Deposition of niobium and other superconducting materials with high power impulse magnetron sputtering: Concept and first results

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Nb-coated SRF Cavities: The promise of very substantial savings

- bulk Nb cavities are very expensive
- so far mixed results for sputtered films
  - adhesion issues on copper
  - relatively low RRR
  - a proven technology for up to about 10 MV/m but $Q_0$-slope at high field
  - Breakdowns observed even at relatively low field
- Nb films on copper were demonstrated and utilized: LEP (CERN, 1996): 216 cavities with sputtered Nb, 6 MV/m with $Q_0 = 3.4 \times 10^9$ at 4.5 K

Fig. 4 of C. Benvenuti et al., Physica C: Superconductivity 351, 421 (2001).
Magnetron Sputtering Concept for Nb (1990s)

J. Langner, et al., 5th Int. Conf. on Modification of Materials with Particle Beams and Plasma Flows, Tomsk, Russia, 2000, pp. 399-401.
1974 - Thorton’s Structure Zone Diagram

Contains the effects of energetic particle bombardment

ARGON PRESSURE (mTorr)

Generalized Structure Zone Diagram including the Effects of Plasma Assistance on Films (“Energetic Condensation”)

derived from Thornton’s diagram, 1974

$T^* =$ normalized temperature and potential energy
$E^* =$ normalized kinetic energy
$t^* =$ normalized film thickness

Zone 1: porous, tapered crystallites separated by voids, tensile stress
Zone 2: densely packed fibrous grains
Zone 3: recrystallized grain structure

Transition from tensile (low $E^*$) to compressive stress (high $E^*$)

Region of possible low-temperature low-energy ion-assisted epitaxial growth

Dense film, reduction of deposition by sputtering

Line separating net deposition and net etching

Cutout to show structure

Fine-grained, nanocrystalline, with preferred orientation
**HIPIMS: A Form of “Ionized Sputtering.”**

One Approach to “Energetic Deposition.”

“What distinguishes HIPIMS from the long-practiced pulsed sputtering?”

**Technical Definition:**
*HIPIMS is pulsed sputtering where the peak power exceeds the average power by typically two orders of magnitude.*

(implies a long pause between pulses, hence the term “impulse”)

**Physical Definition:**
*HIPIMS is pulsed sputtering where a very significant fraction of the sputtered atoms becomes ionized.*

(implies that self-sputtering occurs, which may or may not be sustained by target ions)

Why do we care? Because bias can be applied to affect film-forming ions (not atoms)!


About 500 kW peak
15 cm dia. Cu target

Image from the seminal (but not first) paper:
Copper target
2" magnetron

A. Anders, et al., JAP 103 (2008) 039901
at low pressure, little compression and rarefaction

at large distances, significant differences in ion speed and plasma arrival
HIPIMS without any gas: Pure Self-Sputtering in Vacuum

HIPIMS and Self-Sputtering of Niobium

HIPIMS with Nb target, Ø 5 cm

in 0.25 Pa of Ar

in 0.50 Pa of Kr

Minimizing Argon trapping into the growing film:  
→ We look for the minimum gas pressure at given average power

Preliminary Nb coatings by HIPIMS

- Grains as well as defects in substrate are reproduced in the coatings → this points to the importance to care (worry!) about the substrate.

Observation: Adhesion on aluminum (incl. its oxide) is superior → consider aluminum substrates and cavities!
Our First $T_c$ and RRR Measurements of Nb

- $T_c = 8.35$ K
- RRR = 4.36, seems disappointingly low, but:
  - measurement includes Al underlayer
  - residual gas contamination expected
  - in hindsight: this sample had a very rough sputtered Al underlayer
Niobium Films on Aluminum-Silicon Substrates

Al on Si

Nb on Al on Si
Great Effect of Temperature indicates: Ion Assistance is still insufficient

- XRD of Nb on Al on Si
  - At RT: small crystallites
  - At 200°C: gradually greater grains, stress relaxation
  - At 400°C: major change to larger, aligned crystals

- These films were grown without any bias! → future work.
One more piece of evidence for the importance of the substrate...

- HIPIMS film of Nb on (amorphous!) glass → nanocrystalline film
Custom Movable, Cylindrical Magnetrons for 1.3 GHz Cavities
**Dual Magnetron: Most effective for a Biasing Approach: Affecting Ion Energies and Trajectories**

- **nearly perpendicular incidence!**
- **sheath**
- **plasma**
- **ion trajectory**
- **possible collision**
- **cavity**
- **$V_{\text{bias}}$**
- Magnetron 1
- Magnetron 2
A Dedicated Nb-HIPIMS Chamber @ Berkeley

- with initial LDRD and later DOE-HEP FY10 funding:
  - chamber for 1.3 GHz SRF cavities
  - base pressure in the low $10^{-8}$ range
  - residual gas analyzer
  - 2 small cylindrical, movable magnetrons
  - decoupled substrate heating and biasing
  - pyrometer 100-600 °C
  - 2 SIPP pulsers for dual-HIPIMS and bias
A state-of-the-art HIPIMS system for 1.3 GHz, offering optional dual-HIPIMS and two-material HIPIMS.
HIPIMS Coatings Technology for SRF Cavities

- dual cylindrical magnetron in at relatively low power sputtering mode
  - Dominated by argon emission
- dual cylindrical magnetron in high power mode (above runaway threshold)
  - Dominated by niobium emission

Outlook: besides the single and dual magnetron modes, we should extend our goals to other materials
- Relatively straight forward to include NbN and Nb/NbN multilayers
- One could use two different materials with two asymmetrically operating magnetrons to produce Nb₃Sn, NbTi alloys, Mg₂B, and multilayer structures
Summary / Conclusions

- there is a compelling story for thin Nb-film SRF cavities
- many issues need to be solved, including
  - substrate preparation and
  - coatings technology
- cathodic arc and HIPIIMS are distinct technologies, each delivering an “energetic condensation” approach
- HIPIIMS has the advantage of not generating macroparticles (assuming that arcing is prevented)
- Nb has a relatively low self-sputtering yield \( \rightarrow \) “gasless” self-sputtering in vacuum could not be demonstrated
- low pressure operation works well with optimized pulse frequency.
- a dedicated HIPIIMS system with two cylindrical magnetrons is completed and waiting to be used. Material systems beyond Nb could and should be investigated.