Microstructure evolution and mechanical properties of cold rolled C-103 sheets annealed under different conditions

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Introduction

Niobium C-103 material in the form of sheets are used for the realization of the nozzle divergent section of the rocket engine through metal forming process. Due to unavailability of required larger size sheets, smaller and thicker plate has been imported and developed technology indigenously to cold rolled to desired size to meet strategic requirement of ISRO program. During this process, in order to remove the cold rolling effects, the material needs to be annealed at a high temperature to obtain desired microstructure and mechanical properties [1]. The cold rolled sheets were subjected to annealing at temperatures of 1100, 1200, and 1300 ℃ for 1, 2, and 4 hours each and subsequently characterized through extensive mechanical testing & microstructural characterization to arrive at an optimum combination of temperature and time to achieve desired properties.

Objective

- To study the effect of vacuum annealing temperature and time on microstructural and mechanical behavior of cold rolled C-103 material and understand the recrystallization behavior of material
- Identify the optimum vacuum annealing parameters to obtain YS of 248 MPa, UTS of 372 MPa, % El of 30 %, with grain size of ASTM no 6 or finer.

Material and Method

Table 1: Chemical Composition of C-103 Rolled Sheets in wt. %

Hf	Zr	Ti	Ta	W	N	O	C	H	Nb
9.46	0.02	0.85	0.22	0.21	0.0005	0.012	0.007	0.0003	Bal

Experimental Details

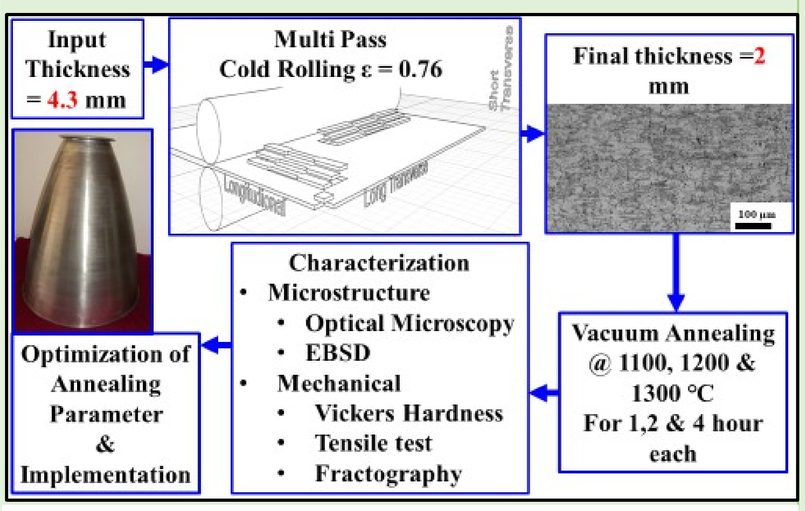

Fig 1: Schematic of experimental plan

Results & Discussions

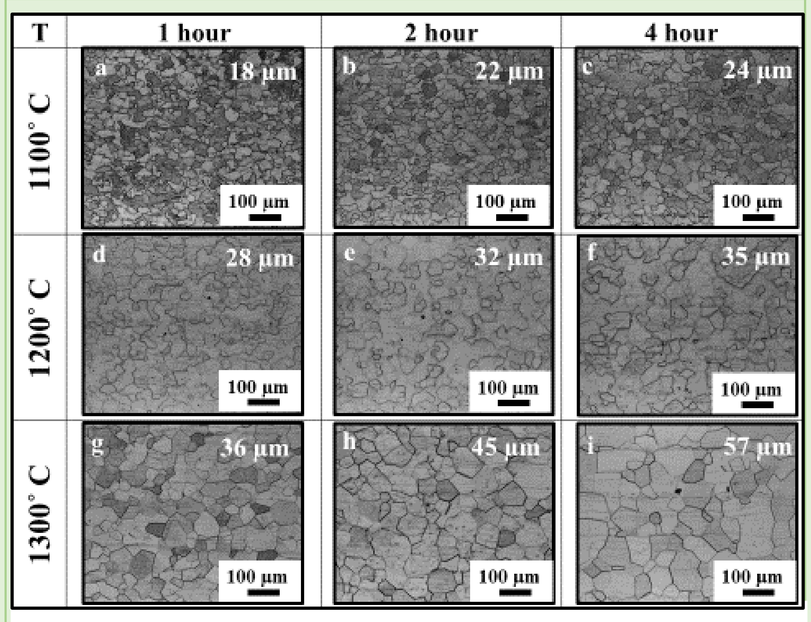

Fig 2: Optical microstructure of cold rolled C-103 sheet after vacuum annealing at varying temperature and time

Vickers Hardness and Grain Growth Kinetics

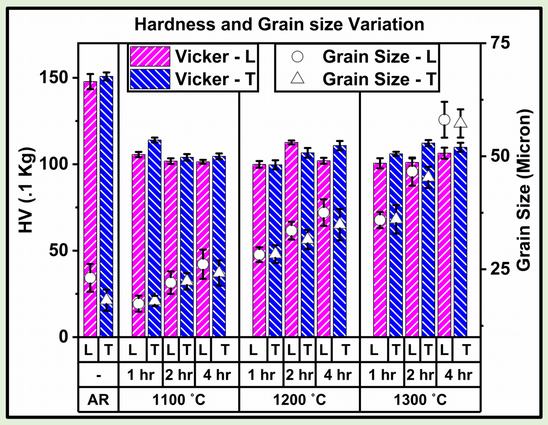

Fig 3. Vickers hardness and grain size variation

- As Rolled (AR) sheet exhibit highest hardness.
- With annealing treatment cold rolling effect has been eliminated and observed with decrease in hardness values. .
- Recrystallized grains increases with annealing temperature and time indicative of removal of cold rolled effects.

Table 2. Grain Kinetics constant

Temp	n	k	R^2
1100	0.21	7.7	0.93
1200	0.14	15.4	0.99
1300	0.33	9.2	1.0

$$D = kt^n$$ [2]

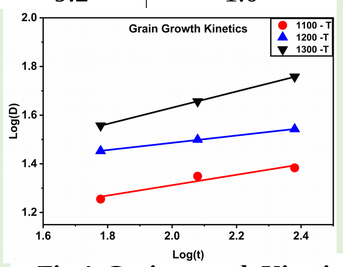

Fig 4. Grain growth Kinetics

Tensile Properties

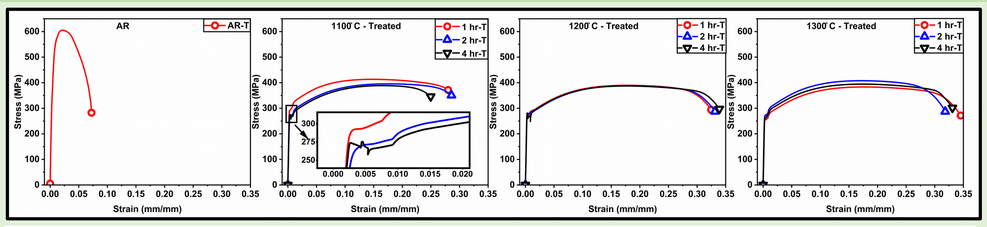

Fig 5. Stress Strain curve in transverse direction with different treatment conditions

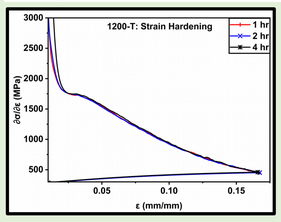

Fig 6. Typical Strain hardening curve in transverse direction at 1200 °C

Table 3. True strain at UTS

ε =	1 hour	2 hour	4 hour
1100 ℃	0.14	0.16	0.15
1200 ℃	0.16	0.16	0.16
1300 ℃	0.16	0.16	0.16

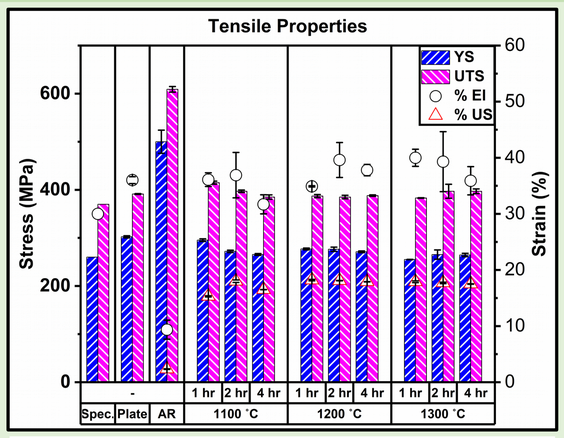

Fig 7. Tensile properties in Transverse Direction

Fig 8. Tensile tested sample

Fractography

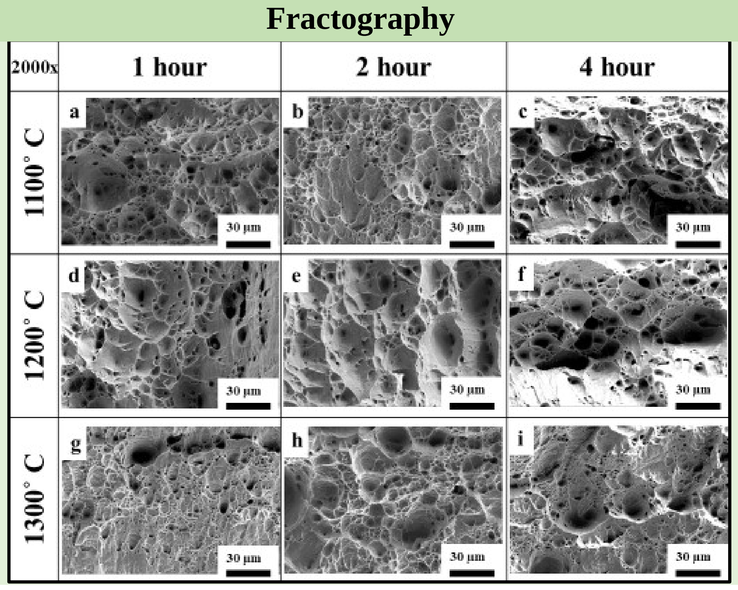

Fig 8. Fractography images of heat-treated samples at 2000x

Electron Backscattered Diffraction (EBSD)

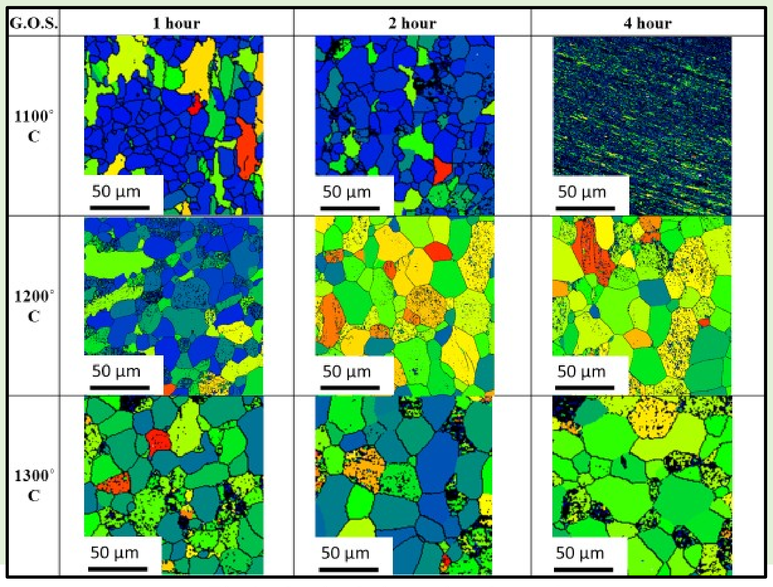

Fig 9: Grain Orientation Spread

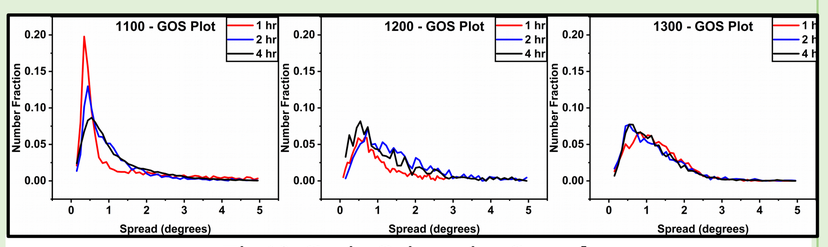

Fig 10. Grain Orientation Spread

The broad peak in G.O.S. Plot indicates a lack of preferred misorientation direction, which reflects the extent of recrystallization.

Conclusion

- Cold rolling leads to a reduction in ductility below 30 %, with raise in YS, and UTS. The effects of cold working have been mitigated by employing vacuum annealing.
- Annealing at 1100 °C shows initiation of recrystallization, however, the effect of cold rolling is not eliminated.
- More than 90 % recrystallization is achieved only after 1200 °C – 1 Hr heat treatment condition, heat treatment condition 1300 °C – 2 and 4 hr leads to grain coarsening beyond ASTM 6.
- Among heat treatment conditions that fulfill the above criteria, 1200 °C -2 and 4 hr conditions showed an identical and best balance of the mechanical property (0.2% YS >250 MPa, UTS > 372 MPa, % El > 30%).
- Thus, optimum heat treatment conditions are 1200 °C –4 hr condition.
- This finding were implemented in actual cold rolled sheets.

References

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2. A Study on the Hall–Petch Relationship and Grain Growth Kinetics in FCC-Structured High/Medium Entropy Alloys; Yung-Chien Huang, Che-Hsuan Su, Shyi-Kaan Wu and Chieh Lin; Entropy 2019, 21, 297

Acknowledgments

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