. Using long resonators coupled to Josephson junctions, we measured propagation velocities for our microstrip-line circuits; this measurement gave us an independent estimate of the NbTiN magnetic-penetration-depth and a qualitative measure of RF-losses in the microstrip-lines.

Introduction

With the upcoming construction of the Far Infrared Space Telescope (FIRST) and of NASA's new airborne observatory (SOPHIA), low-noise heterodyne receivers are needed for the frequency band of 700 to 1200 GHz[2,3]. For frequencies below 700 GHz, niobium-based SIS mixers provide nearly quantum limited performance.[4] However, above 700 GHz, Nb has significant RF-losses, so although Nb-based SIS tunnel junctions are still viable mixer elements, the Nb-tuning circuits used to tune out the junction capacitance do not perform well. One alternative is to use Nb tunnel-junctions with normal metal tuning circuits.[5] A better solution is to use a superconductor with a larger energy-gap that will be nearly lossless, for the tuning circuit. Previously we demonstrated that NbTiN was an excellent candidate for low loss tuning circuits at these high frequencies, and we achieved state-of-the art mixer results at 649 GHz, with a hybrid Nb-mixer using a NbTiN ground-plane.[6] Our next step is to fabricate SIS mixers with all NbTiN tuning circuits. If we use all Nb tunnel junctions in these structures, the NbTiN layers will trap quasi-particles in the junction region,

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which will degrade our mixer I-V characteristics. We would like to replace one or both of the junction electrodes with NbTiN to reduce or eliminate this quasi-particle trapping. To accomplish this, we are fabricating SIS mixers using two distinct junction-technologies (NbTiN/MgO/NbTiN and Nb/Al-AlN_x/NbTiN.)

NbTiN Film Deposition and Characterization

NbTiN films are deposited by reactive DC-magnetron sputtering from a Nb-Ti alloy target in a mixture of argon and nitrogen. The vacuum chamber used is pumped by an 20 cm diameter cryopump and typically has a base pressure of $1x10^{-8}$ Torr. 7.6 cm diameter MAC sputter sources from US Thin Films Inc. are used, and the target to substrate separation is approximately 6 cm. A constant Ar flow of 150 sccm was used for all of our depositions; the N_2 flow was varied between 9 and 15 sccm (typically 13) to optimize the NbTiN's properties. High-purity Nb and Ti starting materials are vacuum-arc-remelted to make the Nb-Ti alloy targets.[7] Despite this, we had some difficulty with metal impurities in some of our targets, and x-ray photoemission spectroscopy was necessary to determine the "good" quality targets. We have evaluated several different ratios of Nb to Ti, however, all of our device work was done with 78% Nb to 22% Ti by weight.

There are many ways to characterize our NbTiN films, however, we are primarily interested in is the RF-losses of our films in the terahertz frequency range. Since it is extremely difficult to measure RF-losses at these frequencies, we instead focus on two DC properties to evaluate our film quality. The transition temperature of the films should be above 15 K, so the energy gap will be greater than 2.5 meV; this is a minimum requirement for use at 1.2 THz. In addition, the resistivity of the films just above the transition temperature (typically measured at 20 K) is also a good indicator of film quality with lower resistivities being desirable.

The highest quality NbTiN films are under compressive stress. Since excessive stress is a problem in device fabrication, it is important to measure stress in our films. Film stress was measured by observing the deflection in long, narrow glass or silicon "beams" with NbTiN films deposited on them[8]. The deflection is measured over several millimeters using a Tencor profilometer. Although this method is only accurate to 20 or 30%, it does give us an adequate way to screen out bad films, which would cause a problem in device processing.

The best quality NbTiN films are made by applying an RF-bias at 13.56 MHz to the substrate during the film growth. We believe this increases the quality of our films through added ion-bombardment and electron heating of the film surface. This technique increases the film Tc by a small amount, and it lowers the low-temperature resistance of the films significantly. The disadvantage of this technique is that these films have higher compressive-stress than films deposited with a grounded substrate. Table 1 shows typical deposition conditions and properties for some of our NbTiN films. Also listed in this table are several films with higher titanium concentrations. These films are interesting because they have lower resistivities, but their lower T_cs mean their energy gaps will be too low for use at 1200 GHz. As our targets erode, the sputter conditions need to be continually optimized, however, several general statements seem to hold: the deposition rate should be approximately 40 nm/min for bias sputtered films and 50 nm/min for unbiased films. If the RF-bias is too large (V_{DC}<-75 V), nitrogen is resputtered from the film. High quality films can be grown with higher RF-bias levels, however the optimal nitrogen level is then a strong function of the RF-bias level. To avoid this complication, most of our films are grown with a DC-bias of –50 V, which corresponds to roughly 4 or 5 Watts of absorbed power. Finally, lower pressures and/or higher deposition rates lead to higher quality films, but also to greater compressive stress.

Table 1

Substrate/ Temp.	Pressure (mTorr)	DC-bias (Volts)	Sputter Current (Amps)	Sputter Voltage (Volts)	Rate (nm/min)	σ (dynes/ cm^2)	T_c (K)	ρ_{20K} (μΩ-cm)
Oxidized Si	6.0	0	1.06	-230	50	$6x10^9$	15.2	81
Glass	5.0	-50	0.90	-246	38	$2.5x10^{10}$	16.3	56
Oxidized Si	5.0	-50	0.85	-261	45	$5x10^9$	15.3	63
Mgo/450° C	5.0	-50	1.25	-266	52	-	17.0	37
Si (Nb-Ti 70-30%)	6.0	-40	1.10	-226	44	-	14.6	46
Si (Nb-Ti 65-35%)	5.0	-50	1.20	-295	68	-	13.8	40

Tunnel Junction Fabrication

Fabrication of NbTiN-based tunnel junctions is similar to that of NbN based junctions[9]. There are several differences however. The biggest difference is in controlling the effects of compressive stress in NbTiN. Bias-sputtered NbTiN has the higher compressive stress, and we use it only as a ground plane. If the stress in the ground plane is less than $5x10^{10}$ dyne/cm^2 and the substrate surface is clean, we have no problems getting our films to adhere to the substrate. Before depositing our films we typically use an RF Ar-ion clean at –150 V and then deposit a thin MgO buffer layer. Stress in the counter electrode can also cause problems. The counter electrode must be kept thin (50 nm) to avoid stress relief after the junction-etch. Finally, to make electrical contact to the tunnel junction and to avoid the wiring layer from deadhering from the devices, we use an Ar-ion clean at –150 V. Stress in the wiring layer is kept below $1x10^{10}$ dyne/cm^2.

Our tunnel junctions are patterned using photolithography and reactive ion etching (RIE). Three different gas mixtures are used in our processing. The first step is generally an Ar pre-clean at 30 mT and 120 Watts. With one exception, Nb and NbTiN films are etched in a mixture of CF_2C_2 (16 sccm) CF_4 (4 sccm) and O_2 (3 sccm), at 30 mT and 133 Watts. This mixture of gases provides an anisotropic etch of both Nb and NbTiN. After using this mixture, the wafer is rinsed in water to remove chlorine salts, which can degrade the device's properties over time. For the last minute of the wire etches, we use a different mixture (CF_4--15 sccm and O_2--2 sccm); this is also done to reduce problems with chlorine-salts.

For the hybrid mixers, the junction etch is extremely important because it must go through the Nb counter-electrode, but not the underlying NbTiN ground plane. A similar problem exists for the Nb/Al-O_x/Nb hybrid junctions with the Al tuning circuits. We use a pair of etch monitor samples to accurately control the etch times during the junction etch. The first monitor has only the NbTiN counter electrode, and the second has the complete tunnel-junction without the ground-plane. The junction etch consists of an Ar preclean, followed by the counter-electrode etch, which is terminated as-soon-as the first monitor clears. It is important to stop this step promptly, or an aluminum-fluoride layer is formed, which is difficult to remove. The Al-AlN_x barrier is removed with an Ar etch followed by the Nb counter-electrode etch; this etch is terminated when the second monitor clears. The final etch step is a 1 minute Ar clean to ensure the SiO layer adheres well to the ground plane.

NbTiN/MgO/NbTiN Trilayer Deposition

Trilayer deposition for NbTiN/MgO/NbTiN junctions is virtually identical to that of NbN/MgO/NbN junctions. The base-electrode of our NbTiN/MgO/NbTiN mixers was typically 280 nm and is deposited with an RF bias. Next, the MgO barrier was RF-sputtered from a pressed MgO target. To reduce the deposition rate and promote more uniform coverage, the substrate is swept over the source in a circular arc. After the MgO deposition, the substrate is exposed to a brief oxygen-plasma glow discharge to cure pinholes in the barrier. Finally a thin (50 nm) NbTiN counter-electrode is deposited with the substrate holder grounded.

NbTiN/Nb/Al-AlN_x/NbTiN Hybrid-Junction Deposition

The ground plane of the hybrid-junctions is identical to the NbTiN/MgO/NbTiN junctions. The Nb base-electrode (typically 30 nm) is deposited immediately following the ground plane. The deposition conditions for this layer are optimized for T_c and to yield a slight compressive stress (2-5x10^9 dynes/cm^2). As is the case with Nb/Al-O_x/Nb junctions, this small amount of compressive stress seems to give smoother Nb films and better quality devices[10]. A thin (6-9 nm) Al layer is deposited on top of the Nb; to promote uniform coverage, the substrate holder is swept over the Al target during deposition. The aluminum-nitride barrier is formed by applying an RF-bias to the substrate for approximately 1 minute while it is in a nitrogen ambient (20 mT.) The DC-bias voltage during the nitridation is typically -50 V. This process is discussed in greater detail elsewhere in these proceedings[1]. Finally the 50 nm NbTiN counter-electrode is deposited with the substrate grounded. In some cases, we heated the substrate to approximately 250° C before depositing the counter-electrode. The quality of these devices seemed to be slightly better than the conventional ones, but the results were not conclusive.

Twin-Slot Mixer Design and Fabrication

We have fabricated a number of mixer chips for use in Caltech's twin-slot receiver.[11] The first mixers we fabricated used a mask set designed for hybrid Nb/Al-O$_x$/Nb tunnel junctions and Al wiring. Fabrication of these junctions went very well, but the mask was not designed properly, so mixer results were not optimal. As a result, a new mask set was designed with several changes. We went from a contact mask-aligner to an I-line stepper. The stepper allowed us to reduce the junction size from 1.1, 1.3 and 1.5 µm to 0.7, 0.8 and 0.9 µm, which was deemed necessary to push the operating frequency above 1 THz. The improved layer-to-layer alignment of the stepper (approximately 0.5 µm) also allowed us to reduce our line widths from 2 to 1 µm, thus reducing the parasitic capacitance at the IF-frequency. Because we were concerned about the adhesion of the wiring layer, the second SiO insulation layer was not used. Figure 1 is an optical photograph of an 1150 GHz mixer chip. An unfortunate result of the above changes was an increased chance of open circuits or series weak-links at places where the narrow (2 µm) wire crossed over the edge of the trilayer. This is particularly a problem because a series link in the second crossover does not show up in the DC I-V characteristic because the ¼ wavelength-short is an open circuit at DC frequencies. In past designs, the second SiO layer, which was deposited while rotating the substrate at a slight angle, made the step edge smoother. The second SiO layer reduced parallel shorts at these crossovers. In addition, Nb-based trilayers are defined with a liftoff stencil, so the trilayer edge is tapered, thus reducing step-edge problems. A new mask using wider lines at the crossovers and a second SiO layer is now being designed. We also intend to increase the thickness of our wiring layer from 400 to 600 nm to reduce this problem.

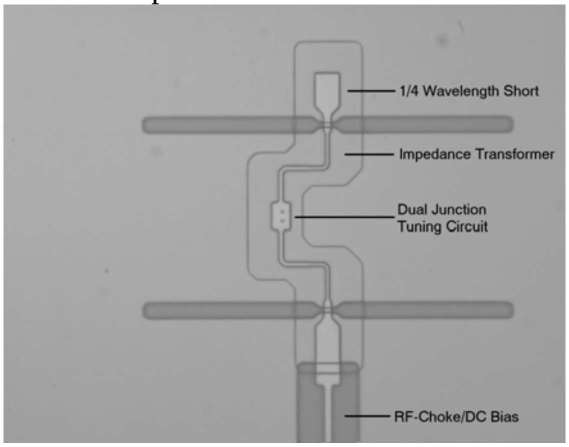

Figure 1: 1150 GHz Twin-Slot Mixer.

Current-Voltage Characteristics of Mixers

Despite the problems we encountered in fabricating mixers using the new mask design, we were able to deliver many devices. Most of these devices exhibited lead switching somewhere between 5 and 10 mV. These devices could be tested in an FTS, where the DC bias is well below the switching threshold, however, the switching-level shifts down in voltage as LO power is applied, so these devices made poor mixers. Detailed mixer and FTS results are given elsewhere in this proceeding.[12]

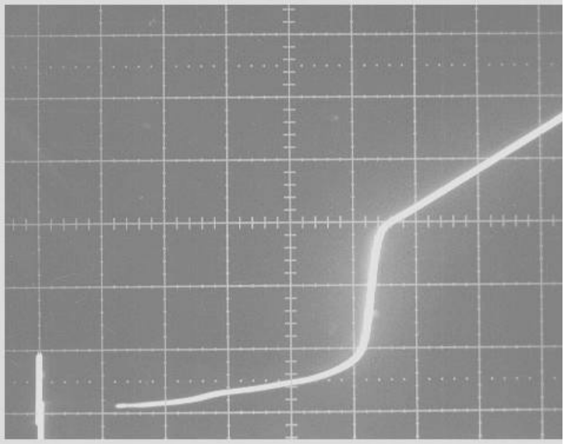

Figure 2: I-V characteristic of a 650 GHz NbTiN/MgO/NbTiN mixer (20 µA, 1mV/div).

Figure 2 is an I-V characteristic of a NbTiN/MgO/NbTiN mixer (two 0.8µm junctions in parallel). The I-V characteristic is nearly identical to an all-NbN device. In high R_NA (100 Ω µm^2) devices, the sum-gap voltage is typically 5.2-5.3 mV. As R_NA decreases, the gap voltage decreases due to self-heating. These I-Vs are fairly "soft" in comparison to a Nb/Al-O_x/Nb device, having excess leakage above half the gap voltage, and a broad turn on at gap voltage. Nonetheless we have achieved respectable mixer results with these devices (T_R=250 K at 638 GHz)[13]. The largest disadvantage these mixers suffer is their increased capacitance as compared to Nb mixers[9], which causes significantly narrower RF-bandwidths, and makes it more difficult to properly design mixer circuits.

Figure 3 is an I-V characteristic of a NbTiN/Nb/Al-AlN$_x$/NbTiN test junction. As compared to the NbTiN/MgO/NbTiN device, this I-V characteristic has much lower leakage and a sharper current-rise at the gap-voltage. In addition, the gap voltage (3.35 mV in this case and typically 3.5 mV) is significantly larger than that of a Nb/Al-O_x/Nb mixer (2.9 mV). An increase in gap voltage of 0.6 mV corresponds to an increase in available DC-bias range of 1.2 mV, which is significant for a THz mixer. The best mixer performance with NbTiN/Nb/Al-AlN$_x$/NbTiN mixers is T_R=195 K at 582 GHz[6], however the conversion loss was 9.6dB, so there may have been some other problem with this mixer.

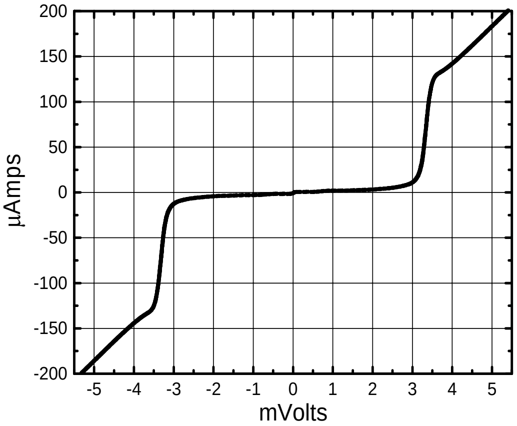

Figure 3: I-V characteristic of a 1.1 µm NbTiN/Nb/AlN/NbTiN test device (R_NA=29 Ωµm^2)

One additional advantage of aluminum-nitride barriers over aluminum-oxide barriers is that they survive much higher temperatures. Niobium/aluminum-oxide junctions begin to degrade at temperatures as low as 130° C. We have baked our completed NbTiN/Nb/Al-AlN$_x$/NbTiN junctions at temperatures up to 294° C in air with only a 10% increase in R_N. The gap voltage of the devices did not change, and the subgap leakage-current did not increase. As a result of the temperature insensitivity, wiring layers can be deposited at elevated temperatures to improve their properties; however, this was not typically done.

Josephson Resonances in NbTiN Circuits

A Josephson junction oscillates at a frequency that is proportional to the voltage applied to it ($\nu = V$x484 GHz/mV, where ν is the frequency and V is the voltage). When a Josephson junction is connected to an electrical circuit with a high-Q resonance, steps will appear in the I-V characteristic at voltages corresponding to the frequency of the resonance. We can use this phenomenon to determine the resonance-frequency of our mixer tuning-circuits. By making circuits with long micro-strip line resonators, we can also measure the propagation velocity of our micro-strip lines[14,15]. Also, since these lines are long, the circuit will not be high-Q, if there is significant loss in the NbTiN films. Thus the presence of a high frequency resonance is an indication of low loss in our NbTiN films.

Figure 4 is an I-V characteristic of a NbTiN/Nb/Al-AlN$_x$/NbTiN mixer with the subgap region expanded to 2 μA/div. There is a resonance at 2.05 mV, which corresponds to 990 GHz. The current rise at the resonance is roughly 10% or the current rise at the gap-voltage, indicating the tuning circuit is low loss.

Figure 5 is an I-V characteristic of a hybrid-junction connected to a 500 μm long NbTiN/SiO/NbTiN microstrip-line. Clear resonances are observable up to 1.9 mV with weaker resonances also observable up to 2.2 mV; this corresponds to frequencies of 920 and 1060 GHz respectively. At 1 THz, the microstrip-line is approximately 20 wavelengths long. Therefore, based on these resonances we are fairly confident our NbTiN films have reasonably low loss at 1 THz. Using on this device and others we estimate the magnetic penetration-depth of our films to be between 200 and 250 nm. It should be noted that this measurement assumes the penetration depth of the base and wire-electrodes are equal, which is not true in general. As a result, the penetration depth we calculate is an average value for the ground-plane and wiring layer. Nonetheless, this data agrees well with calculations using the BCS relation between magnetic penetration-depth, resistivity and Tc.

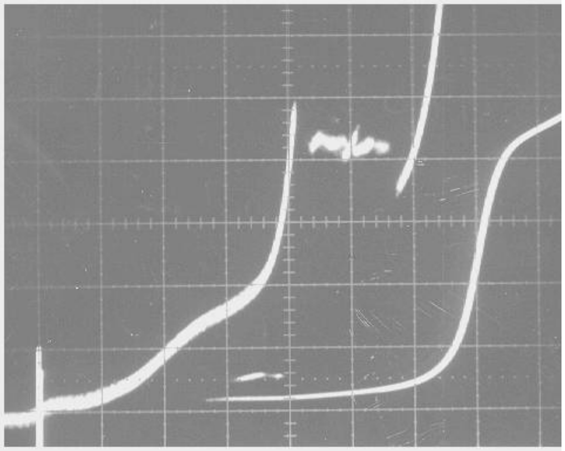

Figure 4: I-V characterisitic of an 1150 GHz, 0.7 μm, NbTiN/Nb/Al-AlN$_x$/NbTiN mixer (20μA, 500 μV/div)

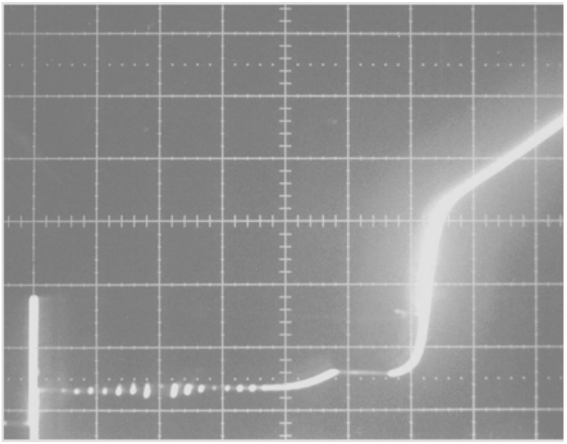

Figure 5: I-V characteristic of a hybrid junction connected to a 500 μm, open-ended microstrip-line stub (20μA, 500 μV/div.)

$$\lambda \approx 100nm \times \sqrt{\frac{\rho(\mu\Omega cm)}{Tc(K)}}$$

Using typical values of T_c=15.2 K and ρ=70 μΩ cm implies that λ=215 nm.

SQUID Measurement of NbTiN Magnetic Penetration-Depth

We can also measure the penetration depth of our films using DC-SQUIDS.[16] The geometry of our SQUIDs is a microstrip-line with two junctions connecting the microstrip-line to the ground-plane. By running a control current along the mircrostrip-line and observing the periodicity of the SQUID modulation, we obtain the inductance of the microtrip-line. We can then calculate the penetration depth of the NbTiN films, although again we are assuming the ground-plane and wiring electrode have the same penetration-depth. These SQUID measurements yield a magnetic penetration-depth of 200-230 nm, which agrees well with our other data. We had hoped to measure the specific capacitance of our devices using these same SQUIDs, but we did not have sufficient time.

Conclusions and Future Work

Mixer results at 600 GHz, and Josephson resonances indicate that NbTiN has very low RF-losses for frequencies below 1 THz. We anticipate excellent mixer performance with both NbTiN/MgO/NbTiN and Nb/Al-AlN_x/NbTiN SIS tunnel junctions at 1 THz. However, we would like to use SIS mixers for frequencies up to 1.2 THz. Based on our all-NbTiN junctions, The gap voltage of our NbTiN films is at least 2.6 mV, so in theory we will be able to get low-loss structures up to 1260 GHz. At this time, we have no data on what our losses are above 1.0 THz. If necessary, we can heat our substrates during the deposition of our NbTiN and we can grow our ground planes on single-crystal MgO substrates to reduce the losses of our NbTiN microstrip-lines.

We are just beginning to design and test Terahertz mixers. One of our primary goals is to get agreement between our theoretical models and our experimental results, so we can design future receivers more accurately. From a fabrication standpoint, we need to produce many devices with identical I-V characteristics, so RF results from several different tuning circuits can be directly compared. In addition, we need to have good DC characterization of $R_N A$, magnetic penetration-depth and junction capacitance. Thus far, we were not able to fabricate NbTiN mixers using our new mask set, which had the correct $R_N A$, high yields and without series weak-links. We are currently redesigning our mask set to reduce problems at ground-plane crossovers. We are also having great difficulty controlling run-to-run variation in the $R_N A$ of Nb/Al-AlN_x/NbTiN junctions[1], and we are looking at a number of possible solutions to this[1].

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[1] B. Bumble, H.G. LeDuc, and J.A. Stern "Fabrication of Nb/Al-N_x/NbTiN Junctions for SIS Mixer Applications Above 1 THz," *This Proceedings*, 1998.

[2] J. Zmuidzinas, G. Blake, J. Keene, T. Phillips, N. Erickson, P. Goldsmith, A. Harris, M . Morris, W. Langer, H. LeDuc, and W. McGrath, "A submillimeter/far-IR heterodyne receiver for SOFIA," *#09.06, Bull. American Astron. Soc.* **191**, Dec. 1997.

[3] T.G. Phillips, J. Zmuidzinas, W.D. Langer, C. Lawrence, and J. Pearson, "The FIRST mission," *#41.21, Bull. American Astron. Soc.* **191**, Ded. 1997.

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[4] J. Carlstrom and J. Zmuidzinas, “Millimeter and submillimeter techniques,” in *Review of Radio Science 1993-1996,* W.R. Stone, ed., pp. 839-82, Oxford University Press, 1996.

[5] M. Bin, M. C. Gaidis, J. Zmuidzinas, T. G. Phillips, and H.G. LeDuc, “Low-Noise 1 THz niobium superconductoring tunnel junction mixer with normal metal tuning circuit,” *Appl. Phys. Lett.***68**, pp. 1714-16, 1996.

[6] J.W. Kooi, J.A. Stern, G. Chattopadhyay, H.G. LeDuc, B. Bumble, J. Zmuidzinas, “Low-Loss NbTiN Films for THz SIS Mixer Tuning Circuits,” *J. IR and MM Waves* , **19**, 1998 (in press).

[7] Targets were supplied by Angstrom Sciences, 1200 Lebanon Rd, W. Mifflin, PA 15122.

[8] G.G. Stoney, *Proc. R. Soc. London Ser.,* **A 82**, p. 172, 1908.

[9] J.A. Stern, B.D. Hunt, H.G. LeDuc, A. Judas, W.R. McGrath, S.R. Cypher, and S.K. Khanna, "NbN/MgO/NbN SIS Tunnel Junctions for Submm Wave Mixers," *IEEE Trans. Magn.*, **MAG-25**, 1989.

[10] T. Imamura, “Fabrication of high quality Nb/AlO$_x$-Al/Nb Josephson junctions: Spttered Nb films for junction electrodes,” *IEEE Trans. Appl. Superconductivity,* **2**, March 1992.

[11] J. Zmuidzinas and H.G. LeDuc, “Quasi-optidcal slot antenna SIS mixers,” *IEEE Trans. Microwave Theory Tech.,* **MTT-40**, 1797-1804, 1992.

[12] J.W. Kooi, J. Kawamura, G. Chattopadhyay, J. Zmuidzinas, J.A. Stern, H.G. LeDuc, and B. Bumble, “Low-loss NbTiN films for THz SIS mixer tuning circuits,” *these proceedings.*

[13] J. Zmuidzinas, J. Kooi, J. Kawamura, G. Chattopadhyay, B. Bumble, H.G. LeDuc, and J.A. Stern, “Development of SIS mixers for 1 THz,”*submitted to the proceedings of the SPIE*, 1998.

[14] R.L. Kautz, “Picosecond Pulses on Superconducting Striplines,”*J. Appl. Phys.,* **49**, pp.308-314, 1978.

[15] A.V. R•is•nen, W.R. McGrath, P.L. Richards, and F.L. Lloyd, “Broad-Band RF Match to a Millimeter-Wave SIS Quasiparticle Mixer,” *IEEE Trans. Microwave Theory and Tech.,* **MTT-33**, 1985.

[16] J.H. Magerlein, “Specific Capacitance of Josephson Tunnel Junctions,” *IEEE Trans. Magn.*, **MAG-17**, pp. 286-289, 1981.