High-Performance Thin-Film Niobium Produced via Chemical Vapor Deposition (CVD)

Ryan Porter

SRF, July 19 2017

Lanzhou, China

CVD Niobium Work at Cornell/Ultramet

- Cornell University has been testing CVD niobium created by the company Ultramet.
  - Produces CVD metals and ceramics for aerospace, medical and energy industries
- Early CVD Nb work focused on making good (pure/high RRR) samples and devising a CVD-based cavity fabrication methodology
  - Successful!
- Made full CVD niobium cavity
  - Had some fabrication issues
- RF tests of CVD sample plates

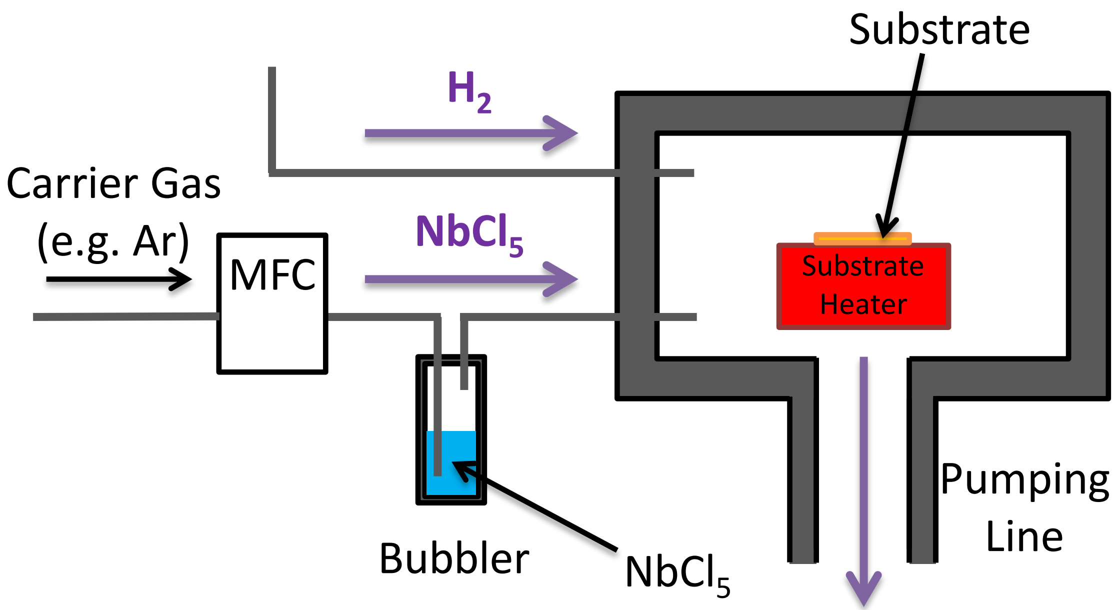

CVD niobium on molybdenum puck

Thin Film Niobium Advantages

- Increased thermal stability
  - Nb: 75 W/m -> Cu: 300-2000 W/m
- Lower cost
  - High RRR Nb ≈ 10x price of Cu
    - CVD process uses commercially available chip Nb start materials (~$50/pound)
  - Can avoid (reduce) expensive electron beam welding
- Decreased Flux Trapping Sensitivity
  - 300 - 500 nΩ/G -> 1 nΩ/G
- Can avoid inclusions caused by machining

What is CVD?

- A substrate is exposed to one of more vapors/gasses and a reaction/decomposition takes places that leaves niobium (or other metal/ceramic) behind



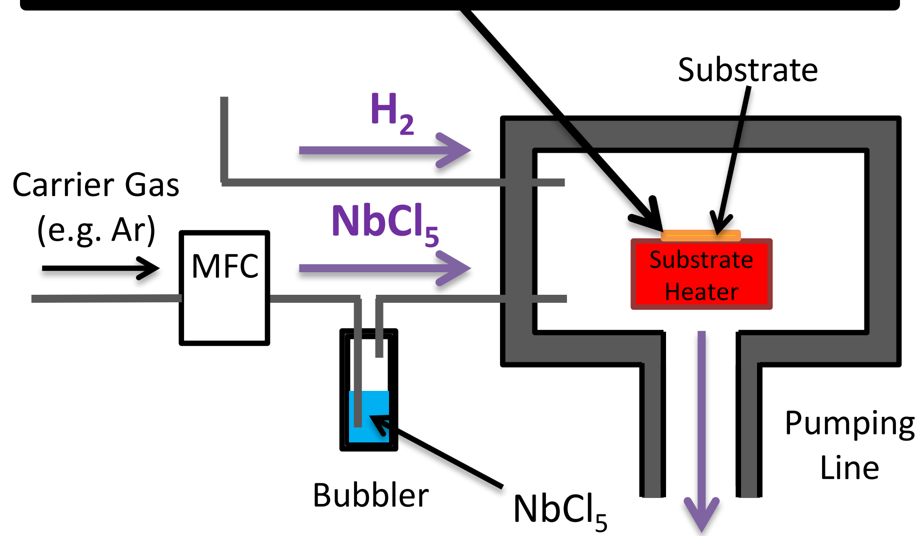
Reactor diagram showing use of $NbCl_5$ to produce CVD niobium (based on diagram by P. Pizzol et al. in Proc. of IPAC 2016, Busan, Koriea, 2016)

What is CVD?

How to deposit niobium?

- Common method:
  - $NbCl_5$ is vaporized
  - Hydrogen is added
  - The substrate is heated (e.g. 700 C)
    - Reaction takes place on substrate
  - Resultant gasses pumped away

2 NbCl5 + 5 H2 -> 2 Nb + 10 HCl



Reactor diagram showing use of $NbCl_5$ to produce CVD niobium

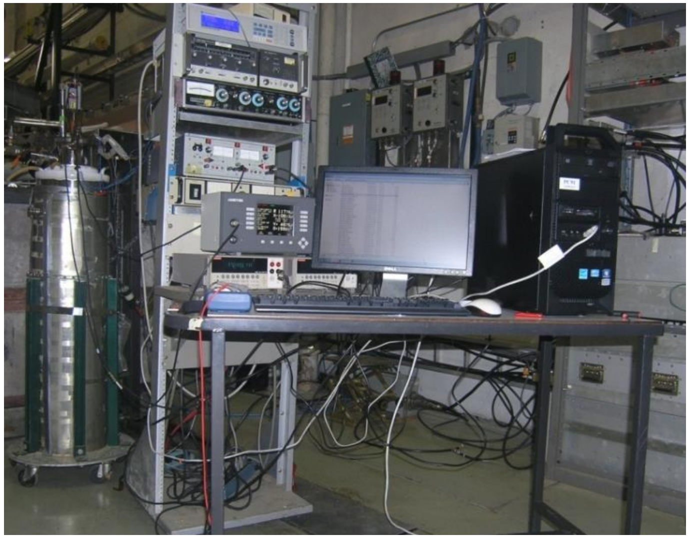
How does it differ from other techniques?

Advantages of CVD:

- Forms a metallurgical diffusion bond
  - Strong enough withstand High Pressure Rinses (HPR) and Centrifugal Barrel Polish (CBP)
  - Creates good thermal contact
- High deposition rate
  - 300 μm/hour
  - Can make both thick and thin films

Sample Studies

- In 2012 we worked with Ultramet to test CVD niobium samples
  - Various CVD parameters (pressure, temperature, etc.)
  - Find process created good niobium
- We tested the RRR and T_c
  - Found T_c to be consistent with niobium
  - Found samples with RRR > 250
- **<u>CVD can make high RRR niobium!</u>**

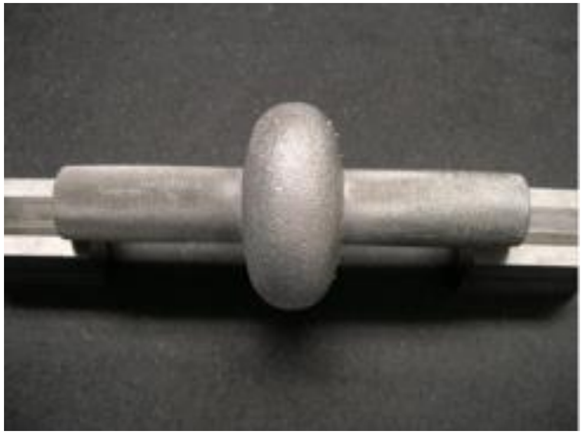


RRR measurement system at Cornell

CVD Niobium Cavity

- Ultramet produced a <u>3 GHz elliptical cavity</u>
  - Graphite mandrel
  - Several microns of high RRR niobium
    - <u>Layer was CNC ground</u>
  - +3 mm of low RRR niobium
  - The niobium machined and the mandrel was removed

Graphite mandrel



Cavity after CVD process

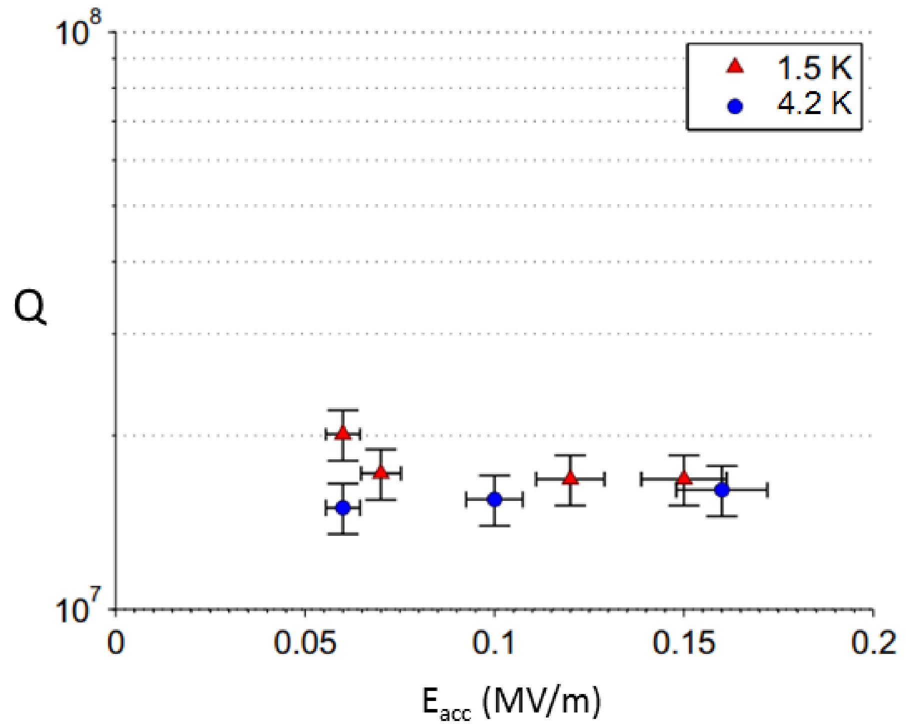
CVD Niobium Cavity

Seamless bulk CVD niobium cavity!

CVD niobium cavity after machining

CVD Niobium Cavity Initial Tests

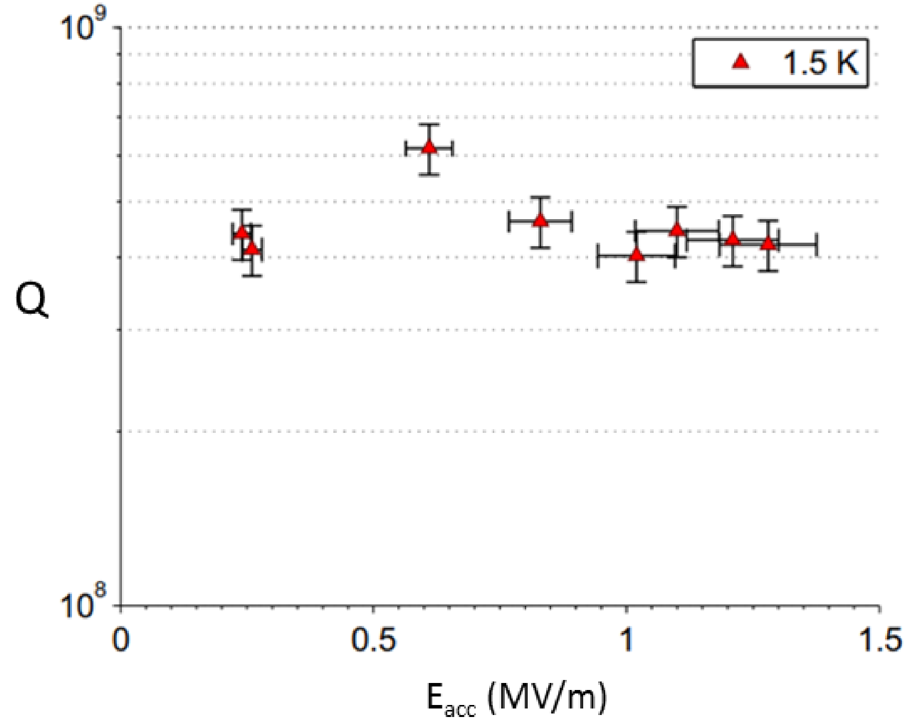
- Initial tests of the cavity showed high surface resistance
  - R_o of 14 Ωm
- Surface contaminants from production
- Needs BCP



CVD Niobium Cavity Results

After 100 µm BCP:

- **R_{BCS} as expected**
- $R_o \sim 650$ nΩ
  - Was 14 µΩ
  - Expect <10 nΩ
- Quench at 1.2 MV/m
  - Pits found on cavity surface
- Low mean free path
  - Contaminants from mandrel outgassing
- Machining may have damaged RF surface
- **Good results!**
  - Need cleaner production



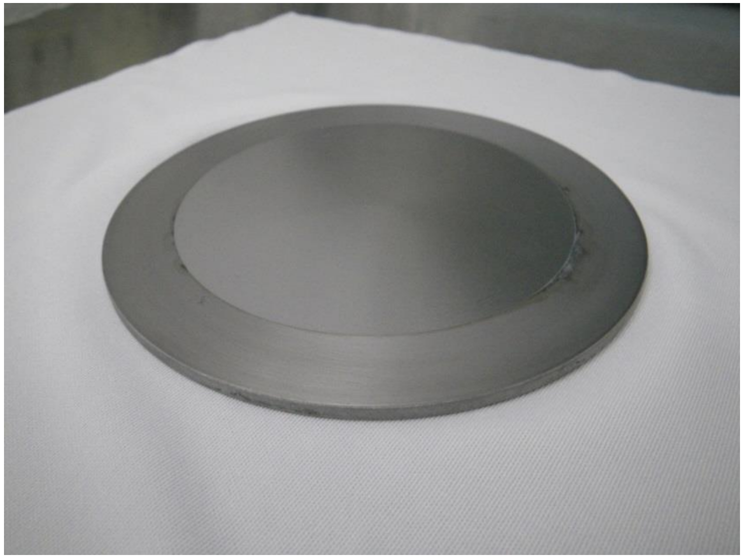
Cornell Sample Host Cavity

- Full cavities is harder and expensive
  - Want films instead of bulk
- Need to test CVD surfaces in SRF conditions
- The Cornell Sample Host cavity can take 5-inch sample plates
  - 3.9 GHz sample host cavity
  - Fairly uniform magnetic field over sample
  - Q of $1 \cdot 10^{10}$ at 2 K with niobium sample

The Cornell Sample Host Cavity

CVD Test Plates

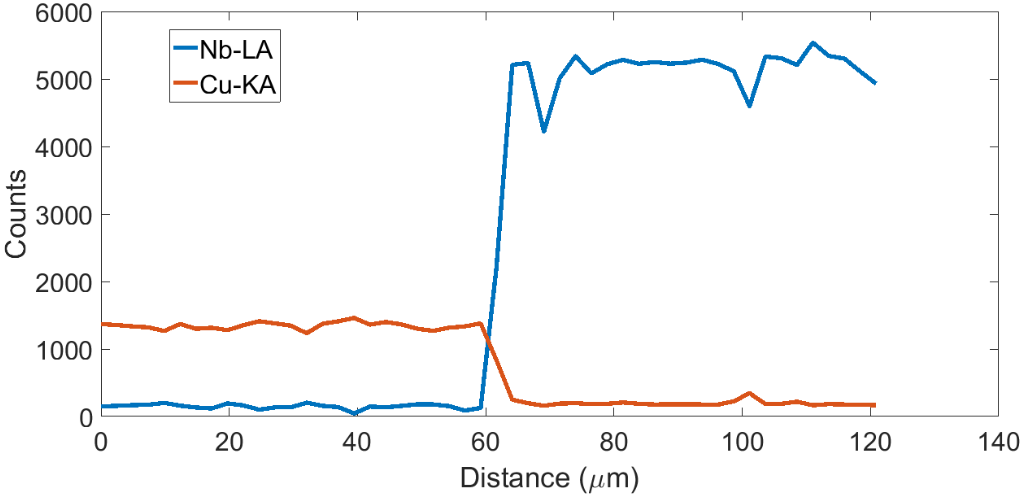
- In 2016 Ultramet made CVD niobium on Cu and Mo samples
- Multiple processes
  - Low and high temperature CVD
- Nominally 150 – 200 µm thick films
- Some mechanically polished
- **<u>Plates tested “as received”</u>**

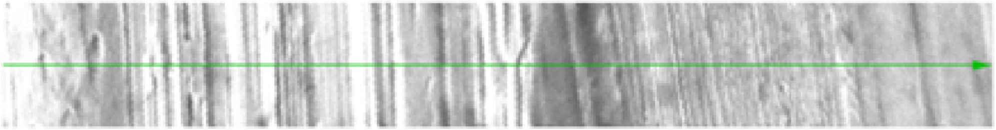


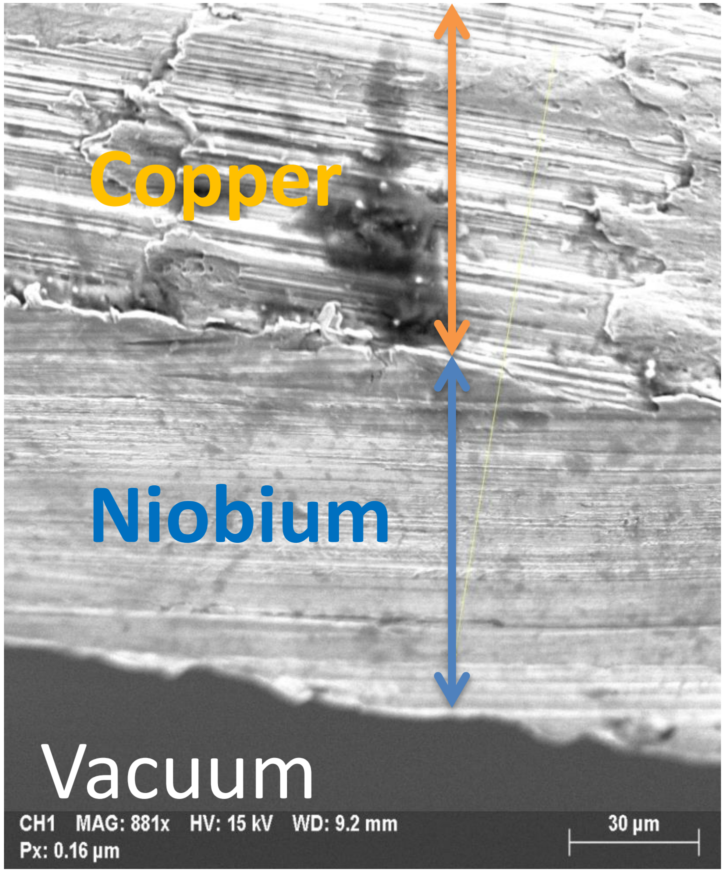
CVD niobium on copper test plate

Surface Cross-Section

- Cross-section view shows a 60 μm Nb surface layer for this sample (on Cu)
- EDX line scan shows fast transition from the substrate to coating
  - <u>Cu has not diffused far (~1 um) into Nb</u>

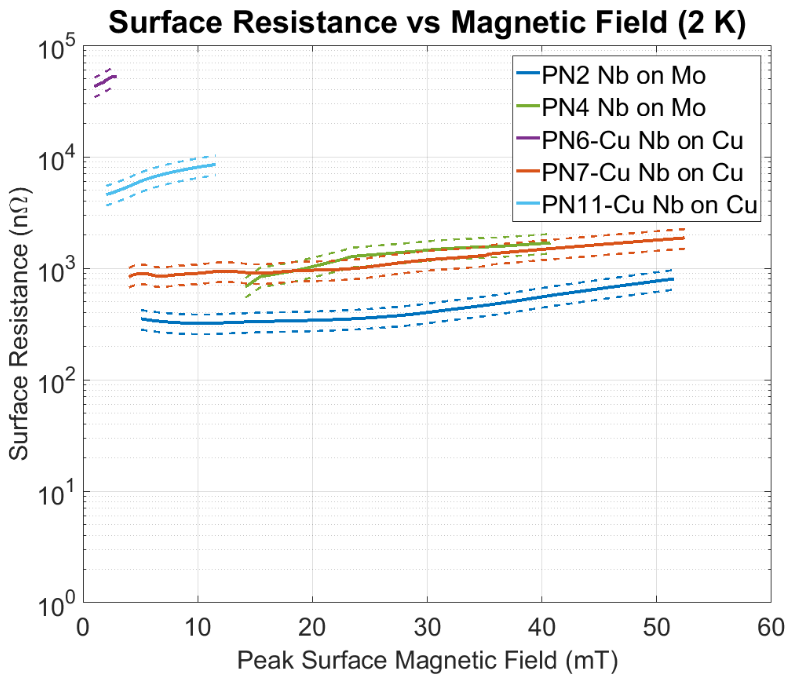






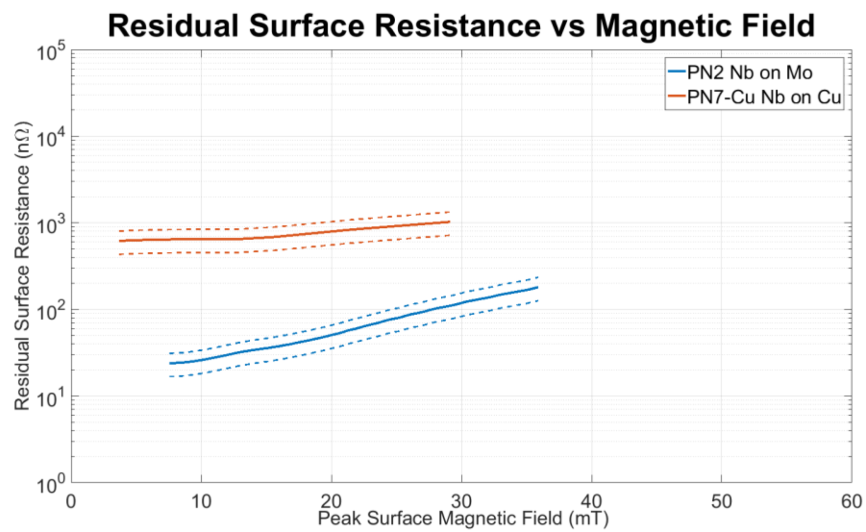
Surface Resistance

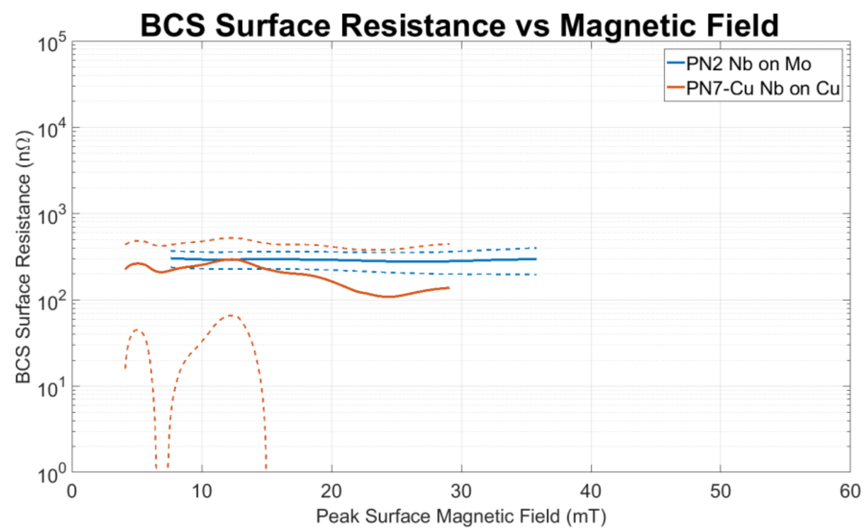
- Tested at 2 K (and other temperatures):
- Medium quench field of 50 mT
  - 12 MV/m for TESLA cavity
  - **10 times increase in quench field!**
- Resistance as low as 300 nΩ
- Resistance rises rapidly with increasing field



Surface Resistance

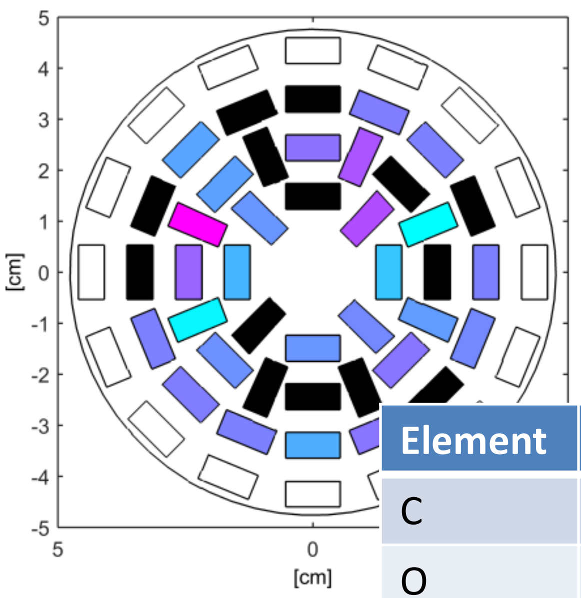
- R_{BCS} at 2 K was 100 to 300 nΩ and flat
  - Expect ~110 nΩ 3.9 GHz niobium
- R_{BCS} is flat
- Best plate had $R_o \sim 20$ nΩ at low fields
  - R_o is <10 nΩ for good niobium
  - High temperature Nb on Mo plate
- R_o is 200 nΩ to 50 µΩ for other samples

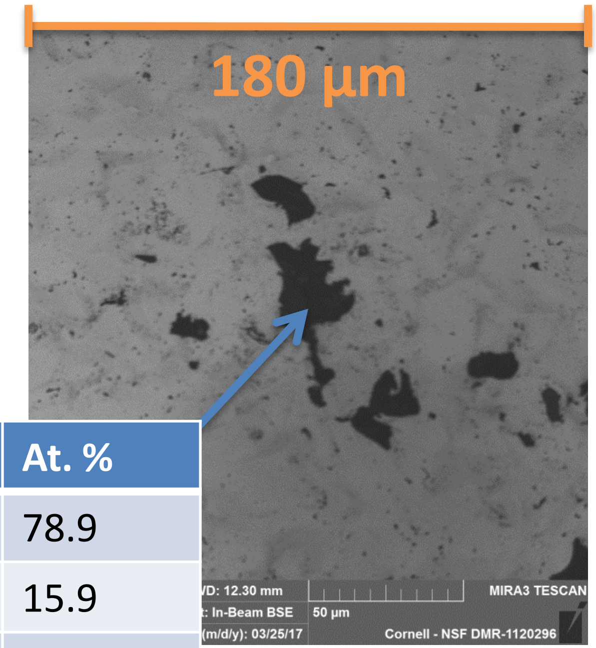




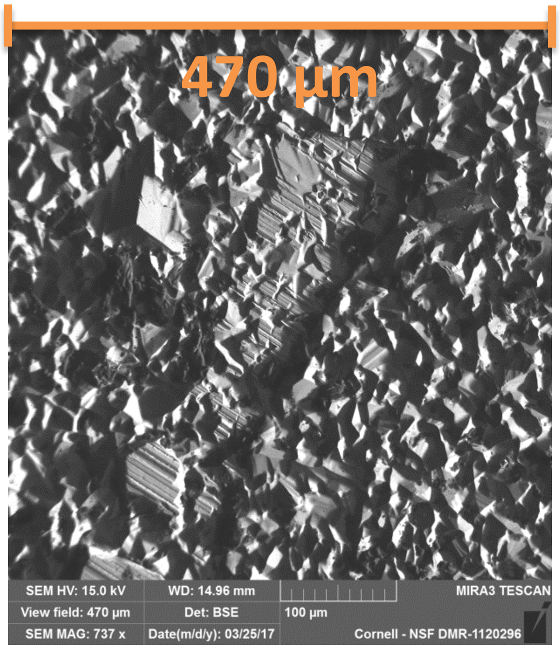
Surface Defects

- Thermometry showed local heating on the sample plates
  - SEM imaging found <u>large pits and inclusions</u>
    - Chemical analysis found inclusions rich in carbon, oxygen and other elements
      - May be caused by condensation forming on roof of reactor dripping onto the sample—not a problem for cavities!





Element	At. %
C	78.9
O	15.9
Nb	2.4



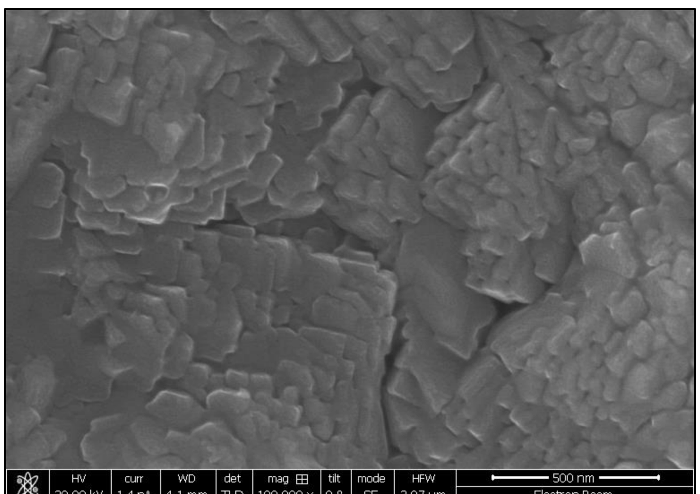
Potential Causes

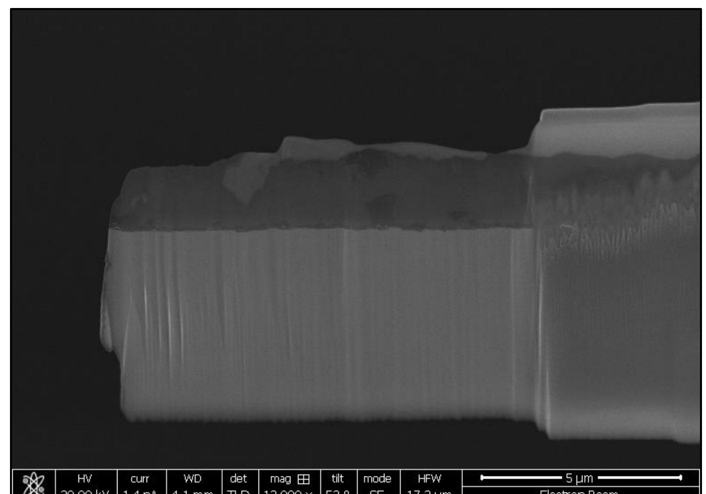
- Bumps, divots, and inclusions in surface
- Glow Discharge Mass Spectrometry (GDMS) of some CVD Nb on Cu plates revealed 200-400 ppm Cu on surface
  - How much is too much copper?
- Q-Disease?
  - Some CVD techniques use hydrogen
  - Previous CVD samples had a high hydrogen concentration
    - Recent plates not tested for hydrogen

Other CVD Niobium for SRF Work

Sample Studies

- Paolo Pizzol et al. at STFC have created Nb on Cu samples
  - used CVD and (Plasma Enhanced) PECVD
- Has successfully created several µm thick Nb thin films
  - Correct structure
  - Still low RRR

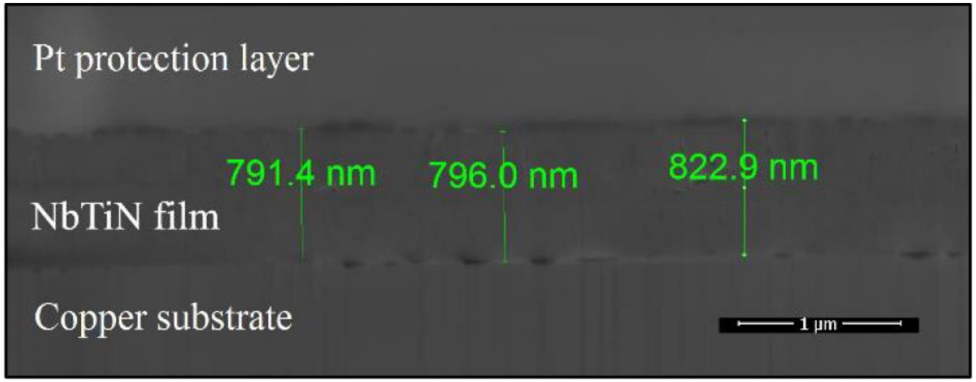


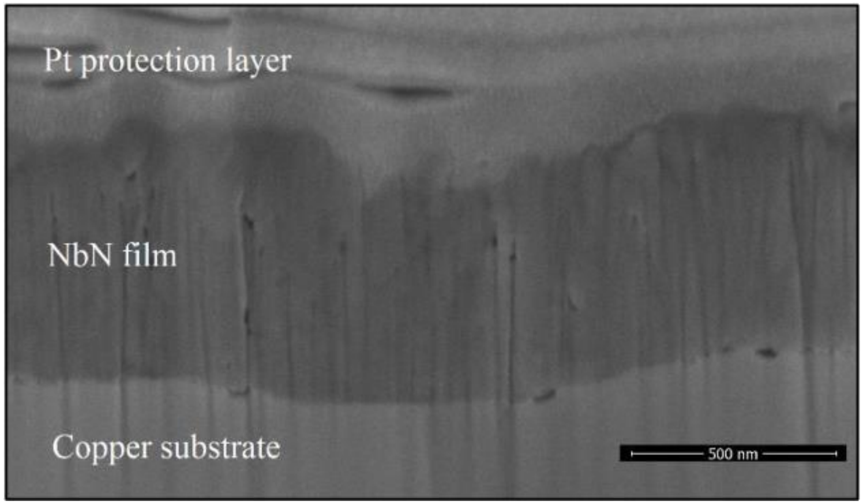


Beyond Niobium

P. Pizzol et al. have also been working on CVD of other niobium based materials.

- Successfully produced CVD NbN and NbTiN





Conclusion

- Can create high RRR niobium on Cu
  - As good as best sputtered niobium
- Robust, chemically-bonded coatings with good thermal interfaces
- Can coat complex geometry

- Fairly low resistances
  - Best case:
    - Q ~ $4.5 \cdot 10^9$ at 2 K for 1.3 GHz TELSA cavity
- Achieve medium field strength

- Need to take measures to consistently minimize preventable contamination issues

- Greatly improved from early work
- CVD niobium is promising
  - <u>Recommend further R&D</u>