# RHEED Image Analysis on Epitaxial Niobium Thin Films

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## Introduction

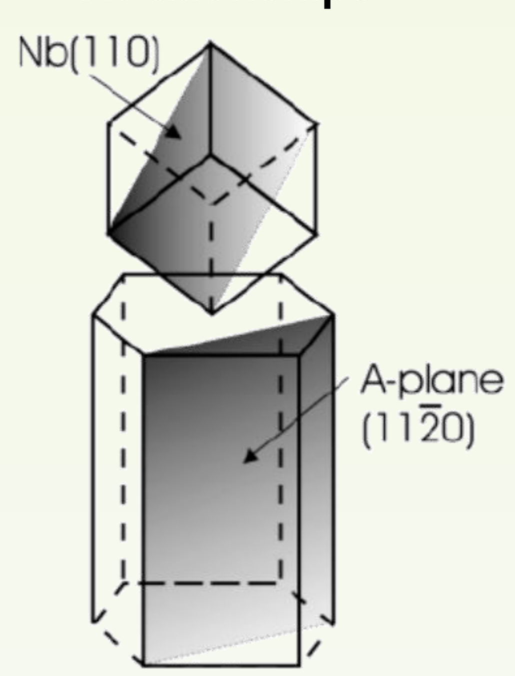
The need to improve superconducting thin film coatings for radio frequency (SRF) cavities used in linear accelerators, has inspired recent niobium thin film research. To better understand the SRF properties in thin film niobium, correlated studies of structure, surface morphology and SRF performance are underway. Here we present our recent work on epitaxial growth of niobium on a-plane sapphire as model system. This includes an in-depth look at the variation in the strain of the film versus its thickness by measuring the lattice parameter of the surface layer using reflection high-energy electron diffraction (RHEED).

## Niobium Thin Films

### Advantages of Niobium Thin Films

- SRF properties are essentially a surface phenomenon (RF penetration depths less than 1 micron). Thin film techniques may offer a degree of fine-control over the active SRF surface leading to improved performance characteristics.
- Next generation films and devices capable of maintaining large field gradients beyond the fundamental limits of bulk niobium (i.e. the multilayer surfaces proposed by A. Gurevich [7]) are reliant upon the understanding and controlled growth of intervening thin film layers.

### Anticipated Epitaxial Relationships



Adapted from A.R. Wildes [5]

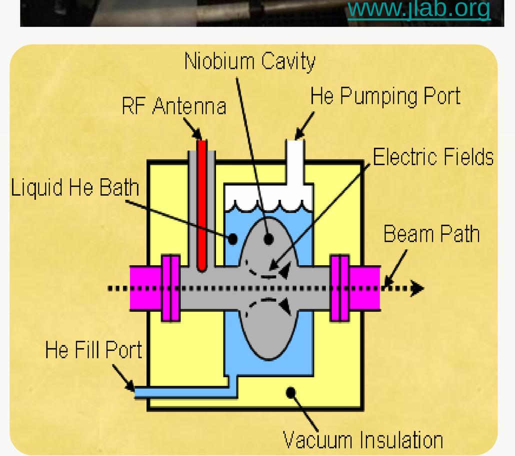
### Growth of Niobium Thin Films

- Our samples are fabricated using UHV DC magnetron sputtering. The epitaxial growth of niobium on various sapphire planes is well reviewed in the literature (A.R. Wildes [5]). A-plane sapphire as a substrate is a suitable proving ground for niobium thin film studies due to the low lattice mismatch (~1.9-12%) and comparable rates of thermal expansion.

## SRF Properties

- It is widely held that scattering (from impurities, lattice defects, etc.) and local field enhancements (from surface topography, surface chemistry, etc.) are the dominant source of failure modes and degradation in SRF cavities. Intuitively, anything that reduces the mean free path of Cooper pairs may diminish performance.
- Performance indicators: A high Residual Resistance Ratio (RRR) – directly proportional to the mean free path of the electrons in BCS theory – is desirable. RRR is the ratio of the resistivity at room temperature to the resistivity at $T_c$ (superconducting transition temperature).

### Niobium SRF Cavity

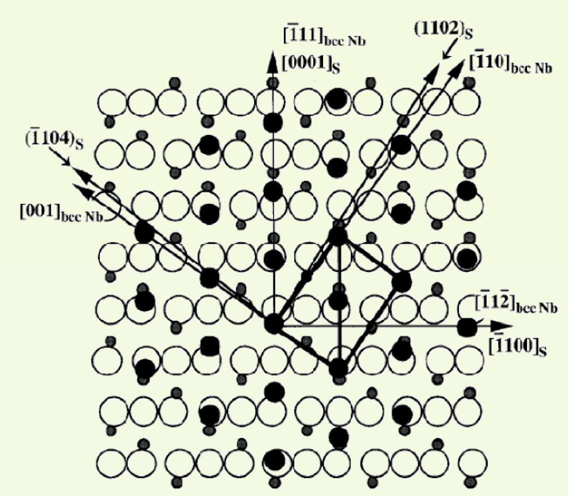



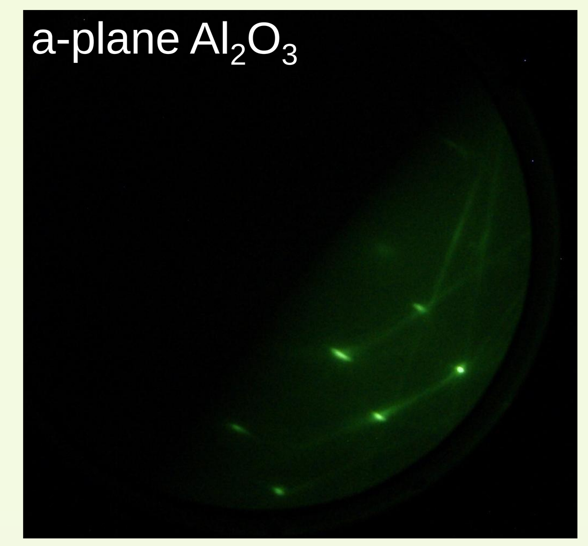

### Comparison of RRR values

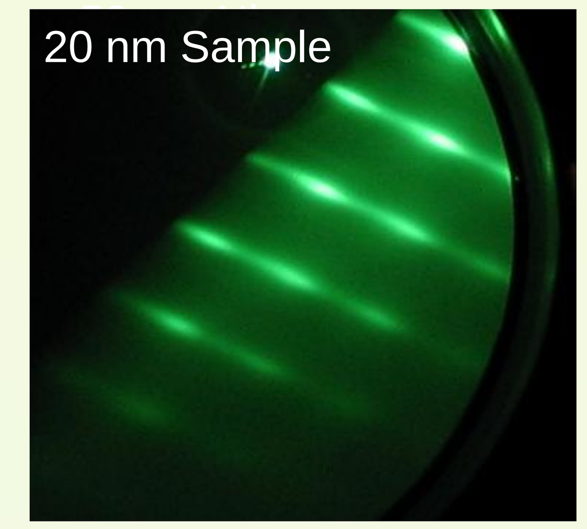
| Group | Thickness (nm) | RRR |
|---|---|---|
| Lukaszew | 600 | 96 |
| S.A. Wolf [2] | 600 | 82 |
| G. Wu [3] | 235 | 50.2* |

* RRR values for niobium thin films is highly dependent on thickness

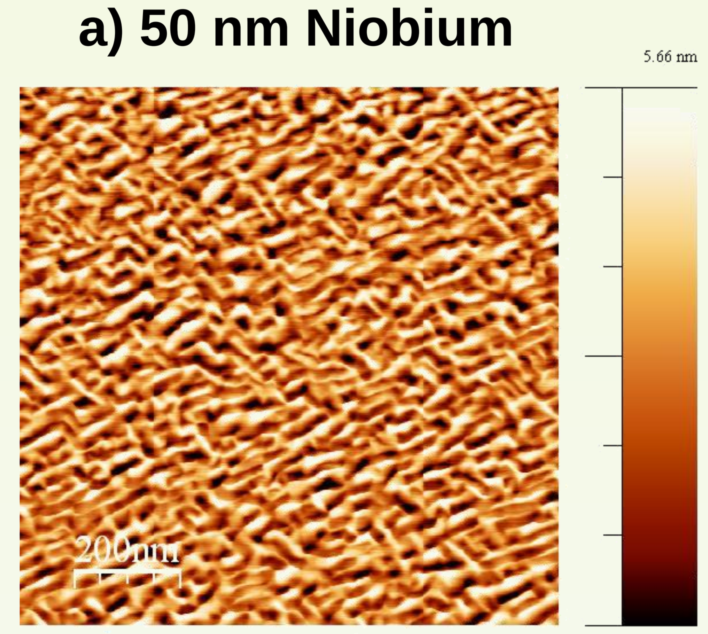
## Surface Morphology and Structural Correlation in: Nb(110)||$Al_2O_3$(11-20)


Atomic view of a-plane epitaxy [1]

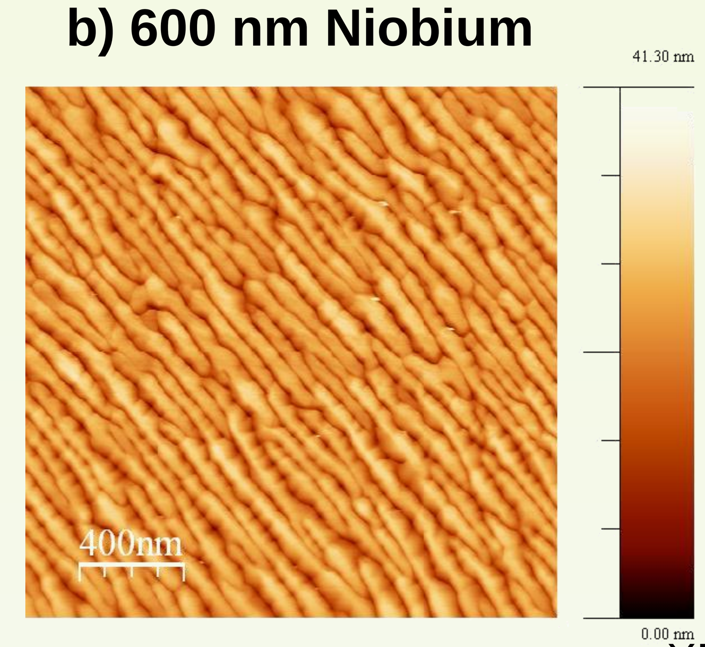


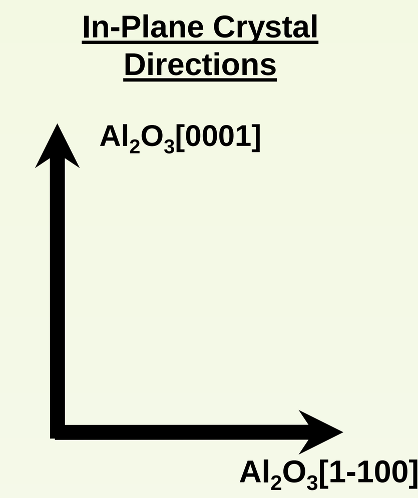



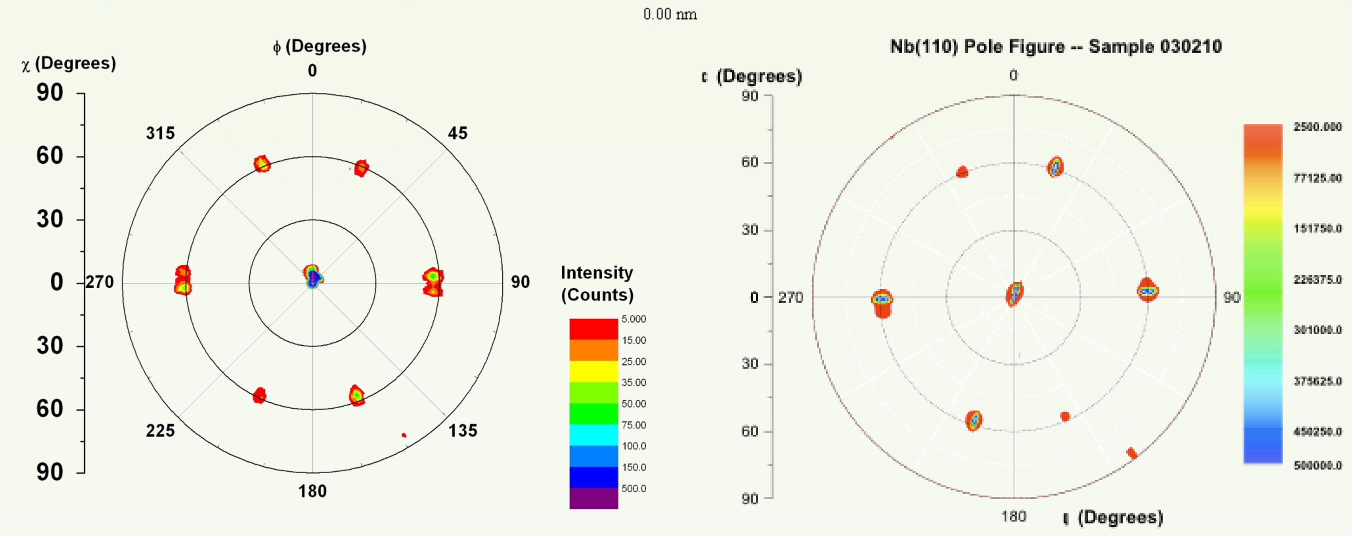

### a) 50 nm Niobium




### b) 600 nm Niobium

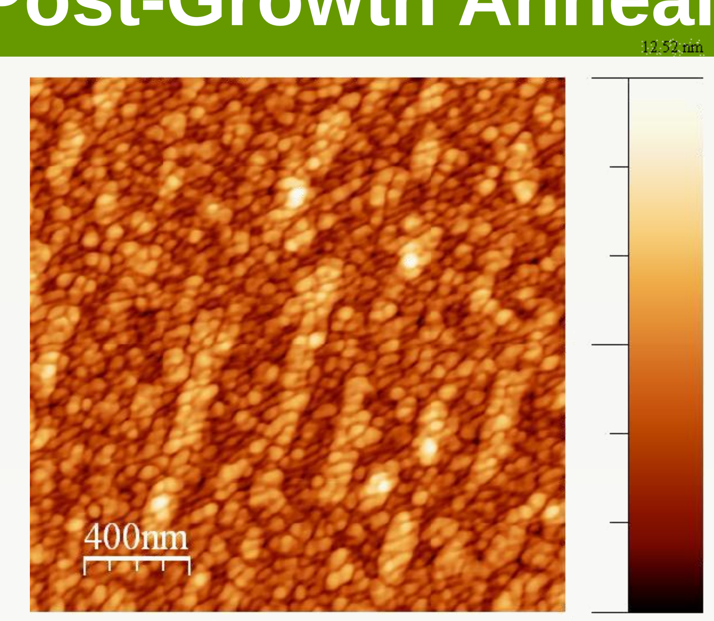



XRD pole figures about the Nb(110) peak indicate (011) texturing along the two possible in-plane orientations in the thinner films while a strong preferred orientation was observed in the thicker film, consistent with the observed surface morphology.

## Post-Growth Annealing




The thickest sample, demonstrating a good RRR value, was placed in a custom built tantalum oven and a4nnealed at a high temperatures (900 °C) for one hour. Spurred by the significant morphological changes, an investigation into the activation energy for this thermally driven process is underway. Further studies into the potential for post-growth annealing treatments to improve the final SRF surface are planned.

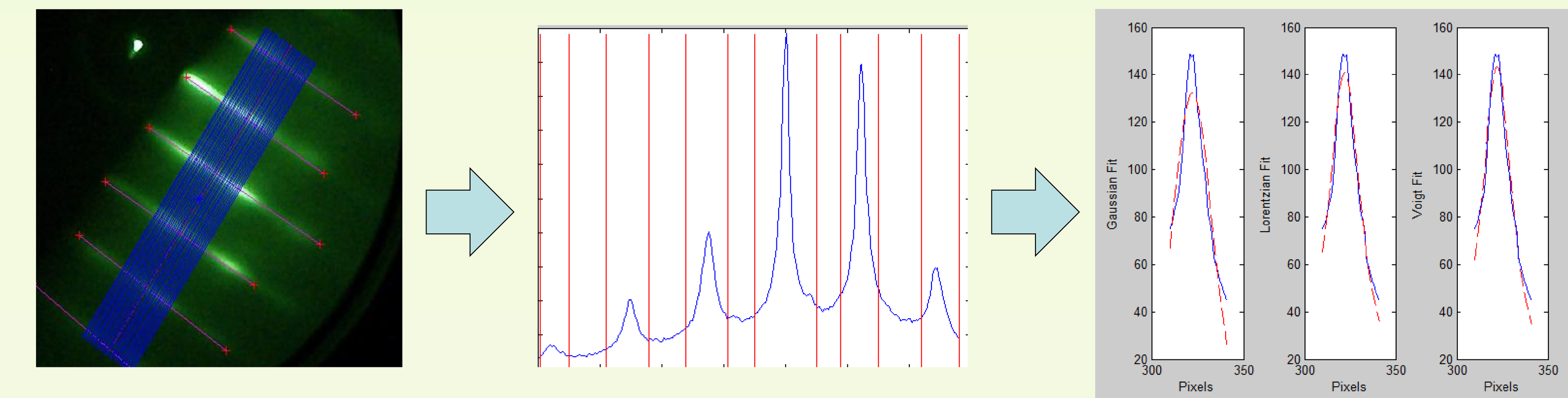
## RHEED Image Analysis

- RHEED images were collected for epitaxial niobium for varying thicknesses and growth parameters.
- Image processing with MATLAB allows for a systematic way to abstract information related to surface crystallinity and morphology.
- A specific image can be used to calculate the lattice parameter corresponding to the topmost layer of the sample using the following equation:

$$a^* = \frac{2\pi W}{\lambda L}$$

- Curve-fitting models are then applied to obtain quantitative information to extract the in-plane strain and lattice parameters.

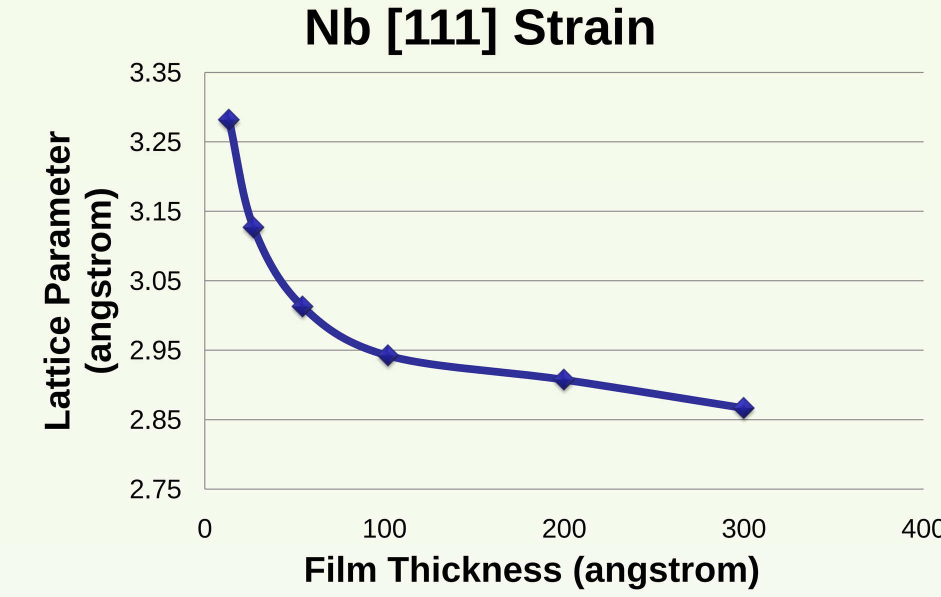
## Strain Evolution and Structure

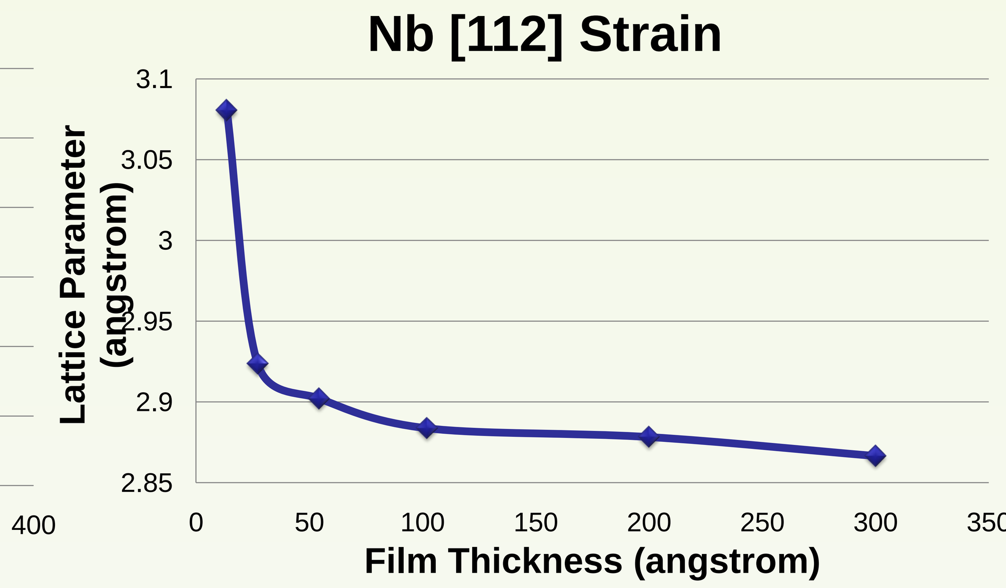


### Strain Evolution

In heteroepitaxial growth, the bulk lattice parameters of the film and the substrate are not commensurate. Thus, strained growth occurs wherein the film's lattice spacing deviates from bulk equilibrium values. As the film grows thicker, the strain may be relieved by the formation of lattice defects such as vacancies or dislocations.

RHEED (Reflection High Energy Electron Diffraction) is a surface sensitive technique that was used to characterize strain evolution in this case. The niobium lattice parameter at the surface can be determined by analyzing the spacing of the characteristic streaks.




## Future Work

- Extend epitaxial growth of Nb to other substrates (c-plane sapphire, synthetic and natural mica, single crystal copper, etc.).
- Systematic studies on the impact of deposition parameters (such as substrate temperature) on the surface morphology and RRR values.
- Investigations into the activation energies required for surface morphological changes arising from high-temperature, post-growth annealing.
- Investigate methods for accelerated growth rates such as HPIMS (High Power Impulse Magnetron Sputtering) with the possibility of a self-sputtering mode.
- Commissioning *in situ* STM for surface characterization.
- Growth studies on other superconducting thin films of interest (e.g. NbN, $Nb_3Sn$, etc.) as well as potential insulating materials, like MgO, for superconducting/insulating/ superconducting multilayer studies.

## References

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