Project 60: Fundamentals of Recrystallization in Binary Nb Alloys

Semi-annual Spring Meeting
April 2022

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Project 60: Fundamentals of Recrystallization in Binary Nb Alloys

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CENTER FOR ADVANCED NON-FERROUS STRUCTURAL ALLOYS

- Student: Will Waliser (Mines)
- Advisor(s): Amy Clarke, Kester Clarke (Mines)

Project Duration
MS: September 2021 to May 2023

- Problem: Hf additions have been shown to increase recrystallization temperatures in Nb alloys, but alternatives that produce similar effects have not yet been identified.
- Objective: Identify binary and/or ternary Nb alloys of interest and experimentally measure recrystallization parameters and microstructural evolution with thermomechanical processing.
- Benefit: Improved performance of superconductors and/or refractory multi-principal element alloys (RMPEAs).

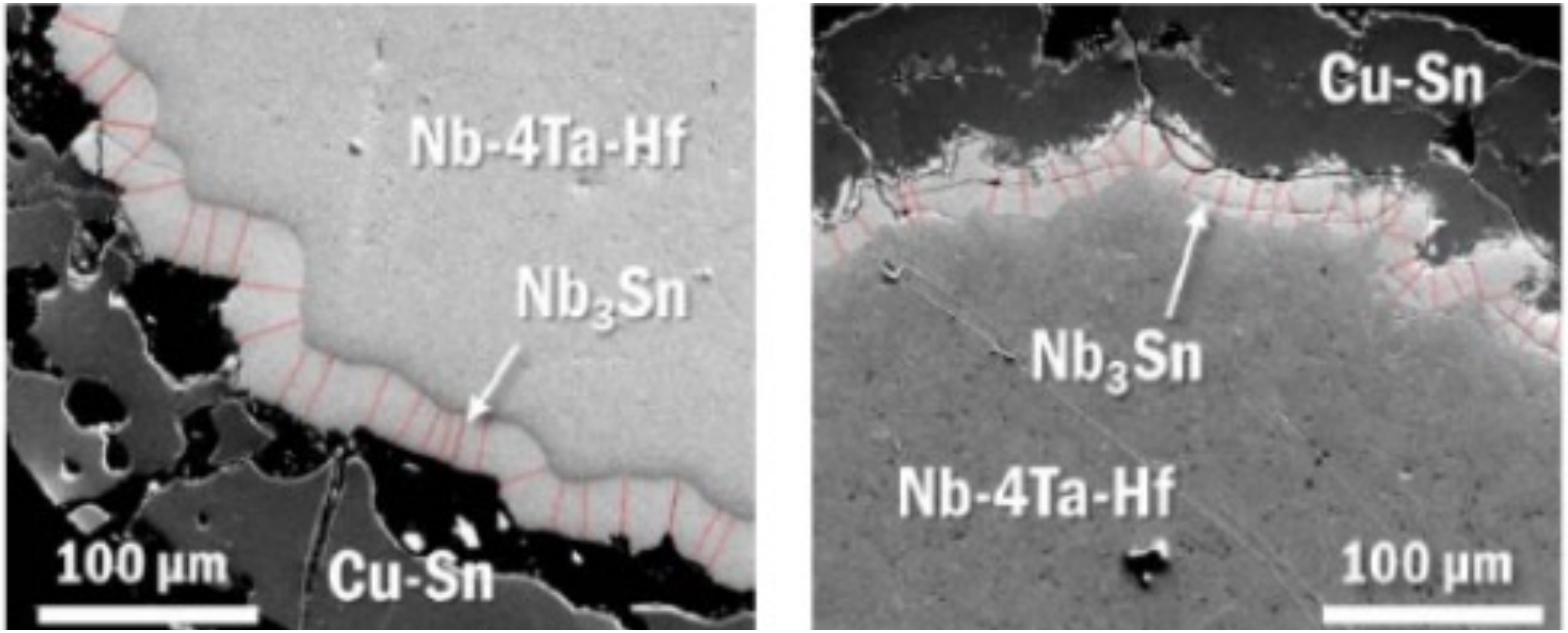
Recent Progress

- Equipment training complete
- Alloy compositions determined
- Materials for producing Ti, Zr, Hf, V, Nb, Ta alloys arrived
  - Remaining materials arriving soon (Mo, W, Re)
- Heat treatment plan for alloy samples has been designed

Metrics

Description	% Complete	Status
1. Literature review	60%	●
2. Select and obtain/produce binary and/or ternary Nb alloys	30%	●
3. Thermomechanical processing and heat treatment of as-cast materials	0%	●
4. Assess recrystallization temperatures and kinetics via heat treatment experiments	0%	●
5. Microstructural characterization of thermomechanically processed samples	0%	●

Industrial Relevance



(a) without intermediate annealing and (b) with intermediate annealing at 1010 ºC/5 h.

[1] Banno, Nobuya, et al Scripta Materialia 199 (2021): 113822.

Superconductor manufacturing

- Nb-Ta-Hf base alloy
  - Wire drawn
- Cu-Sn coating
- Diffusion reaction @ interface
  - 600-800 °C for ~100 h
  - Formation of Nb_3Sn superconductor phase

Literature Review & Element Selection

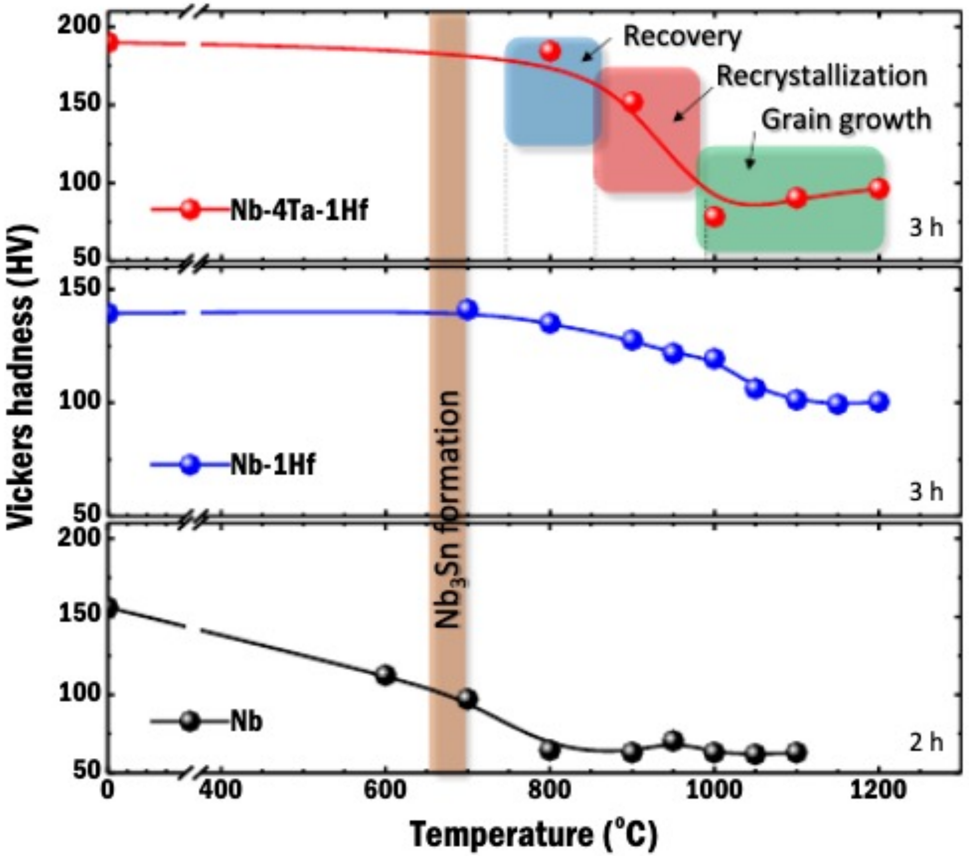


Fig. 2. Vickers hardness of Nb–4at%Ta–1at%Hf as a function of annealing temperature as well as Nb as a reference.

[1] Banno, Nobuya, et al Scripta Materialia 199 (2021): 113822.

Hf effects on Nb microstructure:

- Solid solution strengthening (SSS) and oxide pinning
- Stabilization of grain growth kinetics during recrystallization (Rx)
  - Stabilize heavily worked structures (i.e. Nb wires)
- Increased grain boundary (GB) diffusion paths for Sn into Nb-base alloy during Nb_3Sn reaction
  - Finer grain morphology in final superconducting phase
  - Increased flux pinning -> increased superconductor performance!

Alloying element selection process

- Good solubility
  - Similar strengthening effects
- Minimal loss of workability
- Cost effective

Literature Review & Element Selection

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- 1 wt.% Zr shown to increase Rx temperatures by 100 °C [2]
  - SS interactions and applying dragging forces to high angle GBs
  - No significant effect on work hardening behavior
- Elements with high thermodynamic activity such as V, Zr, and Ti shown to increase Rx temperature of Nb due to the formation of refractory nonmetallic compounds inhibiting Rx [3]
- Re found to be an effective inhibitor of Rx in BCC refractory metals [3]
- Refractory elements such as Mo, W, Ta all have 100% solubility in Nb
  - Well documented SSS effects [3]
  - Sluggish diffusion compared to V, Zr, and Ti, likely to reduce workability of alloy [4]
- Alloying elements selected for initial testing: Ti, Zr, Hf, V, Ta, Mo, W, Re

Literature Review & Composition Selection

Alloy	Maximum amount used	Initial comp to be tested
Nb-Ti	5 wt.% General Electric Cb-2	~2-3 wt.%
Nb-Zr	2.5 wt.% Pratt & Whitney Cb132/Cb132M	~2 wt.%
Nb-Hf	2 wt.% Westinghouse B-88	~2 wt.%
Nb-V	5wt.% Westinghouse B-33/B-66	~5 wt.%
Nb		~100 wt.%
Nb-Ta	100 wt%	~5 wt.%
Nb-Mo	100 wt%	~5 wt.%
Nb-W	100 wt%	~5 wt.%
Nb-Re	~55 wt%	~2 wt.%

- Compositions decided by:
  - Solubility limitations
  - Current industry practices for commonly used Nb alloys [5]
  - Current industry practices for base Nb alloys for superconductor manufacturing
- Maximum amount used: composition/manufacturer/alloy
- Compositions may be subject to change upon initial testing
  - Feedback welcome!

Materials Acquired

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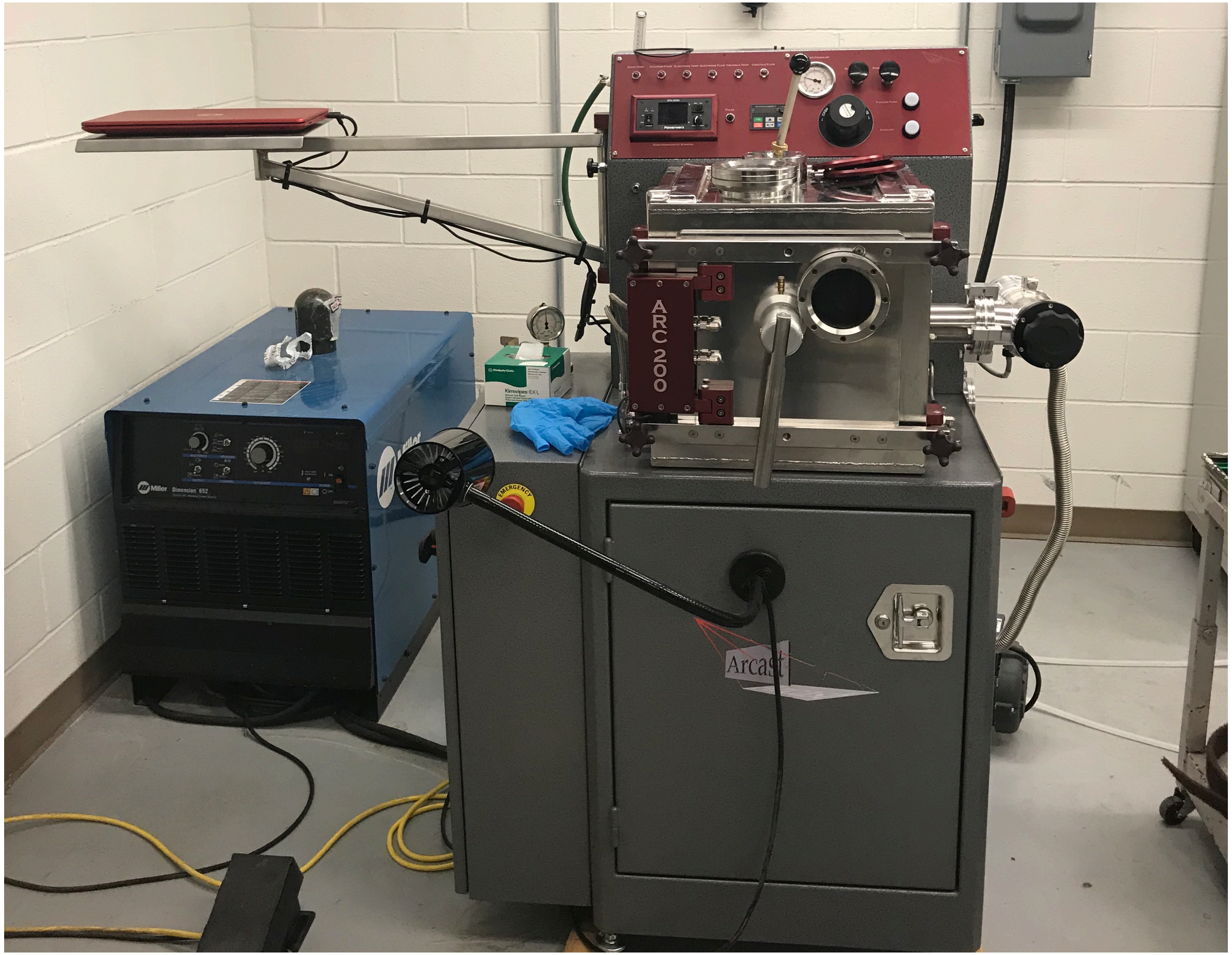
Alloys	Nb-2Ti	Nb-2Zr	Nb-2Hf	Nb-5V	Pure Nb	Nb-5Ta	Nb-5Mo	Nb-5W	Nb-2Re
Parts In stock	X	X	X	X	X				
Parts to be shipped						X	X	X	X

- Materials acquired:
  - Nb chips
  - Hf chips
  - V chips
  - Nb50Zr chips
  - Nb47Ti rod
  - Nb7.5Ta rod
- To be obtained:
  - Nb-Mo, Nb-W, Nb-Re parent alloys
  - Ta chips
- Thank you to **ATI** for the materials!

Manufacturing Alloys



- Arc Melter
  - Vacuum or argon backfill
  - Casting shapes
    - Button
    - Plate
  - Waiting for results of chemical purity before further manufacturing
- Samples to be analyzed using light optical microscopy (LOM)
  - Micro-segregation
  - Cracking
  - Porosity

Deformation Testing: Gleeble

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- Initial deformation to take place in Gleeble
  - Compression test at elevated temperature
    - Workability and flow stress
    - Results will provide a basis for rolling temperatures
- Samples to be analyzed using LOM
- If alloy passes Gleeble testing and LOM analysis, safe for rolling mill

Deformation Testing: Rolling Mill

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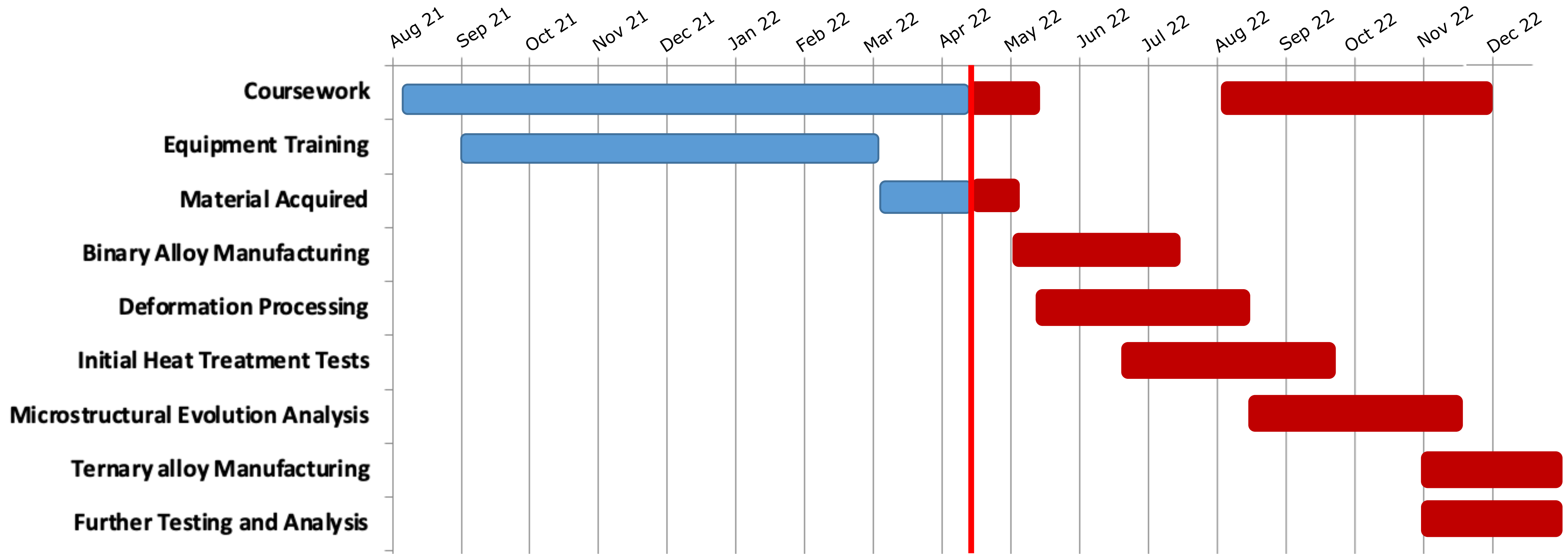
- Samples will be reduced 60% via “warm” rolling
  - Rolling mill capable of 1200 °C hot rolling
    - Rx temperature for Nb ranges between .3-.5 T_m [6]
    - Rolling likely below 745 °C
  - Increments of 20% reduction
    - Hardness measurements to document work hardening behavior
- Fracture during rolling?
  - Workability requirement failed
    - Alloy and element discarded from further testing

Heat treatment plan

Sample: deformation	Nb-Ti 60%	Nb-Zr: 60%	Nb-Hf: 60%	Nb-V 60%	Nb: 60%	Nb-Ta: 60%	Nb-Mo: 60%	Nb-W: 60%	Nb-Re: 60%
Heat treatment #1 (°C, h)	990, 1	990, 1	990, 1	990, 1	990, 1	990, 1	990, 1	990, 1	990, 1
Heat treatment #2 (°C, h)	1050, 1	1050, 1	1075, 1	1050, 1	1100, 1	1100, 1	1100, 1	1100, 1	1100, 1
Heat treatment #3 (ºC, h)	990, 2	990, 2	990, 2	990, 2	990, 2	990, 2	990, 2	990, 2	990, 2

- Static Rx in Ar backfill furnace
- Conservative number of tests to keep the amount of samples manageable
- Standard heat treatment of 990 °C (0.4$T_{m\ Nb}$) for 1 h
- Secondary heat treatment temperatures chosen based on ThermoCalc simulations for alloy melting temperature:
  - 1050 °C (~0.42$T_{m\ Nb}$) → predicted melting temps < 2400 °C
  - 1075 °C (~0.43$T_{m\ Nb}$) → 2400 °C < predicted melting temps < 2477 °C
  - 1100 °C (~0.44$T_{m\ Nb}$) → predicted melting temps > 2477 °C
  - Adjust for differences in diffusion rates for alloying elements
- Subject to change upon testing, input welcome!

GANTT Chart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Coursework
Equipment Training
Material Acquired
Binary Alloy Manufacturing
Deformation Processing
Initial Heat Treatment Tests
Microstructural Evolution Analysis
Ternary alloy Manufacturing
Further Testing and Analysis

Challenges & Opportunities

- Manufacturing alloy samples using the arc melter
  - Remaining materials for Ta, W, Mo, Re alloys soon to be acquired
- Deformation testing
  - Initial testing in Gleeble to gather information on appropriate rolling parameters
  - LOM analysis of samples
  - Reduce samples 60% in rolling mill via hot rolling
    - Work hardening behavior recorded
    - Workability problems?
- Heat treat samples
  - Document microstructural evolution using EBSD and SEM techniques

Thank you!

Will Waliser

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References

[1] Banno, Nobuya, et al. "Influence of parent Nb-alloy grain morphology on the layer formation of Nb3Sn and its flux pinning characteristics." Scripta Materialia 199 (2021): 113822.

[2] Siciliano, Fulvio & Monteiro, Waldemar & Padilha, Angelo. (1995). Comparative study of the recrystallization of pure niobium and a Nb-1 wt.% Zr alloy. 86. 713-718.

[3] Savitskii, 1970, Physical Metallurgy Of Refractory Metals and Alloys

[4] Prokoshkin, D.A., Vasil'eva, E.V. & Vergasova, L.L. Interdiffusion of niobium and some refractory metals. *Met Sci Heat Treat* **9,** 199–201 (1967). https://doi.org/10.1007/BF00653142

[5]Frank, R. G. (1968). Recent advances in columbium alloys. *Refractory Metal Alloys Metallurgy and Technology*, 325–372. https://doi.org/10.1007/978-1-4684-9120-3_9

[6] Wilkinson, W. D. (1969). Chapter 8. In Properties of refractory metals, Gordon and Breach Science, pp. 304–307.