

Project 43-L: Thermodynamics of Refractory Alloys

Semi-annual Fall Meeting October 2021



- Student: Bobby Puerling (Mines)
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This work was funded by the Department of Energy's Kansas City National Security Campus which is operated and managed by Honeywell Federal Manufacturing Technologies, LLC under contract number DE-NA0002839.

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Kenzi



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Project 43-L: Thermodynamics of Refractory Alloys



- Student: Bobby Puerling (Mines)
- Advisor(s): Amy Clarke (Mines), Jonah Klemm-Toole (Mines)

Project Duration
Master's: Jan. 2020 to May 2022

- **Problem:** Gaps exist in the thermodynamic databases where refractory alloys are concerned.
- **Objective:** Compile thermodynamic data for compositions of interest, assess the phase stability of MoNbTa with heat treatments, and characterize microstructures.
- **Benefit:** Improve databases for thermodynamic predictions.

- Recent Progress**
- First heat treatments at 1700 °C are completed and samples are being analyzed
 - First trial heat treatment at 1500 °C using “nesting doll” setup has been performed.
 - Investigating possibility of performing 2000 °C heat treatment at Michigan Tech.

Metrics		
Description	% Complete	Status
1. Literature review	95%	●
2. Comparison of published phase diagrams to Thermo-Calc predicted phase diagrams	100%	●
3. Create heat treatment method utilizing DICTRA and Scheil simulations	100%	●
4. Perform heat treatment, microstructural characterization, and assess phase stability	10%	●
5. Input experimental data into Thermo-Calc and compare new predictions to experimental data	0%	●

Background

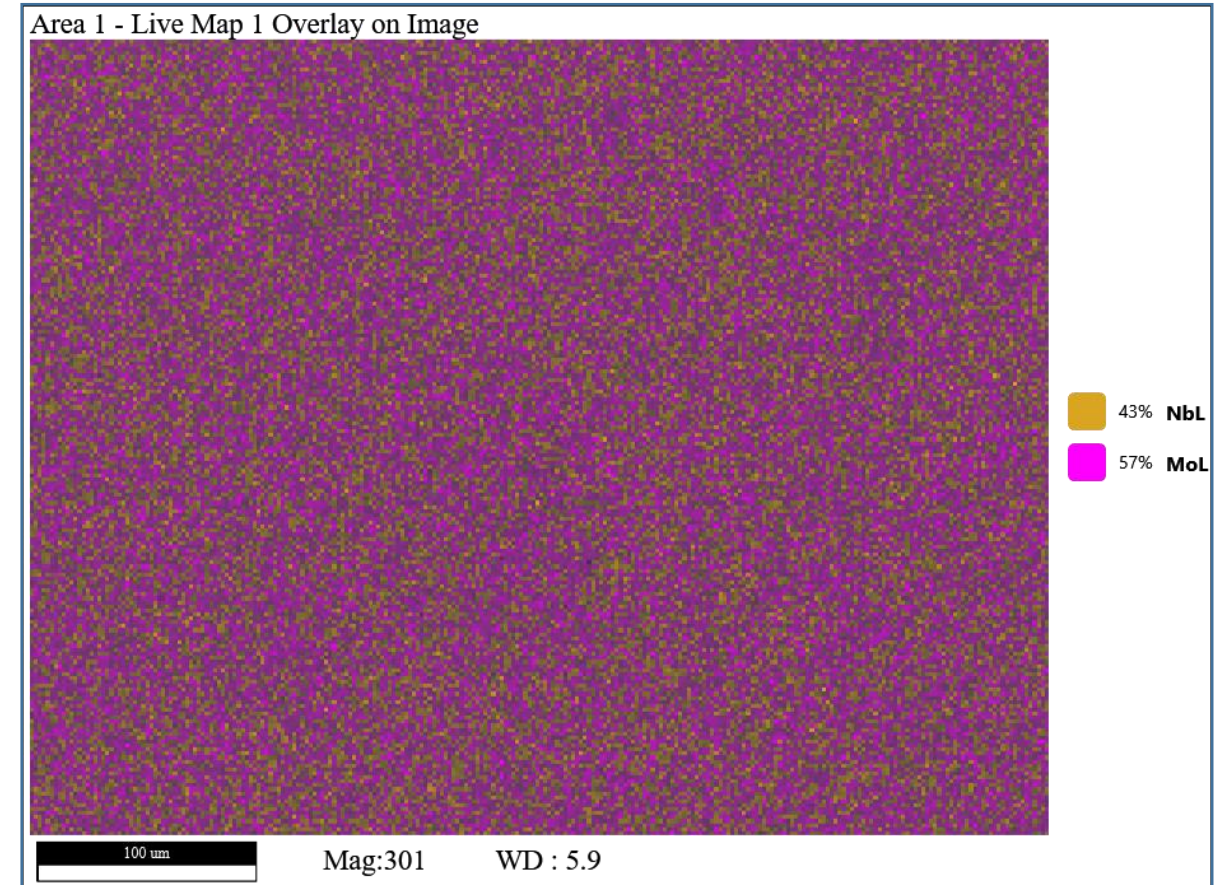
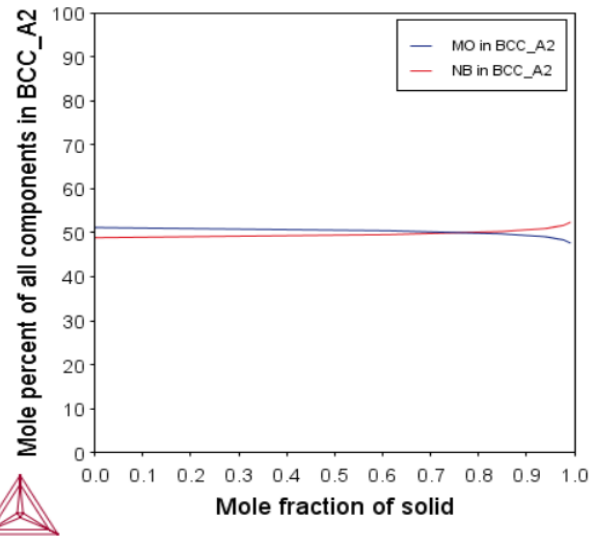


- HEAs/MPEAs/CCAs (High Entropy Alloys/Multi-Principal Element Alloys/Complex Concentrated Alloys) popular for research during last 15 years
- Growing need for advanced structural metallic alloys capable of ultrahigh temperature performance in extreme environments
 - Recent research into MPEAs consisting of only, or primarily, refractory metals (RMPEAs)
- Limited knowledge of refractory alloys beyond binaries
 - Extremely high melting temperatures lead to experimental difficulties
- Fabrication of RMPEAs challenging
 - Need for discovery/development of RMPEAs with capability to be fabricated (some room temperature ductility) while maintaining high temperature strength (thermomechanical processing)
 - Need for discovery/development of RMPEAs for additive manufacturing

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Diffusion Couples

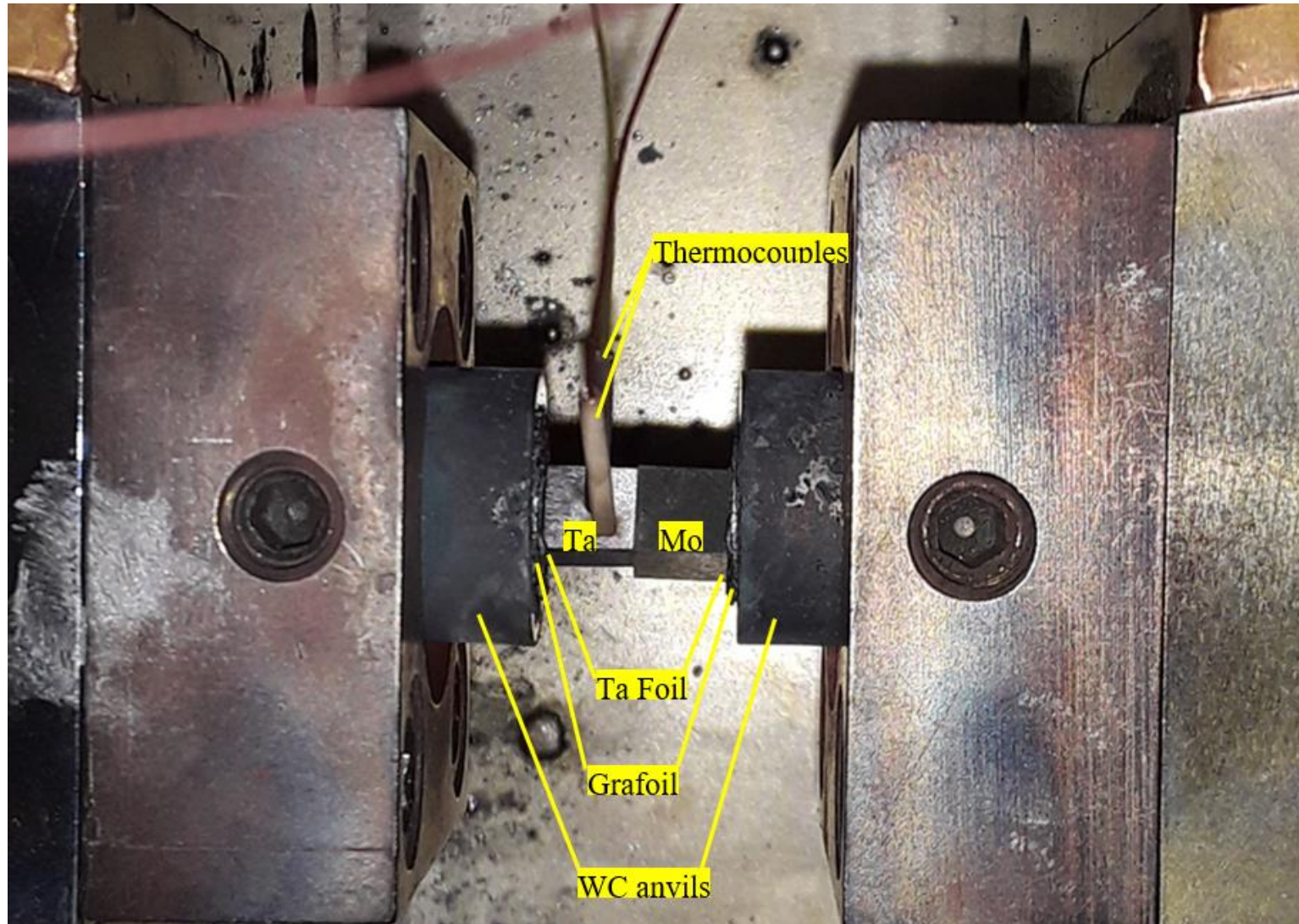
- Pure Ta with MoNb binaries
- Melting Temperatures
 - Mo = 2623 °C
 - Nb = 2477 °C
- Minimal solidification segregation



- **TAKEAWAY:** no segregation observed in MoNb as-cast buttons

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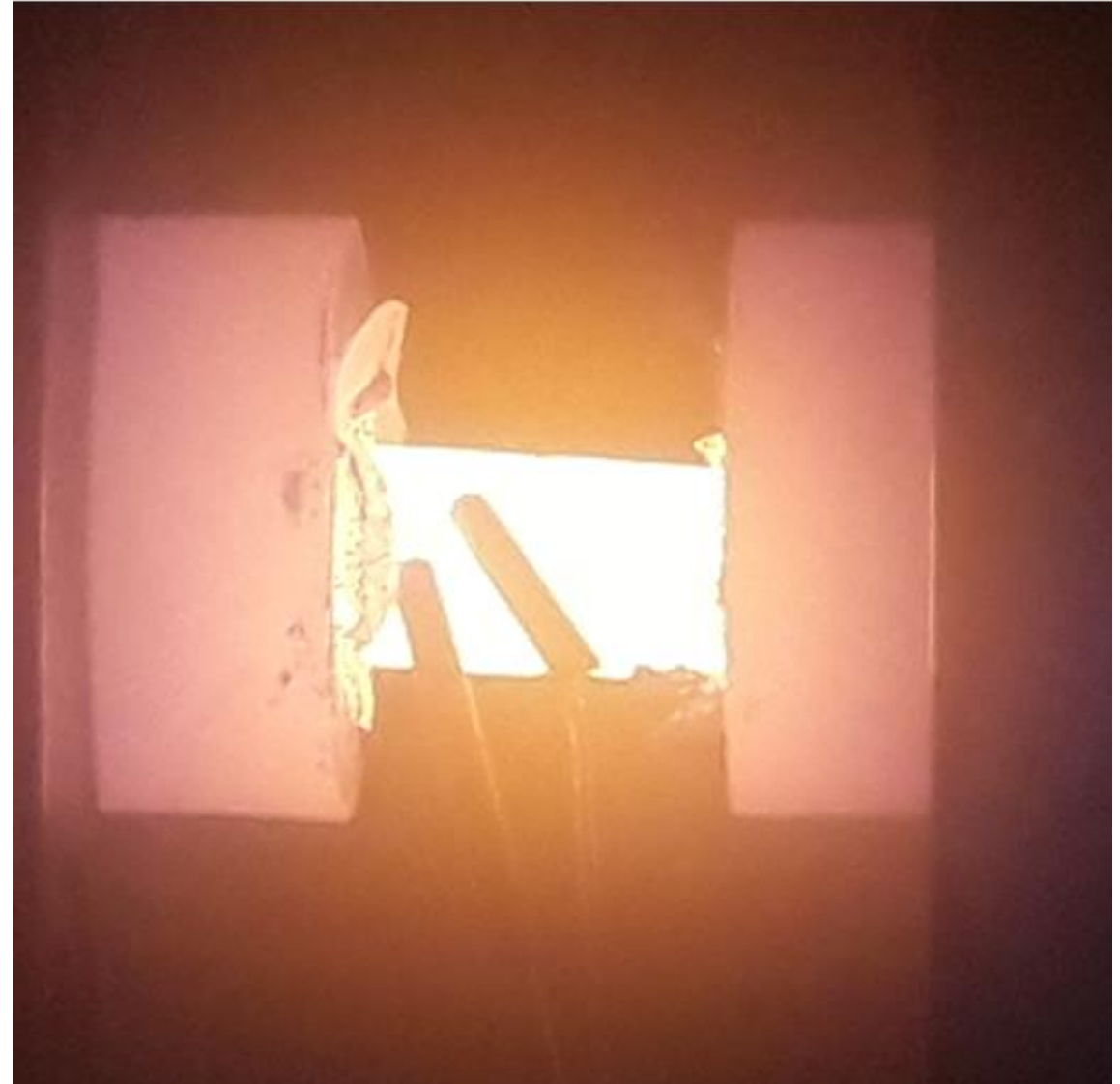
Diffusion Couples



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Diffusion Couples

- Apply 2kN compression
- Ramp to 1200 °C over 4 min
 - Compress 1/4mm during ramp
- Hold at 1200°C for 30 min
 - Compress 1mm during hold



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Trial Heat Treatments in Hot Press



- Two test heat treatments
 - Mo-Ta diffusion couples
 - Attempted 1800 °C
- First:
 - Pulled vacuum then backfilled with Ar at room temp
 - Reached 1790 °C
 - Significant oxidation observed
- Second:
 - Heated to 1600 °C in vacuum then backfilled with Ar
 - Burn off any water or organics
 - Reached 1750 °C
 - No oxidation observed
- **TAKEAWAY:** Performing heat treatments at 1700 °C, in Ar atmosphere after heating to 1600 °C in vacuum

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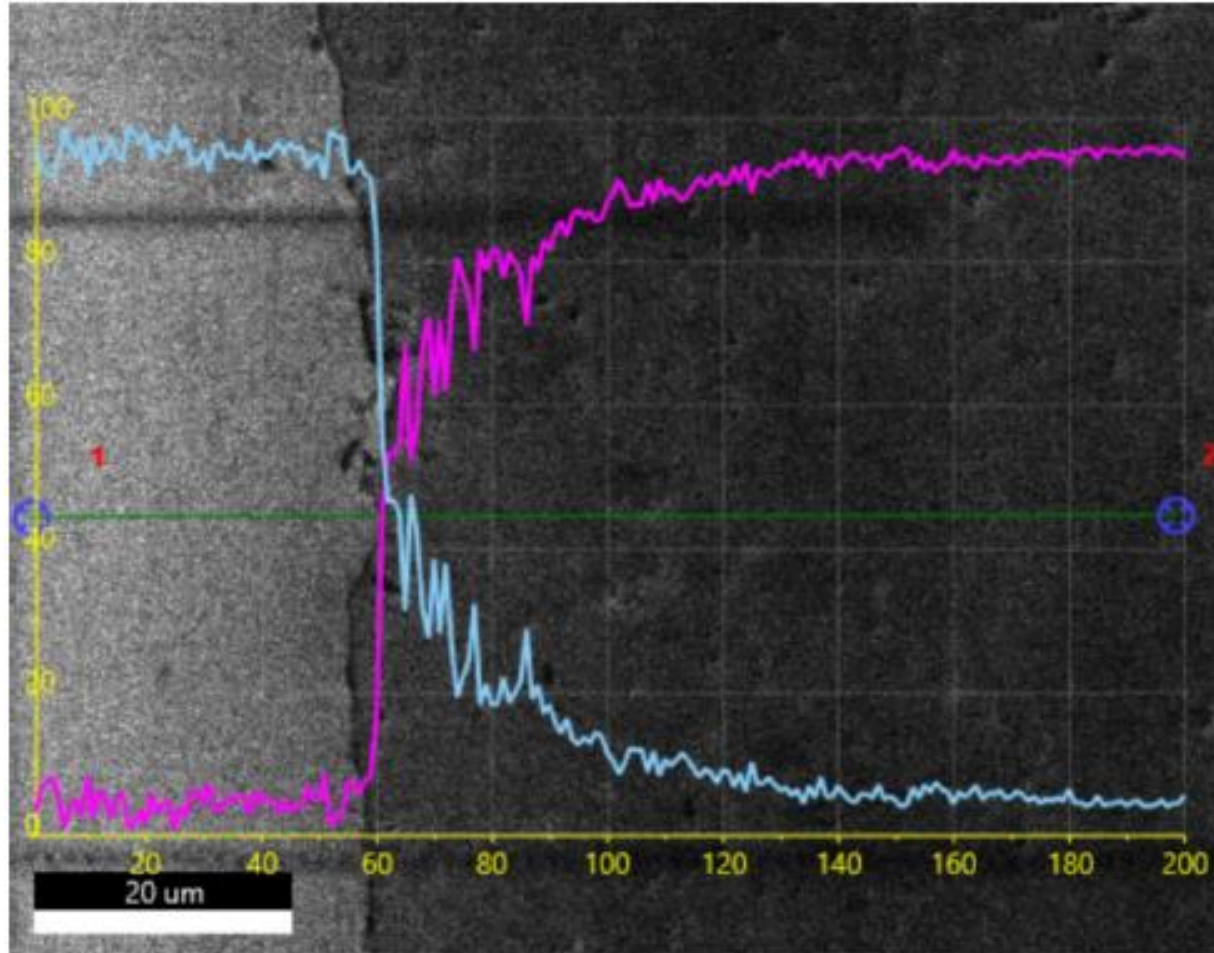
Heat Treatment Progress



- Total 420 hours at 1700 °C
 - 1 sample set
 - Performed in 7 segments
 - Needed to investigate ability of the hot press to hold for long times
 - Samples are being investigated
- Additional heat treatments at 1700 °C will be performed
 - 3 sample sets
 - Different prep methods
 - Total 500 hours

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Mo-Ta Diffusion Couple (420h @ 1700°C)

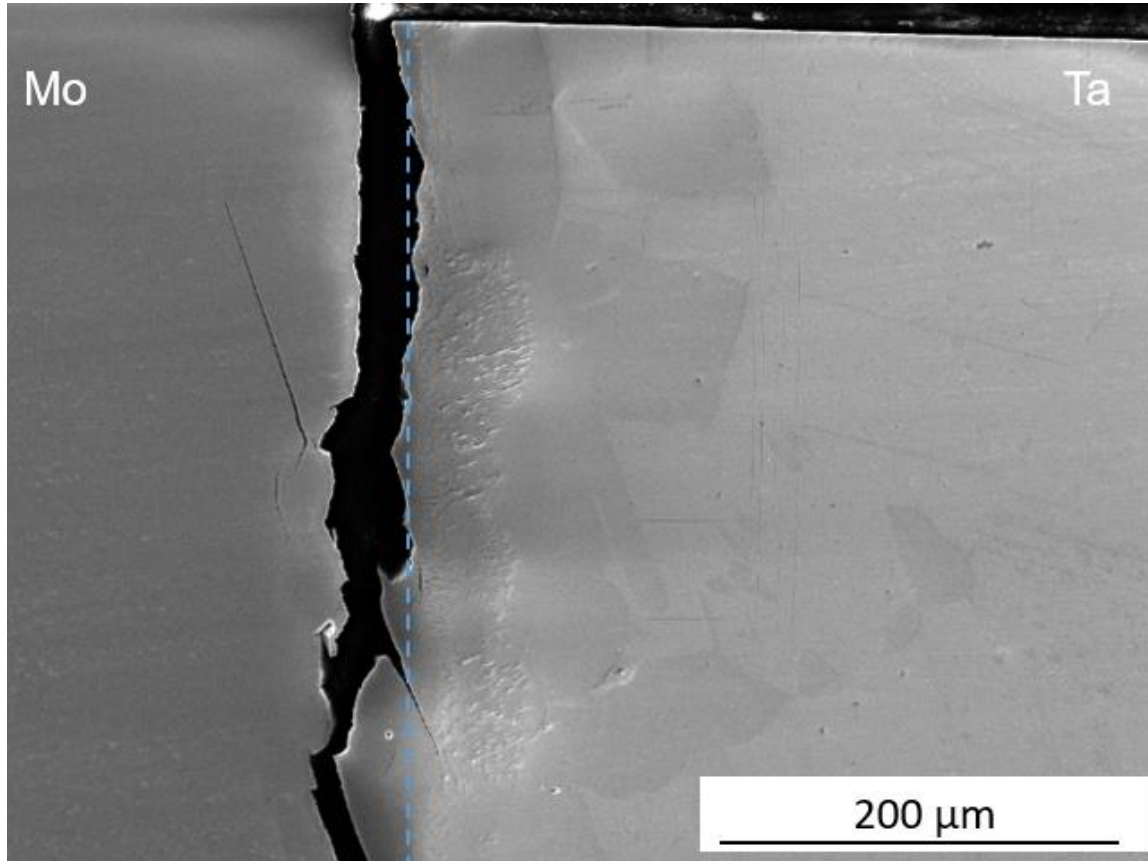


Mo L
Ta L

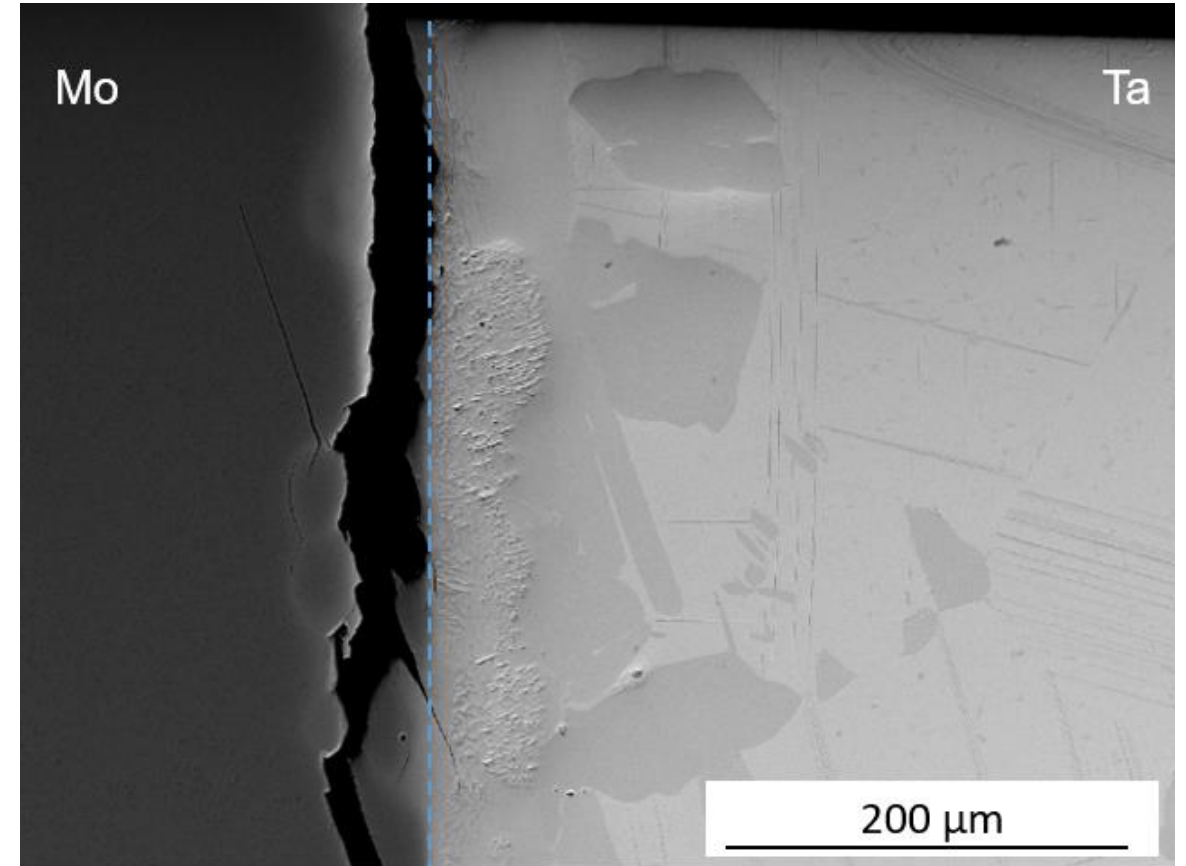
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Mo-Ta Diffusion Couple (420h @ 1700°C)

SE



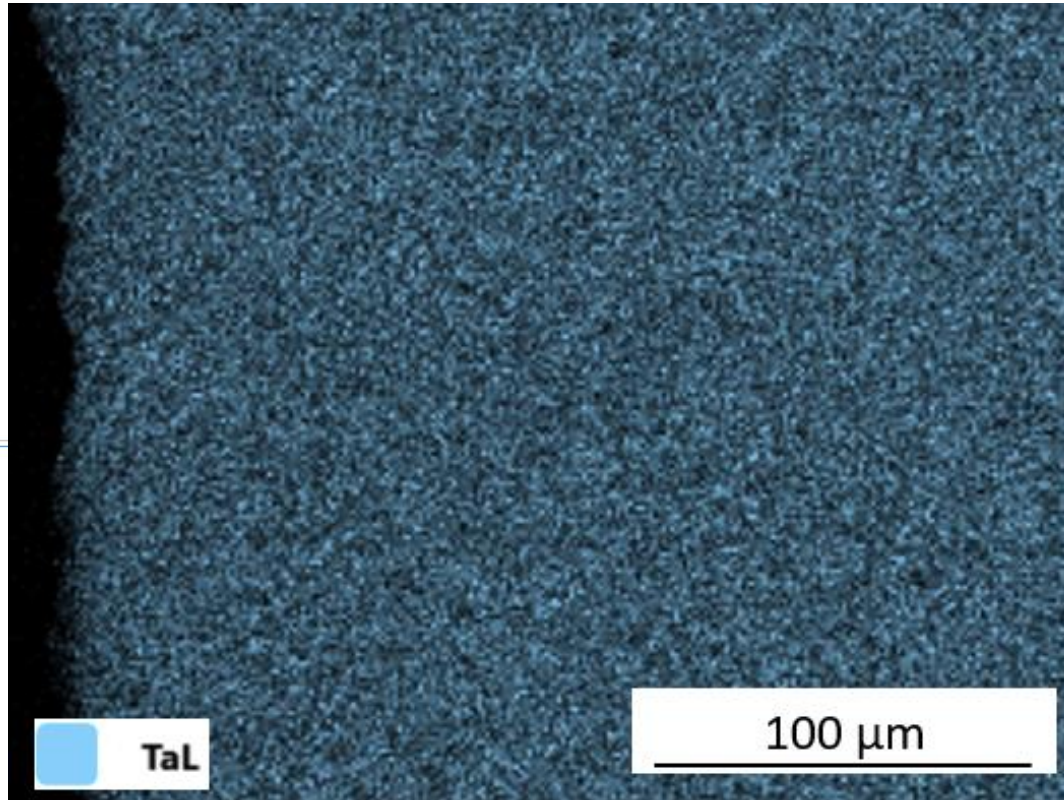
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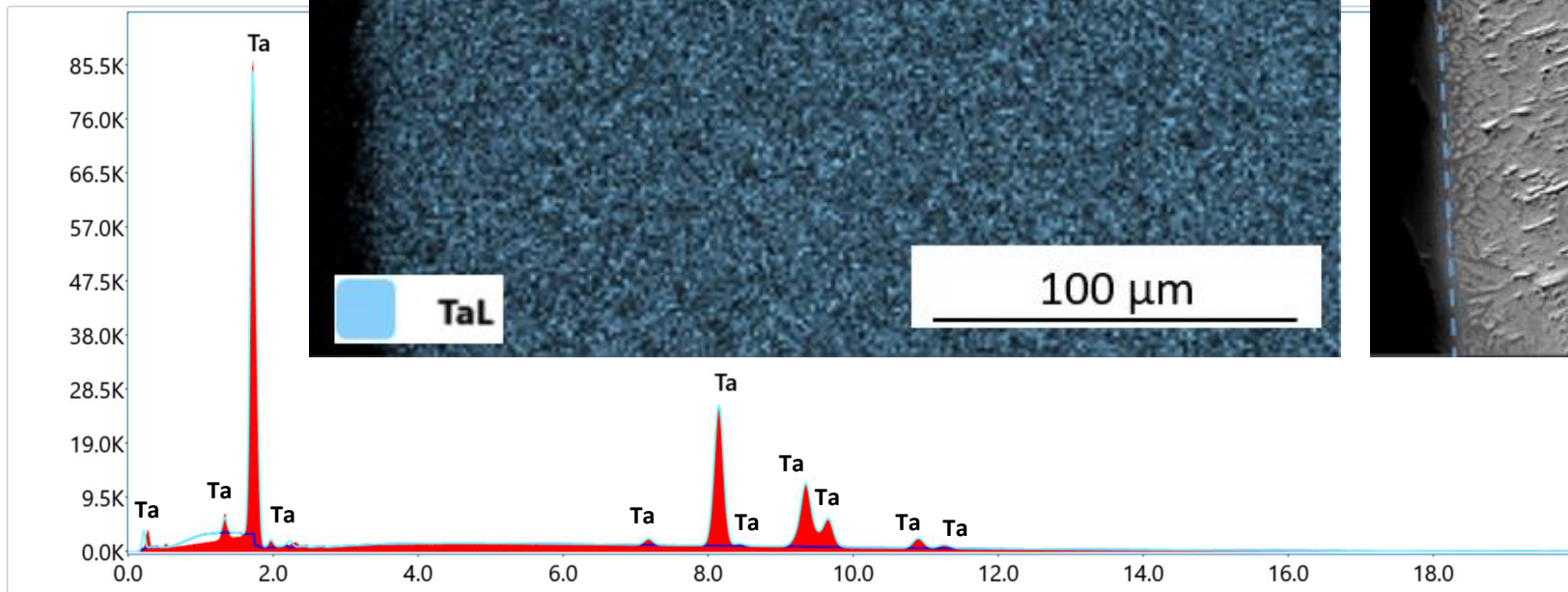
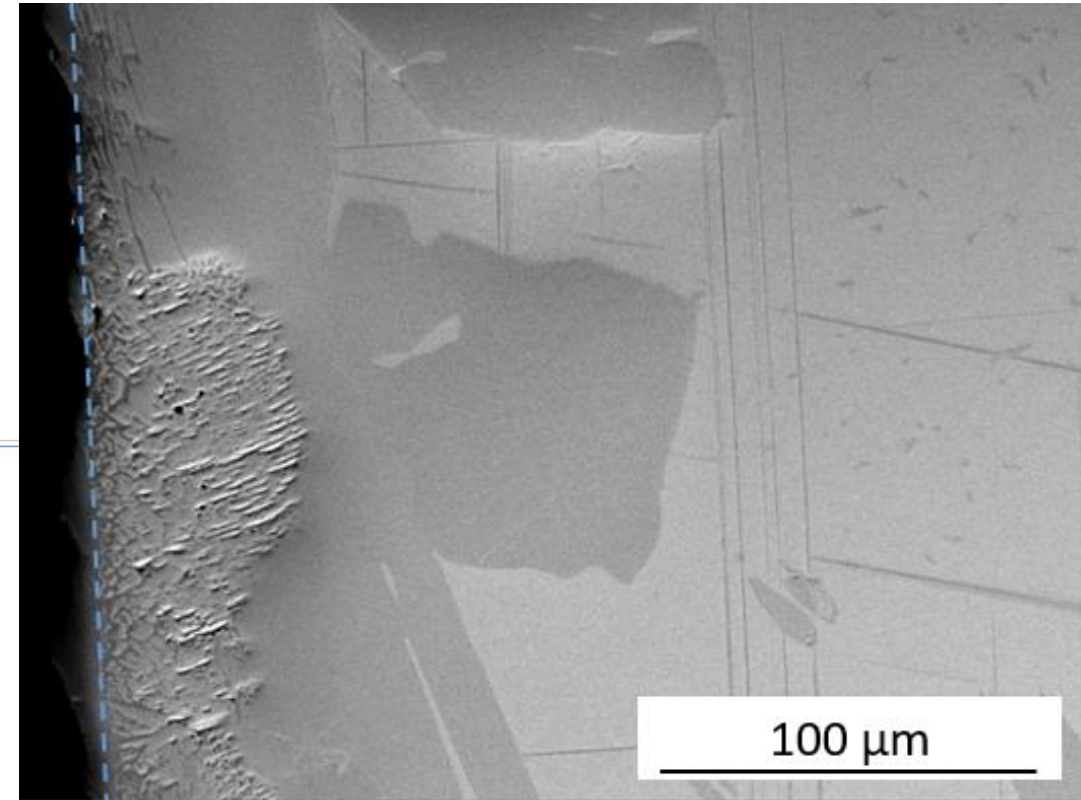
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Mo-Ta Diffusion Couple (420h @ 1700°C)

EDS Map

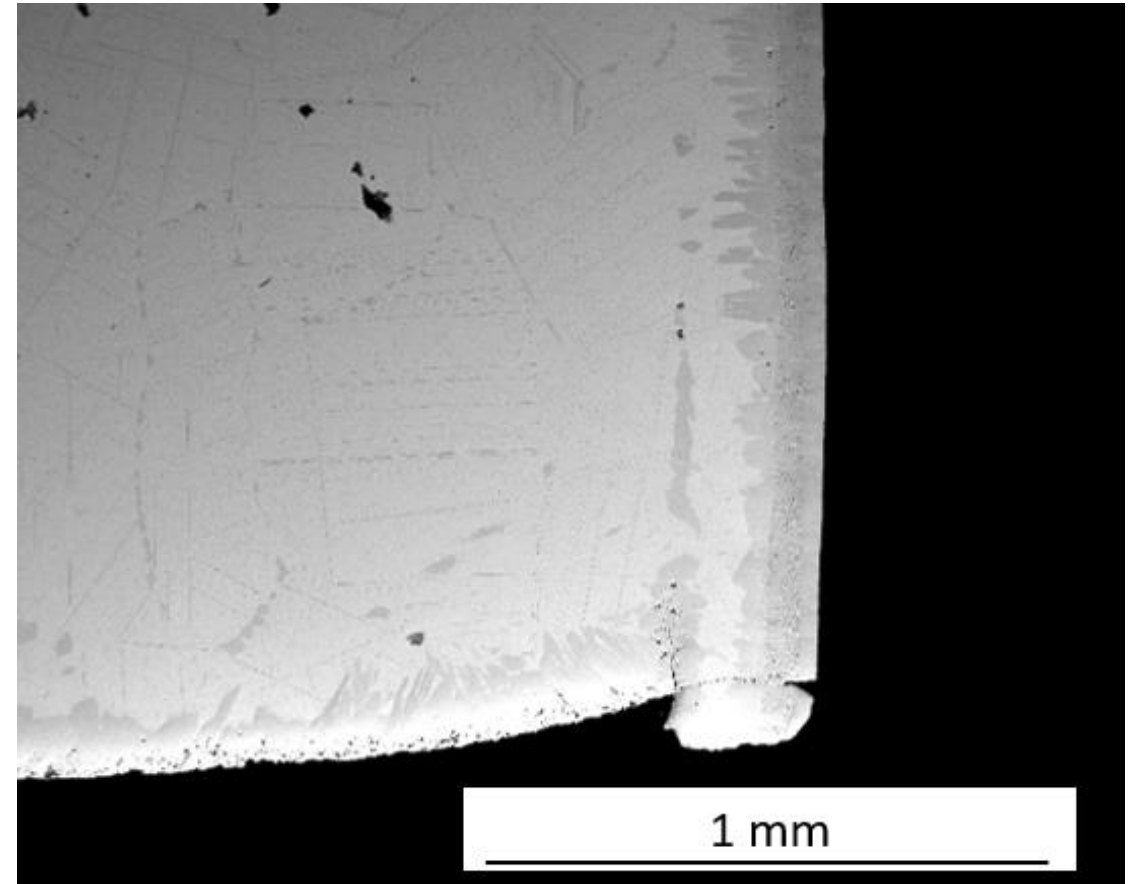
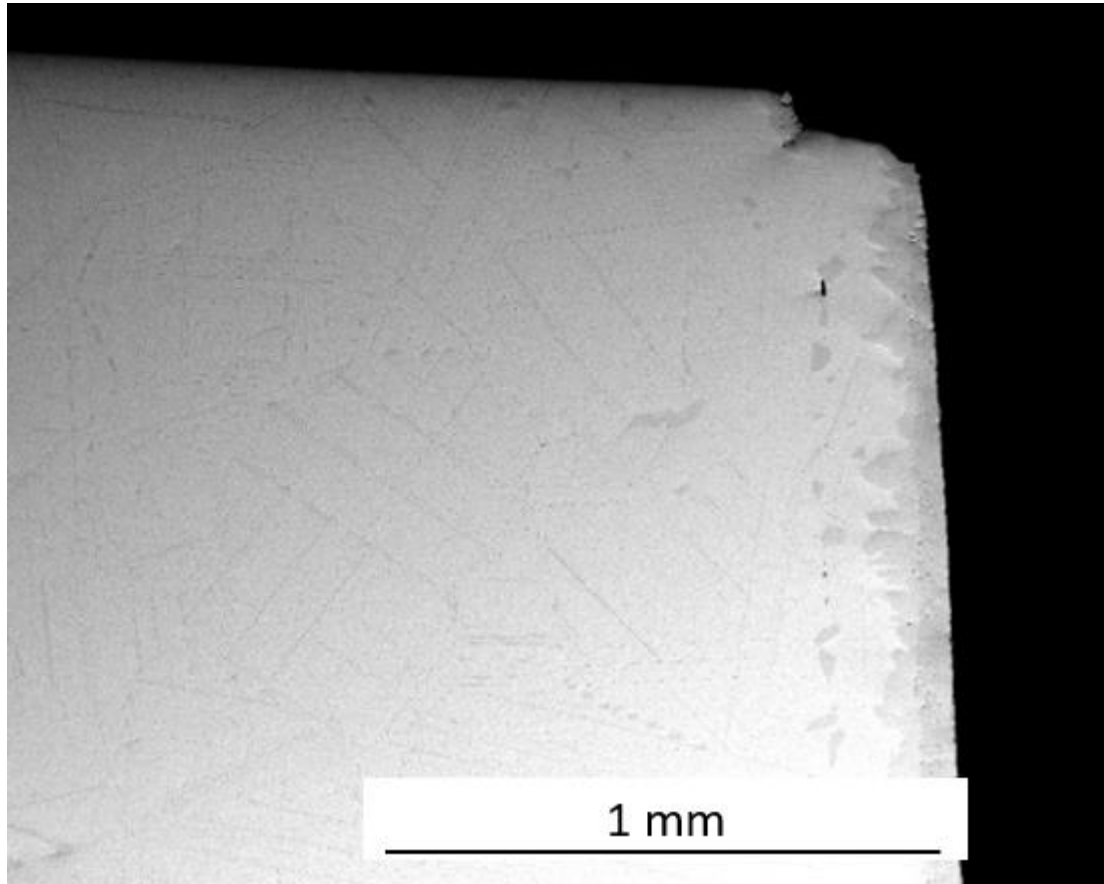


BSE



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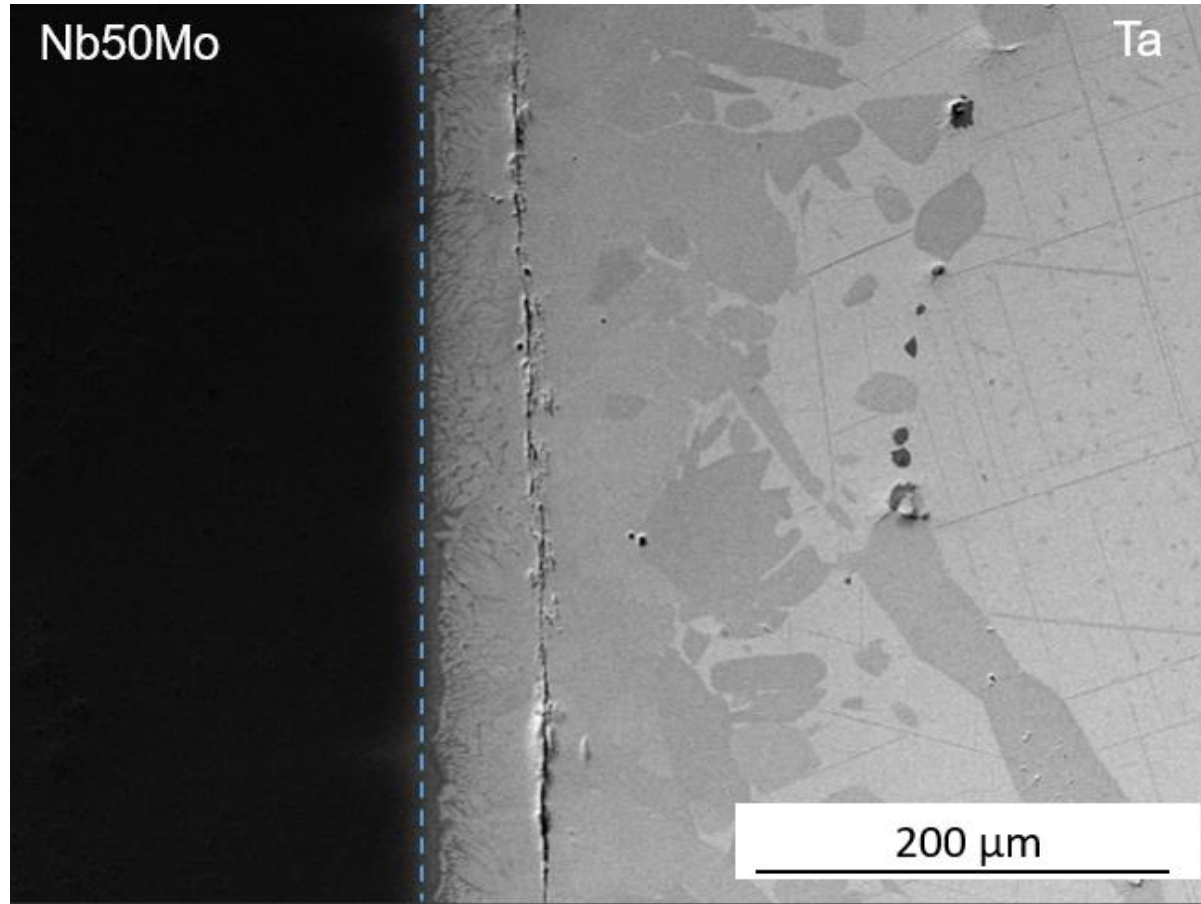
Mo-Ta Diffusion Couple (420h @ 1700°C)



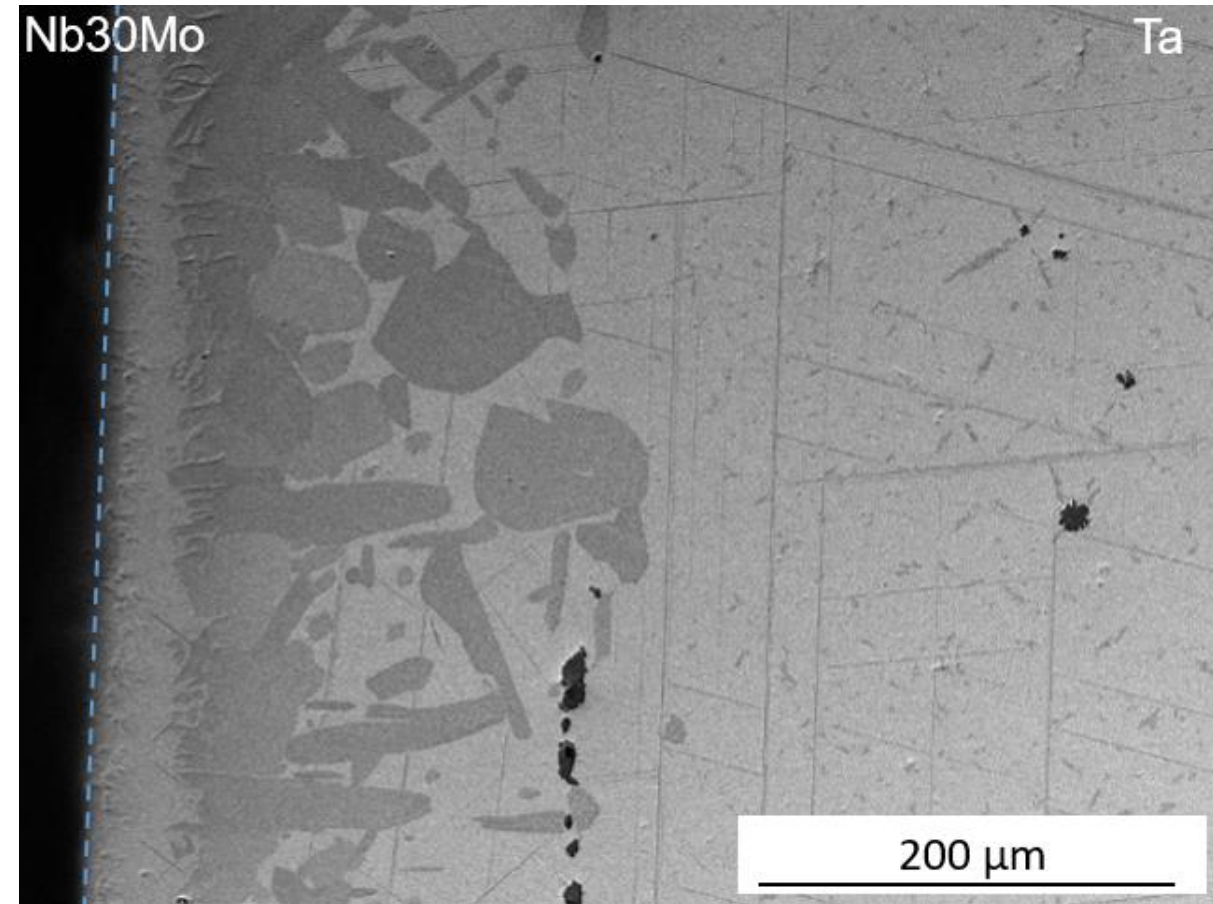
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Diffusion Couples (420h @ 1700°C)

Nb50Mo - Ta

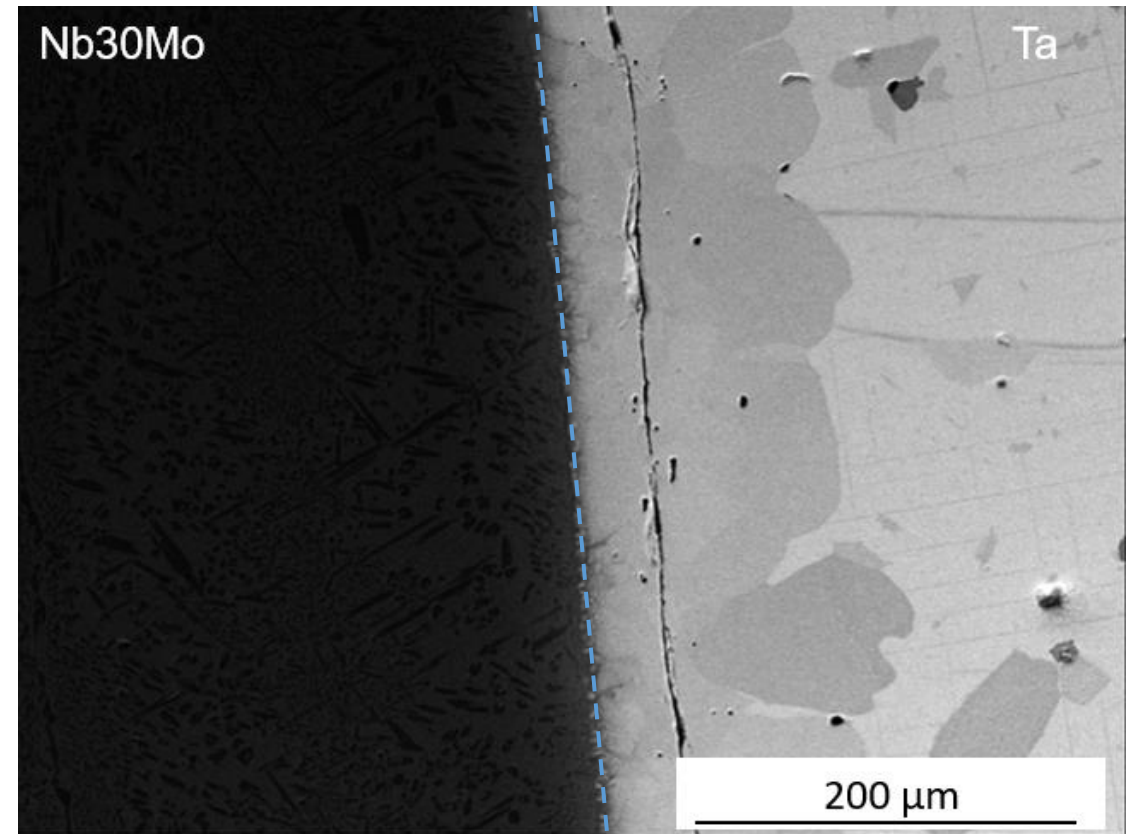
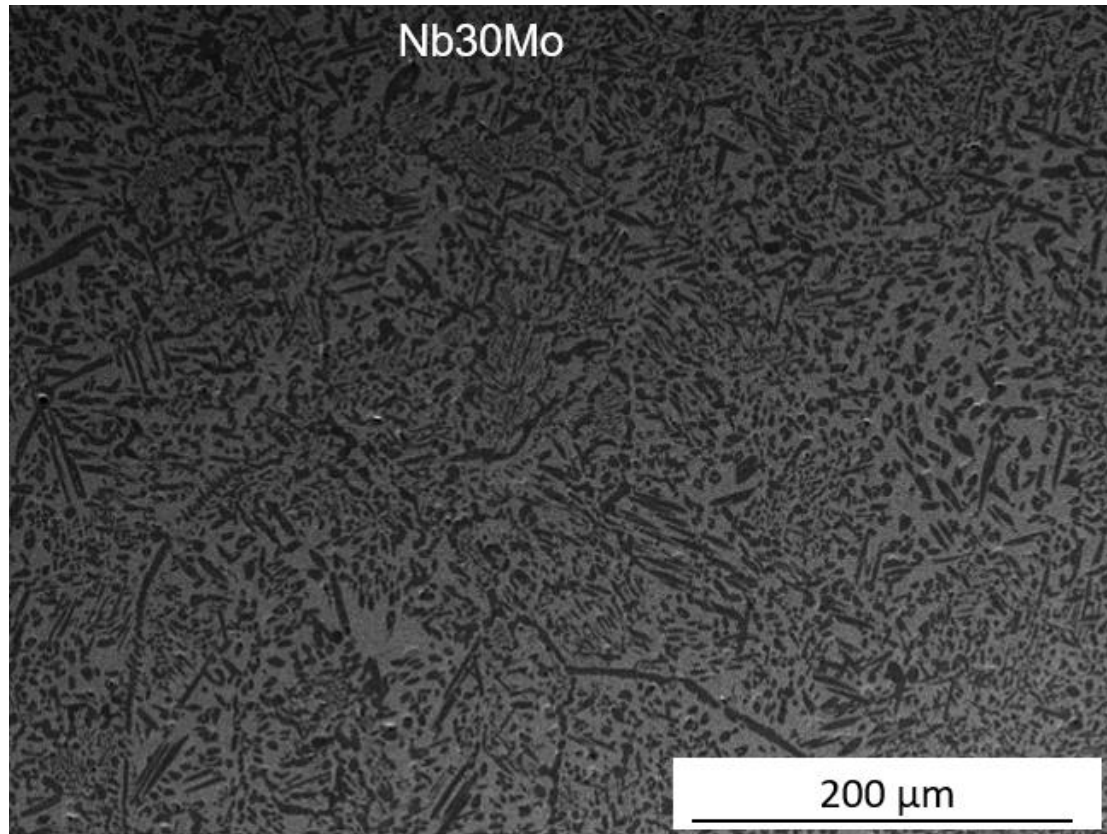


Mo30Nb - Ta



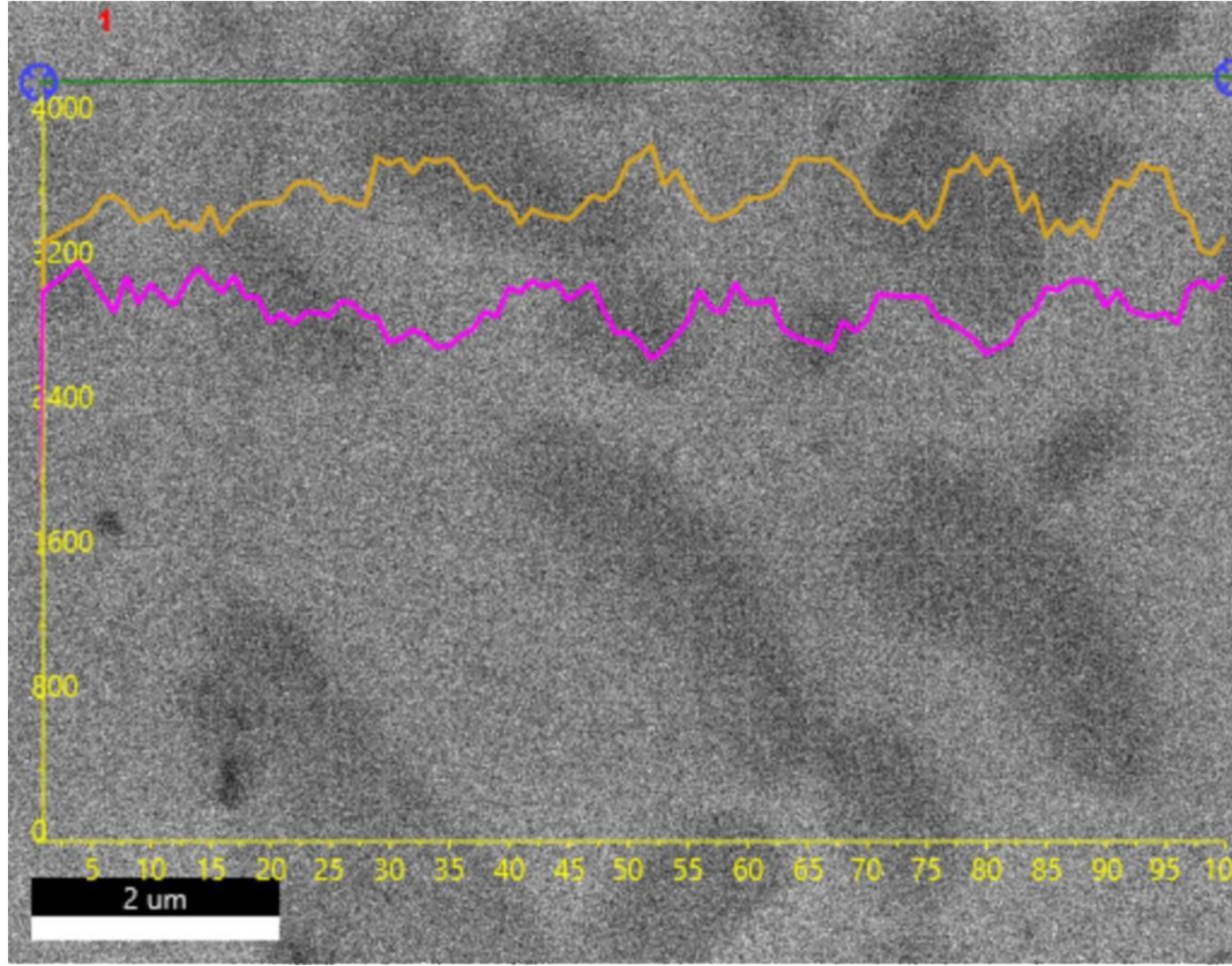
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Ta-Nb30Mo Diffusion Couple (420h @ 1700°C)

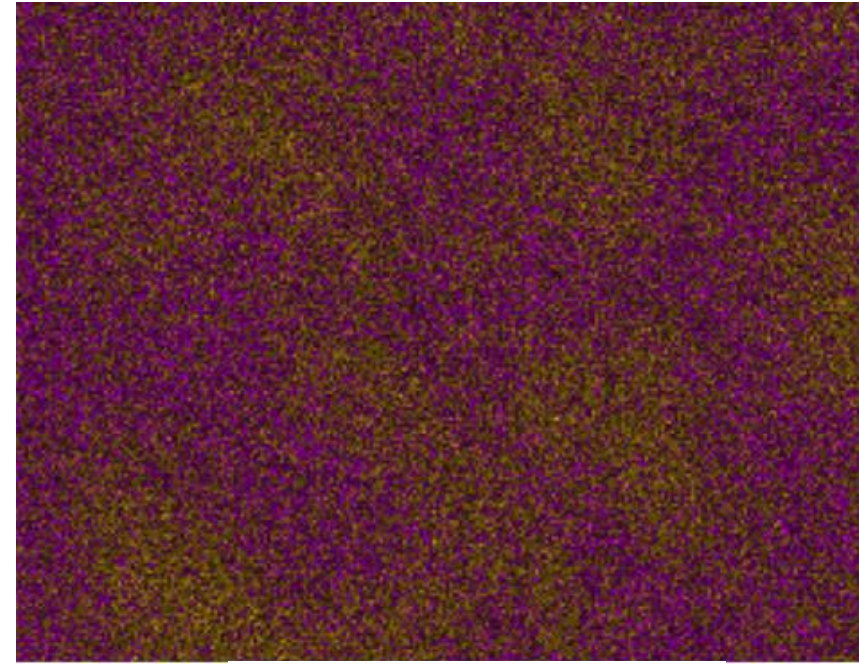


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Ta-Nb30Mo Diffusion Couple (420h @ 1700°C)

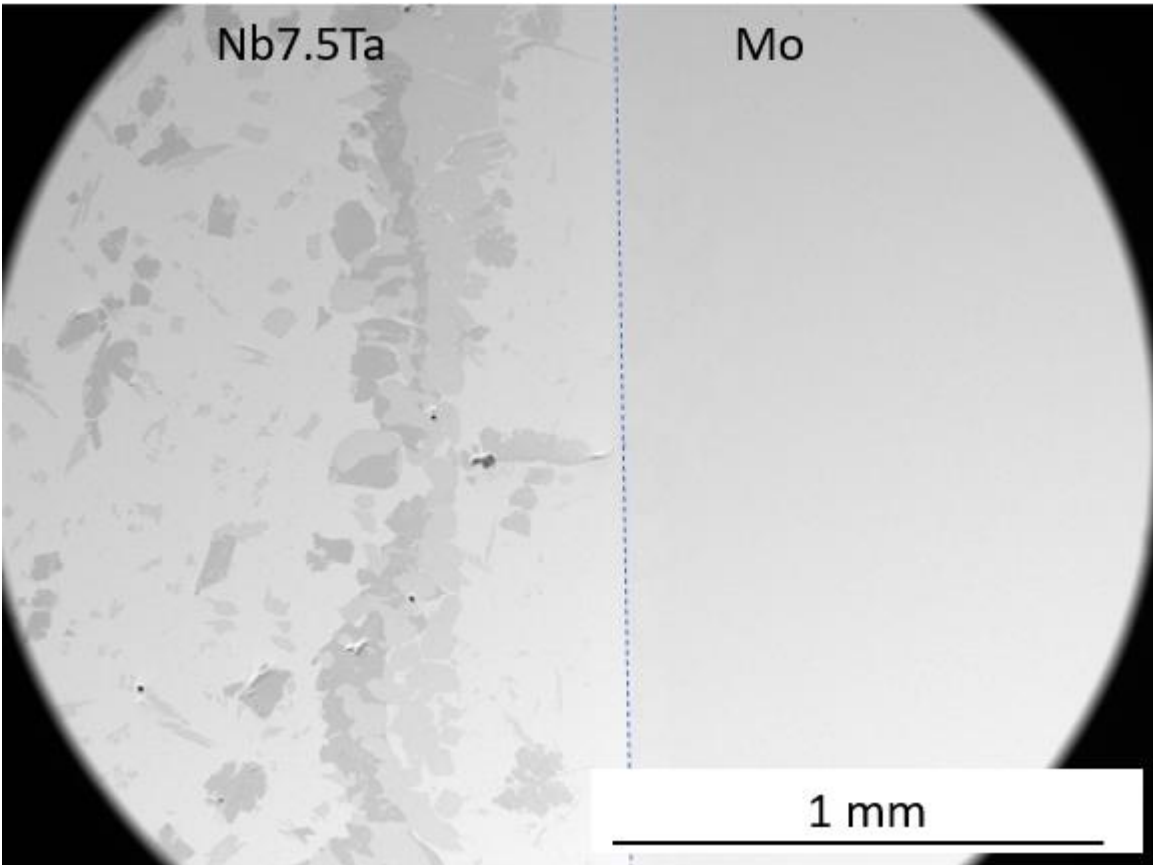
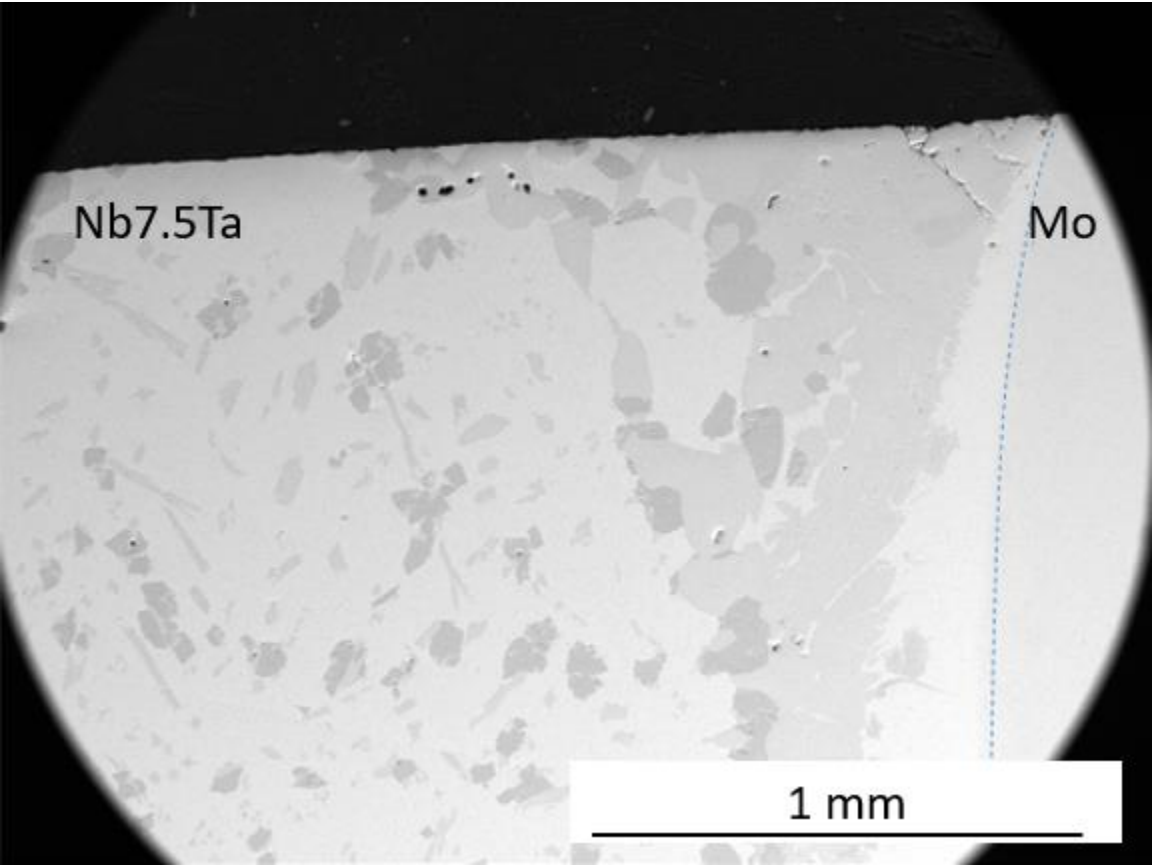


 **Nb L**
 **Mo L**



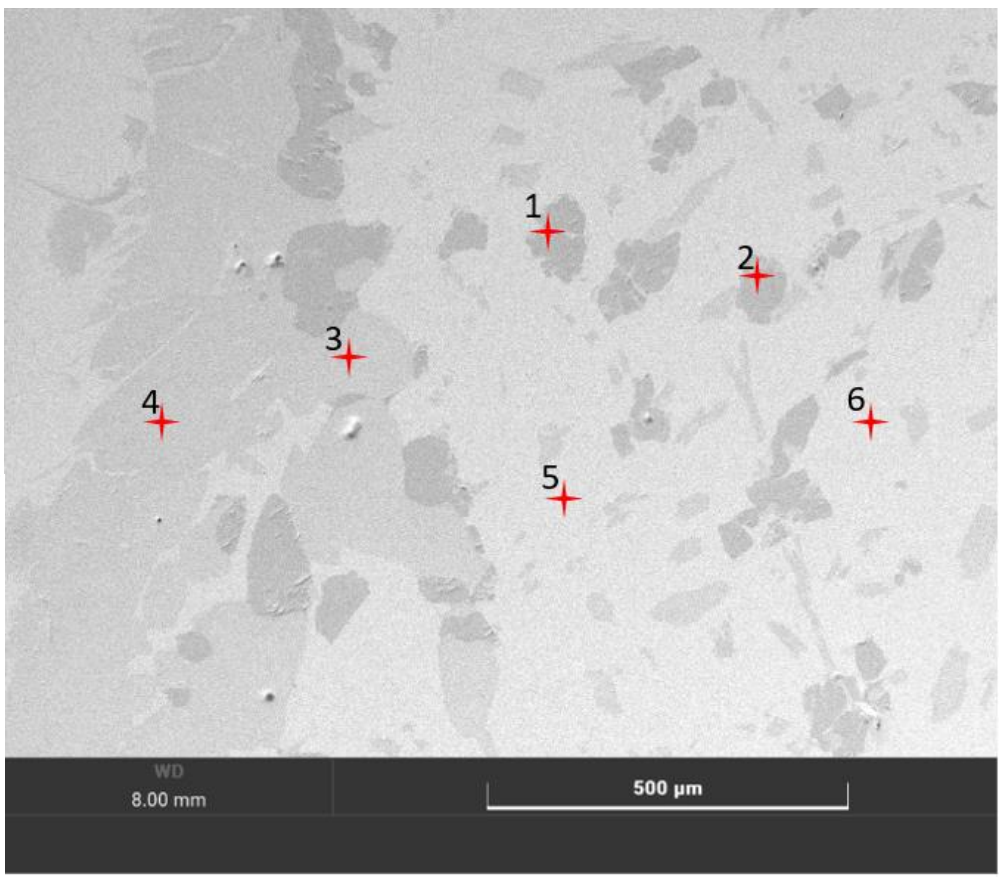
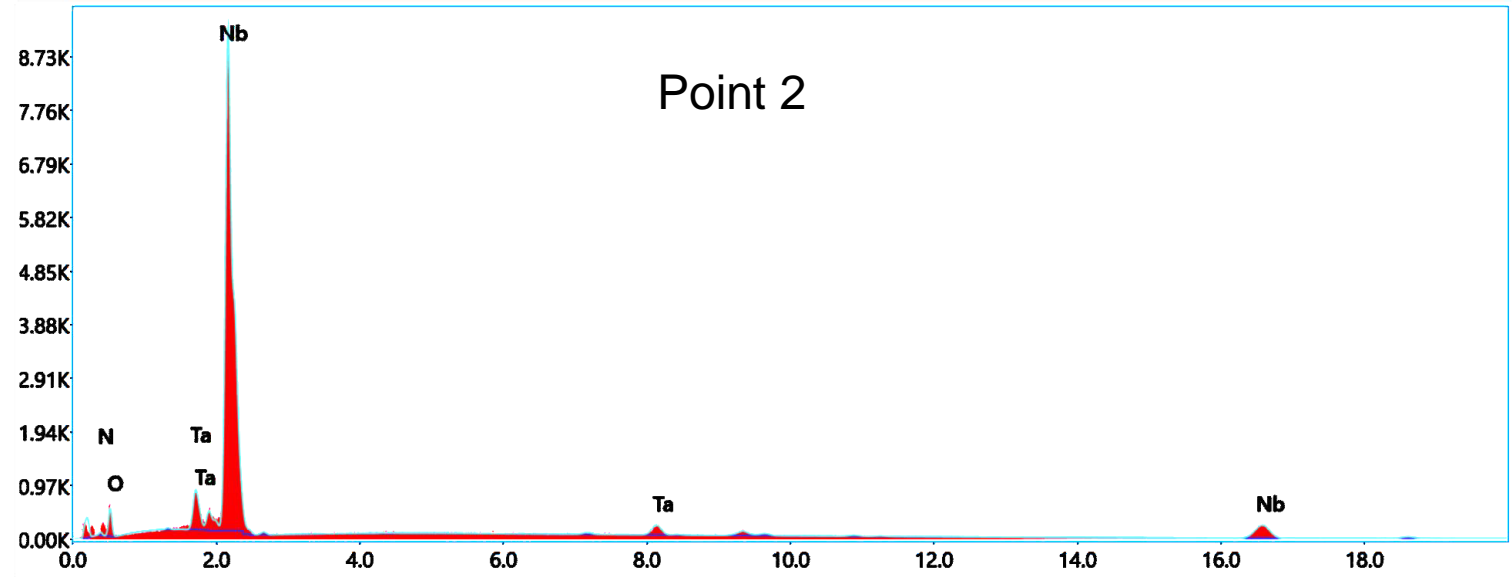
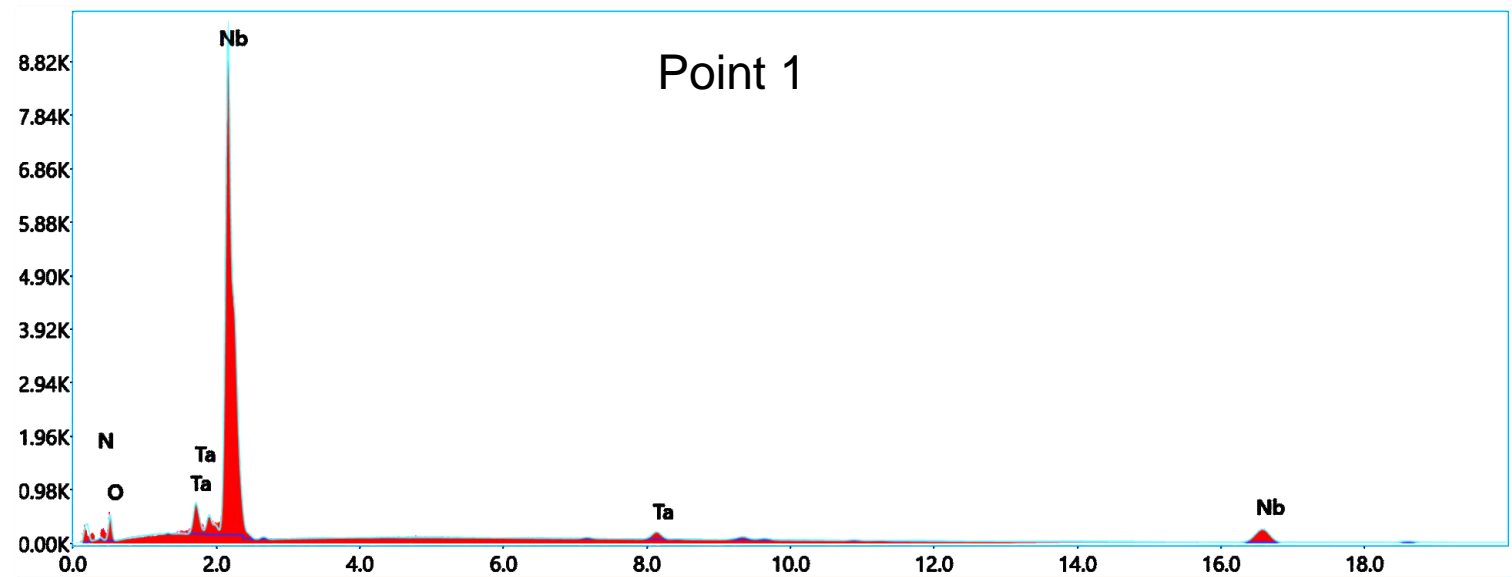
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Mo-Nb7.5Ta Diffusion Couple (420h @ 1700°C)



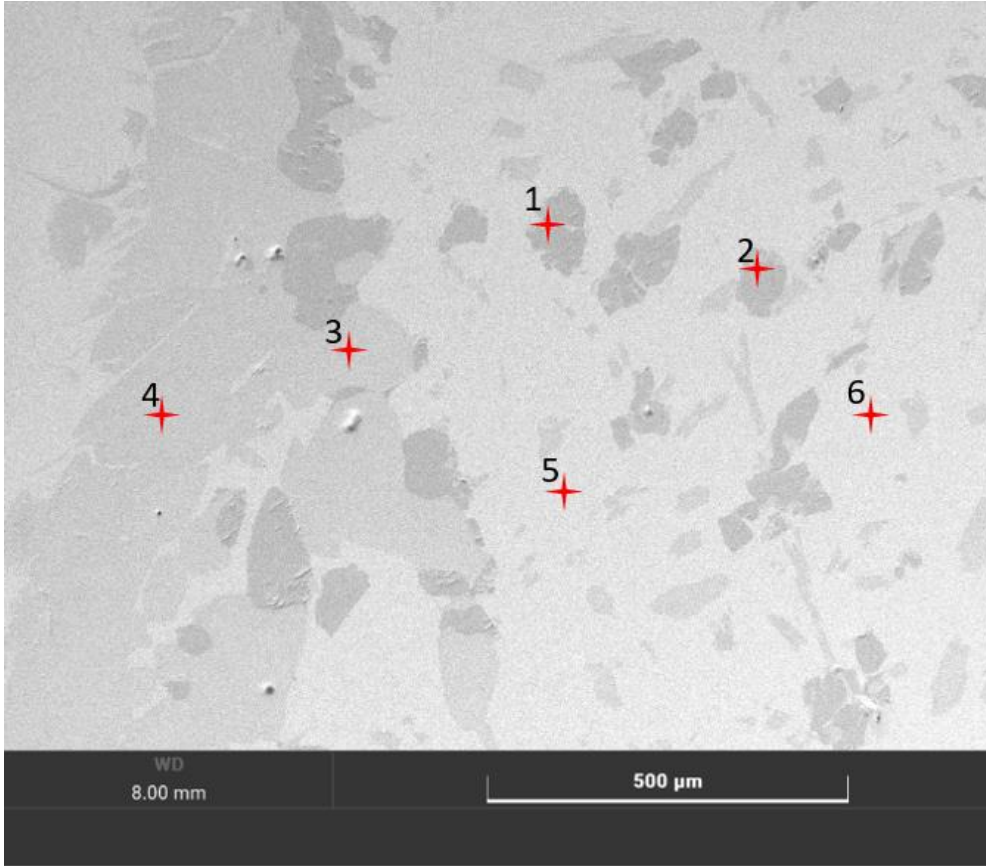
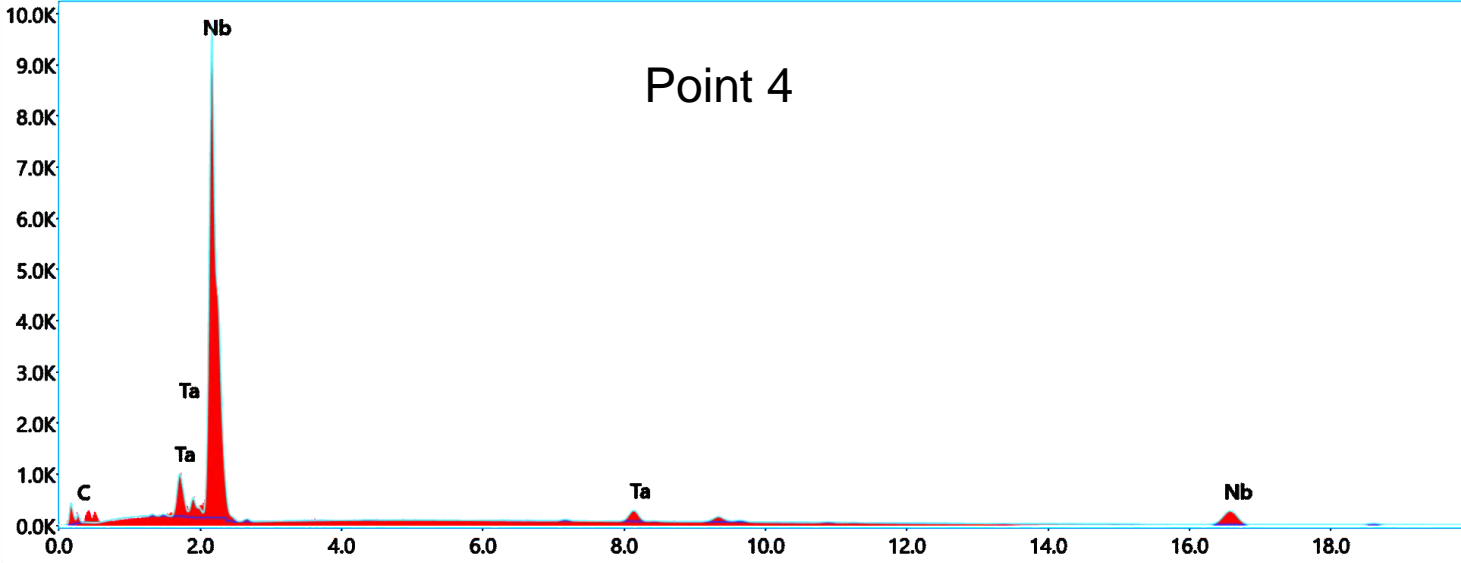
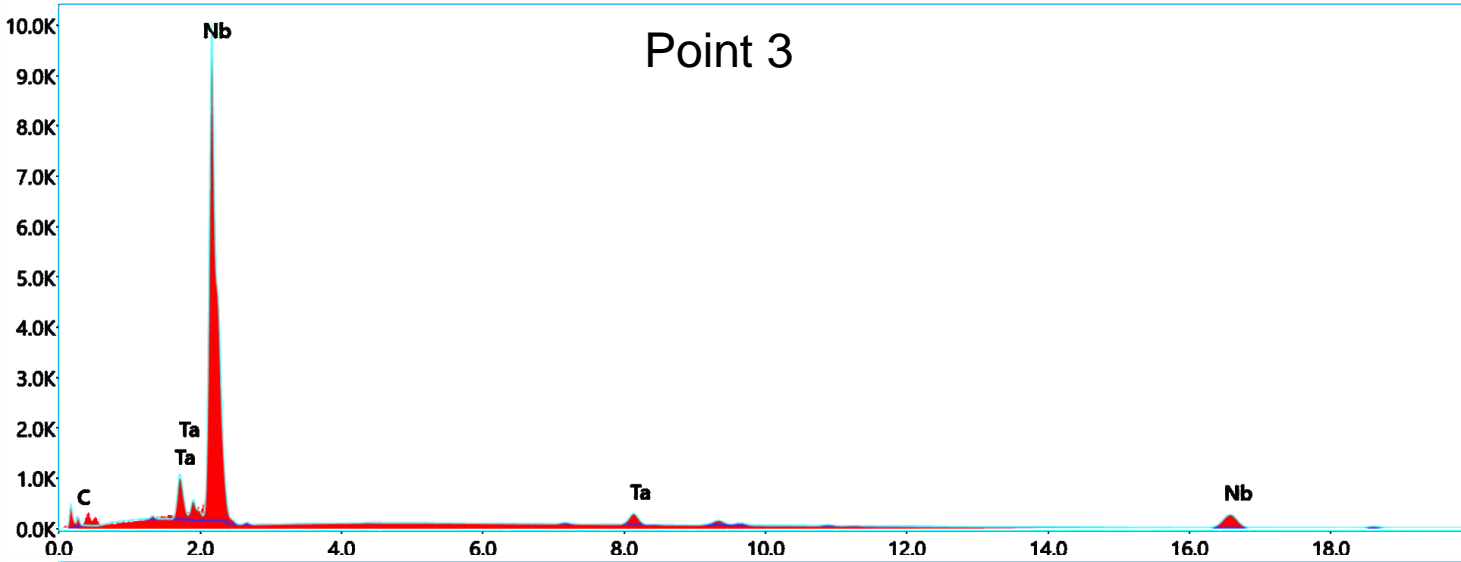
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Mo-Nb7.5Ta Diffusion Couple (420h @ 1700°C)



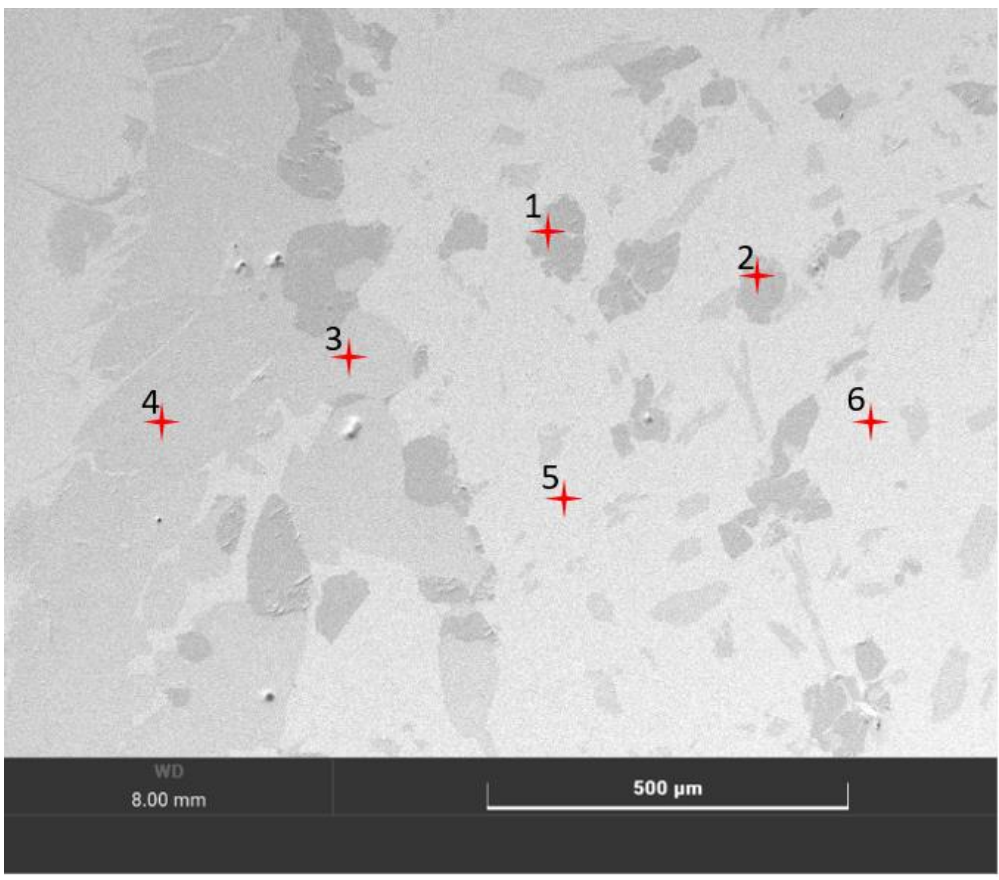
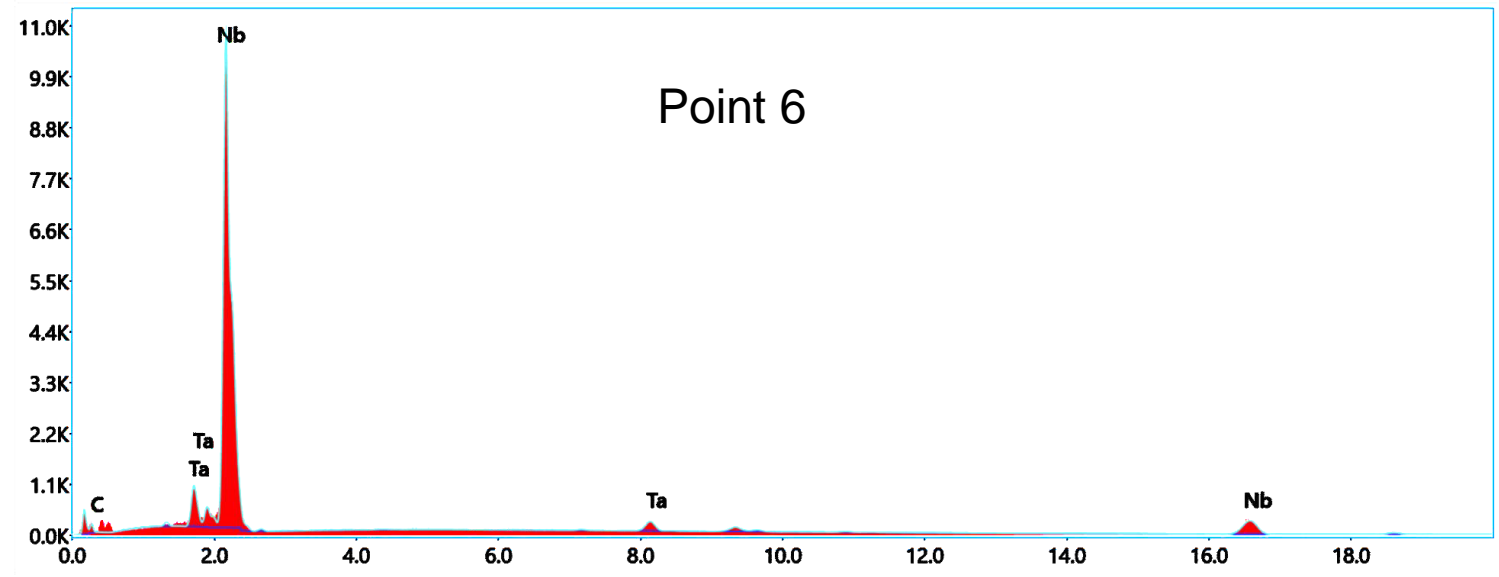
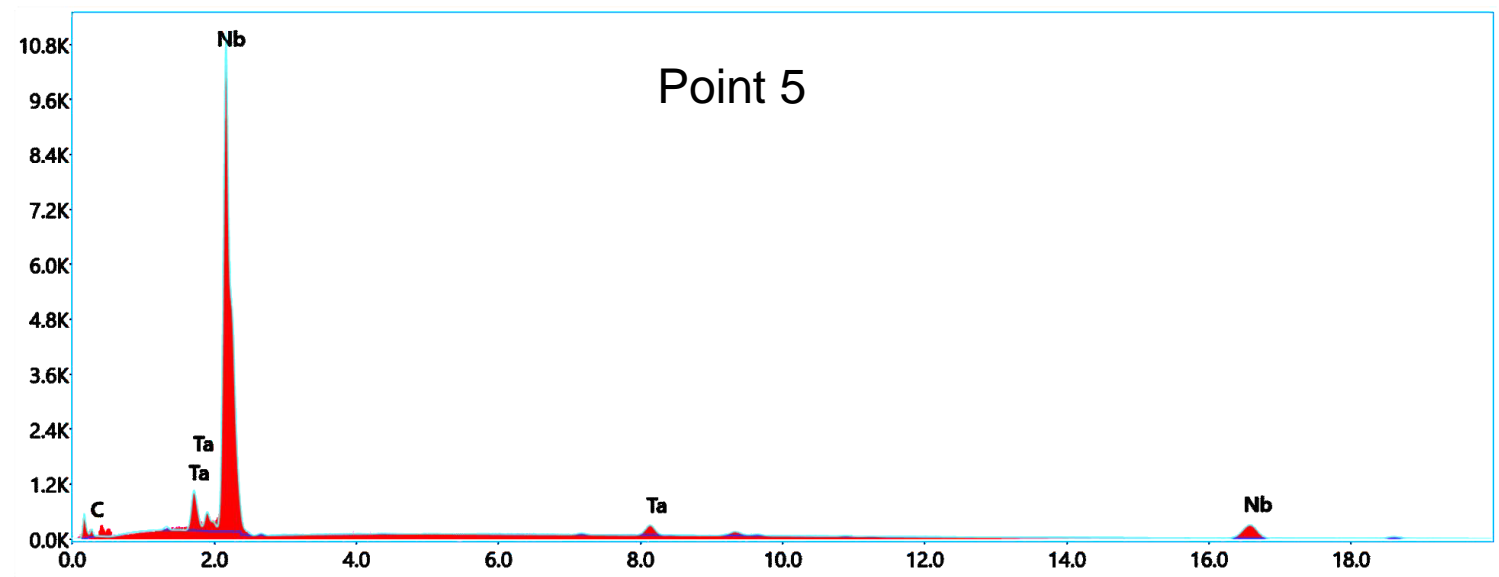
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Mo-Nb7.5Ta Diffusion Couple (420h @ 1700°C)



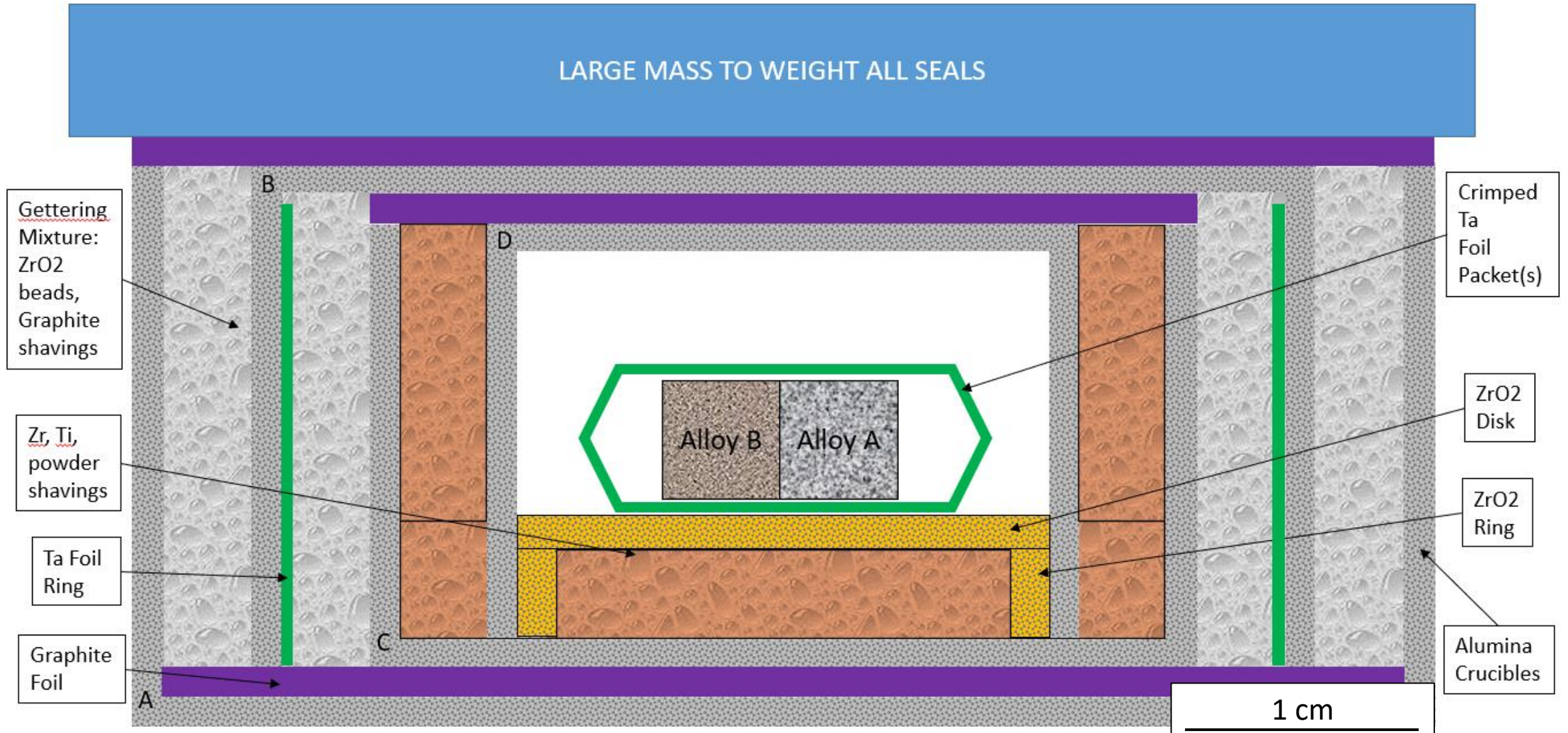
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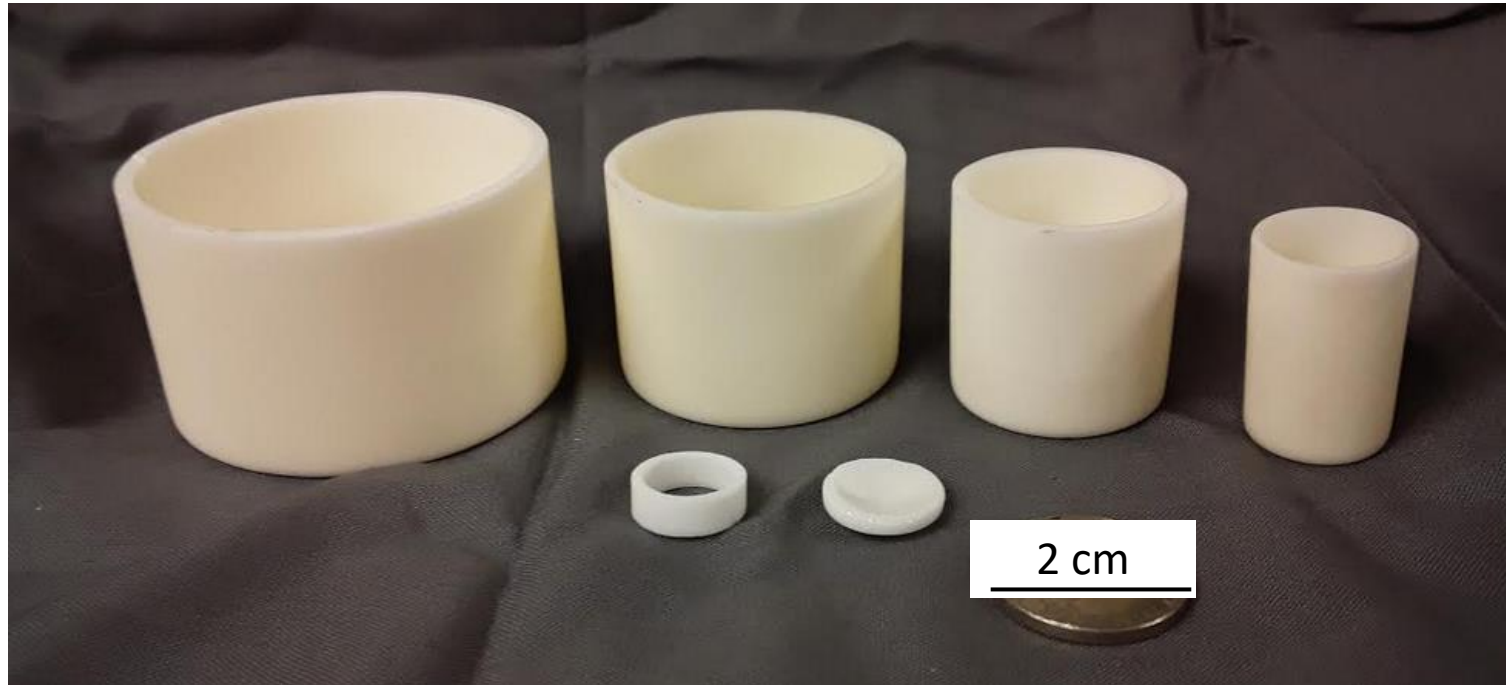
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Nesting Doll Diffusion Chamber



Nesting Doll Diffusion Chamber

- All materials on hand
- Alumina crucibles and Zirconia ring and lid are cut to size
- Test run will be performed with a Mo-Ta diffusion couple

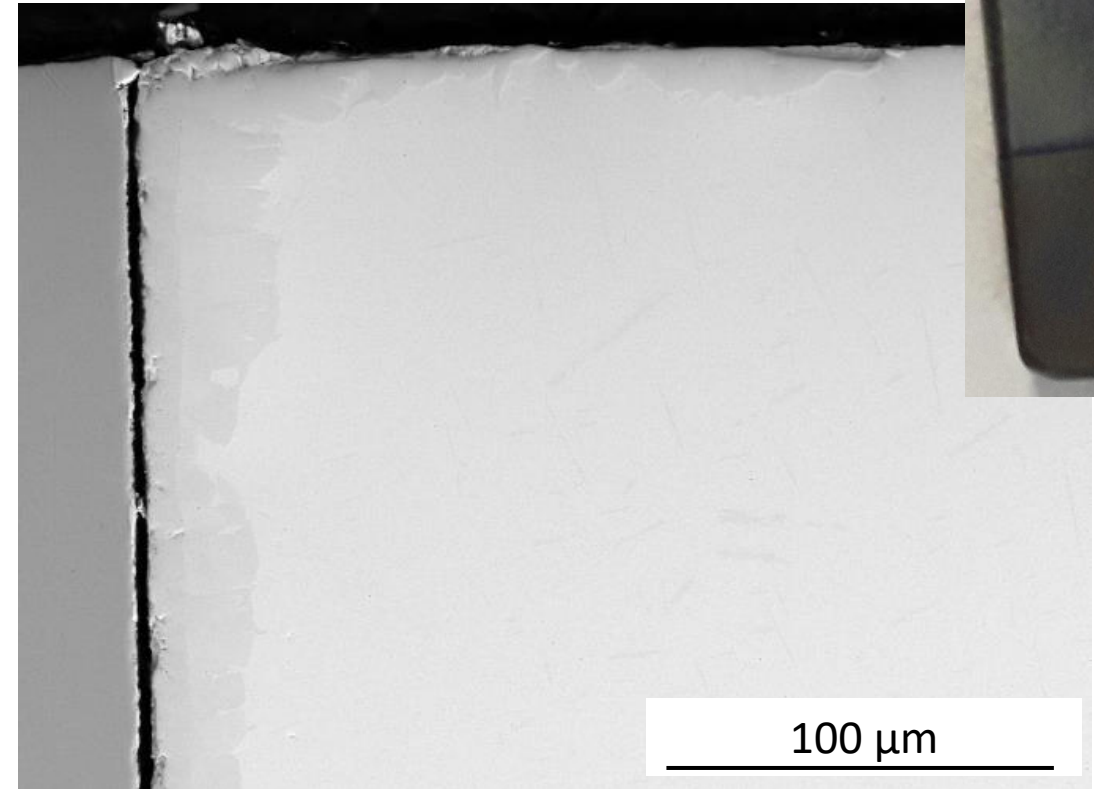
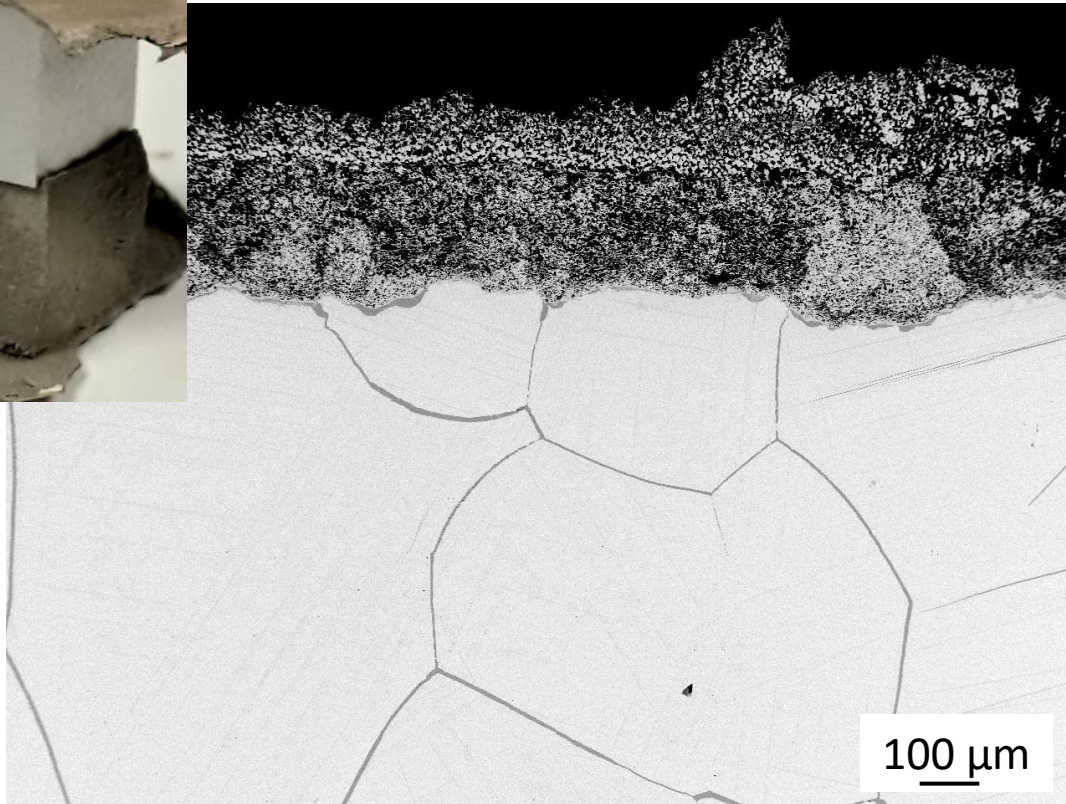
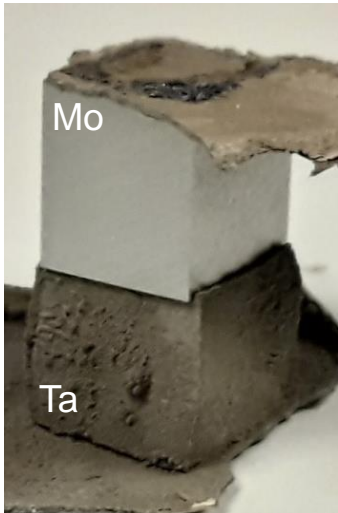


- **TAKEAWAY:** Nesting dolls ready to be made, waiting on furnace power

Nesting Doll Trial Heat Treatment

Hot Press Trial #1

Nesting Doll Trial #1

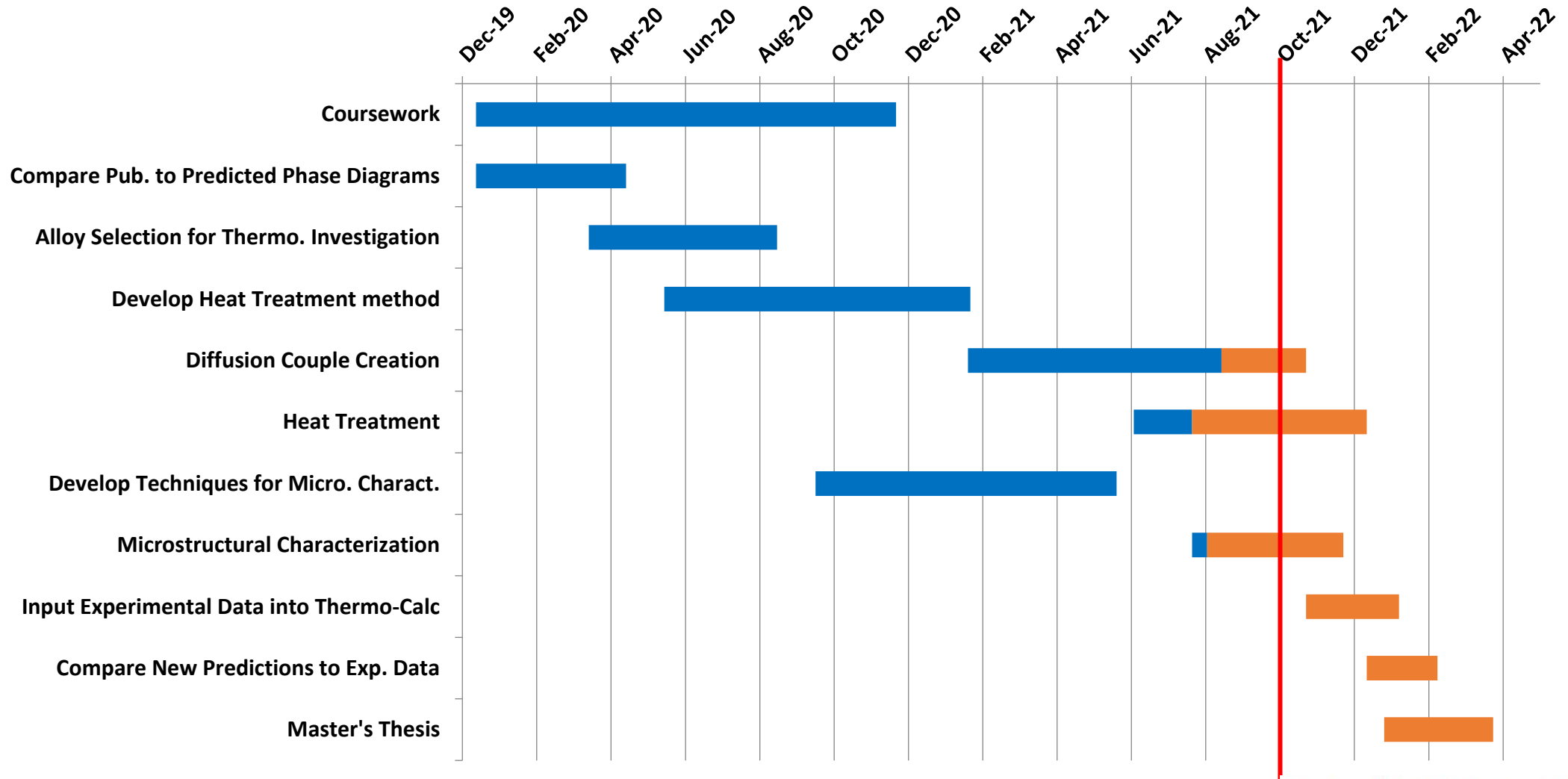


Moving Forward



- Heat treatments
 - Continue heat treatments in hot press
 - Trial heat treatment in Mellen furnace
 - If successful, begin long term heat treatment (6 weeks = 1000 hours)
 - Send samples to Michigan Tech?
- Post heat treatment
 - Assess diffusion lengths
 - Characterize microstructure
 - Assess phase stability
 - If multiple phases are found, additional samples will be made and heat treated
- After characterization
 - Input data into Thermo-Calc database

Gantt Chart



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Challenges & Opportunities



- Challenges so far
 - Heat treatments
 - Hot press had vacuum issues that took multiple weeks to fix
 - Finding furnace for long term heat treatments
 - Finding furnace capable of 2000 °C and available for >100 hours
 - Learning how to input experimental data into Thermo-Calc database
 - Juggling a newborn and research responsibilities
- Opportunities
 - Developing process for heat treating oxygen sensitive materials in open air furnace
 - Learning a new “language”
 - Spending a lot of time with my daughter

Thank you!
Bobby Puerling
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