Advanced Surface Nitriding

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Why Nitride

• Low-temperature process
• No quench requirement
• Minimal distortion
• Resistance to oxidation
• High hardness values
• Same core properties
Basic Theory

• Large voltage frees bound electrons
• Particle acceleration
• Vacuum increase mfp => greater energy
• Ions collide to give off visible light
Initial Design

Initial nitriding chamber design [1].
Problems with Initial Design

• Severe arcing when igniting plasma
• Metallic sputter deposition on all ceramic insulators resulting in electrical shorting
• Sample insolation disc shorting due to sputter deposition
  • Inconsistent nitriding results
• High maintenance due to cleaning ceramic every experiment
Solutions

• Adjustable anode (electrical ground)
• Replaced isolation disc with multiple shielded sample holders
• Added shields to thermocouple feedthrough and stage ceramic stands
Current Experiment Matrix

- Metals Nitrided
  - 316L
  - HT9
  - T91
  - Zircaloy 4
  - Pure Iron

- Gas: N\textsubscript{2}/H\textsubscript{2} (90%/10%)

- Cage: SS316

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<tr>
<th>Pressure (mTorr)</th>
<th>Temperature (°C)</th>
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<td>400</td>
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<tr>
<td>750</td>
<td>1 Hr</td>
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316L Nitride Layer at 375C for 2 Hours

Initial formation of nitride layer [2].
316L:525C, 1Torr Cross Section
Polishing Before Etching

30 mins
1 Hour
2 Hours
316L: 525°C, 1 Torr, 1 Hr

Before and after etching with Marble’s reagent.
316L: 400C, 2 Hours

1 Torr

1.5 Torr
316L: 450C

1 Hour
750 mTorr

2 Hours
1 Torr
316L: 525°C, 1.5 Torr

1 Hour

2 Hour
Diffusion Kinetics: 316L
1.0 Torr
316L: 450°C, 1.5 Torr, 2Hr

Nitride Layer
316L: 450°C, 1.5 Torr, 2 Hr
316L: 525C, 1Torr, 2Hr
Atomic Percentage Vs. Depth
316L: 525C, 1Torr, 2Hr
Hardness Vs. Depth

Hardness (Gpa) vs. Depth (um)
316L: 525C, 1Torr, 2Hr
Reduced Modulus Vs. Depth
T91: 450°C, 1.5 Torr, 2 Hr

Nitride Layer
T91: 450C, 1.5Torr, 2Hr
Atom Percent Vs. Depth

![Graph showing atom percent vs. depth for different elements: N K, Cr K, Fe K.](image-url)
Cathodic Cage Nitriding
Future Work

• Analyze nitrided HT9, Zircaloy4, T91, Pure Iron, Pure Zirconium

• Measure how cage thickness changes Hollow Cathode Effect
Questions?


Glow discharge ion nitriding mechanisms
Hollow Cathode Effect