Advanced Surface Nitriding

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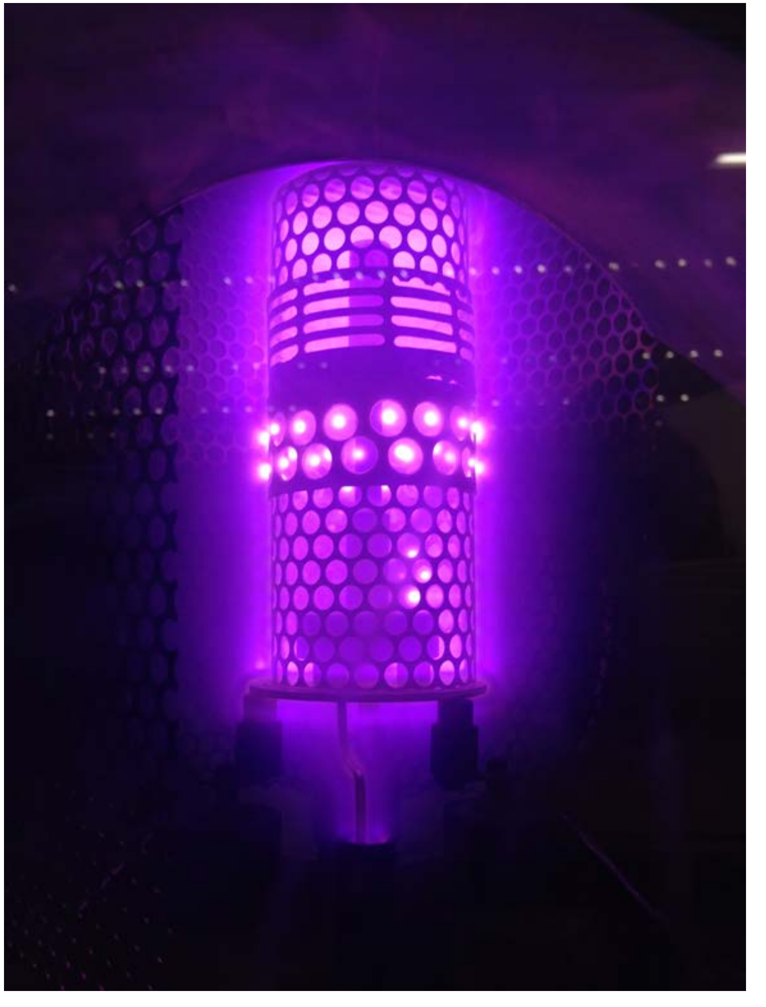
Why Nitride

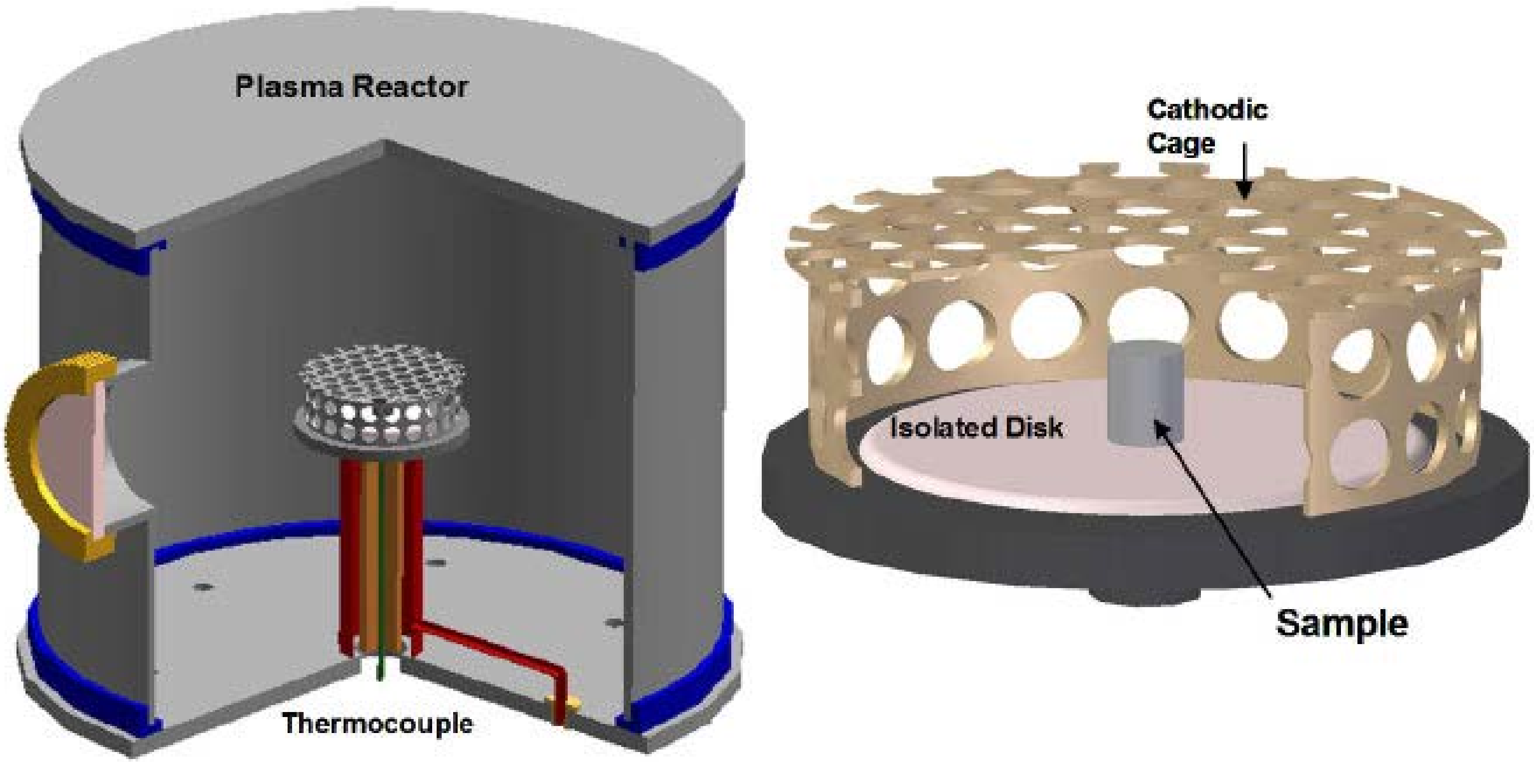
- Low-temperature process
- No quench requirement
- Minimal distortion
- Resistance to oxidation
- High hardness values
- Same core properties

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

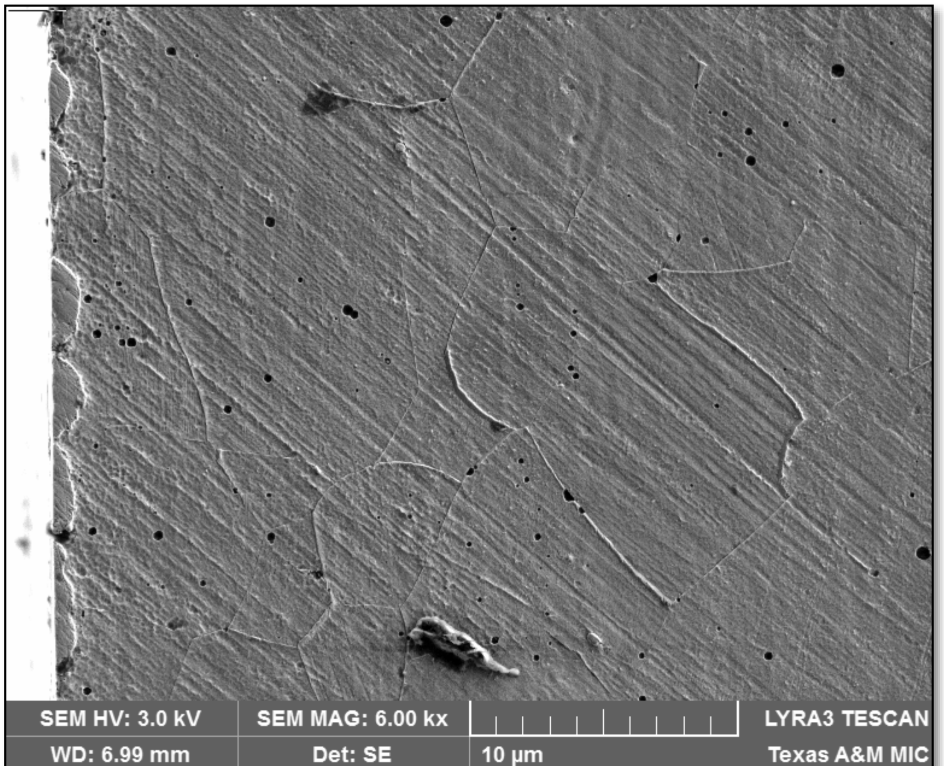
- Adjustable anode (electrical ground)
- Replaced isolation disc with multiple shielded sample holders
- Added shields to thermocouple feedthrough and stage ceramic stands

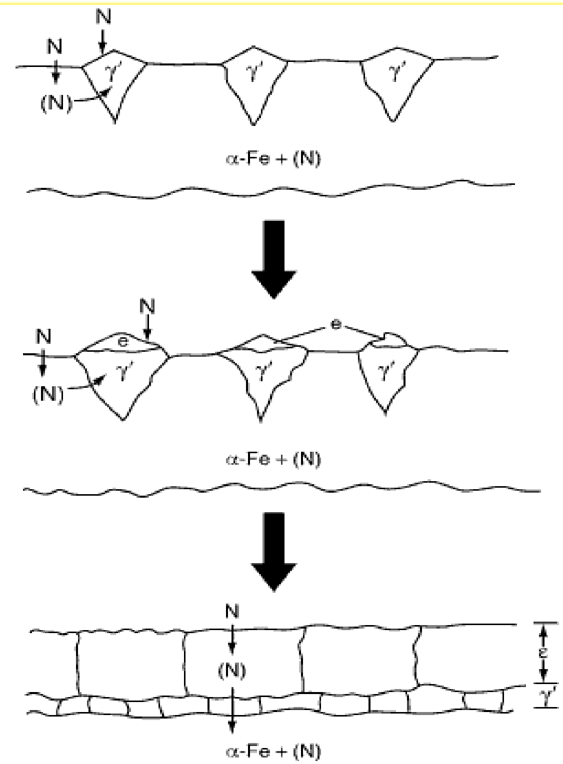
Current Experiment Matrix

- Metals Nitrided
  - 316L
  - HT9
  - T91
  - Zircaloy 4
  - Pure Iron
- Gas: N_2/H_2 (90%/10%)
- Cage: SS316

Pressure (mTorr)	Temperature (°C)		
	400	450	525
750	1 Hr 2 Hr 4 Hr	30 min 1 Hr 2 Hr	30 min 1 Hr 2 Hr
1000	1 Hr 2 Hr 4 Hr	30 min 1 Hr 2 Hr	30 min 1 Hr 2 Hr
1500	1 Hr 2 Hr 4 Hr	30 min 1 Hr 2 Hr	30 min 1 Hr 2 Hr

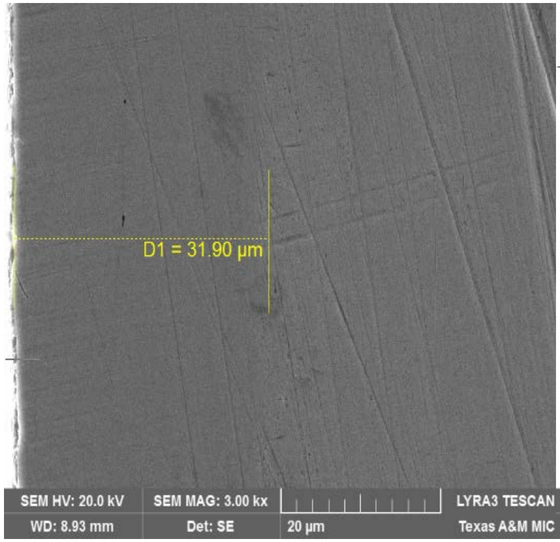
316L Nitride Layer at 375C for 2 Hours

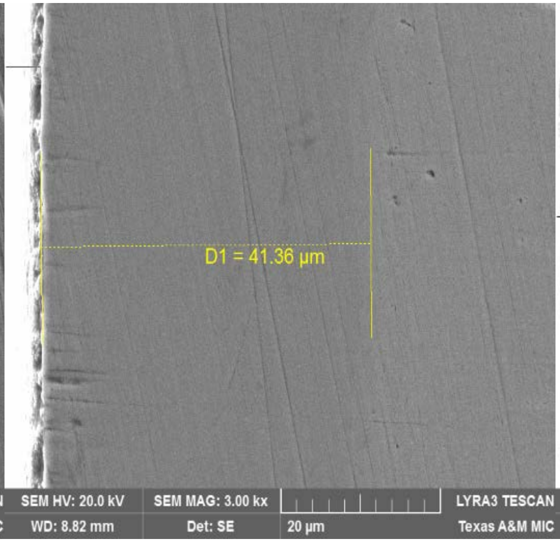


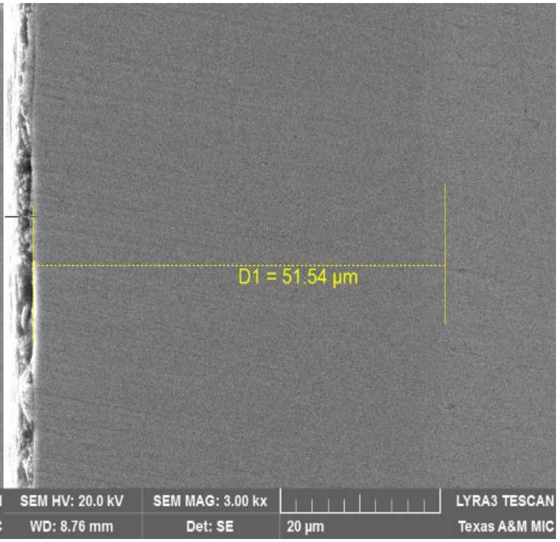


Initial formation of nitride layer [2].

316L:525C, 1Torr Cross Section Polishing Before Etching





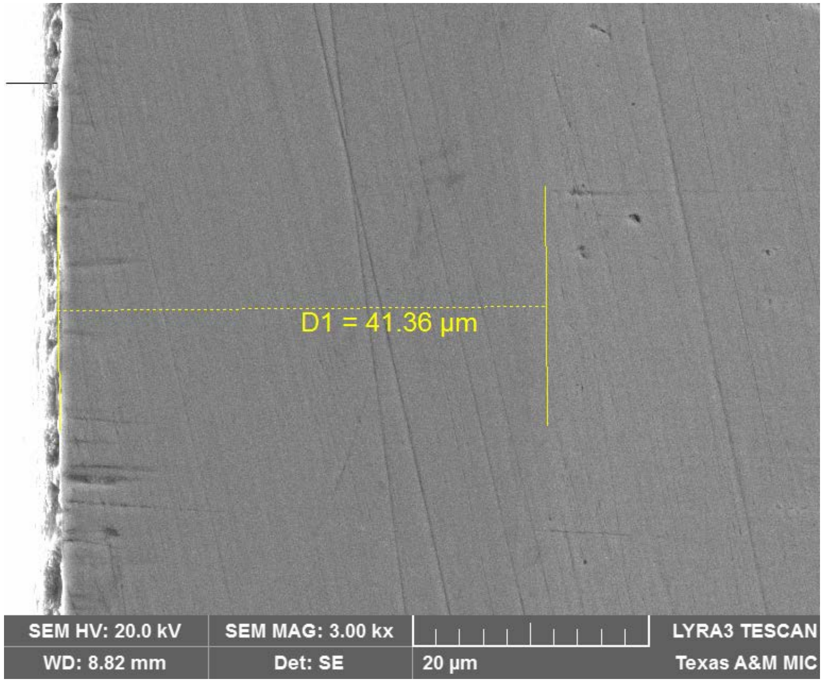


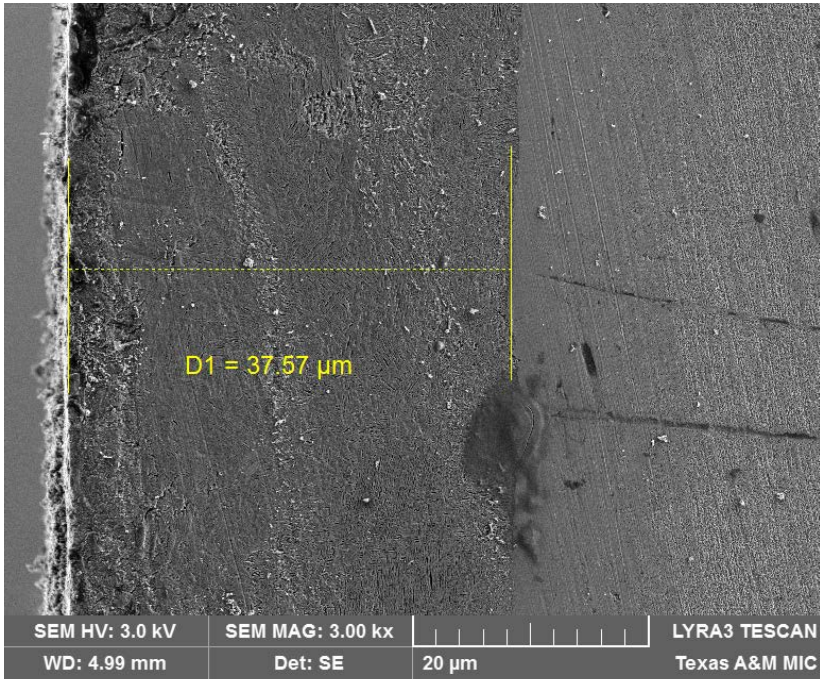
30 mins

1 Hour

2 Hours

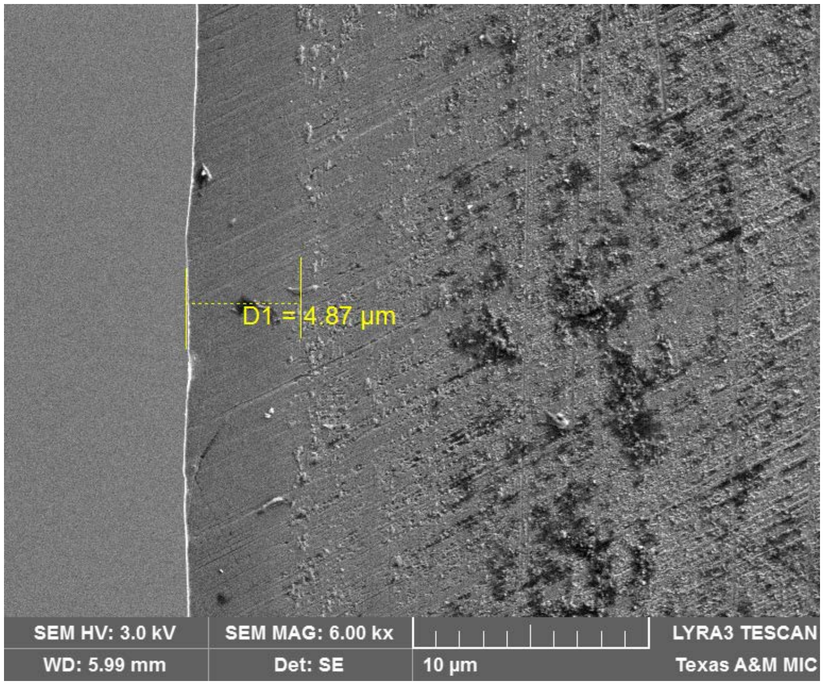
316L: 525C, 1Torr, 1Hr



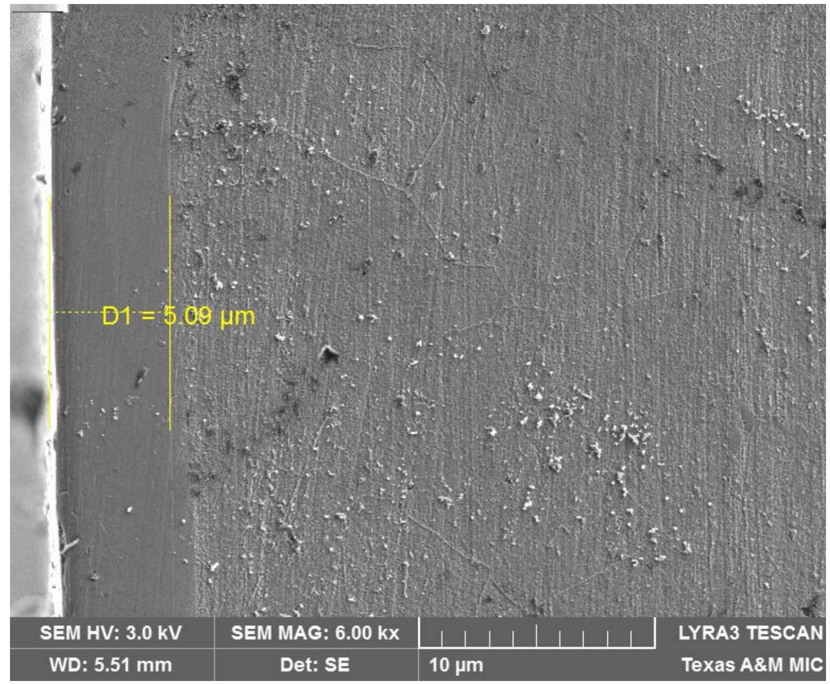


Before and after etching with Marble's reagent.

316L: 400C, 2 Hours

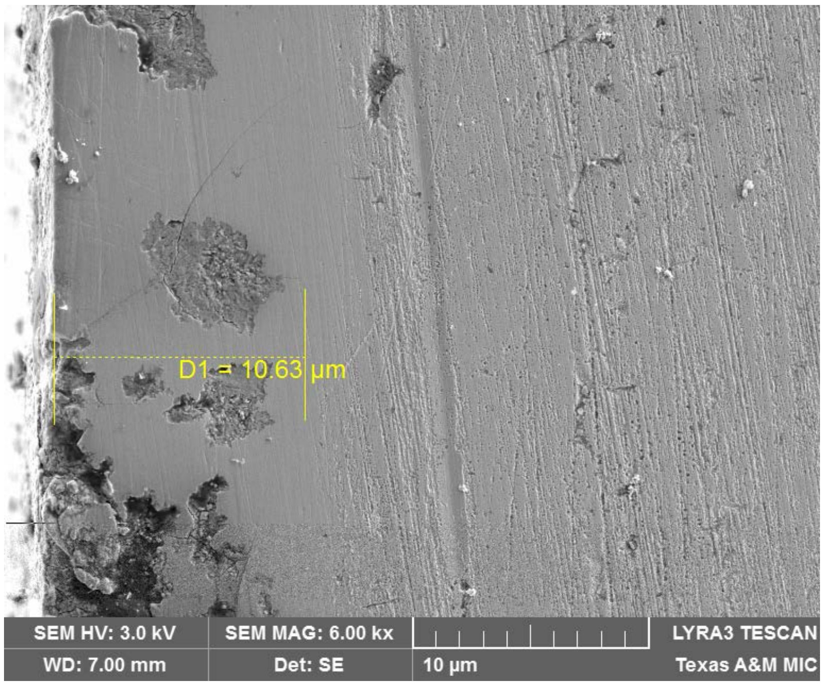


1 Torr

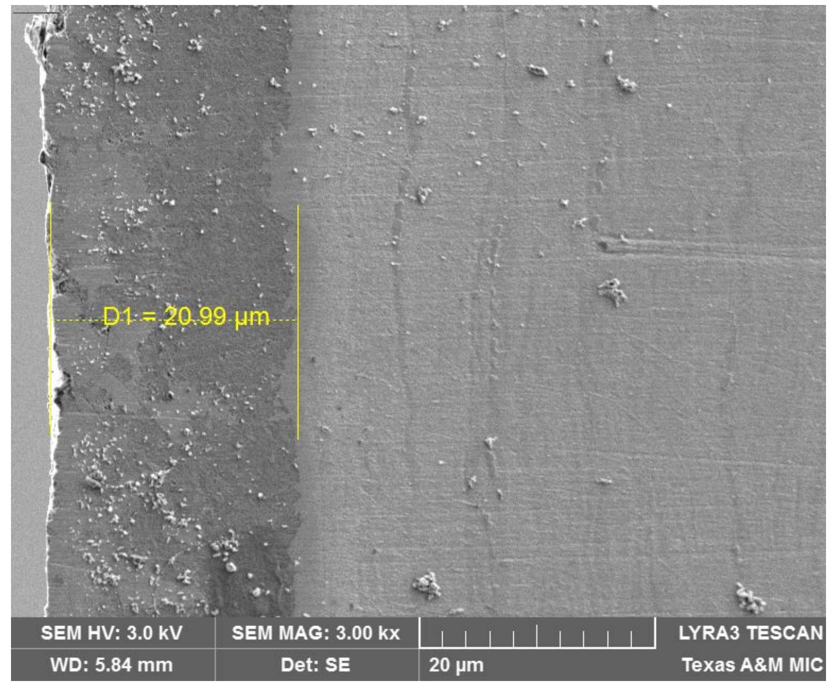


1.5 Torr

316L: 450C

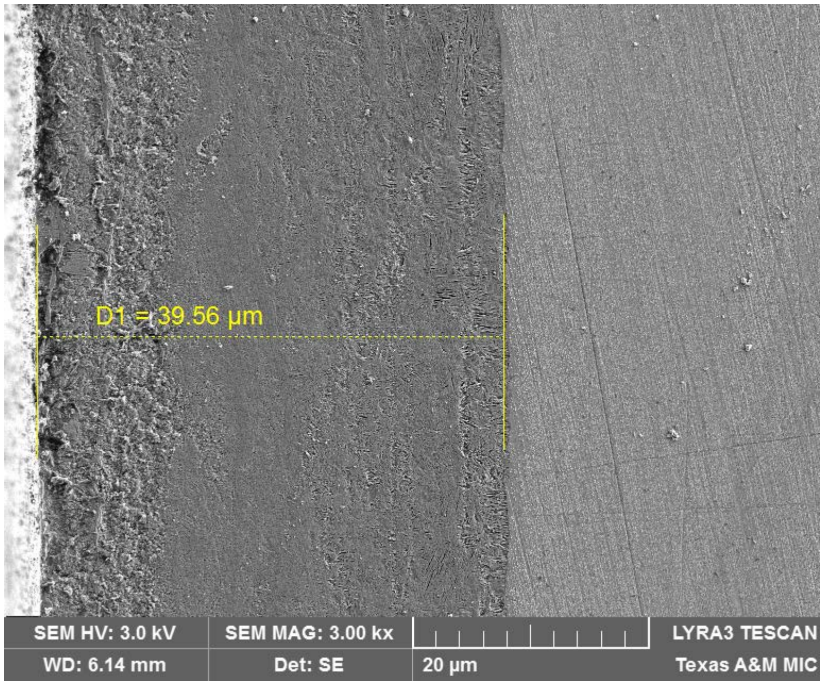


1 Hour
750 mTorr

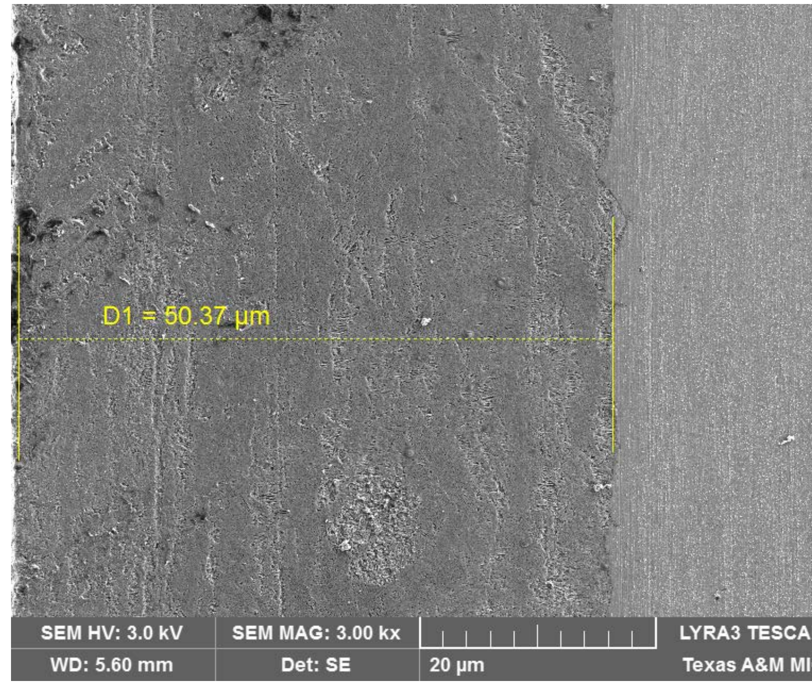


2 Hours
1 Torr

316L: 525C, 1.5 Torr

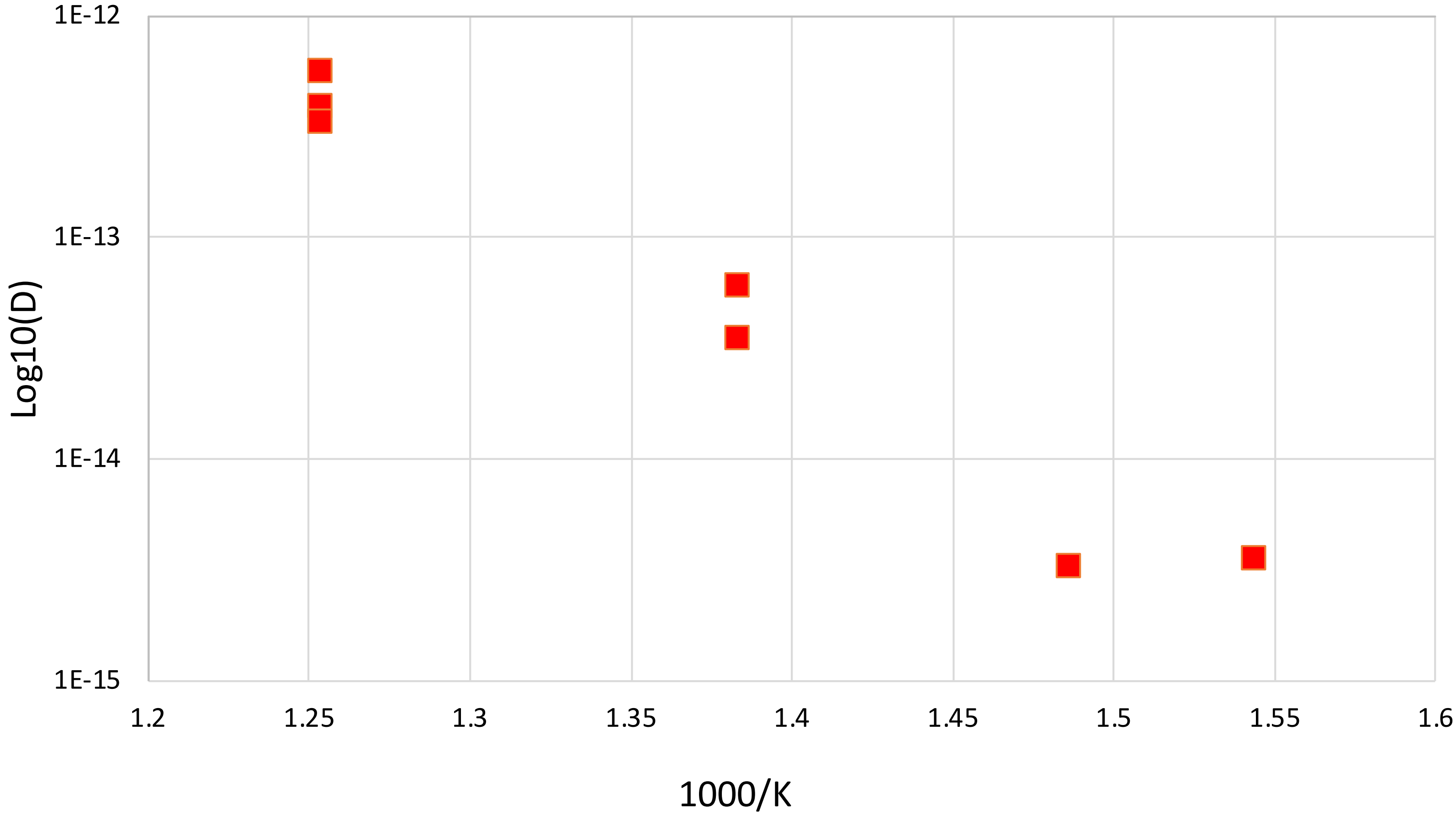


1 Hour

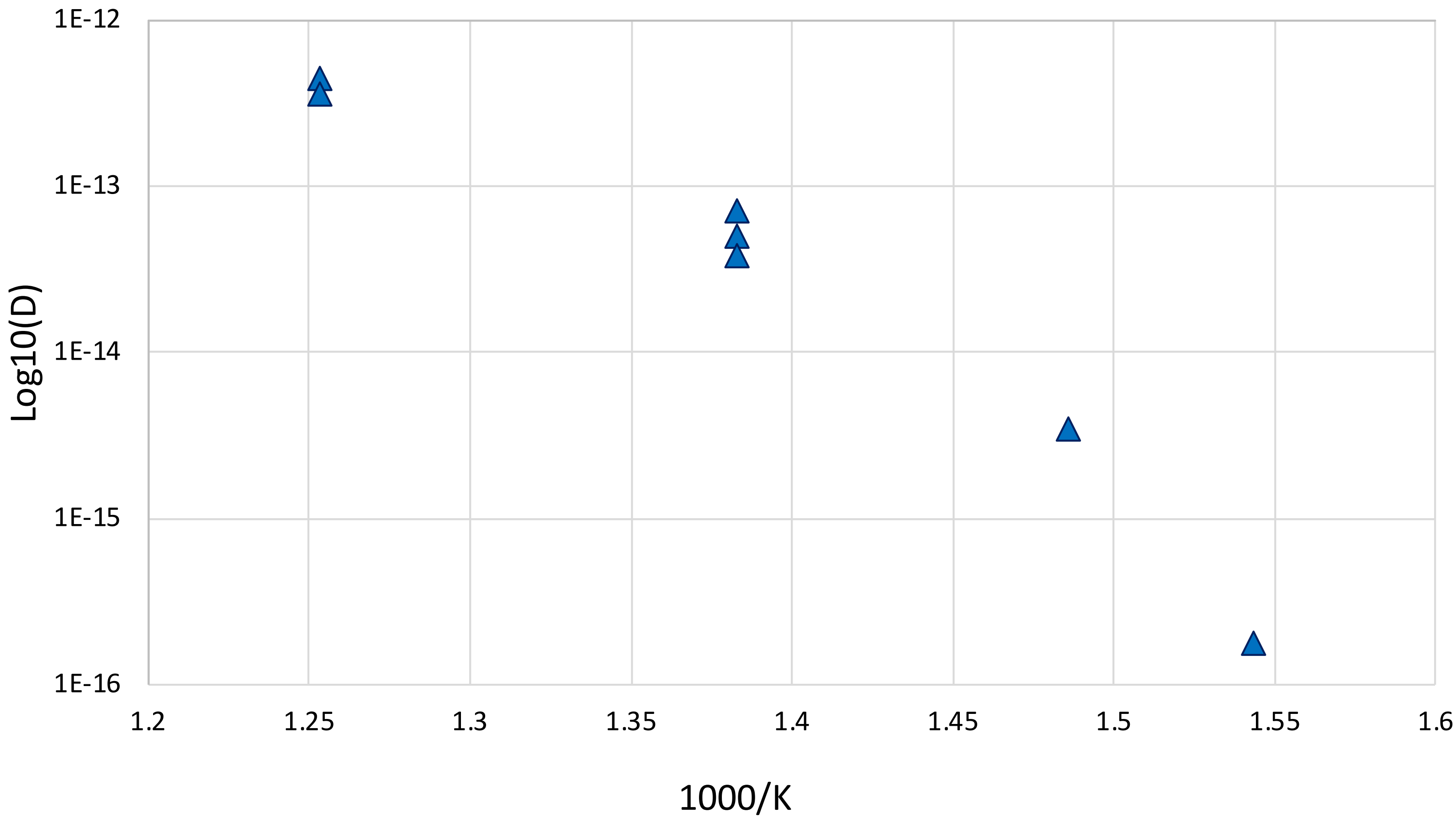


2 Hour

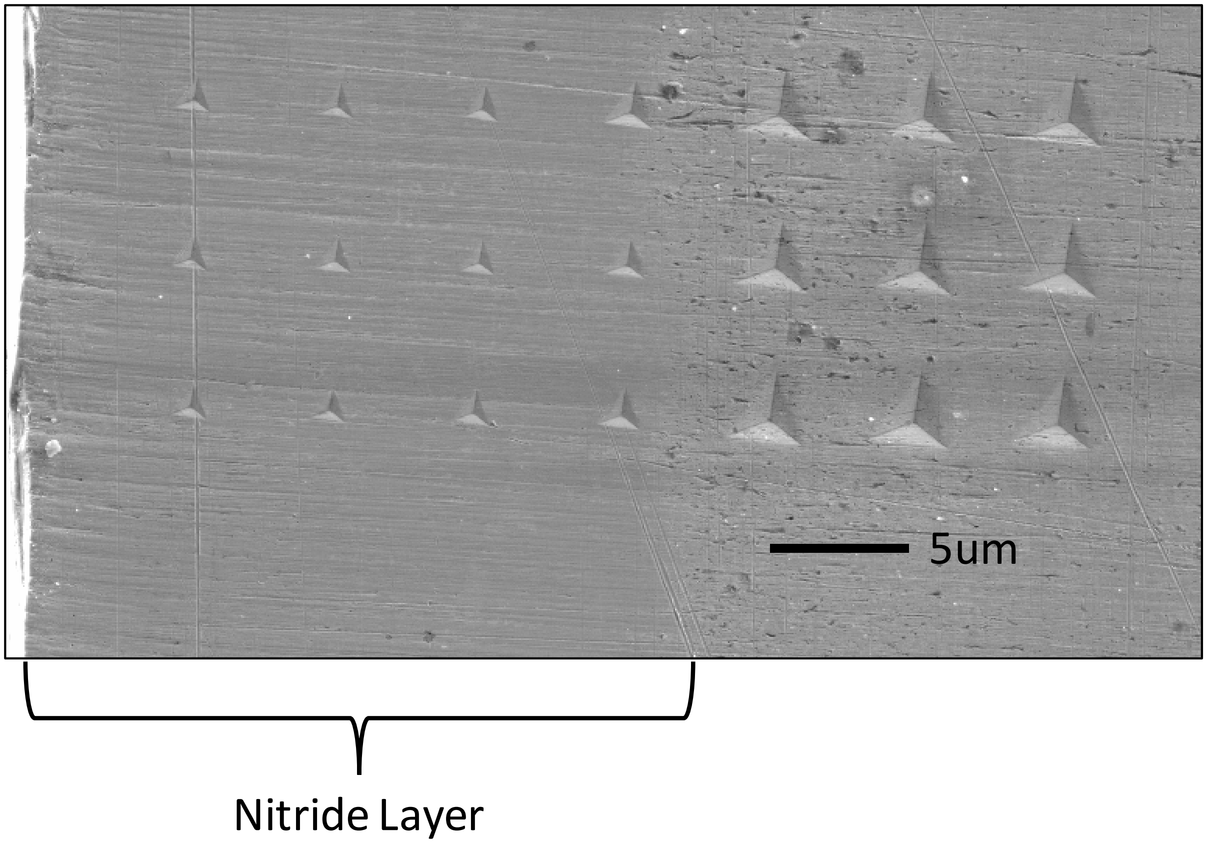
Diffusion Kinetics: 316L
1.0 Torr



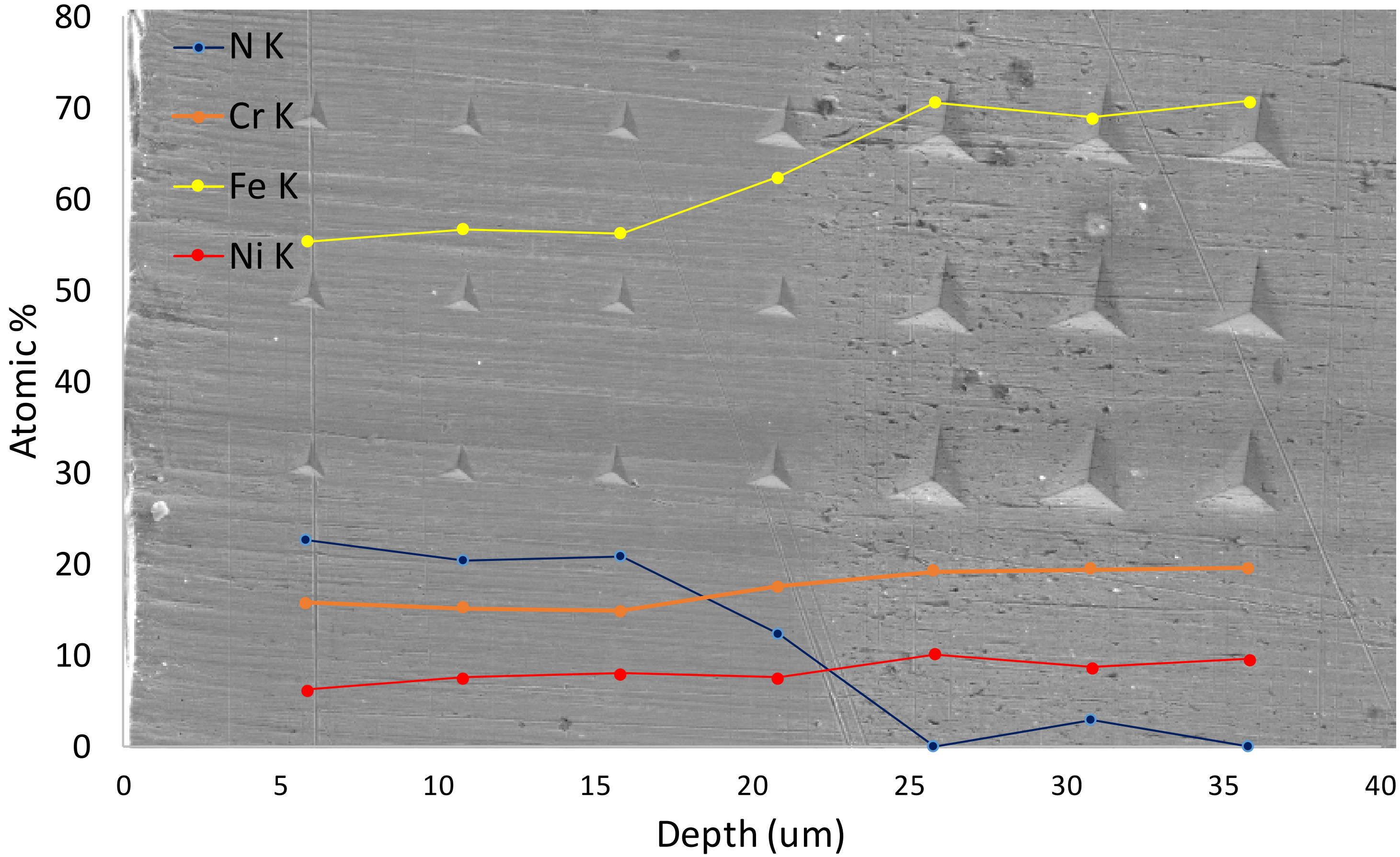
Diffusion Kinetics: 316L
1.5 Torr



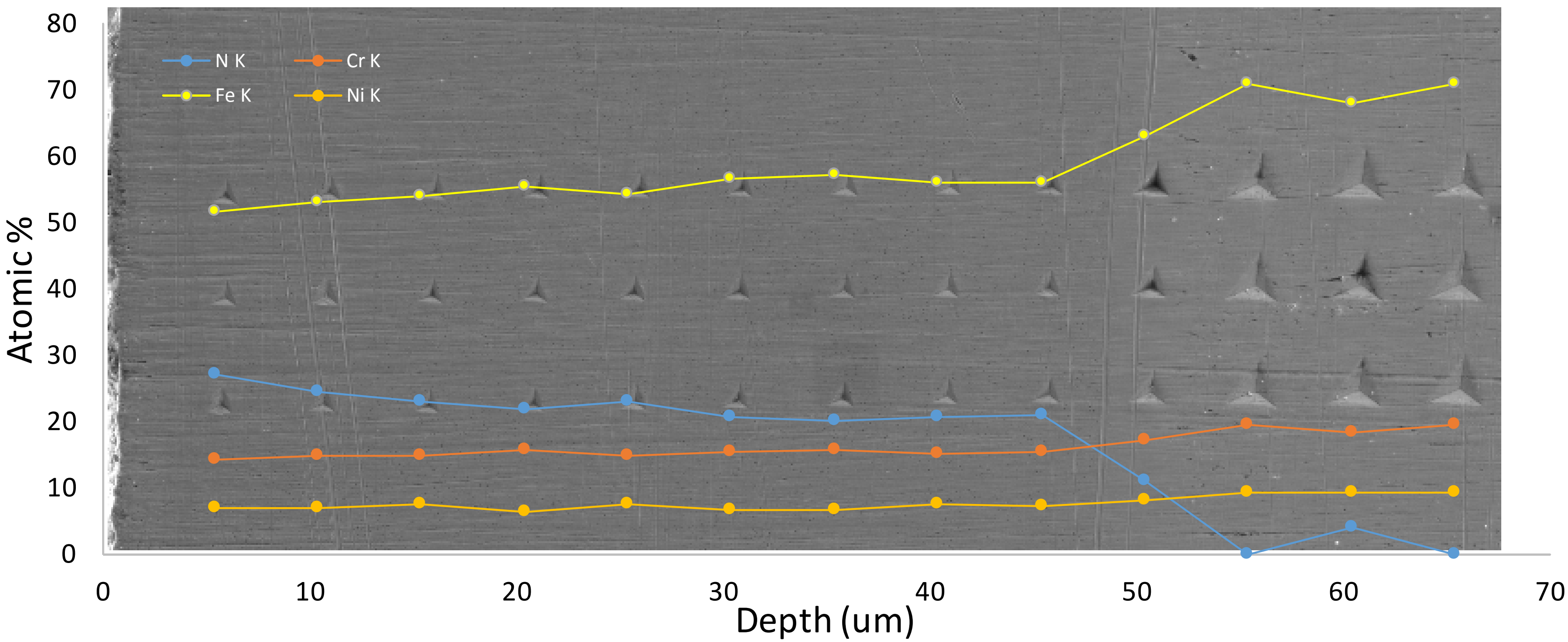
316L: 450C, 1.5Torr, 2Hr



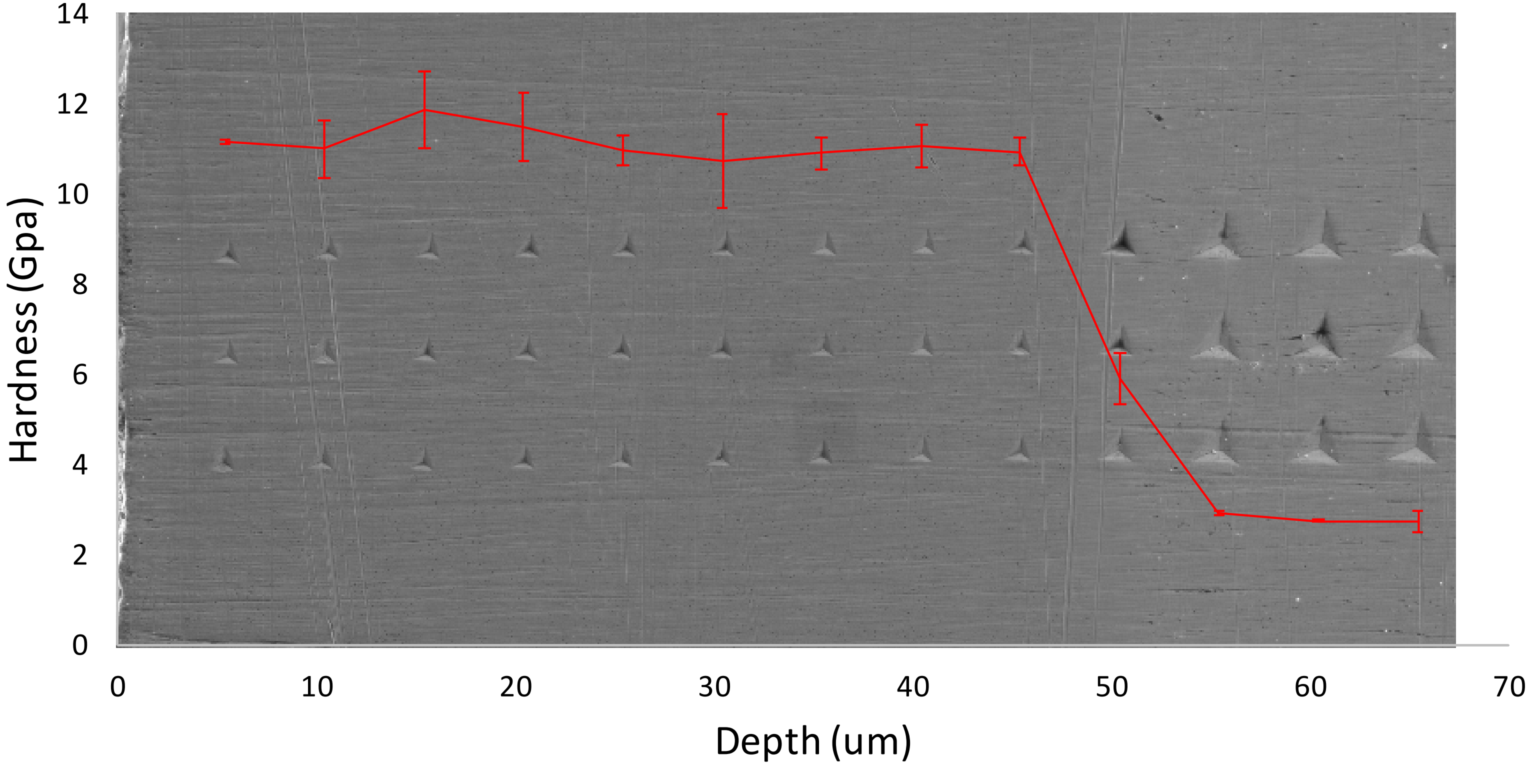
316L: 450C, 1.5Torr, 2Hr



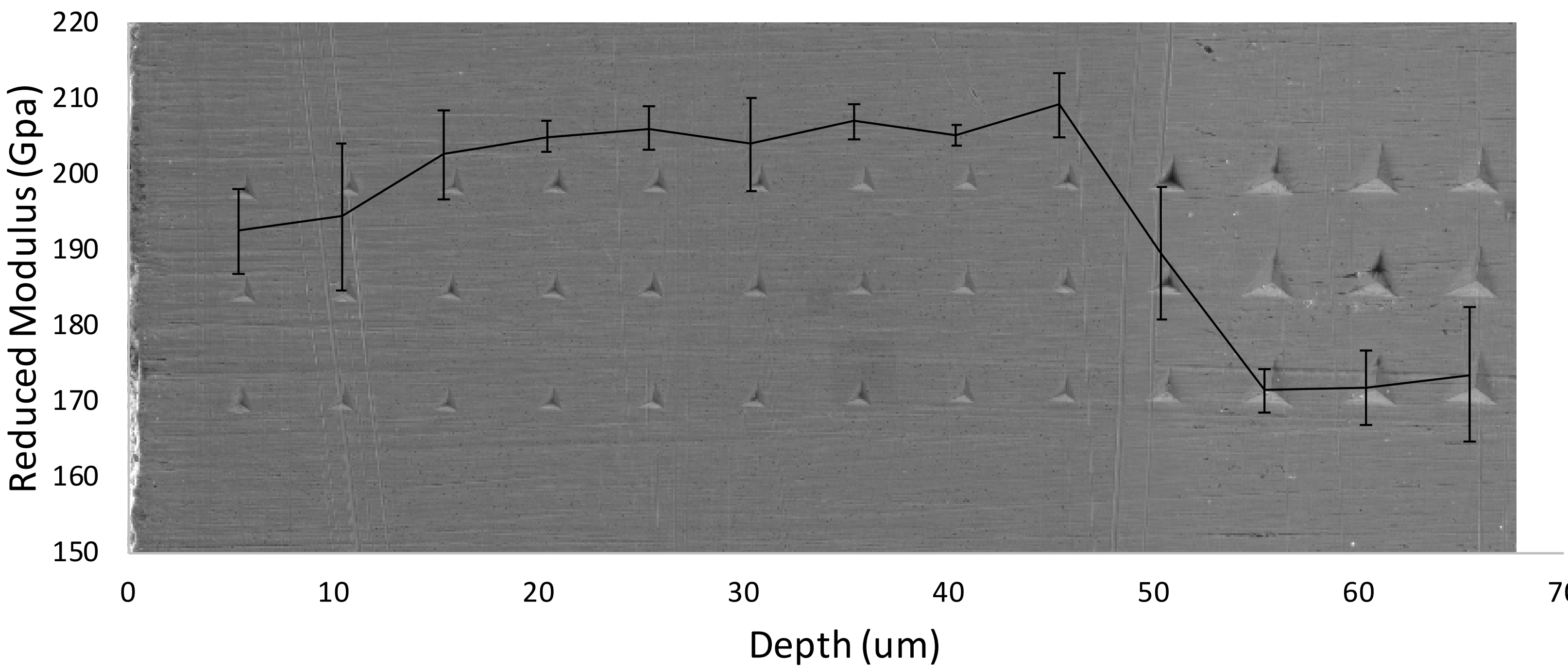
316L: 525C, 1Torr, 2Hr
Atomic Percentage Vs. Depth



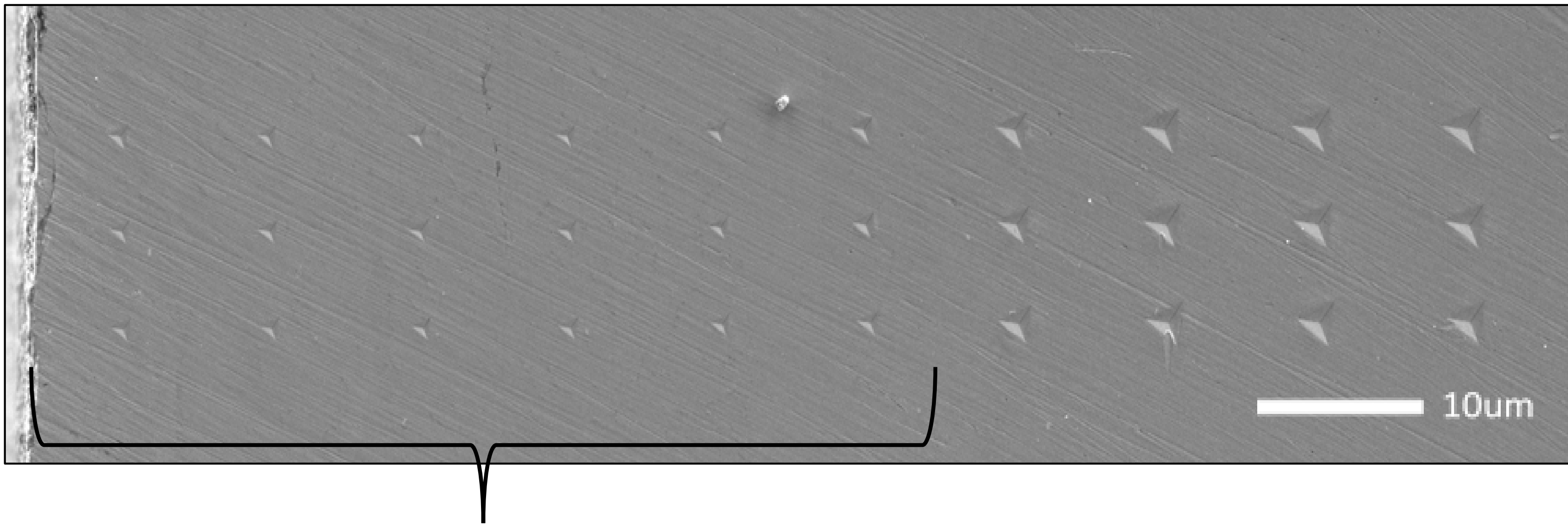
316L: 525C, 1Torr, 2Hr Hardness Vs. Depth



316L: 525C, 1Torr, 2Hr
Reduced Modulus Vs. Depth

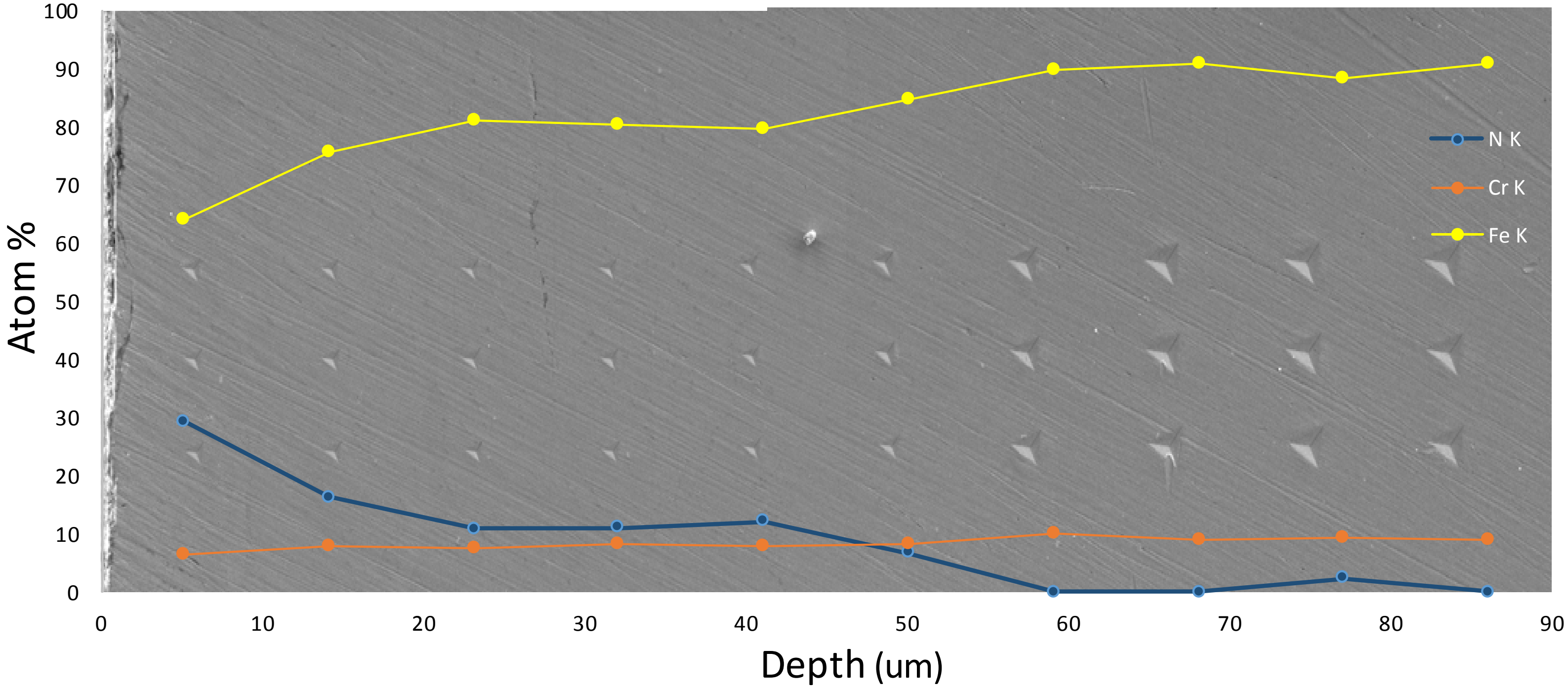


T91: 450C, 1.5Torr, 2Hr



Nitride Layer

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



Cathodic Cage Nitriding

Future Work

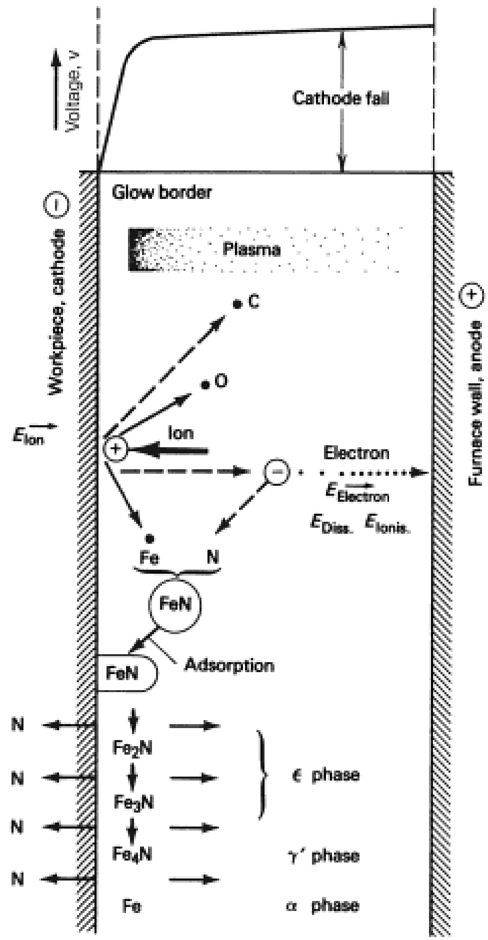
- Analyze nitrided HT9, Zircaloy4, T91, Pure Iron, Pure Zirconium
- Measure how cage thickness changes Hollow Cathode Effect

Questions?

1) R.R.M. de Sousa, et al., *Cathodic cage plasma nitriding of austenitic stainless steel (AISI 316): influence of the working pressure on the nitrided layers properties,* Mater. Res., 17 (2014), pp. 427–433

2) Pye, D., *Practical Nitriding and Ferritic Nitrocarburizing*. 2003, Materials Park, OH: ASM International. 256.

Glow discharge ion nitriding mechanisms



Hollow Cathode Effect