Phase Equilibria in the Nb-Si-B System

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Outline

- Introduction
- Objectives of the Nb-Si-B Project
- Literature
- Experimental Procedure
- Results and Discussion
- Summary
Need to develop materials to work under aggressive conditions (oxidant atmosphere; dynamic loading) at $T > 1200^\circ$C;

Ni-base superalloys limitation: $T_{\text{service}} \approx 0.85 \times T_{\text{melting}} \approx 1150^\circ$C;
✓ High melting point material;
✓ Microstructure: intermetallic phase(s) containing elements for good oxidation resistance in thermodynamic equilibrium with a ductile metallic phase (solid solution) => Me-Si-B systems;
✓ Possible solution should involve a multicomponent Me-Si-B system => need for phase stability information.

- Two-phase field Mo$_{ss}$+Mo$_5$SiB$_2$ ($T_2$)
  ($T_2$ - Cr$_5$B$_3$ prototype)

Mo-Si-B Isothermal Section at 1600°C.
Objectives of the Nb-Si-B Project

- Reevaluation of the Nb-B system;
- Determination of Liquidus projection (Nb-rich region);
- Determination of the isothermal section at 1700°C (Nb-NbSi_2-NbB_2) region;
- Thermodynamic modeling of the Nb-Si-B system.
Nb-B Phase Diagram [1990Mas].

Nb-B Phase Diagram [1969Rud].
Nb-Si System

**Nb-Si Phase Diagram [1993Sch].**

- **1765°C [1980Koc]**
- **1670°C [1991Men]**

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**Literature**

1. [Koc, 1980]
2. [Men, 1991]
3. [Sch, 1993]
B-Si System

B-Si Phase Diagram [1990Mas]
Isothermal Section of the Nb-Si-B system at 1600°C [1960Now]

\textbf{Nb}_5\textbf{SiB}_2 crystal structure
Structural materials: metal–silicon–Boron. The Nb-rich corner of the Nb–Si–B system

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Sub-Solidus projection of the Nb-Si-B system in the Nb-Nb$_5$Si$_3$-NbB region

(1) single-phase; (2) two-phase; (3) three-phases.
 Nb-Si-B System

Structural materials: metal–silicon–Boron. The Nb-rich corner of the Nb–Si–B system

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Vertical section connecting Nb₉₉Si₁ to Nb₅Si₂B
Experimental Procedure

Alloys processing:

Arc Melting
Heat-treatment (1500 – 1800°C)

* Some samples also processed via powder metallurgy
Microstructural Characterization

XRD: powder, room temperature, filtered Cu-Kα radiation; 40kV; 30 mA; (2θ) from 10° to 90°; 0.05° angular step; 2 s/point;

SEM: Back-scaterred electrons image.

Microanalysis via WDS – PET (Nb), TAP (Si), LSM200 (B)
Results: Nb-B System

New Data on Phase Equilibria in
the Nb-Rich Region of the Nb-B System

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Results: Nb-B System

SEM/BSE micrographs of as-cast Nb-B alloys:
(a) 48Nb-52B; (b) 45,45Nb-54,55B

L + Nb₃B₄ ⇌ Nb₅B₆
L ⇌ NbB + Nb₅B₆
Results: Nb-B System

The NbB$_2$-phase revisited: Homogeneity range, defect structure, superconductivity

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Fig. 5. Lattice parameter data of the NbB$_2$-phase from the results of this investigation (AC + HT samples) and results from the literature.

Lattice parameters of NbB$_2$-phase as a function of B contents
Results: Nb-B System

Tridimensional view of the NbB₂ crystal structure
Thermodynamic modeling of the Nb–B system

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Results: Nb-B System

Thermodynamic modeling of the Nb–Si system

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βNb₃Si₃ modeled as (Nb)₄(Nb, Si)(Si)₃

Temperature in Celsius

Mole fraction of Si
Results: Liquidus Projection

SEM/BSE micrograph of as-cast 84Nb-8Si-8B alloy
Results: Liquidus Projection

**SEM/BSE micrograph of as-cast 70Nb-10Si-20B alloy**

- **NbB primary**
- **Nb\textsubscript{ss} + T\textsubscript{2}**
- **T\textsubscript{2}**
Results: Liquidus Projection

$T_2$ primary

SEM/BSE micrograph of as-cast 70Nb-20Si-10B alloy
Results: Liquidus Projection

D$_8^8$ primary

SEM/BSE micrograph of as-cast 62Nb-25Si-13B alloy
Results: Experimental Isothermal sections

SEM/BSE micrograph of alloy # 34 (75Nb-21Si-4B) after HT at 1700°C for 16 h.

Isothermal Section of the Nb-Si-B at 1700°C from this investigation
Results: Experimental Isothermal sections

SEM/BSE micrograph of alloy #33 (80Nb-19Si-1B) after HT at 1700°C for 64 h.

Isothermal Section of the Nb-Si-B at 1700°C from this investigation
Results: Experimental Isothermal sections

$T = 1500^\circ C$

$T = 1600; 1700; 1800^\circ C$
Results: Experimental Isothermal sections

$T = 1600; 1700; 1800^\circ C$
Results: Calculated Liquidus Projection

- L ↔ D8₈ + NbB₂ + NbSi₂
- L ↔ D8₈ + Nb₃B₄ + NbSi₂
Results: Calculated Isothermal Section at 1600°C

Calculated Isothermal section at 1600°C

Isothermal Section of the Nb-Si-B system at 1600°C [1960Now]
Summary

- Experimental Part
  - Nb-B System: Confirmed results from Rudy et al. + insertion of the Nb$_5$B$_6$-phase forming from the liquid;
  - Liquidus Projection for the Nb-rich region was determined: Observation of primary solidification regions of T$_2$ and D$_8$$_8$ phases in addition to those expected from the binary systems;
  - Isothermal sections: Confirmed the general phase equilibria reported by Nowotny et al. (isothermal sections at 1600°C) and Katrych et al.; additional work needed for the Nb$_3$B$_4$-NbB$_2$-NbSi$_2$-D$_8$$_8$ region based on the experiments carried out at 1500, 1700 and 1800°C.

- Thermodynamic Modelling Part
  - Good description for the Nb-B; Nb-Si and Nb-Si-B system; additional work needed to correct the Nb$_3$B$_4$-NbB$_2$-NbSi$_2$-D$_8$$_8$ region.
THANK YOU FOR YOUR ATTENTION!!