# Optimizing Growth of Niobium-3 Tin Through Pre-nucleation Chemical Treatments

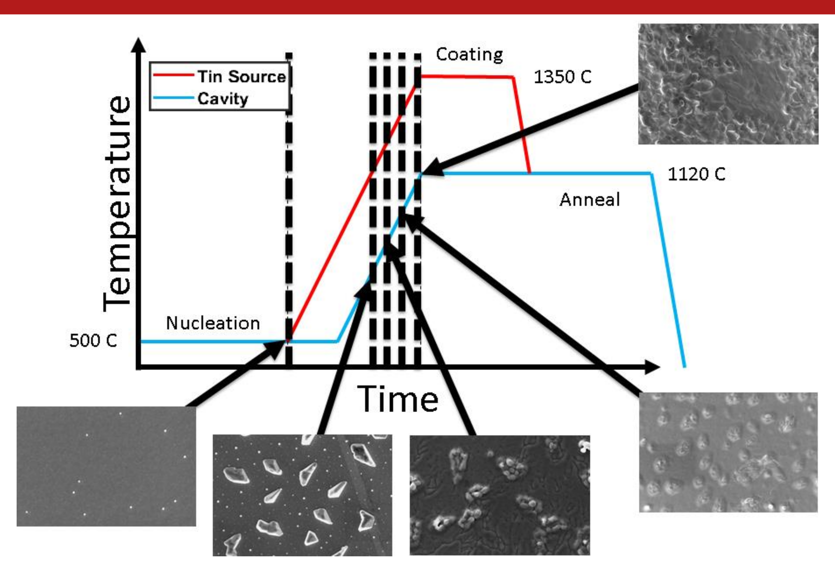
CLASSE — Cornell Laboratory for Accelerator-based ScienceS & Education

SUSPB026

**S. Arnold*, L. Shpani**, T. Arias, G. Gaitan, M. Liepe, N. Sitaraman, Z. Sun (Cornell University, Ithaca, NY)

## **Surface chemistry influences** the **nucleation of $SnCl_2$** on Nb samples during **vapor-diffusion-based $Nb_3Sn$ growth**.

## Introduction and Setup




R. D. Porter, Ph.D. thesis, Phys, Dept., Cornell University, Ithaca, United States, 2021

The **most promising growth method** for $Nb_3Sn$ is **thermal vapor diffusion**, but much improvement is needed for $Nb_3Sn$ grown by this method to reach its full potential. **One key obstacle is achieving a consistently smooth and uniformly thick layer of stoichiometric $Nb_3Sn$.** To address this challenge, this research focuses on **optimizing the initial stage** of this growth process, which involves the **nucleation of tin-rich droplets on the oxide surface** of the niobium substrate.

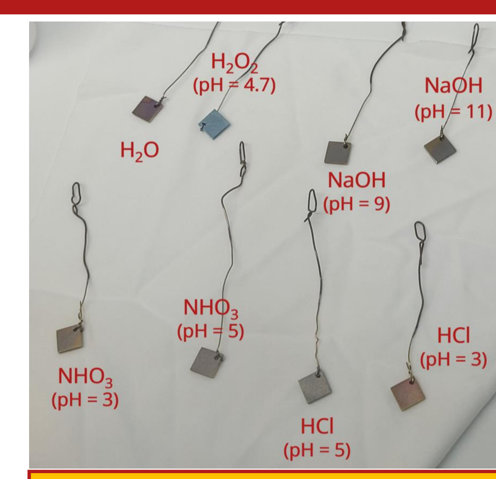


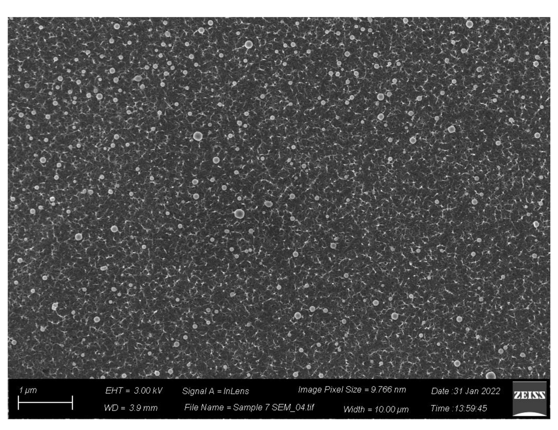

Image of all samples after chemical soaks with treatments (and pH) labeled (control sample not shown).

**The niobium oxide structure plays a crucial role in the binding of $SnCl_2$ to niobium during nucleation.** DFT calculations suggest that acidic solutions which remove OH groups will generate more $SnCl_2$ binding sites, **encouraging more uniform nucleation.**

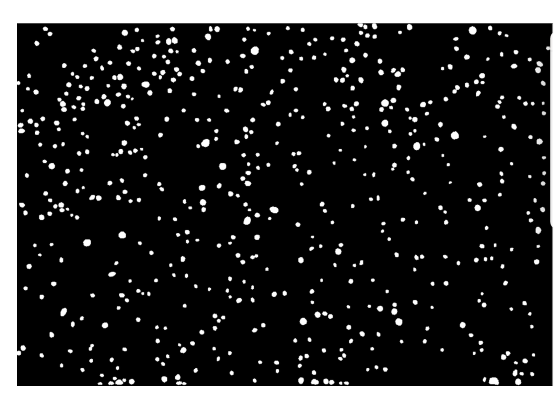
In this sample study, we **compare how different pre-nucleation chemical treatments influence the nucleation** of Sn on Nb samples**, in order to determine the optimal treatment to promote uniform and dense nucleation**.

## SEM Analysis: Nucleation Site Formation

To analyze the uniformity of grown nucleation sites, ImageJ plugins were used to process scanning electron microscopy (SEM) images.



Raw SEM

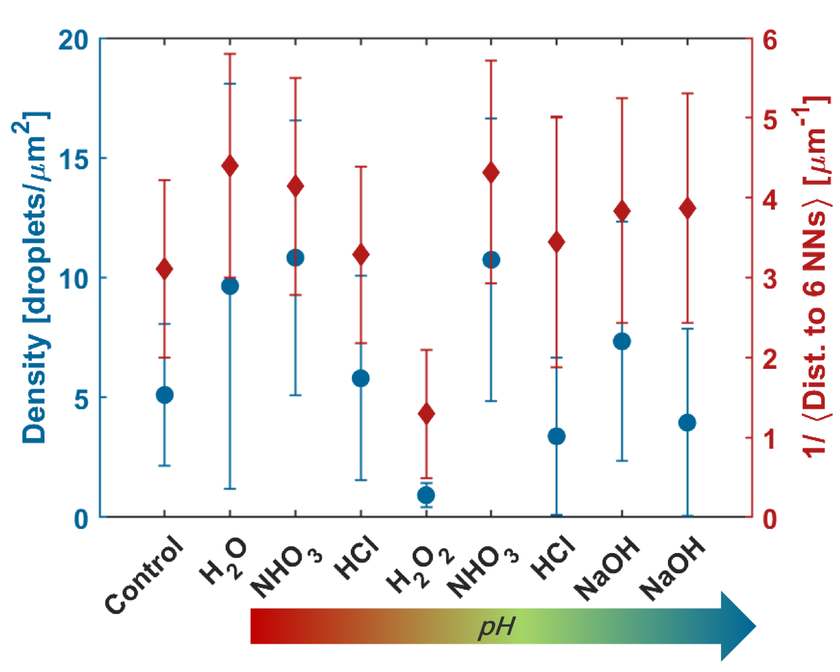


Processed SEM

### Nucleation Site Uniformity Analysis

**Density** and **average six nearest neighbor** calculations were used to **analyze the uniformity of nucleation sites** over many ~65 $\mu m^2$ areas of each sample.

To **minimize tin depleted regions**, we want **dense** and **uniform** nucleation sites. Thus, we want **high density** and a **low average six nearest neighbor values.** Here, we plotted the inverse of the average distance to six nearest neighbors to better observe each sample's performance. A higher value on both plotted metrics corresponds to a more promising nucleation.

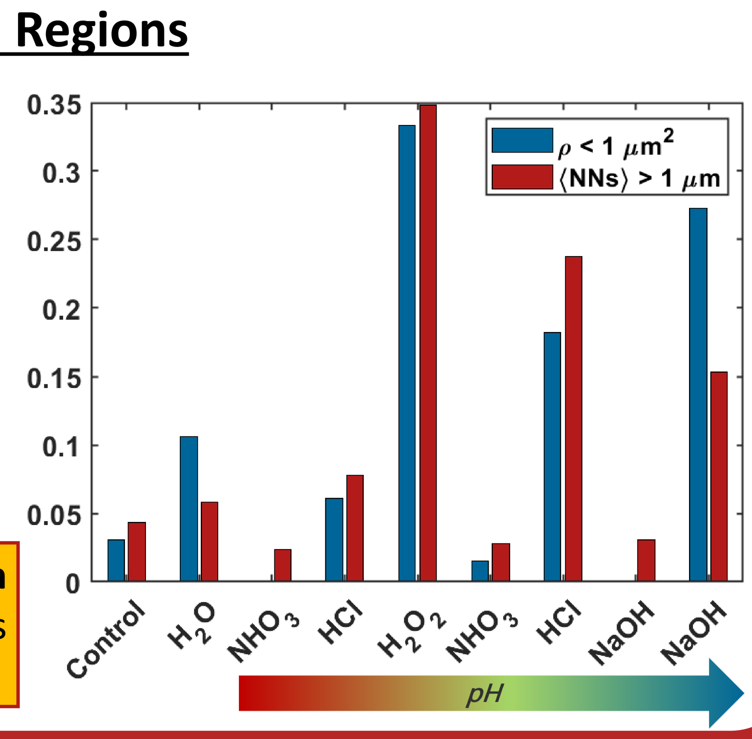



**$H_2O_2$ has the lowest density and largest distance between average six nearest neighbors.**

### Predicted Tin Depleted Regions

$Nb_3Sn$ grains have a roughly 1 μm² area, meaning we want **at least 1 droplet (nucleation site) per 1 μm² area.** To see if each treatment held up to this condition, we considered two metrics:

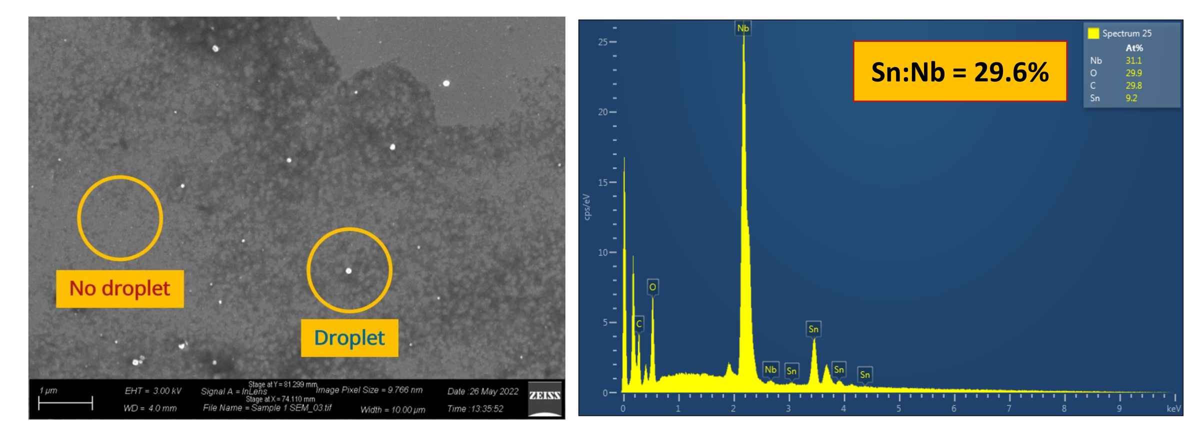
1. How many imaged areas had a **density** of less than one droplet per 1 μm² area?
2. How many **average six nearest neighbor distances** exceed 1 μm?




**$NHO_3$ (low pH)** and **NaOH (high pH)** show **sufficiently high density** and a **uniform distribution**, establishing them as **promising options for potential treatments.**

## EDS Analysis: Atomic Composition of Droplets

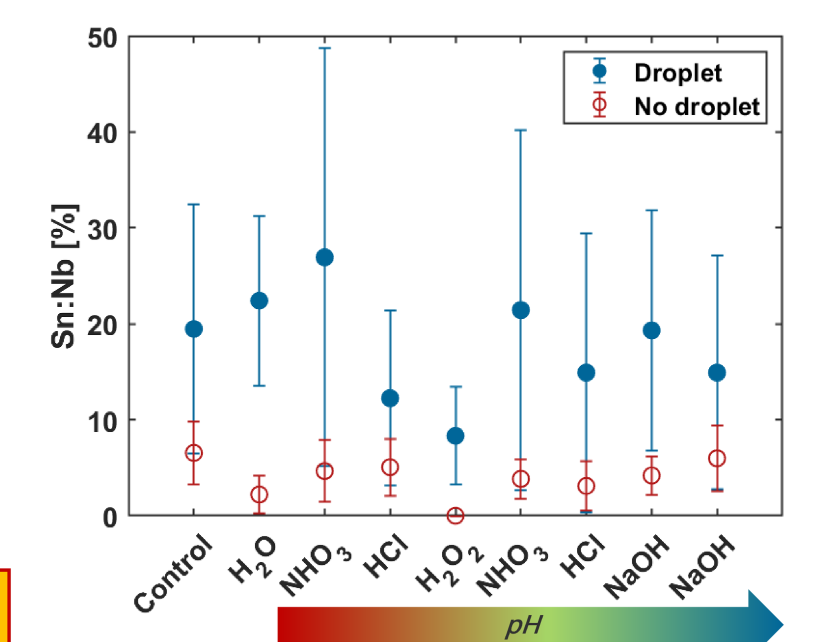
To confirm that the imaged droplets indeed represent tin nucleation sites, we utilize Energy Dispersive Spectroscopy (EDS) to analyze the **elemental composition of the surface of our samples**.




### Point Analysis Scans

Point Analysis scans focus the electron beam on a single point of interest and provide elemental information about the sample at that specific location.

By maintaining a constant accelerating voltage, we can obtain a **relative comparison of the ratio of atomic compositions of Sn and Nb among our samples.**



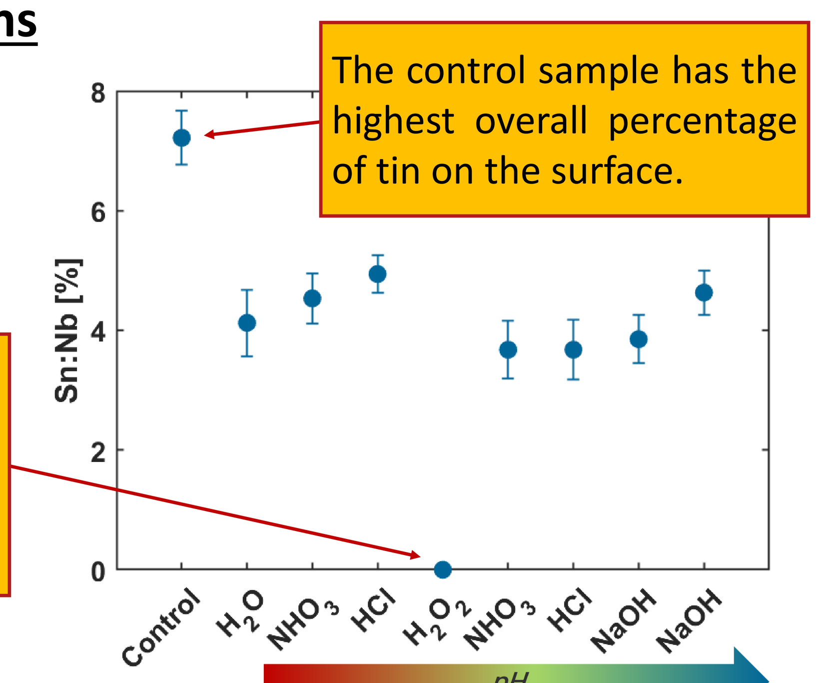

**All samples (except $H_2O_2$) have tin present even when no droplets are seen in the SEM image!**

### Area Scans

In an area scan, the electron beam scans a larger region of the surface and provides a more comprehensive analysis of the elemental composition.

We used area scans of 1800 μm² to **compare the overall composition of tin among our samples**, sacrificing spatial resolution to achieve broader coverage.

$H_2O_2$ has a **negligible amount of tin on its surface**, so we see no tin in any of the area scans!




The control sample has the highest overall percentage of tin on the surface.

## Conclusions and Future Work

From this study, we have found:

→ The chemical treatments influenced the distribution of nucleation sites

★ **The type of chemical treatment had a greater impact on the quality of nucleation compared to the pH of the treatment**

→ $H_2O_2$ treatment showed the **lowest density** of droplets and **lowest overall tin concentration**, suggesting a **significant suppression in nucleation**

→ $NHO_3$ is a **promising treatment** to arise from this study with a **dense and uniform distribution** of nucleation sites after nucleation

**Future work:**

→ Perform another round of sample studies for reproducibility

→ Bring samples to full coat

→ Apply our most promising treatment to a cavity and perform an RF test

## Background

**Niobium-3 tin is a promising alternative material for SRF cavities that is close to reaching practical applications**. To date, one of the most effective growth methods for this material is **vapor diffusion**, yet further improvement is needed for $Nb_3Sn$ to reach its full potential. The major issues faced by vapor diffusion are **tin depleted regions and surface roughness**, both of which lead to impaired performance. Literature has shown that the **niobium surface oxide plays an important role in the binding of tin to niobium**. In this study, we performed various **chemical treatments on niobium samples prenucleation to enhance tin nucleation**. We quantify the effect that these various treatments had through **scanning electron microscopy (SEM)** and **energy dispersive spectroscopy (EDS)**. These methods reveal information on tin nucleation **density** and **uniformity**, and a **thin tin film present on most samples, even in the absence of nucleation sites**. We present our findings from these surface characterization methods and introduce a **framework for quantitatively comparing the sample**s. We plan to apply the most effective treatment to a cavity and conduct an RF test soon.

*sga46@cornell.edu

This research is funded by the National Science Foundation under Grant No. PHY-1549132, the Center for Bright Beams.
This work made use of the Cornell Center for Materials Research Shared Facilities which are supported through the NSF MRSEC program (DMR-1719875).

The Center for Bright Beams — A National Science Foundation Science & Technology Center

SRF 2023 Grand Rapids