HiPIMS Deposition of Nb Coatings with Bias Voltage: Preparation and Characterization at IHEP

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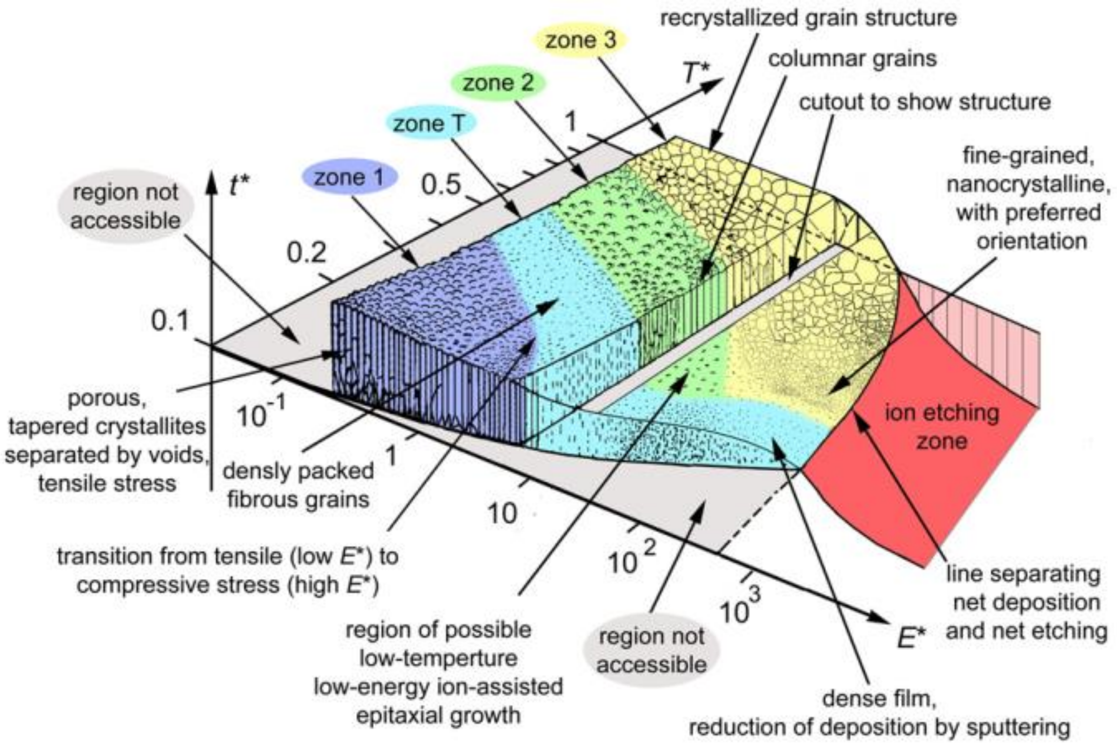
Outline

- **Introduction to HiPIMS Nb Coating Technology**
- **Cylindrical HiPIMS cavity system**
- **HiPIMS Waveform Testing and Analysis**
- **HiPIMS Nb Film Deposition Experiments and Results**
- **Summary and Conclusion**
- **Future plans**

Introduction to HiPIMS Nb Coating Technology

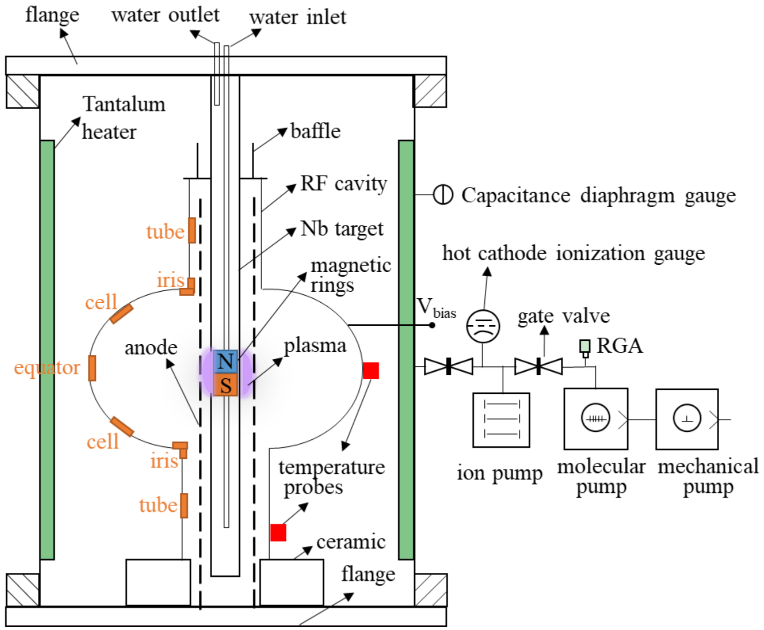
- **What is HiPIMS?**
- An advanced PVD technique that utilizes high-power, short-duration pulses to generate a dense and highly ionized plasma.
- **Why use HiPIMS for Nb Coatings?**
- Increased Film Density
- Improved Crystallinity
- Controlled Film Morphology
- Enhanced Film Adhesion
- Lower Substrate Temperature



Anders A. A structure zone diagram including plasma-based deposition and ion etching[J]. Thin Solid Films, 2010, 518(15): 4087-4090.

Cylindrical HiPIMS cavity system

- chamber for 1.3 GHz SRF cavities
- Niobium cathode RRR~300
- base pressure in the low 10E-9 mbar range
- residual gas analyzer
- movable magnetrons 1mm/s
- Coating temperatures 200℃
- Water cooled
- Substrate preparation is SUBU
- Assembly in clean room
- Coating with Kr

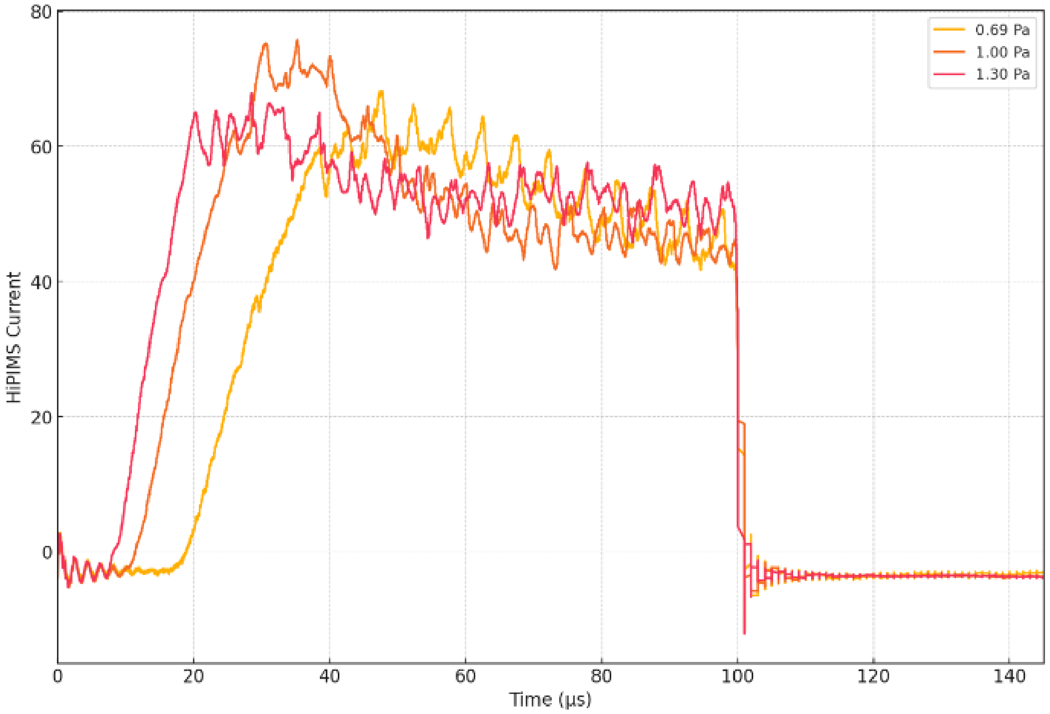


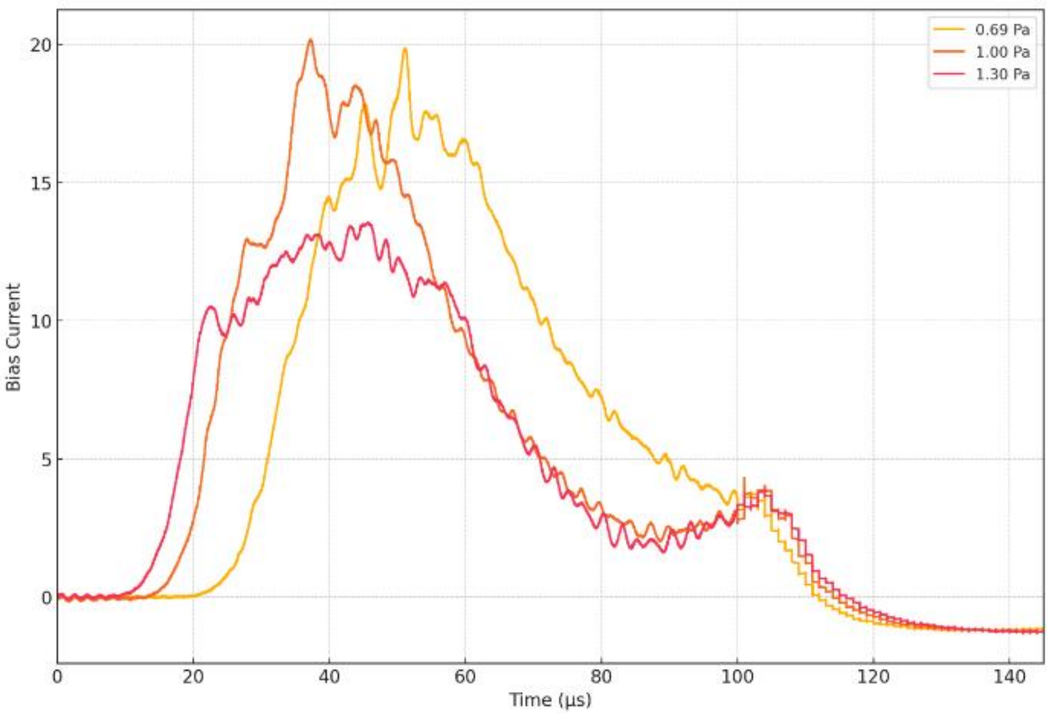
Huttinger Electronics TruPlasma Highpulse DC Unit:

- 10 kW max average power delivered
- voltage up to 1 kV
- pulse width up to 1000 us
- frequency up to 10 KHz

HiPIMS Waveform Testing and Analysis

Effect of Different Gas Pressures on Waveform



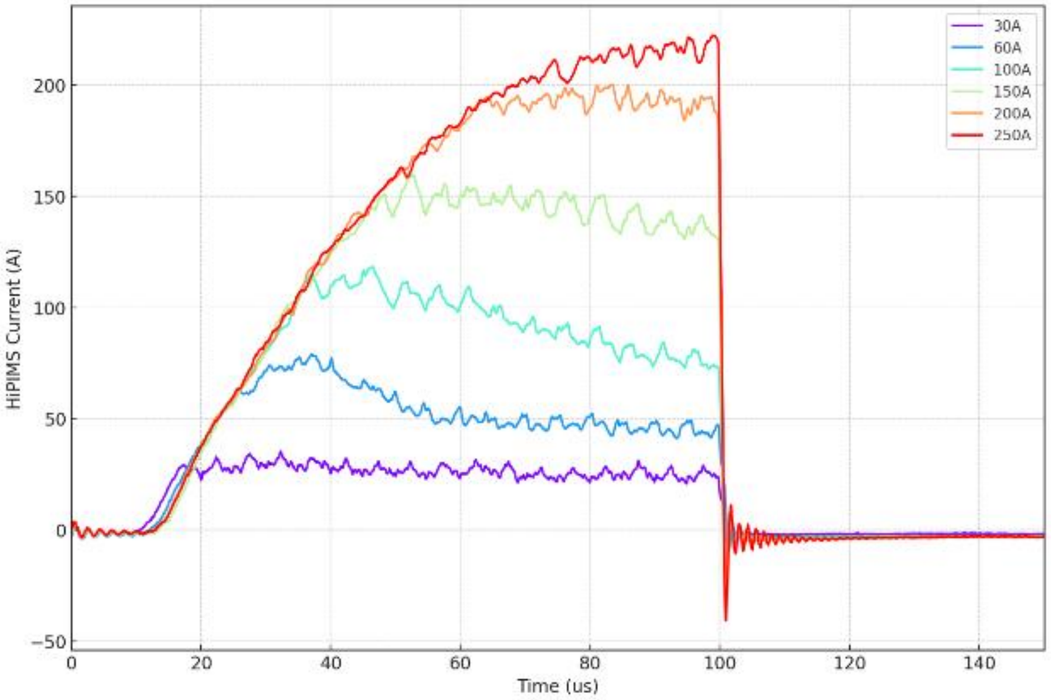


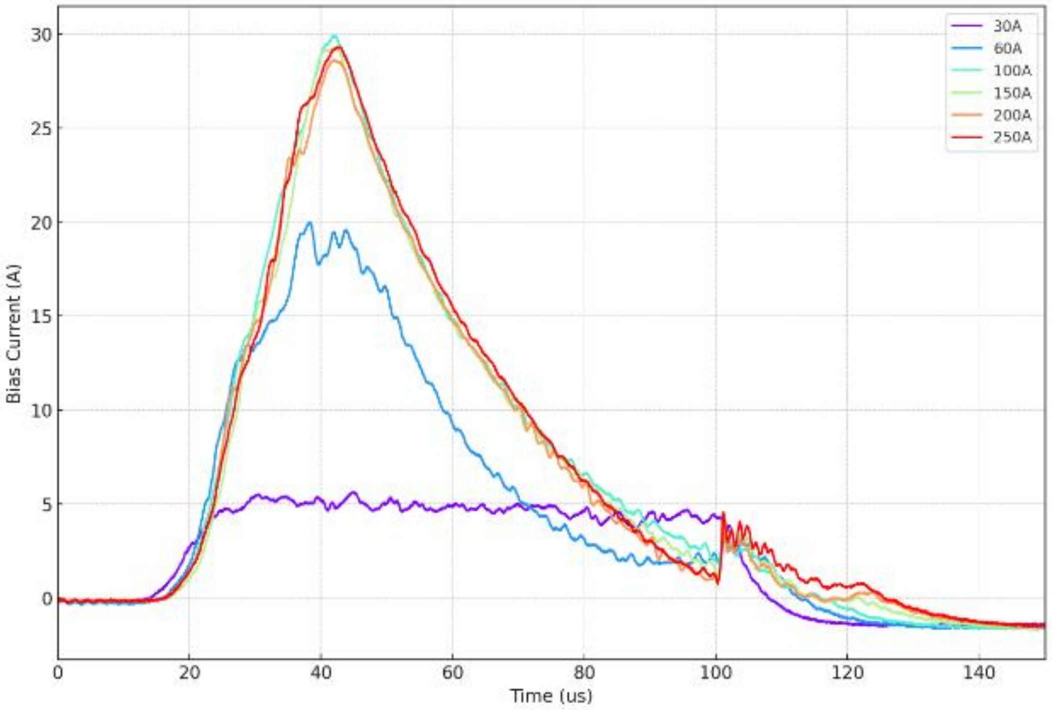
HiPIMS Discharge Transition:
Transition from gas to metal ion discharge
Current Drop:
Post-peak due to lower Nb ion self-sputtering yield.

Ignition Time:
Decreases with higher gas pressure.
Increased Gas Pressure:
Particle collisions become more frequent.
At 1.3 Pa, the peak current value decreases.

HiPIMS Waveform Testing and Analysis

Effect of Different Discharge Currents on Waveform



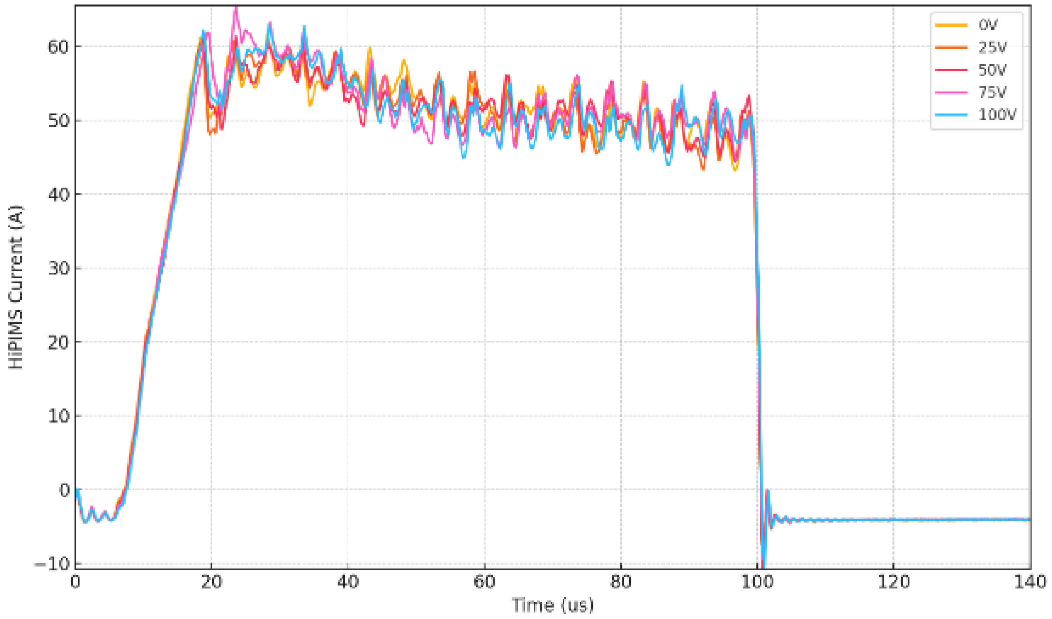


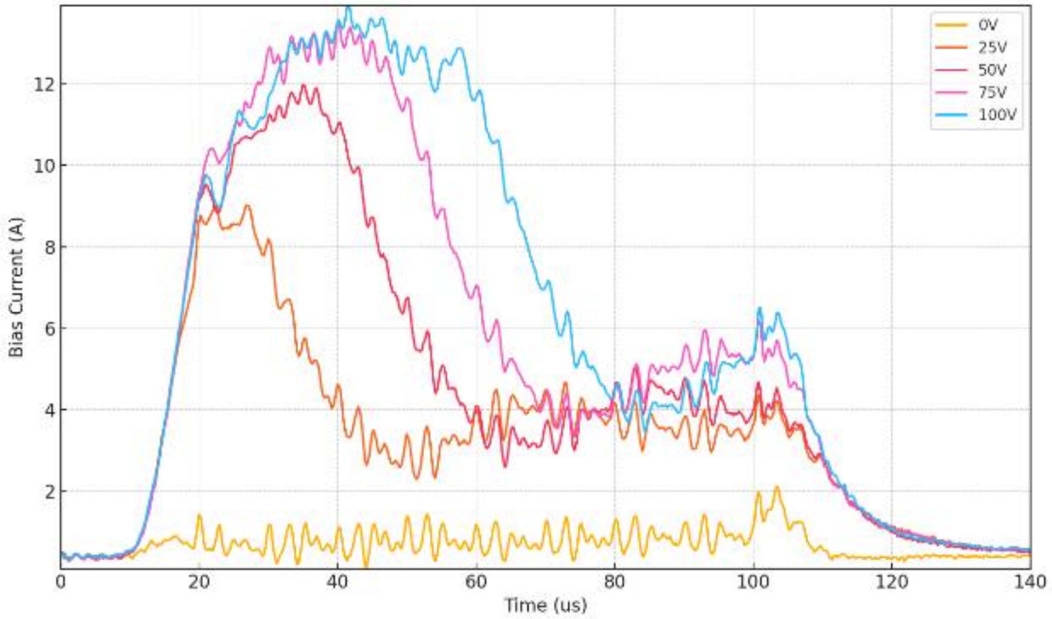
Low Current Waveforms: At lower currents (e.g., 30A), waveforms are rectangular pulses.
High Current Waveforms: At higher currents (e.g., 200A), forming a more pointed triangular waveform.

Bias Current Trends: As discharge current increases, bias currents also adopt a sharper triangular shape, peaking at 100A. Beyond this current, no significant changes are observed in bias current waveforms.

HiPIMS Waveform Testing and Analysis

Effect of Different Bias Voltages on Waveform





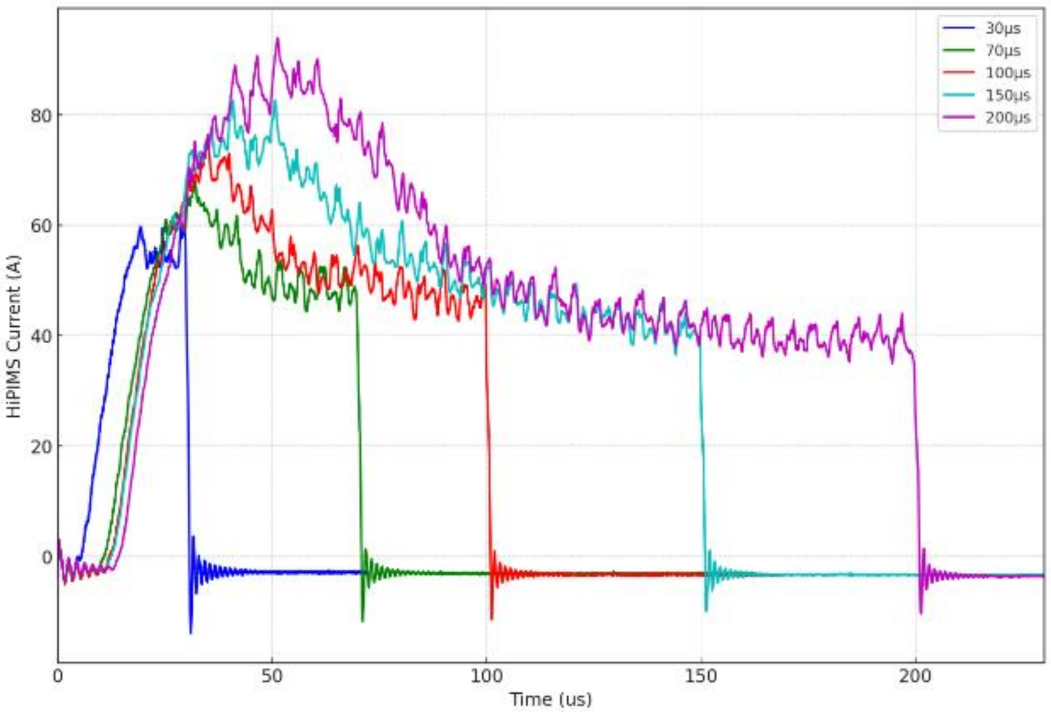
Bias Voltage Effect: No significant impact on the main HiPIMS current waveform; the primary effect is observed in the bias current.

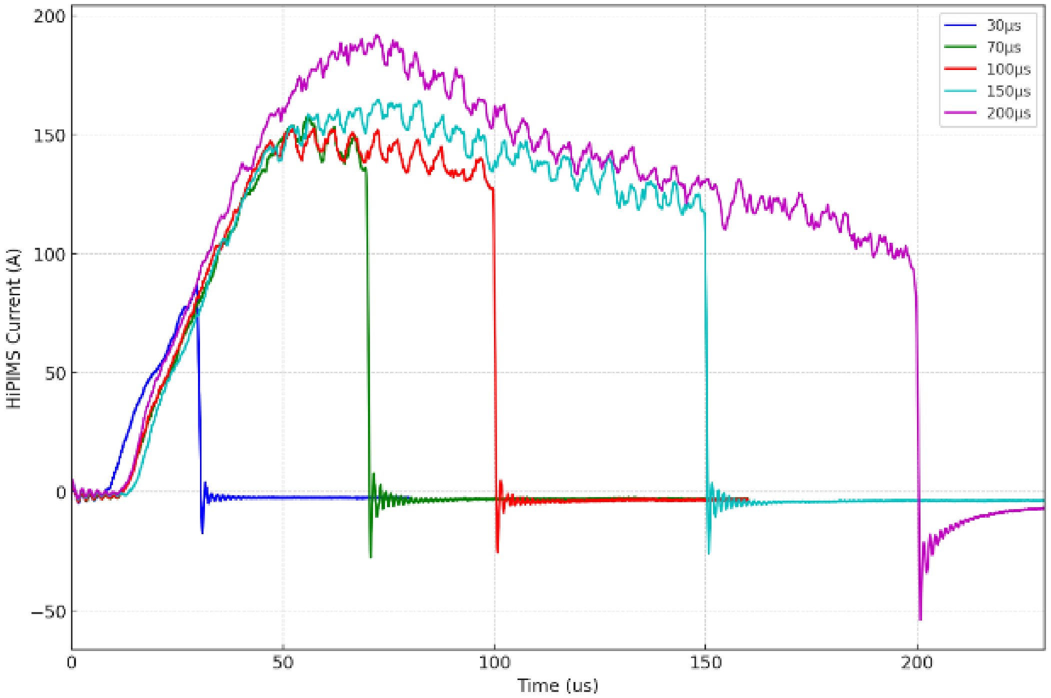
0V Bias Voltage: Almost no distinct pulses in the bias current waveform.

Increasing Bias: As bias voltage increases, the pulses in the bias current waveform become progressively more pronounced. The maximum value of the bias current stabilizes when the bias voltage is raised to 75V.

HiPIMS Waveform Testing and Analysis

Effect of Different Pulse Widths on Waveform under 50V Bias Voltage

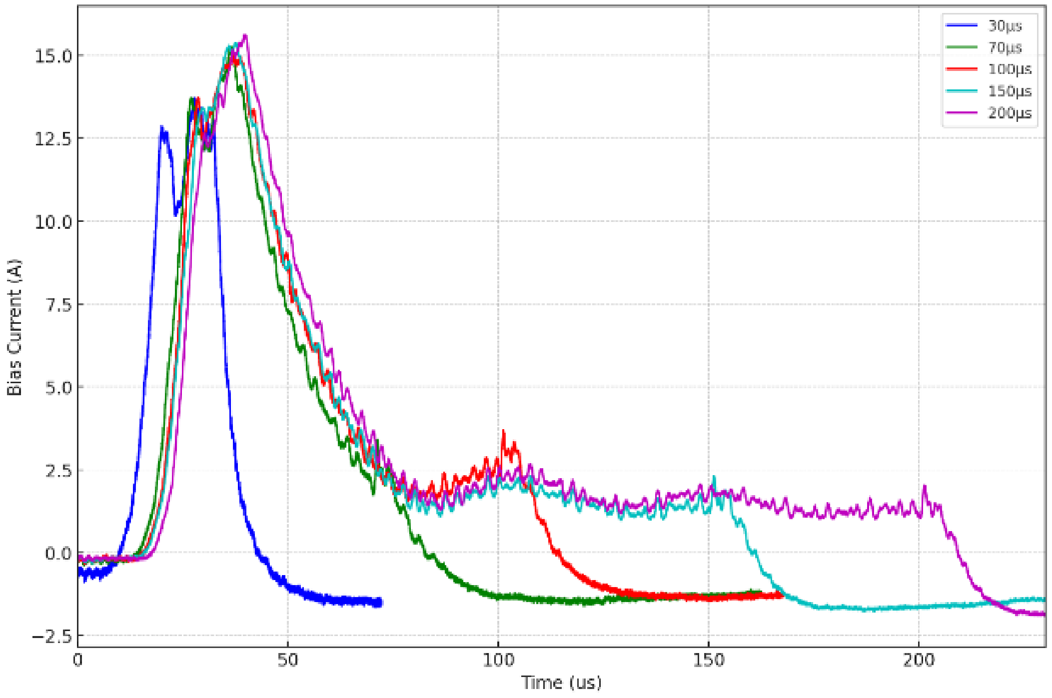


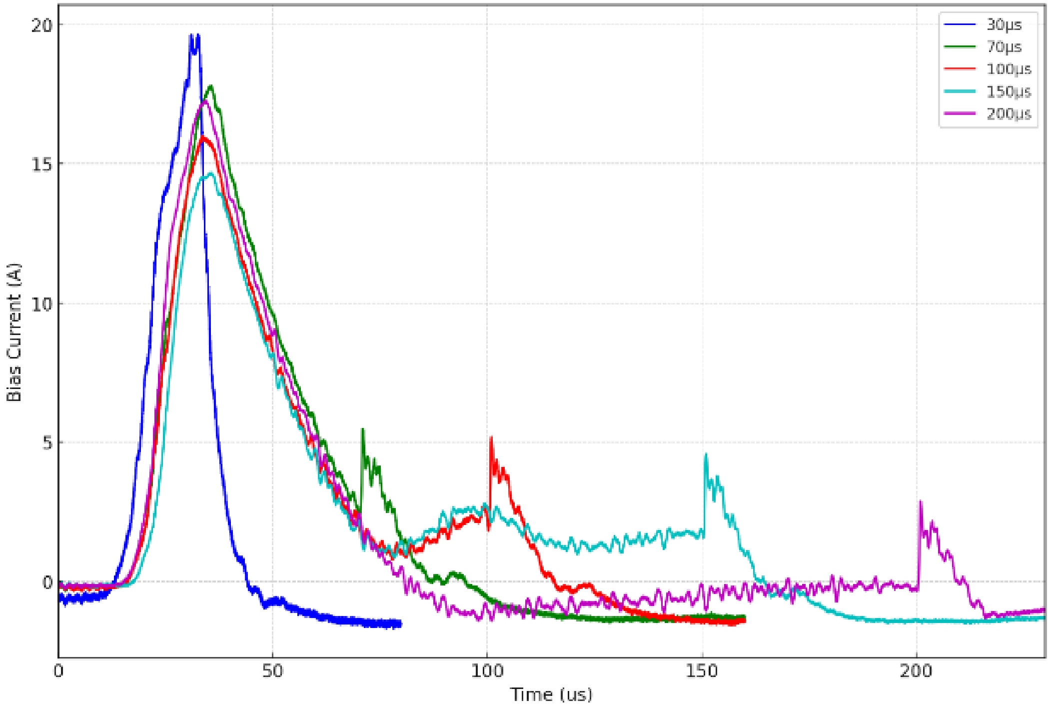


30us Pulse Width: At 60A, the waveform shows a narrow rectangular waveform. At 150A , the waveform exhibits a triangular waveform , indicating gas discharge dominance.
70us Pulse Width: The 60A waveform shifts towards a triangular shape, while the 150A waveform approaches a rectangular shape.

HiPIMS Waveform Testing and Analysis

Effect of Different Pulse Widths on Waveform under 50V Bias Voltage





30us Pulse Width:

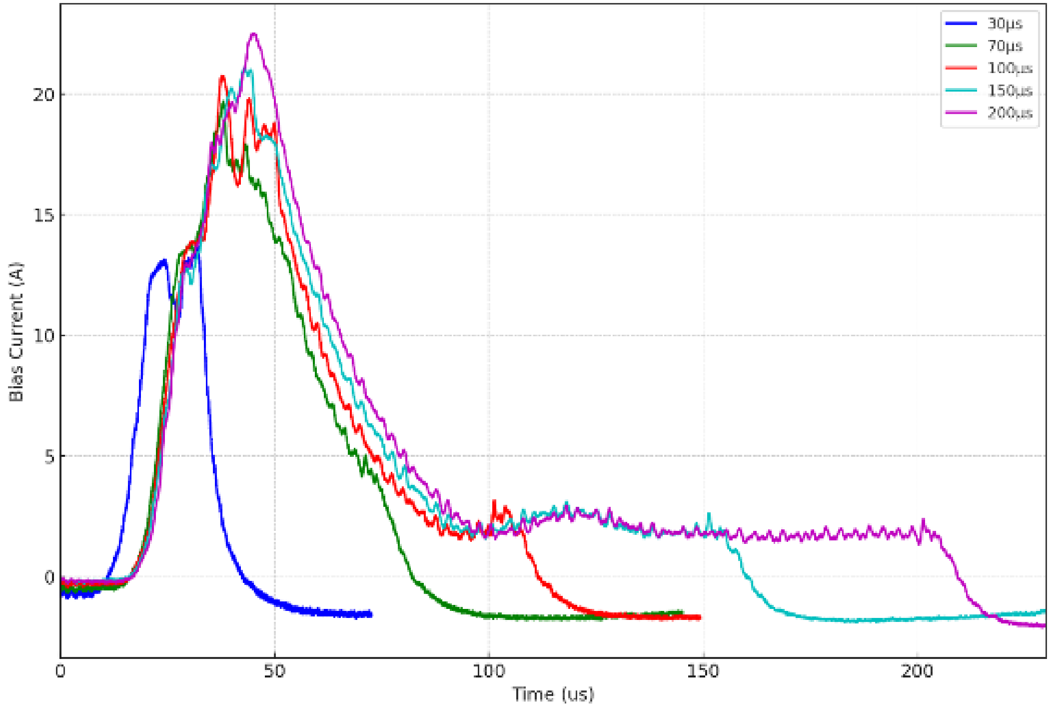
- 60A Bias Current: Double-peak structure observed.
- 150A Bias Current: Triangular waveform with higher peak compared to other widths.

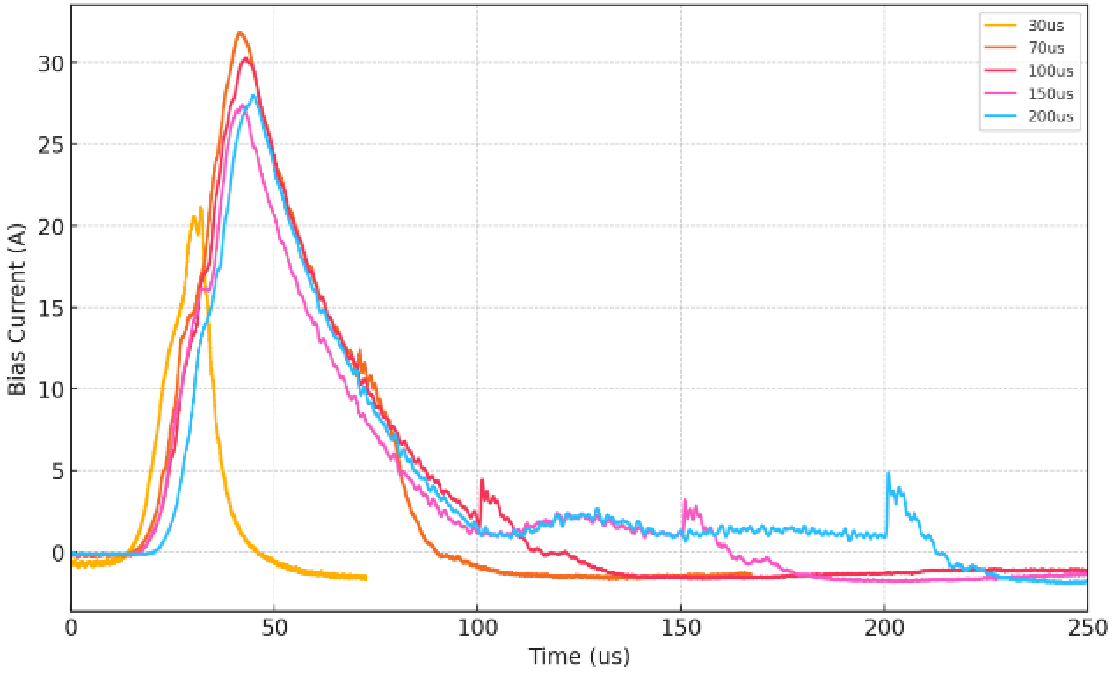
Increased Pulse Width (70us+):

- 60A Bias Current: Peaks stabilize, maintaining similar waveform shape.
- 150A Bias Current: Peaks decrease, with a sudden increase at the end of each pulse.

HiPIMS Waveform Testing and Analysis

Effect of Different Pulse Widths on Waveform under 100V Bias Voltage





60A Bias Current at 100V:

- **30us Pulse:** Similar structure and magnitude to 50V bias voltage.
- **70us Pulse:** Peak current sharply increases.
- **100us Pulse:** Waveform becomes fully formed.

150A Bias Current at 100V:

- **30us Pulse:** Similar structure and magnitude to 50V bias voltage.
- **100us Pulse:** Waveform becomes fully formed, amd peak current starts to decrease,

HiPIMS Nb Film Deposition Experiments and Results

Experiment 1 -50V Bias Voltage and Pulse Width Effects

Experimental Parameters for Nb Film Deposition

Experiment ID	Average Voltage (V)	Peak Current (A)	Discharge Power (KW)	Pulse Width (us)	Frequency (Hz)	Duty Cycle (%)	Peak Power (KW)	Bias Current Average (A)	Discharge Pressure (Pa)
V0-A60-PT100	-560.0	60	1.8	100	615	6.1	37.3	0	0.69
V50-A60-PT30	-617.4	60	1.8	30	2597	7.8	38.5	5.4	0.69
V50-A60-PT100	-593.5	60	1.8	100	636	6.4	44.5	5.1	0.69
V50-A150-PT100	-632.1	150	1.8	100	269	2.7	98.1	4.8	0.69
V50-A150-PT200	-634.8	150	1.8	200	115	2.3	124.6	2.1	0.69

Characterization Results of Nb Film

Experiment ID	Nb(110) Peak Position (°)	Nb(110) Crystal Phase Ratio (%)	Lattice Constant (Å)	Stress (MPa)	Nb Film Thickness (μm)	Surface Roughness (nm)	Tc (K)
V0-A60-PT100	38.8	57.7	3.2821	280.1	3.6	71.1	9.19
V50-A60-PT30	39.0	70.7	3.2472	679.1	2.8	-	-
V50-A60-PT100	38.7	91.2	3.2853	243.8	1.6	31.1	-
V50-A150-PT100	38.4	89.8	3.3098	-36.1	1.9	37	9.32
V50-A150-PT200	38.9	84.8	3.2693	426.4	2.5	21.9	9.40

$$\text{Nb(110)Crystal Phase Ratio} = \frac{I_{(110)}}{I_{(110)} + I_{(200)} + I_{(211)} + I_{(220)}}$$

HiPIMS Nb Film Deposition Experiments and Results

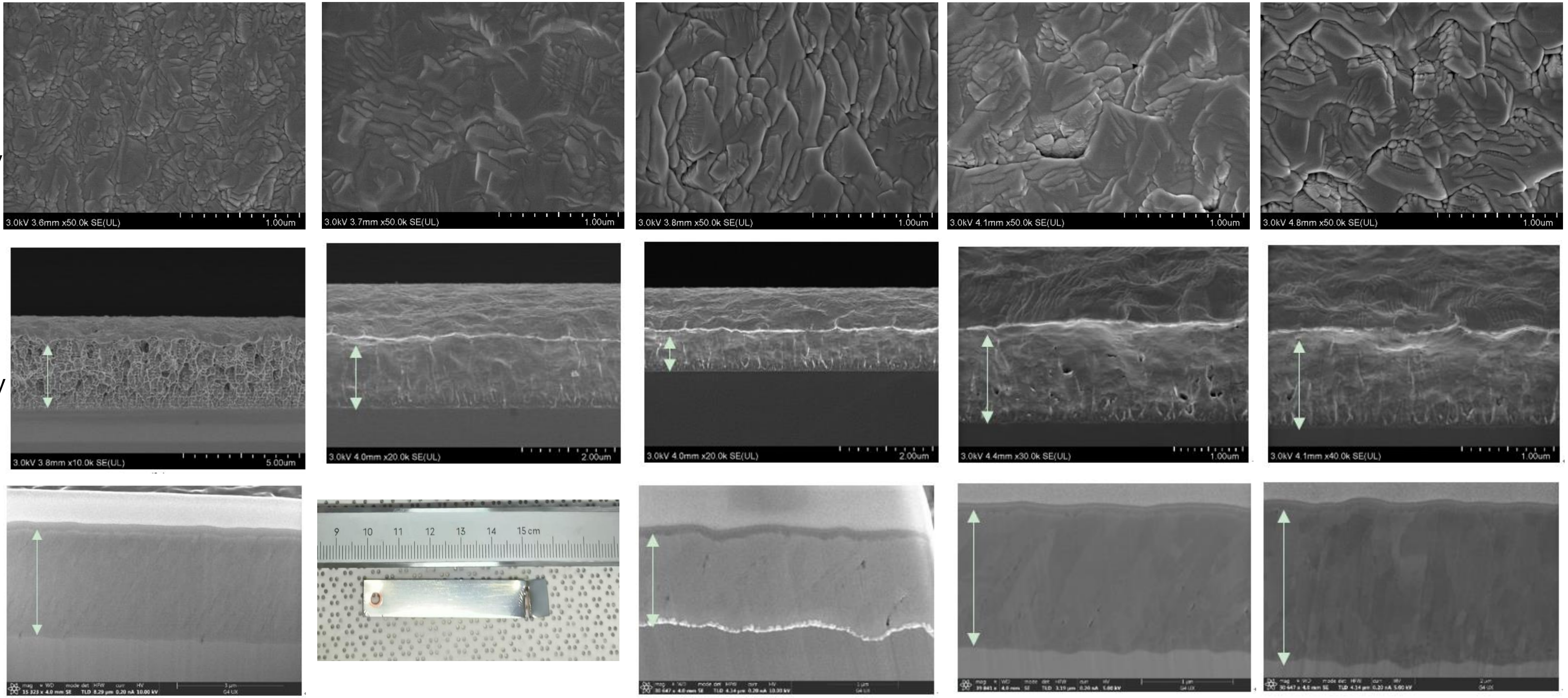
Experiment 1 -50V Bias Voltage and Pulse Width Effects

V0-A60-PT100 | V50-A60-PT30 | V50-A60-PT100 | V50-A150-PT100 | V50-A150-PT200

SEM surface morphology

Cross-sectional SEM morphology

Cross-sectional structure by FIB-SEM



Columnar, porous structure with shadowing defects

Excessive stress

Poor interface quality

Presence of small voids

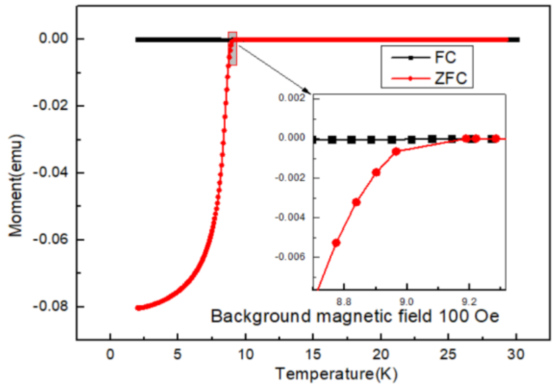
Columnar grain structure, dense

HiPIMS Nb Film Deposition Experiments and Results

Experiment 1 -50V Bias Voltage and Pulse Width Effects

M-T
measurement
curve

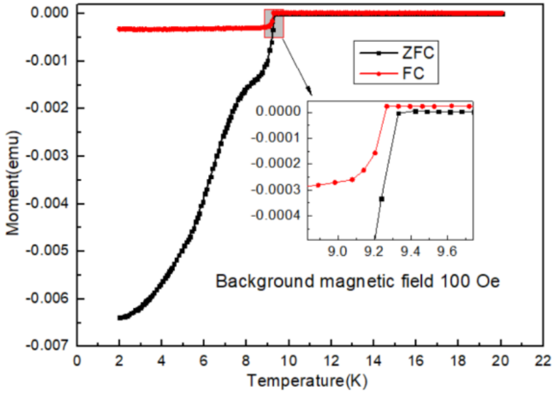
V0-A60-PT100



Tc 9.19K

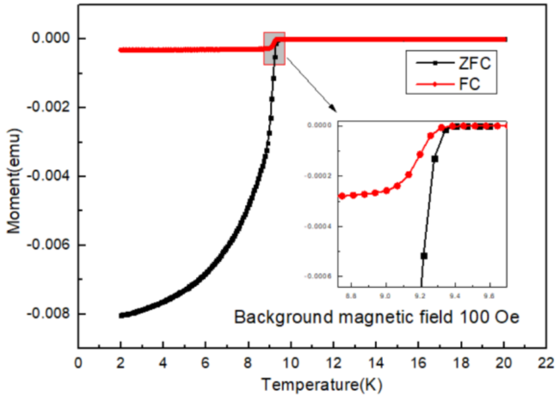
The magnetic response of the FC curve is relatively weak

V50-A150-PT100



Tc 9.32K

V50-A150-PT200



Tc 9.4K

Overall, under a 50V bias voltage, increasing the discharge current and moderately widening the pulse width can improve the microstructure and properties of the Nb film to some extent, but the improvement is still limited.

HiPIMS Nb Film Deposition Experiments and Results

Experiment 2 -100V Bias Voltage and Pulse Width Effects

Experimental Parameters for Nb Film Deposition

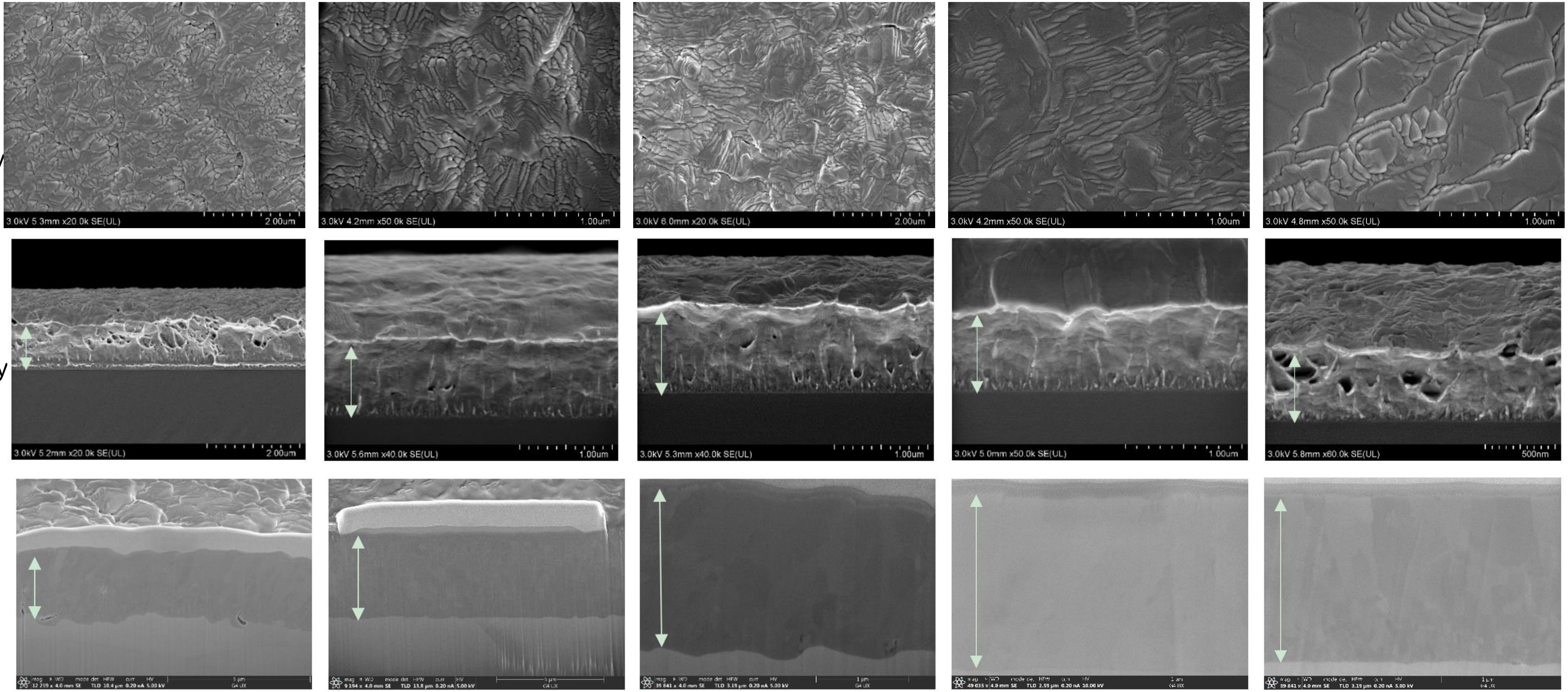
Experiment ID	Average Voltage (V)	Peak Current (A)	Discharge Power (KW)	Pulse Width (us)	Frequency (Hz)	Duty Cycle (%)	Peak Power (KW)	Bias Current Average (A)	Discharge Pressure (Pa)
V100-A30-PT100	-540.8	30	1.8	100	1000	10	18.8	3.8	0.69
V100-A60-PT100	-597.4	60	1.8	100	631	6.3	45.7	7.2	0.69
V100-A60-PT200	-595.3	60	1.8	200	284	5.7	55.1	5.1	0.69
V100-A150-PT30	-657.7	150	1.8	30	2607	7.8	49.8	4.4	0.69
V100-A150-PT100	-635.3	150	1.8	100	269	2.7	104.8	10.7	0.69

Characterization Results of Nb Film

Experiment ID	Nb(110) Peak Position (°)	Nb(110) Crystal Phase Ratio (%)	Lattice Constant (Å)	Stress (MPa)	Nb Film Thickness (µm)	Surface Roughness (nm)	Tc (K)	$H(M_{max})$ (Oe)
V100-A30-PT100	38.4	89.7	3.3096	-34.4	3	36.4	-	-
V100-A60-PT100	38.4	90.4	3.3100	-38.9	2.7	15.7	9.33	3600
V100-A60-PT200	38.7	88.4	3.2842	255.9	2.2	32.4	9.34	2100
V100-A150-PT30	38.4	85.8	3.3097	-35.1	2.2	13.9	9.34	1350
V100-A150-PT100	38.7	89.1	3.2885	206.5	1.8	22.4	9.35	1600

HiPIMS Nb Film Deposition Experiments and Results

Experiment 2 -100V Bias Voltage and Pulse Width Effects



V100-A30-PT100 | V100-A60-PT100 | V100-A60-PT200 | V100-A150-PT30 | V100-A150-PT100

SEM surface morphology

Cross-sectional SEM morphology

Cross-sectional structure by FIB-SEM

Columnar, porous structure

Dense

Dense

Dense

Dense

HiPIMS Nb Film Deposition Experiments and Results

Experiment 2 -100V Bias Voltage and Pulse Width Effects

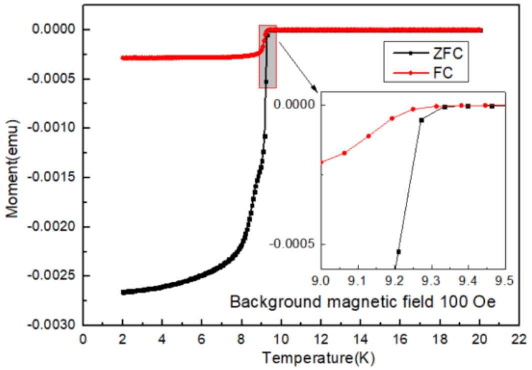
V100-A60-PT100

V100-A60-PT200

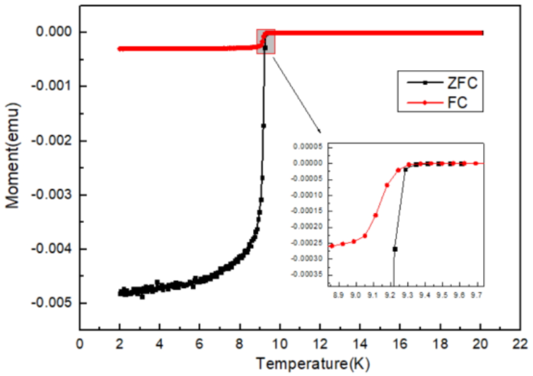
V100-A150-PT30

V100-A150-PT100

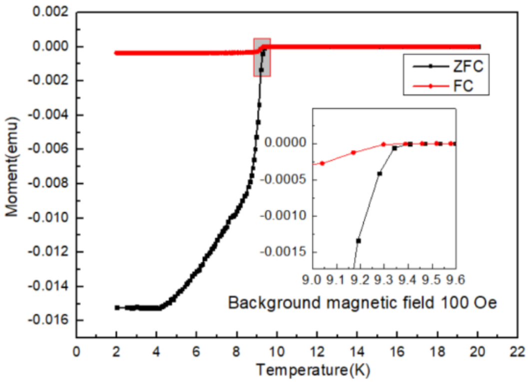
M-T measurement curve



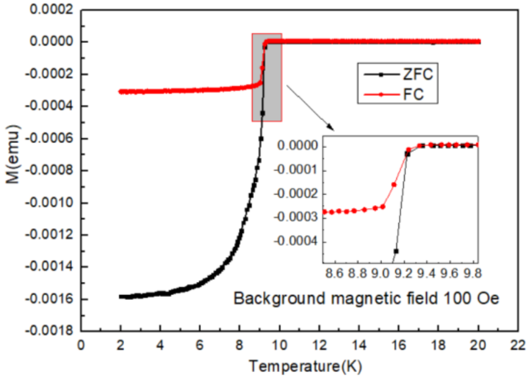
Tc 9.33K



Tc 9.34K

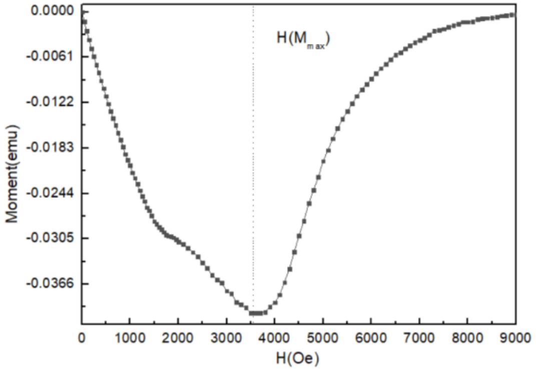


Tc 9.34K

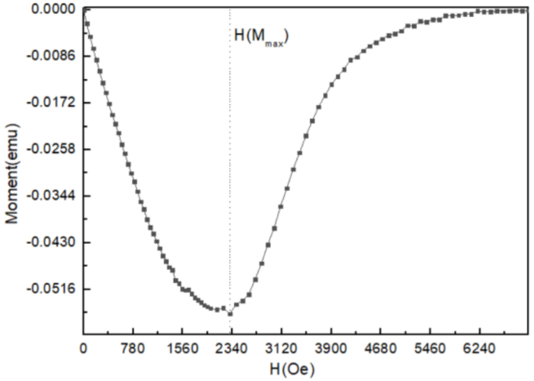


Tc 9.35K

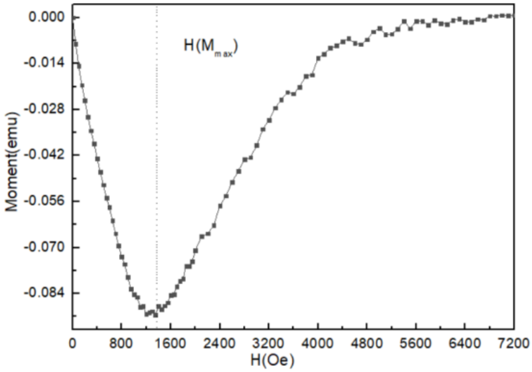
M-H measurement curve



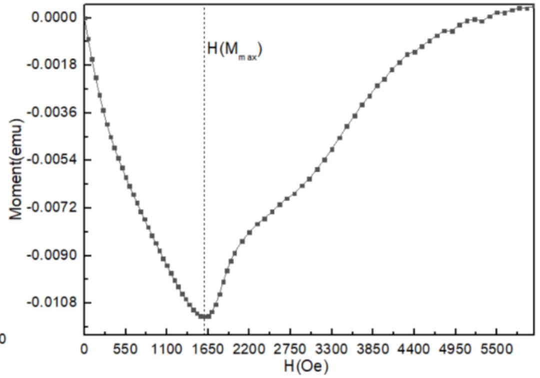
H(M_{max})=3600 Oe



H(M_{max})=2100 Oe



H(M_{max})=1350 Oe



H(M_{max})=1600 Oe

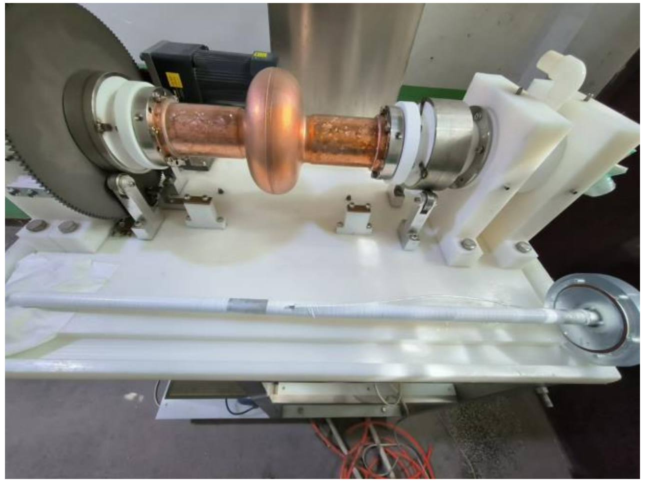
In summary, the Nb film under the conditions of V100-A60-PT100 exhibits the highest H(M_{max}) value (3600 Oe), which is closely related to its dense structure, low internal stress, and reduced surface roughness.

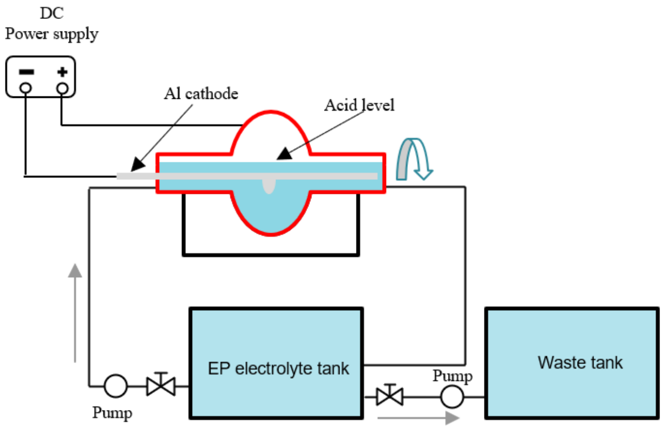
Summary and Conclusion

- Nb/Cu dummy cavities have already been coated with encouraging results.
- HiPIMS with bias voltage can effectively improve the quality of Nb film.
- For obtaining Nb films with excellent surface densities, few internal defects, and fine superconductivity in different regions of the cavity along the axial direction, it is desirable to conduct Cu cavity coating with -100 V bias voltage, 100 μs pulse, and 60 A peak current.

Future plans

- To conduct 1.3GHz copper cavity electro-polishing (EP) experiment, followed by the deposition of Nb film via HiPIMS





- HiPIMS coating (NbN/Cu、NbN-Nb/Cu、Nb3Sn/Cu)