

# Investigation of NbTiN Thin Films and AlN Tunnel Barriers with Ellipsometry for Superconducting Device Applications

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## ABSTRACT

High-energy-gap superconductors with low resistive loss and high-current-density tunnel barriers are important materials to increasing the operating range of THz electronics. This poster presents the development of superconducting NbTiN thin films and AlN tunnel barriers using ellipsometry analysis. NbTiN thin films are deposited by reactive DC magnetron sputtering of NbTi in an argon and nitrogen plasma onto a water cooled, grounded substrate. The effects of total sputtering pressure, gas ratio, target to substrate distance, and source current were examined. AlN tunnel barriers were formed from DC magnetron sputtered thin films with the unique use of an ICP plasma source. The results of this ellipsometry analysis using both discrete in-situ and spectroscopic ex-situ ellipsometers will be discussed.

## NbTiN Thin Films

### Deposition

NbTiN films were deposited by reactive DC magnetron sputtering, using an unbalanced sputtering gun with NbTi target (78 wt% Nb). Ar and N<sub>2</sub> gas flow rates were controlled by independent mass flow controllers. A gate valve before the vacuum pump was throttled to maintain sputtering pressure. Films were deposited with varying pressures, Ar:N<sub>2</sub> ratios, source currents and target to substrate distances. To evaluate the quality of films deposited under a variety of conditions, stress, resistivity, and critical temperature (T<sub>c</sub>) were measured.

### Optical Characterization

The index of refraction (n) and extinction coefficient (k) of the NbTiN films were measured using a UVISSEL spectroscopic ellipsometer from HORIBA Jobin Yvon. Ellipsometry measures the change in polarization of light reflected from a material surface. This data is used to calculate material properties such as film thickness, index of refraction, and extinction coefficient.

Measurements were taken at an incident angle of 70° over the wavelength range of 232.8nm to 732.8nm with a 10nm step size. Because the thickness of the films is greater than the penetration depth of the light in the films, the films were treated as a single substrate layer.

### Results

The comparison between optical properties taken at a wavelength of 632.8 nm and stress, resistivity, and critical temperature is shown in the figures to the right. The relationship between film stress and optical properties is shown in Figure 1. There appear to be two distinct relationships in the plot shown by dashed and solid lines, but no distinct correlation between stress and optical properties.

The relationship between resistivity and optical properties is shown in Figure 2. The figure shows a roughly logarithmic relationship with the index of refraction decreasing and extinction coefficient increasing as resistivity decreases. Remarkably there are no large discontinuities in the curve despite the large variety of conditions used to deposit the films.

The relationship between critical temperature and optical properties is shown in Figure 3. As with resistivity, a strong relationship between T<sub>c</sub> and optical properties is seen with the index of refraction decreasing and extinction coefficient increasing as the T<sub>c</sub> increases. For T<sub>c</sub> above 14K, it becomes difficult to distinguish between the optical properties of the films. Improved ellipsometry models including a layer of surface roughness may help to distinguish between these films.

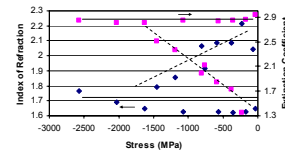


Figure 1. Index of refraction and extinction coefficient at 632.8nm as a function of stress. The films along the dashed line were deposited at varying pressures and Ar:N<sub>2</sub> ratios. The films lying along the solid line were deposited at a pressure of 4 mTorr at varying Ar:N<sub>2</sub> ratios.

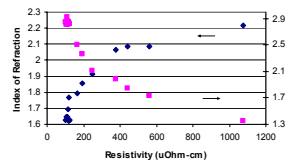


Figure 2. Index of refraction and extinction coefficient at 632.8nm as a function of resistivity.

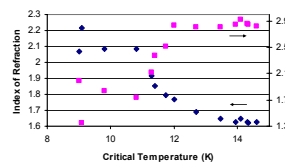


Figure 3. Index of refraction and extinction coefficient at 632.8nm as a function of Critical Temperature.

## AlN Tunnel Barriers

### Plasma Nitridation

AlN films were formed using an Al overlayer process. 100nm of Al was sputtered on a 50nm SiO<sub>2</sub>/Si wafer and then exposed to a nitrogen plasma. The plasma was formed using an inductively coupled plasma (ICP) source from CCR Technology. The use of an ICP allows for independent control of ion energy and ion current density that is not possible using parallel plate sources. The manual energy setting on the ICP source ranges from E(0) to E(10). Although the exact ion energy of the plasma is not known at this time, it is believed to vary linearly from ~20eV to ~175eV. The ICP source can operate at RF powers as low as 10W and up to 600W.

### Thickness Measurements

The thickness of the AlN layer was measured in-situ using a Digisil discrete ellipsometer from HORIBA Jobin Yvon with a wavelength of 632.8nm at an incident angle of 75°. For film thicknesses below ~100Å, the change in ellipsometer data is proportional to the product of the refractive index and thickness [1]. The data presented here was modeled with a fixed index of refraction and varying thickness. The real-time capabilities of in-situ ellipsometry allow the evaluation of AlN barrier thickness without the need to fabricate finished devices.

### Results

Figure 4 shows the change in AlN thickness with varying ion energy and ion current density settings. An increase in AlN thickness is seen with increasing energy setting. At first glance it appears that the thickness of the AlN layer is increasing with RF power, but further examination suggests that the thickness is saturating. Although the thickness of films deposited at higher RF powers increases rapidly at first, it quickly levels off while the thickness of films deposited at lower RF powers increases slowly at first but does not level off as quickly. Figure 5 shows the thickness of AlN films formed at E(2) and 150W with plasma exposure times of 1min, 5min, and 10 min with thicknesses of 10Å, 18Å, and 20 Å respectively. This figure further illustrates a saturation of the film thickness with plasma exposure time and also illustrates the repeatability of this process.

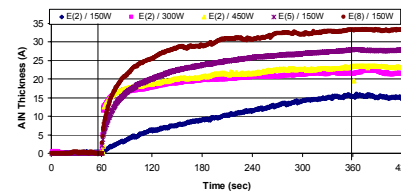


Figure 4: AlN thickness as a function of ion energy setting and RF power. The film was exposed to the plasma for the five minute time period between the two vertical lines.

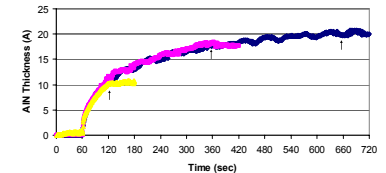


Figure 5: Saturation of AlN thickness over time. Plasma exposure times of 1 minute, 5 minutes, and 10 minutes at E(2) and 150W. Plasma exposure started at 60 sec and ended as indicated by arrows.

## SUMMARY

We have shown that ellipsometry can be used to monitor the deposition process for both NbTiN and AlN films. For NbTiN films the change in optical properties directly reflects the change in the materials critical temperature and resistivity. This can be used to monitor the quality of the film over time and can even be used to monitor films used in devices. In-situ discrete ellipsometry has been used to measure the thickness of AlN layers formed by plasma nitridation. The use of an inductively couple plasma source has allowed us to control ion energy and current density independently. This has allowed for a direct study of the effects of changing ion energy and ion current density on the thickness of the films. We have found that the thickness of the films is strongly dependent on the ion energy and the ultimate thickness of the films has a negligible dependence on the ion current density with the thickness saturating over time. If a plasma with sufficiently low ion energy, corresponding to the desired film thickness, is used, the sample can be exposed to the plasmas for an extended time without increasing barrier thickness. This may allow for formation of films with fewer vacancies and smaller leakage currents in finished devices.