A New Method for Grain Texture Manipulation in Post-Deposition Niobium Films

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Experimental Strategy

➢ Employ energetic condensation (EC) to maximize film deposition density

➢ Use cold substrate to control surface energies during nucleation; dense amorphous film is created which possesses a much larger energy density than the re-crystallized state.

➢ Re-crystallization of the film surface results in a crystalline structure resembling bulk Nb. Grain growth is driven by a pulsed laser, or other forms of heating.

➢ Controlled heating for surface re-crystallization is provided by a pulsed UV HIPPO laser, raster-scanned over the film surface, avoiding excessive substrate heating.

➢ Also, since the surface processing is capable of being performed in the vacuum chamber, any chance for native oxide layer buildup, or interstitial contamination can be eliminated.
Process Stages

Produce a thick (~ 1um) Nb film on “cold” substrates, beginning with silicon, and then copper (2” coupon).  $77 \text{ K} < \text{“Cold”} < 300 \text{ K}$

Energetic condensation (deposition), via MPPMS

- Cool substrate (LN2)
- Cold substrate discourages (abnormal) grain growth during the deposition of thin fim
- Establish graded Nb/Cu interface via high energy (> kV) ions for initial deposition (stitching)
- Deposit thick (1 um) nearly-amorphous film via EC; minimize voids, maximize energy density

HIPPO-Laser induced re-crystallization

- Precise fluence delivery accurately drives grain growth
- Short pulses produce local heat at RF surface for re-crystallization.
- Exploit short pulse timing and Nb thermal diffusivity to avoid overheating substrate.
Deposition Schematic

- Minimize substrate energy via 77 K
- Maximize internal energy of condensate
- Control film thickness during deposition
- Provide controlled temperature conditions for re-crystallization.
Deposition Chamber
Deposition Chamber (cont.)

Magnetron Housing (2” Aja)  Sample Holder (insulated)
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. Power density is ~kW/cm².

Films created with 0% (a), 45% (b), and 100% (c) HiPIMS. Density is clearly evident for 100%.

Modulated Pulse Power Magnetron Sputtering

MPPMS is an attractive alternative to HiPIMS

Micropulse = 100 uS HiPIMS pulse
Macropulse = up to dozens of micropulses
Ignition + extinguish + re-ignition stimulates ion-rich plasma of well-defined energies.

Relative Nb ion production for HiPIMS, MPPMS, and DCMS, obtained by optical spectra (Hala, et al.)

Parametric comparison of EC methods, with resulting Nb film SEMs.

MPPMS films strongly resemble HiPIMS, while also exploiting a higher growth rate (via duty factor).

Prototypical pulse structure obtained by parametric study......

500 J MPPMS Modulator (IGBT, FPGA)

1200 V @ 1500 A / pulse
Kr pressure = ~10 Torr (1 Pa)….Niobium's nearest inert neighbor
Each macropulse = 8 micropulses:

First pulse = 100 us
50 us for subsequent pulses

V = 650 V
P_{ave} = 300 W
F_{rep} = 94 Hz
P_{pk} = 7 kW / macro
W_{pulse} = 3 J / macro
I_{ion} = 35 mA

Starting pressure = 1.7 \times 10^{-7} \text{ Torr}
Deposition time = 5 hrs
Measured thickness = 765 nm (SEM)
Raster-Scanned 355 nm HIPPO Laser

...in situ capability....
Singaravelu, et al., have performed bulk niobium annealing ..... we have a starting point!

Absorption vs Wavelength, niobium

Simulation of 8 ns, 1064 nm Fluence Pulses

Spectra-Physics HIPPO 355-5

~1200 K ($T/T_m = 0.5$) => 240 mJ / cm²

Laser pulse ~12 ns
Repetition rate ~ 50 kHz
T_dwell = 20 us

1-D model OK for now..... 100 um >> $D_{Nb} \tau \approx 700 \text{ nm}$

Bechtel Square Laser Pulse Model*

\[
\Delta T(0, t) = \begin{cases} 
0, & t < 0 \\
\frac{2I_m(1 - R)(Dt)^{1/2}}{K} \frac{\pi^{1/2}}{\tau}, & 0 \leq t \leq \tau \\
\frac{2I_m(1 - R)(Dt)^{1/2}}{K} \frac{\pi^{1/2}}{\tau} \left[ \left( \frac{t}{\tau} \right)^{1/2} - \left( \frac{t}{\tau} - 1 \right)^{1/2} \right], & t > \tau
\end{cases}
\]

K = initial temperature
D = thermal diffusivity
R = reflectivity
I = irradiance

Raster-scanning for precise fluence control

12 ns pulses raise local surface area to $\sim 1200^\circ C$

Substrate is at 77 K, and protected from surface temperature

Spectra Physics HIPPO Laser (5W @ 355 nm)
Anticipated Analyses

XRD
Grain Size (bulk)
Compression strain

SEM
Film thickness
Surface texture, roughness

TEM
Evidence of ion stitching
Re-crystallization, depth, crystal structure

EBSD
Grain structure (surface), orientation
Strain

Tc, Surface Impedance Characterization Cavity*

XRD, $T_c$ Analysis of First Films (Scherrer)

**XRD vs Fluence for Nb Films**

77K Substrate (Si)

(laser processed at 300 K)

<table>
<thead>
<tr>
<th>Fluence</th>
<th>$T_c$</th>
<th>Grain Size</th>
<th>Strain</th>
</tr>
</thead>
<tbody>
<tr>
<td>77K, Raw</td>
<td>9.37</td>
<td>4.4</td>
<td>0.025</td>
</tr>
<tr>
<td>300 mJ</td>
<td>9.29</td>
<td>8.8</td>
<td>0.0125</td>
</tr>
<tr>
<td>308 mJ</td>
<td>9.27</td>
<td>9.16</td>
<td>0.012</td>
</tr>
<tr>
<td>283 mJ</td>
<td>9.27</td>
<td>8.8</td>
<td>0.0124</td>
</tr>
<tr>
<td>260 mJ</td>
<td>9.29</td>
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<td>230 mJ</td>
<td>9.28</td>
<td>7.72</td>
<td>0.0141</td>
</tr>
<tr>
<td>203 mJ</td>
<td>9.29</td>
<td>7.59</td>
<td>0.0144</td>
</tr>
<tr>
<td>107 mJ</td>
<td>9.32</td>
<td>6.98</td>
<td>0.0157</td>
</tr>
<tr>
<td>10 mil Foil</td>
<td>9.27</td>
<td>43.98</td>
<td>0.0025</td>
</tr>
</tbody>
</table>

< 500 mJ!

Resistance vs. Temperature - LT

$T_c = 9.27$
Delta $T_c = 0.03$
Conclusion

Grain texture manipulation facilitates detailed studies

MPPMS is suitable for producing high density films

“Simplistic” thin film deposition greatly improves manufacturability (minimize parameters)

Potential Grain texture control from localized heat source

In situ processing eliminates oxide layer

Ion stitching maximizes film adhesion to substrate (thermal)

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