# Stoichiometry and Thickness Dependence of Superconducting Properties of Niobium Nitride Thin Films

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# Collaborators & Acknowledgements

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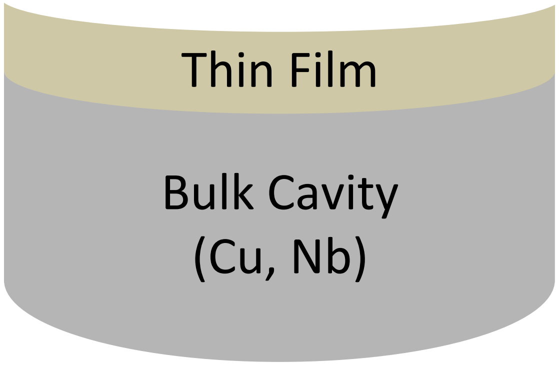
# Overview

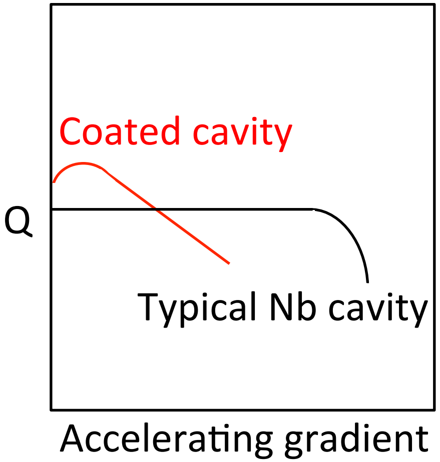
- Motivation
- Niobium Nitride Thin Films
  - Growth and Characterization
  - DC Superconducting Properties
- Issues in Thin Film Growth
- Conclusions and Future Work

# Motivation

- Current Nb cavities are expensive, difficult to shape, and have poor thermal conductivity.
- Other metal cavities (e.g. Cu) are less expensive, so coat the interior of these with a superconducting thin film.
  - Performance issues – Q slope, etc.



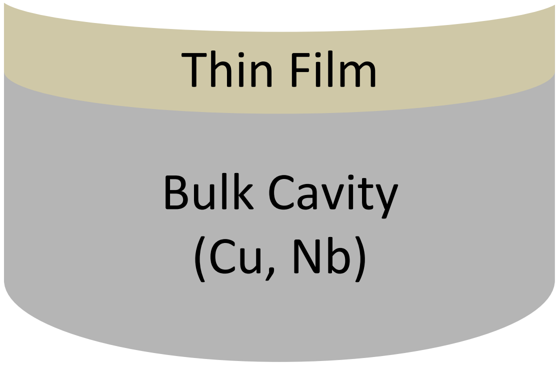
**Schematic of coated cavity**



**Representative Q slopes**

# Motivation

- There is obviously a great deal of interest in improving the quality of the thin films used.
- For films thinner than their London penetration depth ($d < \lambda_L$), there is an enhancement in $H_{C1}$.
  - Previously shown in $MgB_2$.[1]



**Schematic of coated cavity**

$$B_{C1} = \frac{2\phi_0}{\pi d^2} \ln\left(\frac{d}{1.07\xi}\right)$$

**Lower critical field of a superconducting thin film**

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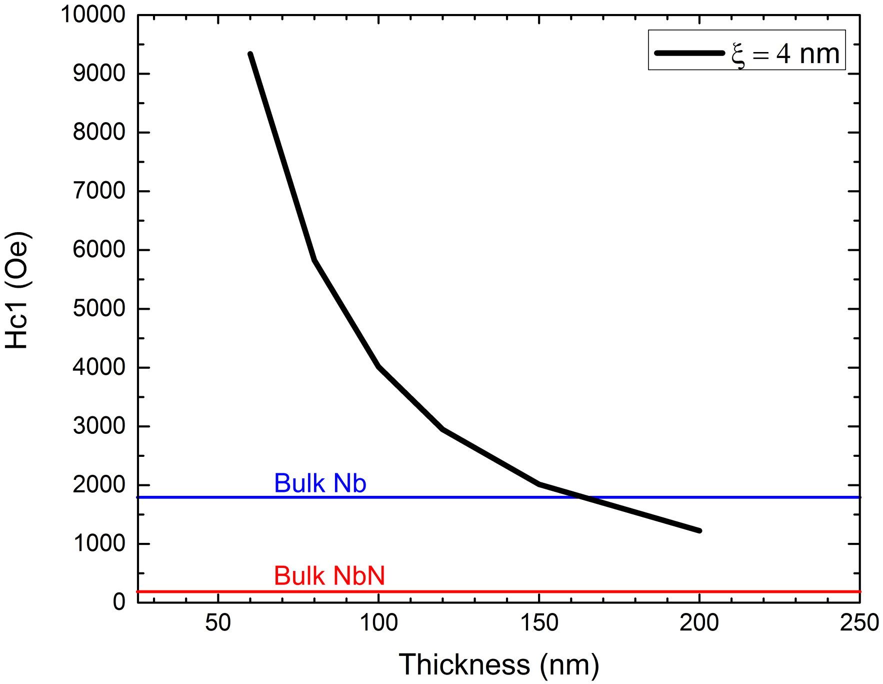
[1] D. B. Beringer *et al. IEEE Trans. Appl. Supercond.* **23**, 7500604 (2013).

# Motivation

- Niobium nitride (NbN) is a promising material for thin film coatings.
  - Higher $H_C$ and $T_C$ than niobium (Nb)
  - Binary compound, achievable growth temperature



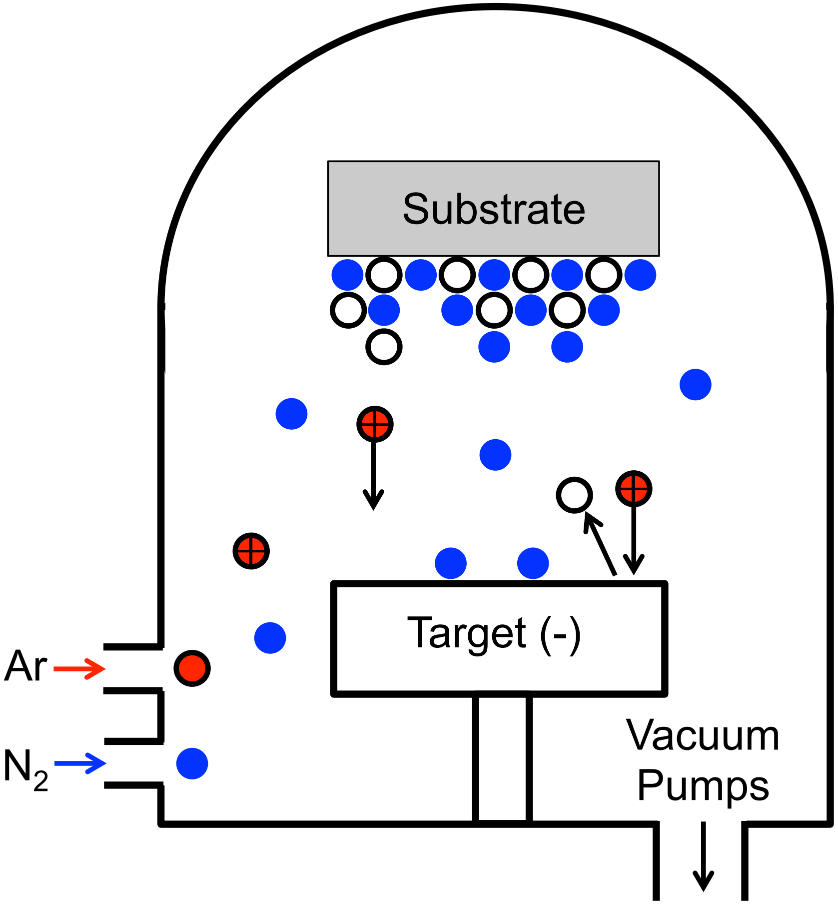
**Predicted $H_{C1}$ enhancement for NbN thin films with thickness less than their London penetration depth.**

M. R. Beebe *et al. J. Vac. Sci. Technol., A* , **34**, 021510 (2016).

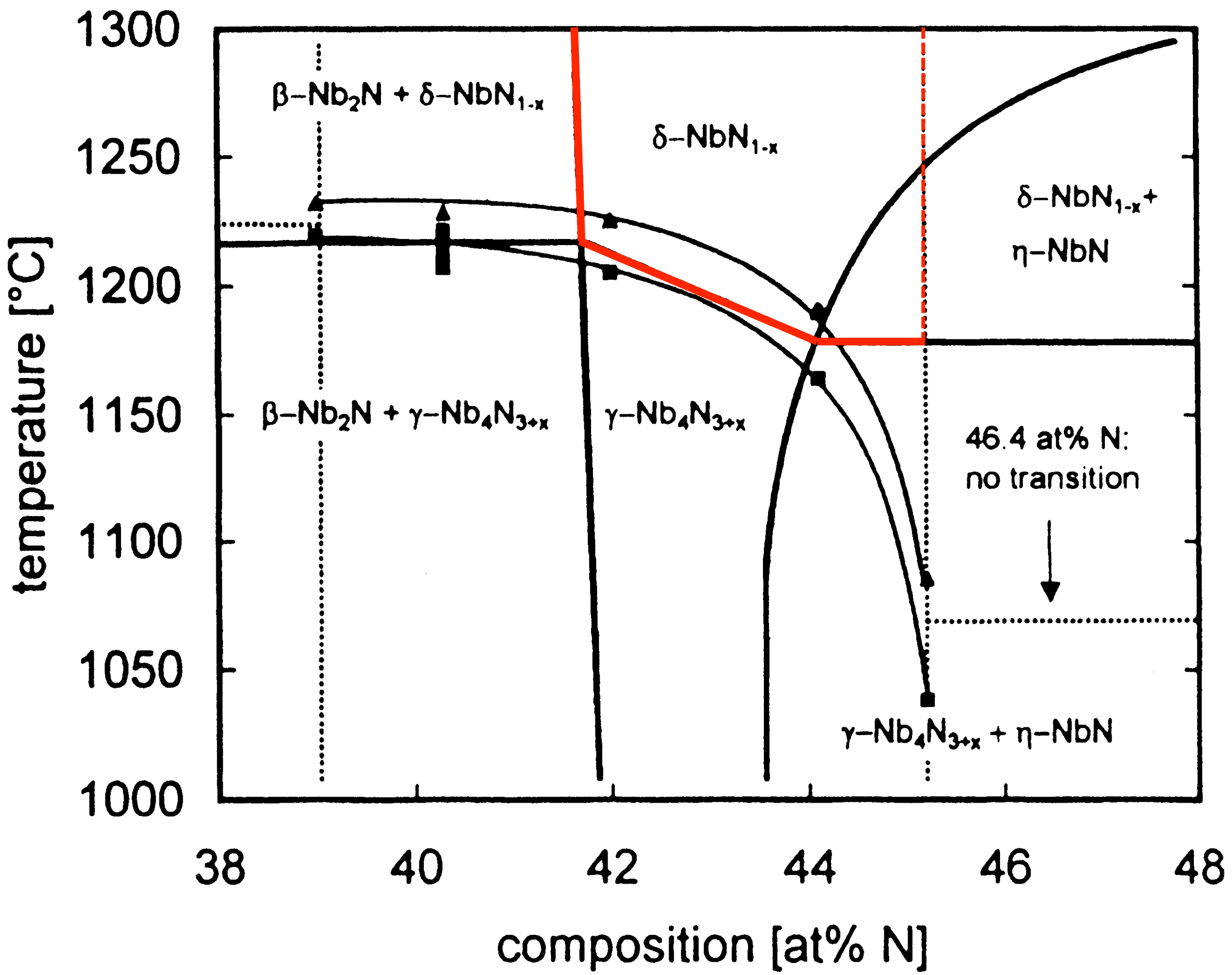
| | $H_C$ | $H_{C1}$ (Oe) | $T_C$ (K) |
|---|---|---|---|
| **Nb (bulk)** | 2000 | 1800 | 9.23 |
| **NbN (bulk)** | 2300 | 200 | 16.2 |

# NbN Thin Film Growth

- A thickness series of NbN thin films were reactively sputtered on MgO(100) substrates.
- NbN grows epitaxially on MgO, making the characterization of the NbN thin films much more straightforward.



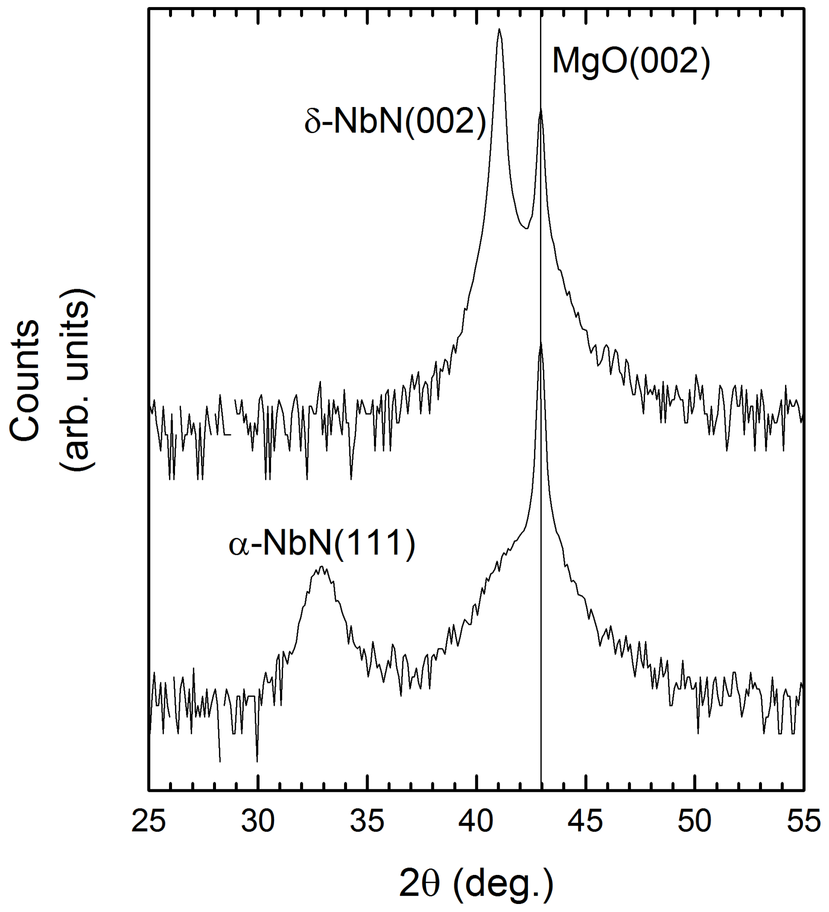
# NbN Thin Film Characterization



W. Lengauer, M. Bohn, B. Wollein, and K. Lisak, *Acta Mat.* **48**, 10 (2000).

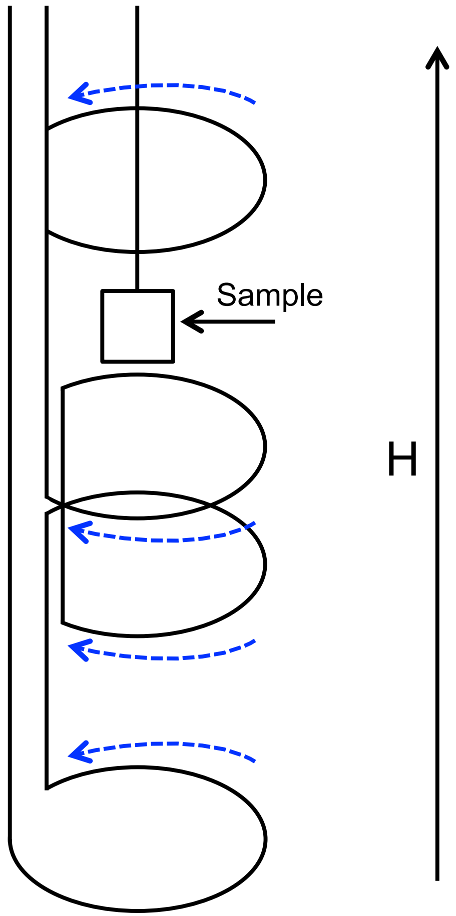
# NbN Thin Film Characterization

- The stoichiometry of each phase determines the corresponding lattice parameter.
- X-ray diffraction was used to determine the lattice parameter (and thus phase), mosaic structure, and average grain size for each film.



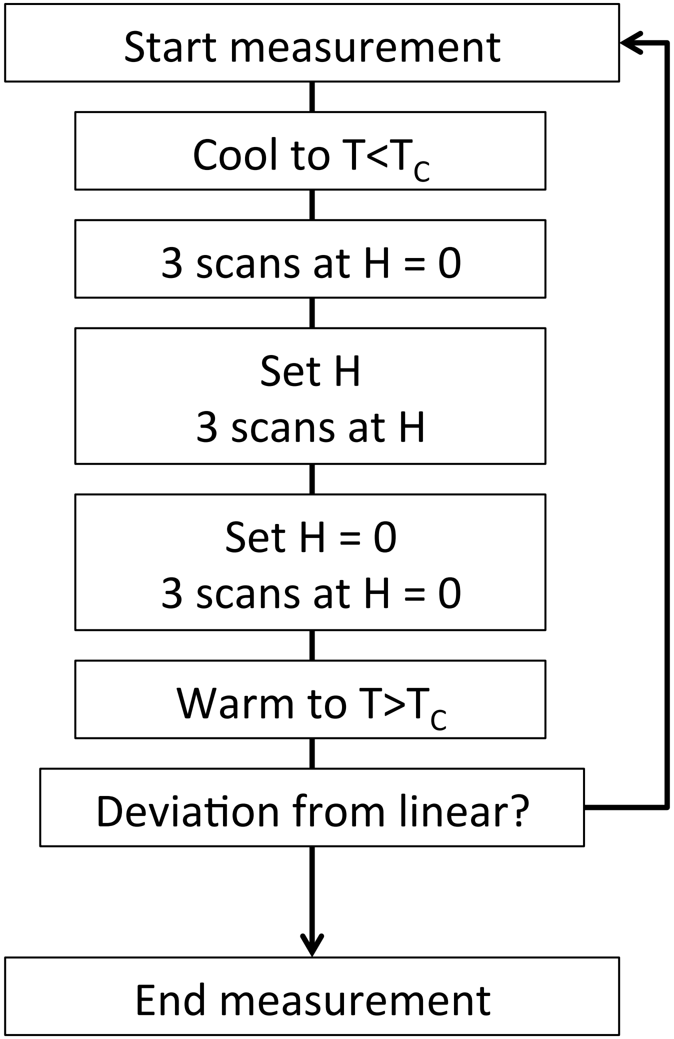
# NbN Thin Film Characterization

- Using a Quantum Design MPMS, DC measurements were made of the $T_C$ and $H_{C1}$ of each NbN film.
- Sample alignment is critical, as is the quality of the film surface.
  - Any field not parallel to the film surface will introduce vortices.



# NbN Thin Film Characterization

- All $H_{C1}$ measurements thus are at least a slight underestimate.
- Two methods for $H_{C1}$:
  - When M vs. H deviates from the Meissner slope
  - Method outlined by C. Bohmer *et al.*, *Supercond. Sci. Technol.* **10**, A1-A10 (1997), which removes contributions from trapped M



# NbN Thin Films

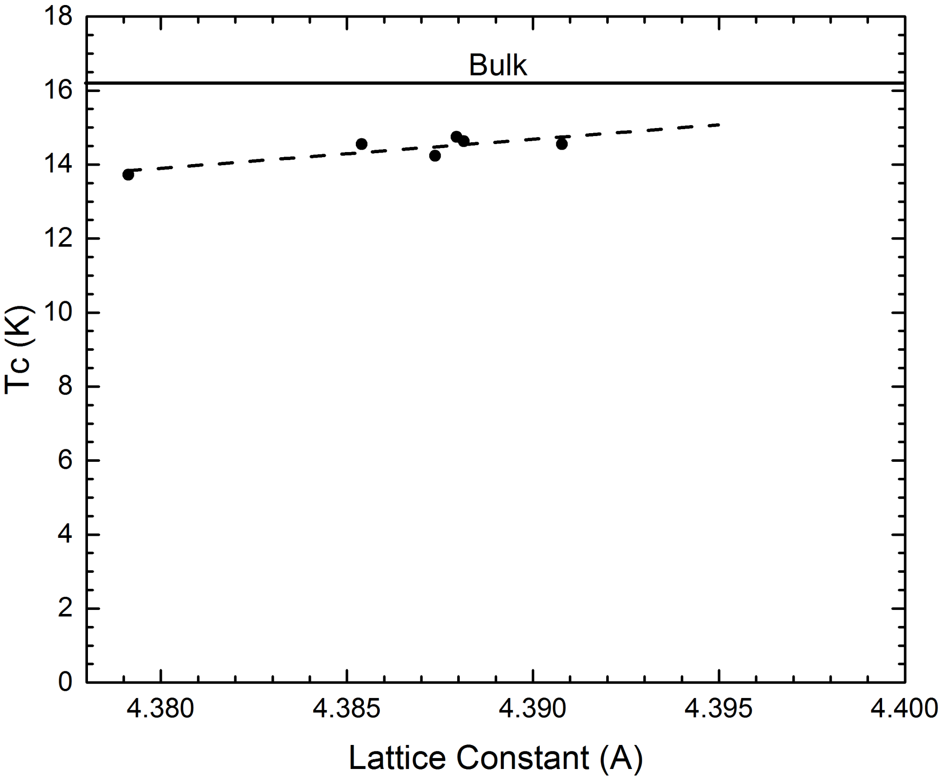
| **Sample thickness (nm)** | **a (Å)** | **$T_C$ (K)** | **$H_{C1}$ (Oe)** |
|---|---|---|---|
| 36 | 4.349 | 10.98 | 76 |
| 120 | 4.357 | 9.6 | 140 |
| 121 + 5 Au | 4.361 | 13.2 | 1600 |
| 85 | 4.379 | 13.72 | 500 |
| 150 | 4.385 | 14.55 | 270 |
| 100 | 4.387 | 14.24 | 350 |
| 120 | 4.388 | 14.75 | 950 |
| 80 | 4.388 | 14.62 | 1000 |
| 60 | 4.391 | 14.55 | 700 |
| Bulk | 4.395* | 16.2 | 200 |

*Reported bulk values range from 4.38 Å to 4.42 Å.[2]

[2] E.I. Isaev *et al*. *J. Appl. Phys.* **101**, 123519 (2007).

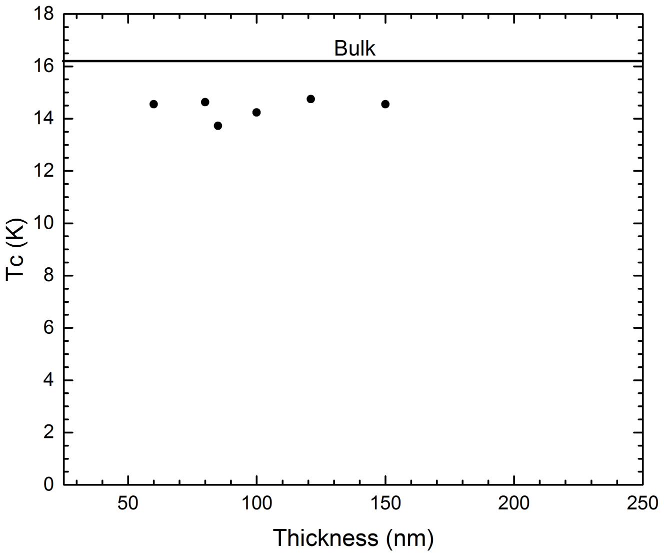
# NbN Thin Films

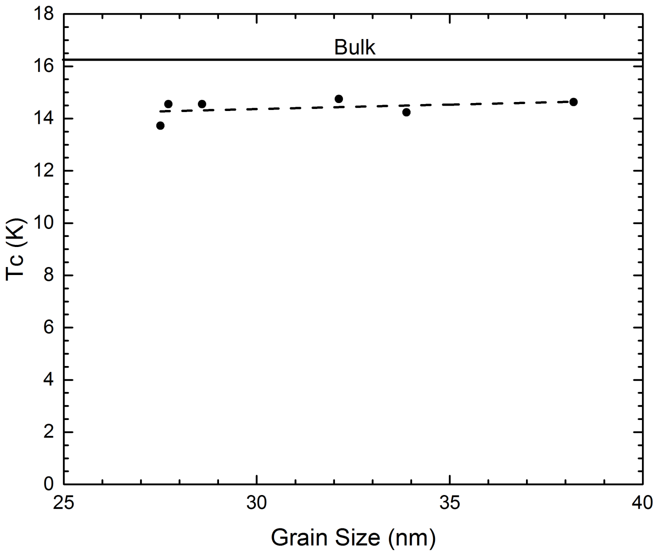
- In the bulk-like regime, $T_C$ shows a linear increase with increasing lattice parameter, although a thin film with the bulk lattice parameter a = 4.395 Å would not quite have bulk $T_C$ due to the defects inherent in all sputtered thin films.



# NbN Thin Films

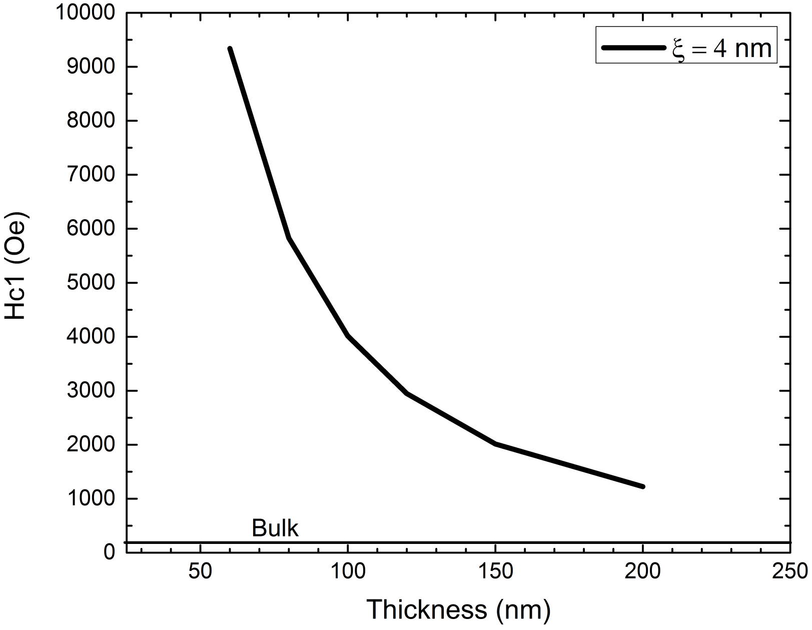
- There is no real correlation between film thickness and $T_C$ - as long as the films are bulk-like, they will have roughly the same $T_C$.
- Increased grain size, however, does lead to a slight increase in $T_C$.
  - Fewer intergrain boundaries lead to less electron scattering.

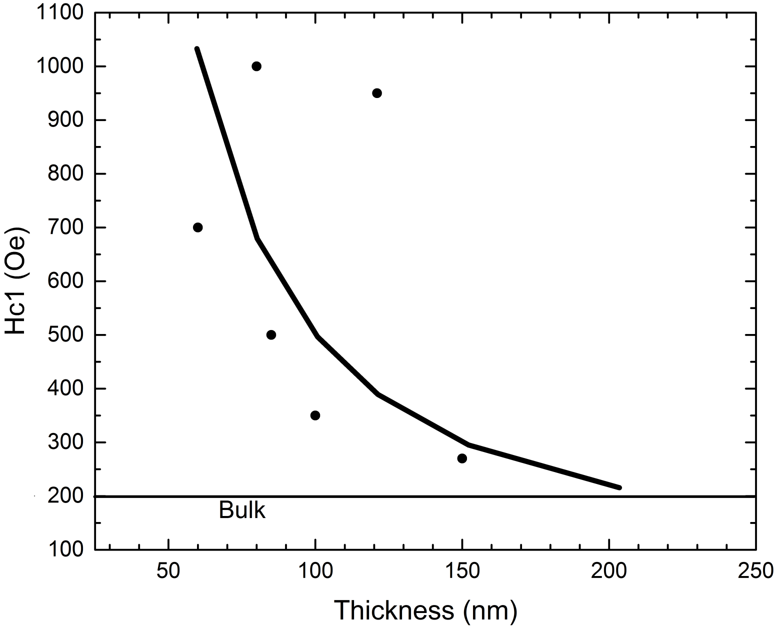




# NbN Thin Films

We see the expected trend in $H_{C1}$ with respect to thickness, although the measured values are about one order of magnitude smaller than expected. This difference can be explained by both film defects and the difficulty of the $H_{C1}$ measurements.





# Issues in Thin Film Growth

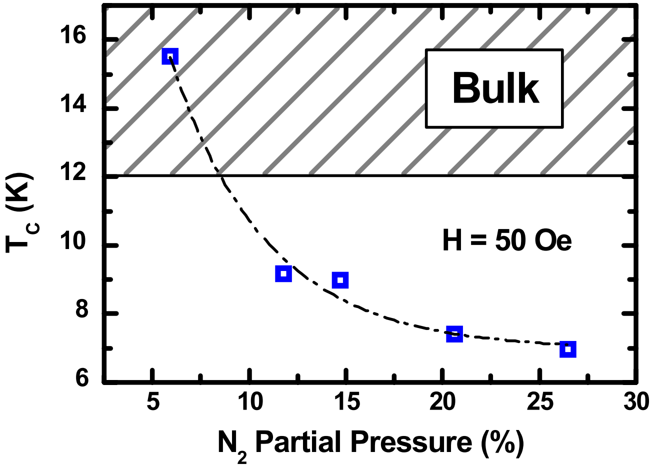
- Because the δ phase is such a small part of the overall NbN phase diagram, finding the right set of deposition parameters is crucial. These parameters include
  - system geometry
  - Ar/$N_2$ ratio (partial pressure)
  - sputtering power
  - substrate temperature

# System Geometry

- System geometry heavily affects deposition – parameters that work for growing a coupon sample on a flat heater can be very different from those required to sputter coat a cavity.
- Changes in our deposition system led to a recalibration of growth parameters.
  - New heater installed to allow for growth of larger samples
  - Sputtering gun repaired and Nb target replaced

# Partial Pressure

- Previous work[3] had shown that plasma with 5.9% $N_2$ gave optimal δ-NbN in our system. This percentage still gives the best results.



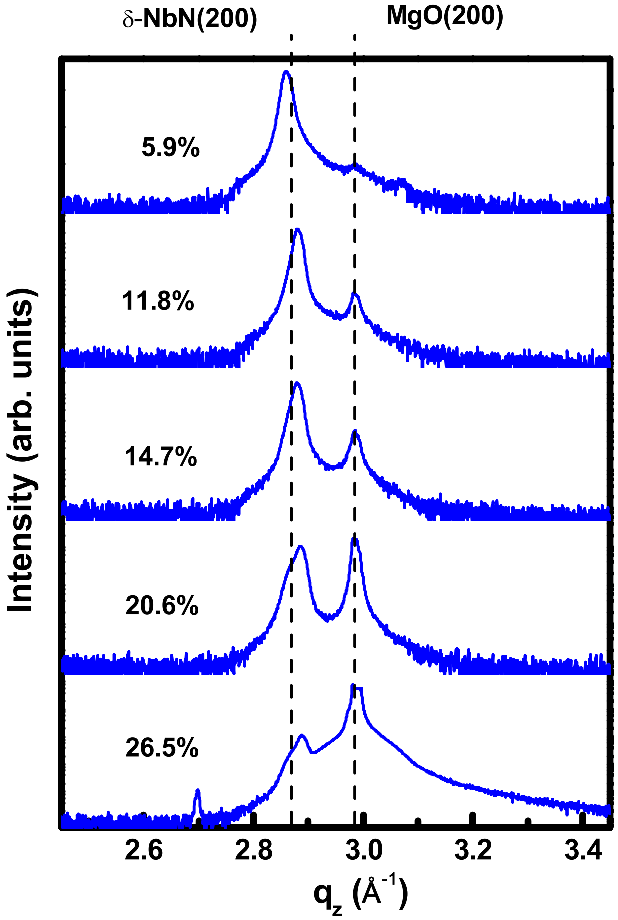


**Figure 3.** XRD scans for varying nitrogen partial pressure. The vertical dashed lines represent the positions for bulk δ-NbN (lower end of range presented in text) and MgO.

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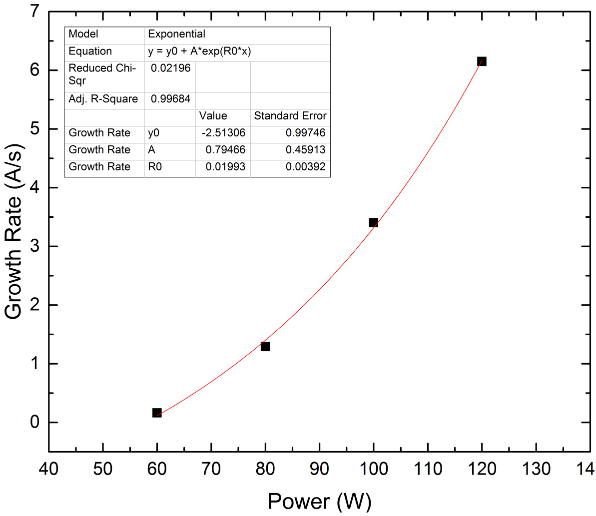
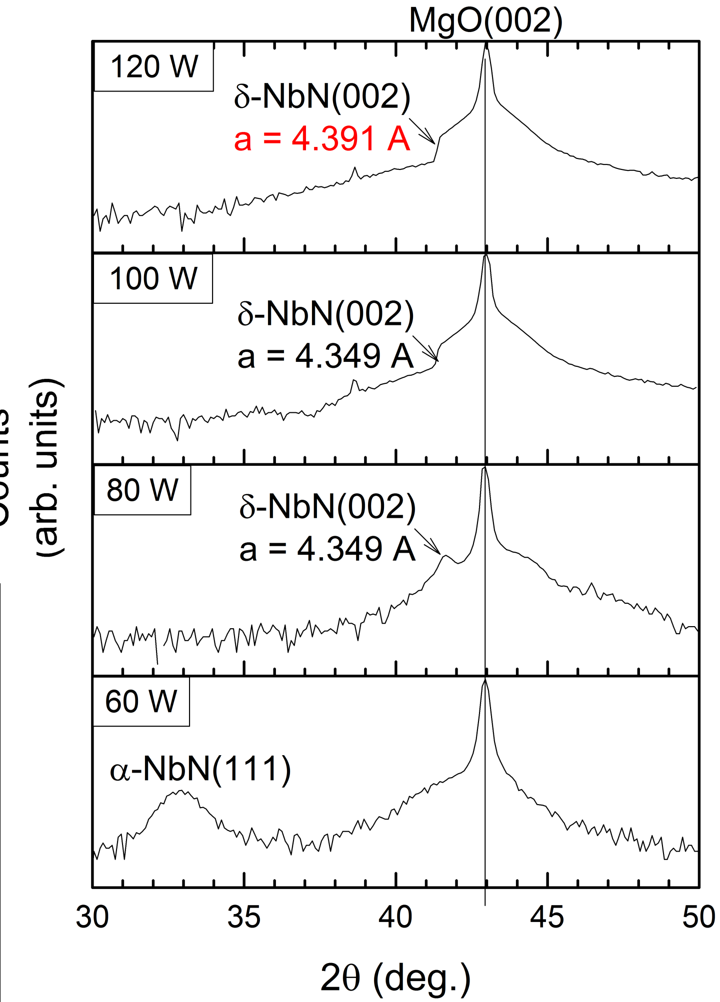
[3] W. M. Roach *et al. Supercond. Sci.Technol*. **25**, 125016 (2012).

# Sputtering Power

- Previous samples were grown at 60 W, which no longer gives δ-NbN.
- Increased power yields a better lattice parameter and a higher growth rate.

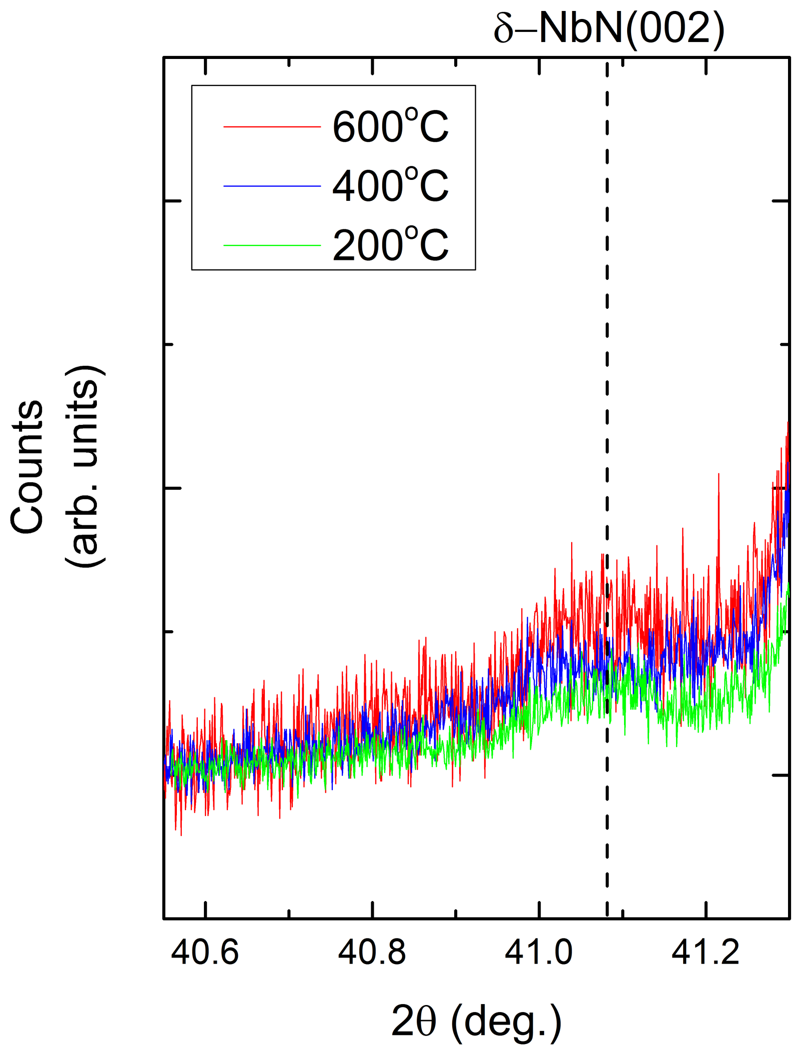


| Model | Exponential | | |
|---|---|---|---|
| Equation | y = y0 + A*exp(R0*x) | | |
| Reduced Chi-Sqr | 0.02196 | | |
| Adj. R-Square | 0.99684 | | |
| | | Value | Standard Error |
| Growth Rate | y0 | -2.51306 | 0.99746 |
| Growth Rate | A | 0.79466 | 0.45913 |
| Growth Rate | R0 | 0.01993 | 0.00392 |



# Substrate Temperature

- Higher substrate temperatures yielded more crystalline films.
- As the temperature increased, we saw more substrate nitridization.
  - Lack of control of thickness, phase of NbN which forms could negatively affect cavity performance.

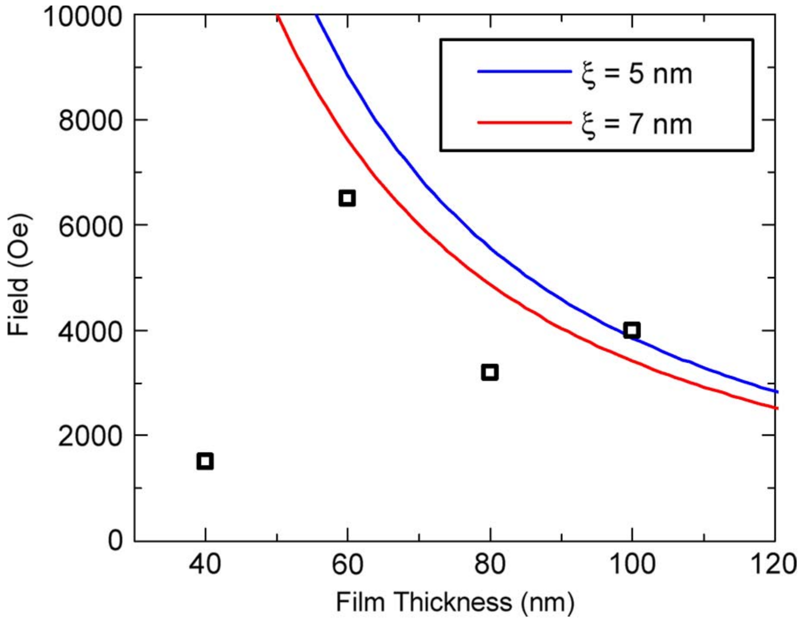


# Conclusions and Future Work

- Understanding the effects of deposition conditions on the stoichiometry of NbN thin films, and in turn the effect of the stoichiometry on the DC superconducting properties, allows films to be tailored for specific applications (cavities, detectors, etc.).
- Correlating DC and RF properties will then allow predictions of RF performance from structural characterization of NbN films.

# Conclusions and Future Work

- Current and future work with NbN thin films should explore more energetic deposition methods (pulsed DC sputtering, HIPIMS, ECR).
  - More energetic plasma yields denser films, which in general perform better.
  - $MgB_2$ films grown via HPCVD showed an $H_{C1}$ enhancement that matched theoretical predictions.



D. B. Beringer *et al. IEEE Trans. Appl. Supercond.* **23**, 7500604 (2013).