# Characterization of Heated and Room Temperature Dual Off-Axis and Confocal Sputtering Techniques for Thin-Films in AJA International Quantum Materials Deposition Systems



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**Abstract:**

As industry interest in quantum device fabrication grows, the Cornell Nanoscale Facility (CNF) has expanded its thin-film deposition capabilities with two AJA International sputtering systems: AJA Q and AJA Q2 [1]. This report primarily explores Q2, which features confocal sputtering (Nb and Ti targets) and a specialized Dual Off-Axis (DOA) deposition mode (Ta targets) [See Figure 1]. DOA utilizes two opposing RF plasma sources to deposit heavy atoms at lower-energy collisions, with added capability for in-chamber IR heating, up to 800°C, to promote the formation of thermodynamically stable alpha-tantalum (α-Ta) [See Figures 2 and 4]. α-Ta —where literature defines its range between 15 to 60 μΩ·cm [2] — can also form at room temperature when a 5nm Nb seed layer is deposited before the tantalum deposition, provided chamber pressure (P) approaches 3 mTorr from the right. The DOA adjustability (x) allows the plasma sources to move in and out of the deposition chamber, starting arbitrarily outside the chamber and increasing the distance from that point to 150mm. Both sources are mirrored from one another; thus, their 0mm origins are respective. In this study, confocal targets were tested at five pressures (3, 7, 10, 15, and 20 mTorr), while DOA targets were tested at three pressures (3, 7, and 20 mTorr) and three source distances (50, 75, and 100mm). Heated DOA depositions were performed at x=75, P=3, at 350°C, 450°C, 600°C, 700°C, 750°C, and 800°C, in addition to x=100, P=3 and P=7, at 750°C. Characterization was conducted using a variety of CNF tools: P-7 Profilometer for average film thickness, FleXus Film Stress Measurement for wafer stress, and Filmetrics R50 for resistivity. Ultimately, the data collected from these characterizations illustrate the effectiveness of AJA Q2, while highlighting its potential for generating α-Ta crystal structure thin-films for superconducting electronics with observable quantum properties.

**Summary of Research:**

To characterize heated and Nb seed layer depositions for α-Ta formation, deposition rate (DR), average thickness, resistivity (ρ), and film stress (σ) were measured for all chamber targets. Due to elemental property differences, deposition times varied: Ta (1000 s) and Ti (1500 s) from previous AJA sputter tool data, and Nb (1800 s) estimated from previous Mo deposition data.

Substrates were MOS-cleaned and BOE-etched before deposition. Three wafers were masked with photoresist, diced into witness samples, used one at a time for each substrate, and measured at a 6-point average for film thickness via profilometry following lift-off. DR was calculated as thickness/time, and σ from FleXus film stress measurements [See Figure 3 for the master dataset].

**Deposition Rates (DR, nm/s):**

- Ti: Decreased with pressure: 0.046 (P=3 mTorr) → 0.022 (P=20 mTorr).
- Nb: Increased with pressure: 0.090 (P=3) → 0.120 (P=20).
- Ta: Increased with both DOA distance (x) and pressure:
  - x = 50 mm: 0.080 (P=3) → 0.210 (P=20)
  - x = 75 mm: 0.180 (P=3) → 0.250 (P=20)
  - x = 100 mm: 0.190 (P=3) → 0.300 (P=20)

**Stress (σ, MPa):**

- Ti: Transitioned from compressive at low P to tensile at mid P, returning to compressive at high P:
  - P=3: −570; P=10: +80; P=20: −250
- Nb: Low residual stress at mid P, more compressive at high P:
  - P=3: +110; P=10: −10; P=15: −4; P=20: −130
- Ta: Higher x and P reduced compression, often neutralizing σ:
  - P=3: x=50: −300; x=75: −100; x=100: −900 ± 100
  - P=7: x=50: −130; x=75: −50; x=100: +20
  - P=20: x=50: −60; x=75: −6; x=100: −10

**Resistivity (ρ, μΩ·cm):**

- Ti: Stable (170–320) for P=3–15, spiking at P=20 (590).
- Nb: ~230 for P=7–20, with large variance at P=3 (18) and P=7 (710).
- Ta: Higher x reduced ρ, while higher P increased it:
  - P=3: x=50: 600; x=75: 300; x=100: 210
  - P=7: x=50: 1400; x=75: 660; x=100: 130
  - P=20: x=50: 3200; x=75: 2000; x=100: 700

**Heated Ta Depositions:**
Room-temperature data identified x=75 mm (P=3) and x=100 mm (P=3, P=7) as optimal for low ρ. At x=75, P=3, σ averaged −410 MPa and ρ=310 μΩ·cm before 750 °C, improving to σ≈−50 and ρ≈31 μΩ·cm at 750–800 °C, within the α-Ta range. At x=100, P=3, σ=−580 and ρ=110 at 750 °C; at P=7, σ=−350 and ρ=60. Both x=100 substrates exhibited frosted silicide formations that hindered α-Ta phase transitions due to their higher energies.

**Nb Seed Layer Depositions:**

While standalone Nb depositions showed that increasing P raised ρ, Nb seed layer effects could differ when followed by Ta deposition. To characterize this interaction, Nb was deposited first confocally while Ta was deposited at its optimal DOA distance (x=75 mm) across different pressures. Deposition times were calculated as t = 5nm/DR using the respective DR values for each P value. At P=3, ρ was low (57 μΩ·cm) with σ=−390 MPa (compressive). At P=7, ρ increased to 88 μΩ·cm with σ=−87 MPa (low residual stress). At P=20, ρ sharply increased to 2250 μΩ·cm while σ remained low at −47 MPa. Overall, ρ rose steeply with pressure, while σ trended toward neutral values.

**Conclusions and Future Steps:**

Characterization of Ti, Nb, and Ta targets established that optimal conditions for forming **α-Ta** with low resistivity and neutral stress, critical for quantum electronics, require low-pressure (3–7 mTorr) deposition at a specific distance (75–100 mm). While heating to 750–800°C promoted these properties for Ta, close deposition distances risked silicide formation, which could be mitigated by lowering the temperature at optimal distances like 87.5 mm. Seed layer experiments revealed that although stress remained low across pressures, resistivity increased sharply at high pressures, indicating potential electrical degradation. Consequently, seed layers for Nb may be more favorable under low-pressure, unheated conditions. These preliminary results confirm that a combination of low chamber pressure and optimized deposition distance is key to producing desirable α-Ta films, though further data repetition may be necessary to minimize variability.

# References:

[1] Hideki Tomoshige. (2024). *Innovation Lightbulb: Private Investment in Quantum Technology*. Csis.org. https://www.csis.org/analysis/innovation-lightbulb-private-investment-quantum-technology

[2] Myers, S., Lin, J., Souza, R. M., Sproul, W. D., & Moore, J. J. (2013). The β to α phase transition of tantalum coatings deposited by modulated pulsed power magnetron sputtering. *Surface and Coatings Technology*, *214*, 3845. https://doi.org/10.1016/j.surfcoat.2012.10.06

[3] **(FIGURE 2)** Choi, J., Seong, J., Park, S., Kim, H., Kim, S., Kim, K. H., & Hong, J. (2023). Effect of the Working Pressure and Oxygen Gas Flow Rate on the Fabrication of Single-Phase $Ag_2O$ Thin Films. *Coatings*, *13*(6), 1061. https://doi.org/10.3390/coatings13061061

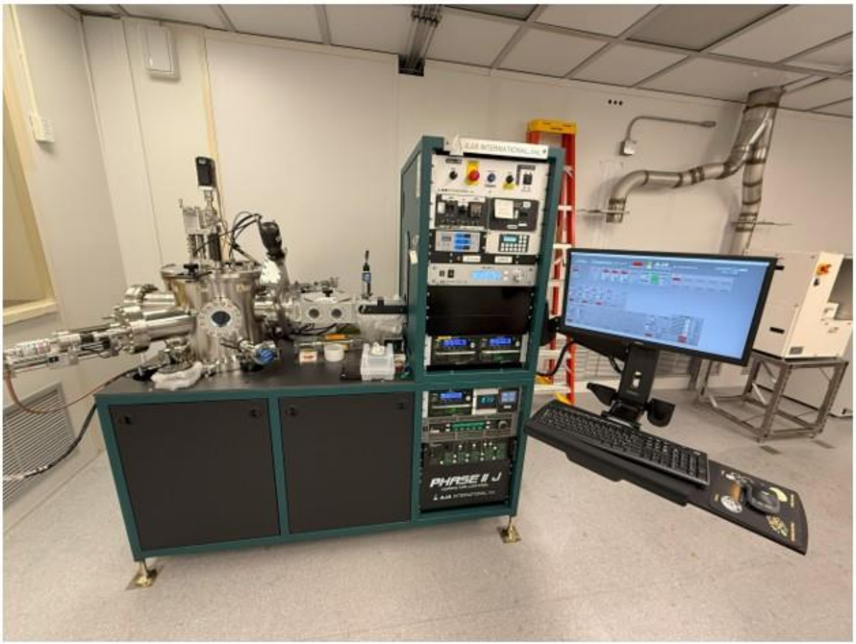


*Figure 1 - AJA International Q2 Deposition System*

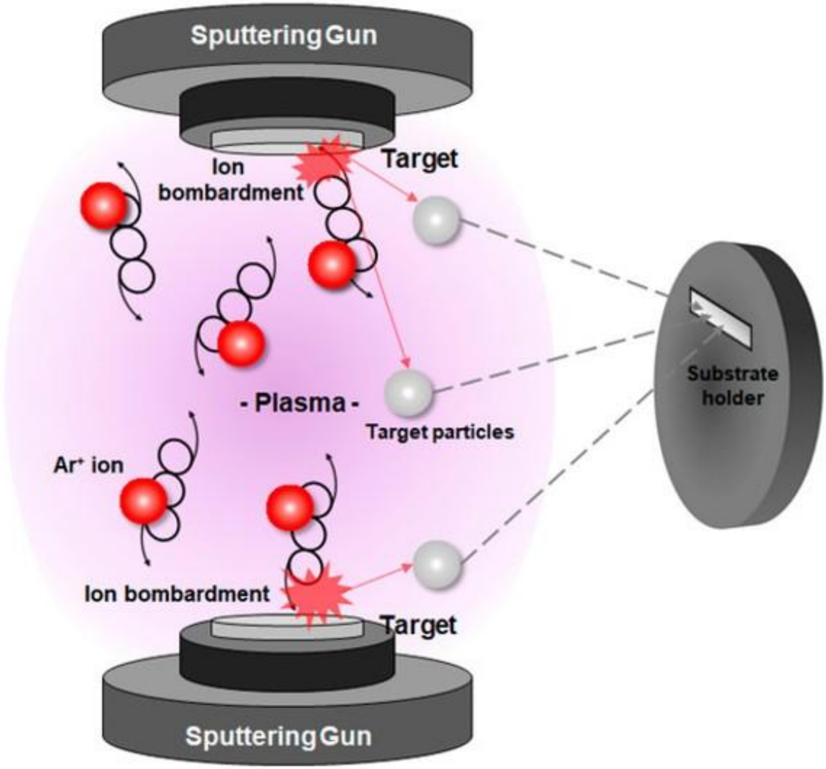


*Figure 2 - Diagram of Dual Off-Axis Sputtering*

| MATERIAL | Wafer Serial # | DOA Distance (mm) | Temp (°C) | Pressure (mTorr) | Sputter Duration (s) | Thickness (nm) | Deposition Rate (nm/s) | Film Stress (MPa) | Resistivity (uOhms·cm) |
|---|---|---|---|---|---|---|---|---|---|
| Ti | #0337 | Confocal | RT | 3 | 1500 s | 70 | 0.046 | -565 | 190 |
| | #0333 | | | 7 | | 74 | 0.049 | -305 | 316 |
| | #0195 | | | 10 | | 55 | 0.037 | 80.2 | 170 |
| | #0196 | | | 15 | | 42 | 0.028 | 0.611 | 206 |
| | #0087 | | | 20 | | 33 | 0.022 | -250 | 590 |
| Nb | #0150 | | | 3 | 1800 s | 175 | 0.097 | 110 | 18 |
| | #0184 | | | 7 | | 150 | 0.083 | -13.6 | 708 |
| | #0185 | | | 10 | | 201 | 0.112 | -9.45 | 231 |
| | #0188 | | | 15 | | 174 | 0.097 | -4.01 | 248 |
| | #0180 | | | 20 | | 214 | 0.119 | -133 | 199 |
| Ta | #0245 | 50 mm | | 3 | 1000 s | 80 | 0.080 | -273 | 632 |
| | #0141 | | | 7 | | 120 | 0.12 | -129 | 1440 |
| | #0332 | | | 20 | | 210 | 0.21 | -63.1 | 3150 |
| | #0116 | 75 mm | | 1.5 | | 111 | 0.111 | -400 | 241 |
| | #0189 | | | 3 | | 180 | 0.18 | -131 | 324 |
| | #0238 | | | 7 | | 190 | 0.19 | -51.4 | 665 |
| | #0241 | | | 20 | | 250 | 0.25 | -6 | 2049 |
| | #0310 & #0311 Averaged | | 350 | 3 mTorr | | 184 nm | 0.184 nm/s | -561 | 340 |
| | #0317 | | 450 | | | | | -153 | 333 |
| | #0315 | | 600 | | | | | -441 | 297 |
| | #0112 | | 700 | | | | | -465 | 280 |
| | #0318 | | 750 | | | | | 107 | 30 |
| | #0123 | | 800 | | | | | -583 | 31 |
| 5nm Nb Seed | #0320 | | RT | 3 | 51s, 1000s Ta | 1425 | 0.097 | -393 | 57.2 |
| | #0287 | | | 7 | 60s, 1000s Ta | 1625 | 0.083 | -87 | 88 |
| | #0284 | | | 20 | 45s, 1000s Ta | 2657 | 0.119 | -46.7 | 2245 |
| Ta | #0288 | 100 mm | | 3 | 1000 s | 1700 | 1.70 | -982 | 180 |
| | #0309 | | | 7 | | 1500 | 1.5 | 17.5 | 130 |
| | #0308 | | | 20 | | 3000 | 3.0 | -8.61 | 720 |
| | #0090 & #0092 Averaged | | 750 °C | 3 | | 1700 | 1.7 | -583 | 106 |
| | #0123 | | | 7 | | 1500 | 1.5 | -347 | 59 |

***Figure 3 - Master Dataset***

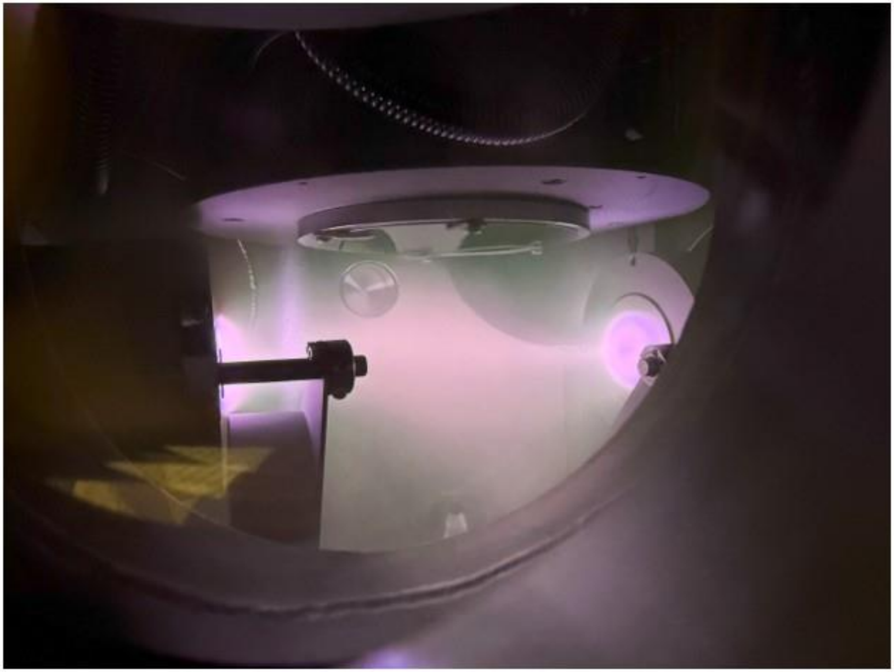

Figure 4 - In-Chamber DOA Plasmas