Deposition of Superconducting Nb Thin Films by Recrystallization and Abnormal Grain Growth

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Outline

- Acceleration basics
- Cavity theory / parameters
- Superconductivity “survival”
- SC material criteria
- Thin films
  - Growth
  - Techniques
- Research
Particle Acceleration

\[ \nabla \cdot \mathbf{E} = \frac{\rho}{\varepsilon_0} \]

\[ \nabla \cdot \mathbf{B} = 0 \]

\[ \nabla \times \mathbf{E} = -\frac{\partial \mathbf{B}}{\partial t} \]

\[ \nabla \times \mathbf{B} = \mu_0 \mathbf{J} + \mu_0 \varepsilon_0 \frac{\partial \mathbf{E}}{\partial t} \]

\[ \mathbf{F} = q \mathbf{E} + q \mathbf{v} \times \mathbf{B} \]

\[ \lambda = \frac{h}{p} = \frac{h}{mv} \sqrt{1 - \frac{v^2}{c^2}} \]

\[ \rightarrow E = hf = \frac{hc}{\lambda} = mc^2 \]
e− Bunch Cloud

1/20,000,000,000 second later
(notice how far the bunches have moved)

Electric Field
(toward the right)

(amount of energy boost)

Positive particles

Negative particles

(ahead on time)

behind the bunch

(ahead on time)

behind

Position

GROWING TRAVELING WAVE

OUTPUT

ELECTRON GUN

ELECTRON BEAM

COLLECTOR
Cylindrical Cavity

\[
\frac{\partial^2 E_z}{\partial r^2} + \frac{1}{r} \frac{\partial E_z}{\partial r} - \frac{1}{c^2} \frac{\partial^2 E_z}{\partial t^2} = 0
\]

\[E_z(r) = E_0 J_0 \left( \frac{\omega r}{c} \right) \rightarrow E_0 J_0 \left( \frac{\omega r}{c} \right) \cos(\omega_0 t)\]

\[H_\theta(r) = i \frac{E_0}{Z_0} J_1 \left( \frac{\omega r}{c} \right) \rightarrow -\frac{E_0}{Z_0} J_1 \left( \frac{\omega r}{c} \right) \sin(\omega_0 t)\]

\[f = \frac{2.405 c}{2 \pi a} \text{ Hz}\]
Cavity Design Parameters

\[ \delta = \sqrt{\frac{2}{\mu_0 \omega \sigma}} \quad \rightarrow \quad R_{\text{surf}} = \sqrt{\frac{\pi \mu_0 \mu_r f}{\sigma}} = \frac{1}{\sigma \delta} \]

\[ G = \frac{2.405 \mu_0 c}{2(1 + \frac{a}{h})} \]

\[ Q_o = \frac{G}{R_{\text{surf}}} \quad \rightarrow \quad Q_o = \frac{2\pi U f_o}{P_{\text{diss}}} = \frac{f_o}{\Delta f} \]

\[ \frac{R}{Q} = \frac{R_{\text{shunt}}}{Q_o} = \frac{4h \mu_0 c}{\pi^3 (J_1(2.405))^2 2.405 a} = \frac{|V_{rf}|^2}{2 \omega U} \]

This is identical to the TL intrinsic impedance, \( \sqrt{\frac{L}{C}} \)
Superconductivity

Hyper-abrupt change in resistivity vs. temperature

Applied H-field destroys SC state:

\[ B = 0 \text{ (SC)} \]
\[ \frac{\partial B}{\partial t} = 0 \text{ (NC)} \]

Also, SC is a change in state, w/ associated relaxation time constant.

Meissner Effect

Magnetic field not only excluded, but expelled!
SC Critical Fields and Vortices

Two flavors of SC:

- **Type I**
  - Mercury
  - $B_c = 0.041 \text{T}$
  - $T_c = 4.15 \text{K}$

- **Type II**
  - Niobium-Tin
  - $B_{c2} = 24.5 \text{T}$, $T_c = 18 \text{K}$

Quantized magnetic flux:

$$\Phi_0 = \frac{h}{2e} = 2.07 \times 10^{-15} \text{T} \cdot m^2$$

Silsbee Hypothesis:

If current in SC wire produces a field greater than $H_c$, the material becomes a normal conductor.

A. Bezryadin, et al.
**London Theory**

**Phenomenological Approach**

Defines a super-current, via super-electrons

Adds 2 more equations to Maxwell's list....

Charge carriers (e-) experience no "friction" and actually accelerate:

\[
 m_e \dot{v} = e \vec{E} - \frac{m \vec{v}}{\tau}
\]

*Drude Model*

Modified Ohm's Law:

\[
 \vec{J} = \frac{n e^2 \tau}{m} \vec{E} \quad \sigma = \frac{n e^2}{\omega m}
\]

Taking curl & Faraday's Law results in...

\[
 \nabla \times \vec{J} = \frac{n_s e^2}{m_e} \vec{B}
\]

*Eq. #2*

B.C.s resolve Meissner condition...

\[
 \frac{d^2 B_y}{dx^2} - \frac{1}{\lambda_L^2} B_y = 0
\]

PDE solution provides....... **London Penetration Depth**

\[
 \lambda_L = \sqrt{\frac{m_e c^2}{4 \pi n_s e^2}} = \sqrt{\frac{m_e}{\mu_0 n_s e^2}} = \sqrt{\frac{1}{\mu_0 \sigma_s \omega}}
\]

Empirical temperature relationship......

\[
 \lambda_L(T) = \frac{\lambda_L(0)}{\sqrt{1 - \left(\frac{T}{T_c}\right)^4}}
\]

Extensive references to Tinkham and Padamsee......
London Implications

Currents deep within the bulk are not allowed, due to induced B-fields, excluded by Meissner Effect. This results in a very thin current on outer skin (or an infinite surface current!), particularly for Type I SC; a vulnerable condition for high-field applications.

As T approaches $T_c$ (from LHS), $\lambda_L$ becomes infinite, consistent with normal conductivity.

$\lambda_L$ is much less than skin depth:

Skin Depth $\sim$ 2000 nm

$\lambda_L \sim$ 40 nm

For normal metals.....

$R_{surf} \propto \sqrt{\frac{\omega}{\sigma_n}}$

(intuitive)

For SC metals.....

$R_{surf} \propto \omega^2 \sigma_n$

(non-intuitive)

Treatment works well for Type I SC, but is miserable for Type II and AC applications

Also, we simply know it's wrong since it is continuous / analog, not “quantum.”


Ginzburg-Landau Theory

\[ n_s \approx n, \ T \ll T_c \]
\[ n_s = 0, \ T > T_c \]

Contrary to quantum “mechanism”

Connects \( n_s \) with a Schroedinger-like wave eq.:

\[ n_s = |\psi(x)|^2 \]

Spatial distribution of \( n_s \), vs. London's unitless

A **characteristic length** emerges, for which lowest energy state is preserved:

\[ \xi_0 = \frac{\hbar \nu_F}{k T_c} \]

\((E_F = 1/2 \nu_F^2 = \text{Fermi Velocity})\)

Pippard modification for mean-free P.L.:

\[ \frac{1}{\xi} = \frac{1}{\xi_0} + \frac{1}{l} \]  

**Effective C.L.**

The G-L Parameter is used to discern Type I and Type II SC:

\[ \kappa = \frac{\lambda_L}{\xi} \]

G-L Parameter

\[ \kappa < \frac{1}{\sqrt{2}} \]  

Type I SC

\[ \kappa > \frac{1}{\sqrt{2}} \]  

Type II SC

Empirical temperature relationship......

\[ H_c(T) = \frac{\Phi_0}{2\sqrt{2} \xi(T) \lambda_L(T)} \]

\[ H_{c-max} = \sqrt{2} \kappa H_c = \frac{\Phi_0}{2 \pi \xi^2} \]
G-L Implications

In addition, maximum field is now connected to flux quanta and C.L.

G-L strictly valid at Tc; extremely low temps can be problematic.
Electrons coherently “condense” into *Cooper pairs*

Mutual Coulombic attraction facilitates momentum exchange between otherwise “uninterested” electrons

Quantum-physical derivation of Coherence Length:

\[ \xi_0 = \frac{\hbar \nu_F}{\Delta}, \quad 2\Delta \approx 3.5 T_c \]

Binding energy of a Cooper pair ~ 3 E-3 eV for niobium

Electrons circulate (like ice skaters!), screening magnetic field (Meissner)

\[ n_c = \frac{n_s}{2}, \quad m_c = 2m_e \]

\[ e_c = 2e \]

Complete description, holding steady after 50+ years; requires patience and persistence!
Tedious, but necessary to extract best performance from SC materials

Phonons, lattice vibrations, affect a pair, with zero net loss/gain. Also, major source of SC conduction

Pairs are large ~ 10-1000 nm!
Related to shortest phonon wavelength (Debye Frequency)

Pairs break and re-form every ~0.05 ns
2-Fluid Model

AC behavior is quite different than DC!

Normal electrons as a fluid, with conductivity, \( \sigma_n \)

Cooper Paired (accelerated) electrons are a “superfluid”

\[
J_n = \sigma_n E_0 \exp(-j\omega t)
\]

\[
m_c \dot{v}_c = -2e E_0 \exp(-j\omega t)
\]

\[
J_s = j \frac{2n_ee^2}{m_e \omega} E_0 \exp(-j\omega t)
\]

\[
J = J_n + J_s = \sigma E_0 \exp(-j\omega t)
\]

\[
\sigma = \sigma_n + j\sigma_s \rightarrow \sigma_s = \frac{2n_ee^2}{m_e \omega} = \frac{1}{\mu_0 \lambda_L^2 \omega}
\]

Surface resistance is real part of surface impedance:

\[
R_{surf} = \Re \left[ \frac{1}{\lambda_L (\sigma_n + j \sigma_s)} \right] = \frac{1}{\lambda_L} \cdot \frac{\sigma_n}{(\sigma_n^2 + \sigma_s^2)} \approx \frac{\sigma_n}{\lambda_L \sigma_s^2}
\]

For normal conductor:

\[
R_{surf} = \frac{1}{\sigma \delta}
\]
Surprisingly, surface resistance is proportional to normal conductivity!

Subsequent steps yield the BCS resistance (for \( l \gg \xi \)):

\[
R_{BCS} \propto \lambda_L^3 \omega^2 l \exp\left(-1.76 \frac{T_c}{T}\right)
\]

- Proportional to exponential increase in temperature
- Proportional to frequency^2

Finally, make substitution alla G-L for the effective penetration depth:

\[
\lambda_L \sqrt{1 + \frac{\xi}{l}}
\]

Experimental data results in a “pedestal” or residual resistance, \( R_{res} \).

RF Surface Resistance

\[
R_{surf} = R_{BCS} + R_{res}
\]

Residual Resistivity Ratio is:

\[
RRR \overset{\text{def}}{=} \frac{\rho(T = 300 \, K)}{\rho(T = 4.2 \, K)}
\]
Surface Impedance

Surface Resistance - Nb - 1500 MHz

$R_{BCS} \propto \frac{1}{\sqrt{l}}$

$R_{BCS} \propto l$

dirty $\xi \approx l$

clean

Captive Audience!

https://writescience.wordpress.com/tag/bicep2/

http://www.vanyaland.com

Courtesy J. R. Delayen
Sergio Calatroni - CERN - Nb Coatings

In the “very clean” limit, a constant $R_{BCS}$ is predicted; computation is more severe....
Superconducting Material Criteria

\[ R_{BCS} \propto \frac{1}{T} \sigma_n \lambda^3 \omega^2 \exp\left[\frac{-1.8kT_c}{kT}\right] \]  
(as presented by Bonin, et al.)

Competing parameters:

1. Large \( T_c \) (minimizes \( R_{BCS} \))
2. Large \( \xi \) (lattice defect tolerance)
3. Large \( l \)
4. Small \( \lambda \) (minimizes \( R_{BCS} \))
5. Large \( H_{RF} \)

RF Hc has empirical relationship to standard Hc:

\[
H_{RF} \approx \begin{cases} 
0.89 \sqrt{\kappa} H_c & \text{For } \kappa << 1 \text{ (Type I)} \\
1.2 H_c & \text{For } \kappa = 1 \\
0.75 H_c & \text{For } \kappa >> 1 \text{ (Type II)} 
\end{cases}
\]

Number of candidates is finite:

- Type I SCs satisfy 2 & 4, but not 1
- BCS requires inverse relationship between 1 & 2

Conflicts, conflicts......A compromise is necessary

Combined with #5, Type II SC is the only clear choice.

<table>
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<th>Material</th>
<th>Tc, K</th>
<th>λ, nm</th>
<th>ξ, nm</th>
<th>H_{RF}, A/m</th>
<th>Misc</th>
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<td>16</td>
<td>1600</td>
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<tr>
<td>In</td>
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<td>24</td>
<td>360</td>
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<tr>
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<td></td>
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<tr>
<td>Nb</td>
<td>9.26</td>
<td>39</td>
<td>38</td>
<td>1.6x10^5</td>
<td>Bingo!</td>
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<tr>
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<td>Nb₀.₆Ti₀.₄</td>
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<td>250-320</td>
<td>4</td>
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<td>200-350</td>
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<td>YBCO</td>
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<td>140</td>
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<tr>
<td>NbTiN</td>
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<td></td>
<td></td>
<td>6.4x10^4</td>
<td></td>
</tr>
</tbody>
</table>

Bonin, B., “Materials for Superconducting Cavities,” CERN Technical Note, cds.cern.ch
Comparison Summary

Normal Conductor

\[ R_{\text{surf}} \propto \sqrt{\omega} \]

\[ R_{\text{surf}} \text{ independent of } T \]

\[ \delta \propto \sqrt{\frac{1}{\omega}} \]

\[
\begin{align*}
\text{Cu @ 300 K (1.5 GHz)} & \quad R_{\text{surf}} \sim 10 \text{ m}\Omega \\
\text{Nb @ 2 K (1.5 GHz)} & \quad R_{\text{surf}} \sim 10 \text{ n}\Omega
\end{align*}
\]

Superconductor

\[ R_{\text{surf}} \propto \omega^2 \sigma_n \]

\[ R_{\text{surf}} \propto \exp\left(-\frac{T_c}{T}\right) \]

\[ \lambda_L \text{ independent of } \omega \]
Bulk Niobium

- Good purity, but ultra-pure not necessarily required
- Surface treatments are leading us to ultimate limit
  - Bulk still has strain, interstitials*, defects, rolling strain, etc.
- Poor thermal conductivity
- Hates to be welded, soldered, etc.

- Currently, S.O.A.
  - RRR ~ 300
  - 40 MV/m
  - $Q_0 \sim 10^{10}$
  - $H_c \sim 200$ mT

*Nb used to be known as columbium. Renaming (1949) followed from the confusion with Tantalum; Niobe was the daughter of Tantalus. Ta and Nb are hopelessly mixed, in nature.
Thin Films

Since penetration depths are << 1um, why are we using bulk?!?

Pros
• Cost! Cavities can be cast from Cu, Al, etc.
• Better thermal conductivity
• Integrate with He transport/cooling structures
• Mechanical amenities
• Precise growth control = parameter control
• Reduction of oxide layer via additional coatings
• Current State-of-art bulk Nb is difficult to deal with
  • Costly annealling
  • BCP, EP!!
  • E-beam welding
• Low $I$, hence high $Q_0$
• Precise control via purification OR degradation*

Cons
• $Q_0$ decreases with increasing RF field, aka “Q-slope” (at least for DC sputtering)
• We haven't figured out the recipe!

Why NOT Thin Films?!?

Single worst impediment is “Q-Slope”

Contaminants, substrate diffusion, etc.

Magnetic field / Energy gap interaction

Critical velocity / density of superfluid, ala LHe$_2$

\[ \text{V. Palmier, SRF 2005} \]

H-field enhancement at grain boundaries
Local heating, NC “hot spots”

In fact, nobody knows.......!!

\[ \text{RRR not likely cause, but symptom} \]
Thin Film Techniques

- CVD
- ALD
  - Way too slow!! > 400 nm
- PVD
  - Sputtering
  - Evaporation
  - PLD
- Electrochemistry
  - Too dirty!

Try to control:
- Grain size
- Purity
- Texture
- Defects & inclusions & dislocations
- Boundaries (diffusion)
- Oxide layer

Thin film technology is very mature: Intel i5, 5th gen. > 1.3 billion transistors!
Growth Stages

- Condensation (phase change)
- Adsorption / thermal accommodation
- Surface diffusion
- Nucleation
- Growth of Islands
- Coalescence “coarsening”
- To infinity and beyond!
  - eg. Abnormal Grain Growth

Figure courtesy of www2.warwick.ac.uk.
Nucleation (Homogeneous)

Change in energy from liquid to solid:

\[ \Delta G(r) = \frac{4}{3} \pi r^3 \Delta G_v + 4 \pi r^2 \gamma \]

\( \Delta G_v \) = Gibbs energy change for solidification
\( \gamma \) = surface energy

\[ \frac{d \Delta G(r)}{dr} = 4 \pi r^2 \Delta G_v + 8 \pi r \gamma = 0 \]

Critical Radius

\[ r^* = \frac{-2 \gamma}{\Delta G_v} \]

Nucleation barrier for solidification

\[ \Delta G(r^*) = \frac{16 \pi \gamma^3}{3 \Delta G_v^2} \]

\[ r_0 = \frac{-3 \gamma}{\Delta G_v} \rightarrow \Delta G(r_0) = 0 \]

\( \Delta G \) is not a minimum; nucleation continues.....

Nucleation (Heterogeneous)

Young's Sessile Drop

\[ \gamma_f \cos \theta_y = \gamma_{sv} - \gamma_{ls} \]

Total Energy

\[ \Delta G_{total} = a_3 r^3 \Delta G_v + a_1 r^2 \gamma_f + a_2 r^2 \gamma_{fs} - a_2 r^2 \gamma_{sv} \]

Critical Radius (\( \sim \) T)

\[ r^* = \frac{-2 (a_1 \gamma_f + a_2 \gamma_{fs} - a_2 \gamma_{sv})}{3 a_3 \Delta G_v} \]

Critical Energy

\[ \Delta G^* = \frac{16 \pi \gamma_{fv}^3}{3 (\Delta G_v)^2} \left[ \frac{2 - 3 \cos(\theta) + \cos^3(\theta)}{4} \right] \]

Collision rate

\[ \Phi = \frac{3.51 \times 10^{23} P}{\sqrt{MT}} \]

P = pressure, M = atomic weight

Monolayer growth rate

\[ t_m \sim \frac{3 \times 10^{-4}}{P} \]

(1 sec for \( 10^{-6} \) Torr)

**Island Growth**

**FVDM - 2D**
- Substrate-vapor free energy prevails
- Individual monolayers
- Arriving atoms seek perimeter

**VW - 3D**
- Atomic Tetris!
- Ultimate film growth mechanism
- Nucleation rate ~ arrival rate (fluence)

**SK - 2D/3D hybrid**
- Individual monolayers
- Nucleation after ~ 5 layers
- Ultimately follows VW

Niobium “cannonballs” prefer a BCC \{1,1,0\} crystal formation

Coarsening

Surface Diffusion

- Diffusion distance can be ~100 um+!

Grain Boundaries

- Atomic “superhighways!”

Ostwald Ripening

- Urban growth?!
- Bubbles in carbonated beverages

Courtesy www.Nonast.net
Abnormal (Secondary) Growth

Force diagram of a cylindrical cap (r, h) with top surface energy, $\gamma_p$, edge surface energy, $\gamma_e$, and bottom surface energy, $\gamma_{ps}$. Interface energy is defined as the difference between substrate energy, $\gamma_{gs}$ and bottom surface energy, $\gamma_{ps}$:

$$\gamma_i = \gamma_{gs} - \gamma_{ps}$$

Surface energies are manipulated so as to drive secondary grain growth. Minimization of interface, surface, and strain energies.

Achieved via substrate T, and film thickness (strain ~ 1/surface)

2-D radial growth is encouraged when h ~ r.


Thompson – Floro Growth Rate Eq.

$$i = M \left[ \frac{(\gamma_i - \gamma_i)}{h} + \gamma_{gb} \left( \frac{1}{r} - \frac{1}{r} \right) \right]$$

Energy which drives growth

$$\Delta F = -\frac{2\Delta \gamma}{h} - \frac{1.15 \gamma_{gb}}{r_n} + \frac{2 \gamma_{gb}}{r_s}$$

In lieu of 3-D energy minimization, surface and interface energies drive grain growth

BCC {1,1,0} crystals grow fastest

Ag crystals have been grown on Ni, having > 100x initial diameters (~ 1µm!).
Floro notes that films grown at LN2 temperatures are inherently defective, and contain a large amount of strained micro-structures, leading to preferred orientation. This resembles the bulk orientation of cold-worked single crystals, which ultimately experience re-crystallization to lower energy configurations when warmed to annealing temperatures, as confirmed by other experimenters. This is precisely the mechanism sought for Nb film growth, for which is predicted by the Ag/Ni experiment.

In addition, randomized islands were observed at 77K Ag substrate; warming resulted in rapid growth along boundaries. Growth was much slower away from grain boundaries.
Argon gas is ionized by HV (initiated by stray cosmic ray)

Electrons are trapped by ExB fields

As electrons migrate along field lines, they ionize additional atoms

Resulting Ar ions are accelerated towards electrode

Target atom(s) is ejected by ballistics of collision

Target atom is sent on it's way to substrate
Fibrous, columnar growth
Produces tons of small grain boundaries, large $R_{\text{surf}}$
Energetic Condensation

High ratio of hyper-thermal particles (10-100 eV)

Energetic ions facilitate diffusion on a cooler substrate

Density, composition, and crystal orientation

Grain competition moderation

Fibrous growth is suppressed (mountaintop removal)

Higher density, less stress

Ion “stitching” (~10 Å / 100eV)

ECR
e- plasma Radiation
Pulse magnetron Bias sputter Direct ion

Courtesy www.swri.org
As an alternative to DC magnetron sputtering, the unit is pulsed with large energy (>10x), but with low duty factor (~1%). The ion ratio is quite extreme, and sufficient for direct ion bombardment. T ~ 200us, F ~ 10 kHz.

Scheme #1

Produce a thick (~ 1μm) Nb film on 77 K Cu substrate (> 10 $\lambda_L$)

Energetic condensation, via MPPMS
  Amorphous film is far from equilibrium
  High density
  Cold substrate discourages post-condensation diffusion, grain growth

HIPPO-Laser induced re-crystallization
  Facilitates film energy minimization by producing large grains
  Strive for electron mean-free paths of > 200nm

Since “thick” requires minimal growth control, it is the easiest path to a re-crystallized film.
Scheme #2

An engineered template is created by controlled amorphous Nb film growth onto 77 K copper,

Create a film, having \( l > 200 \) nm, extending several \( \lambda_L \) depths

2-D abnormal grain growth is encouraged (i.e. Surface energy dominates the bulk film energy).

This will entail multiple thin film growth periods, followed by laser processing, and selective re-crystallization.

If large grains are grown, use the film as an epitaxial template to produce thicker films, beyond the range where grain competition is complete.
Pump Cart

LN2 operation @ 77K
MPPMS Modulator

1200V @ 1500 A peak-pulse
500 Joules / pulse

IGBT-Based bi-polar pulser
Laser Processing / Recrystallization

Raster-scanning for precise fluence control

12 ns @ 50 kHz pulses raise local area to ~ 900°C

Diffusion results in large crystals

Industrial HIPPO 355 nm Laser, 5W / 8 kW

Film Morphology
Film Analysis w/ SEM

300K Substrate \( \sigma_r = 17 \text{ nm} \)

77K Substrate \( \sigma_r = 7 \text{ nm} \)
Residual Resistance Ratio (RRR)

\[ RRR = \frac{\rho(T = 300 \, K)}{\rho(T = 4.2 \, K)} \]


\[
Z_s = \frac{P_{rf}}{kB_{pk}^2} + i \omega \mu_0 \left( \frac{\gamma_{ref}}{\lambda_{ref} + \frac{f - f_{ref}}{M}} \right)
\]

Connects surface morphology to RF performance
What's Next?!

5” Simard Magnetron

5000+ Joule Modulator

Laser Table

Close proximity, remove guide coil

Copper substrate

Proceed with laser recrystallization
Thank You!!