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Sandia National Laboratories

Kinetics of Phase Transformations in Boron-containing 304L Stainless Steel

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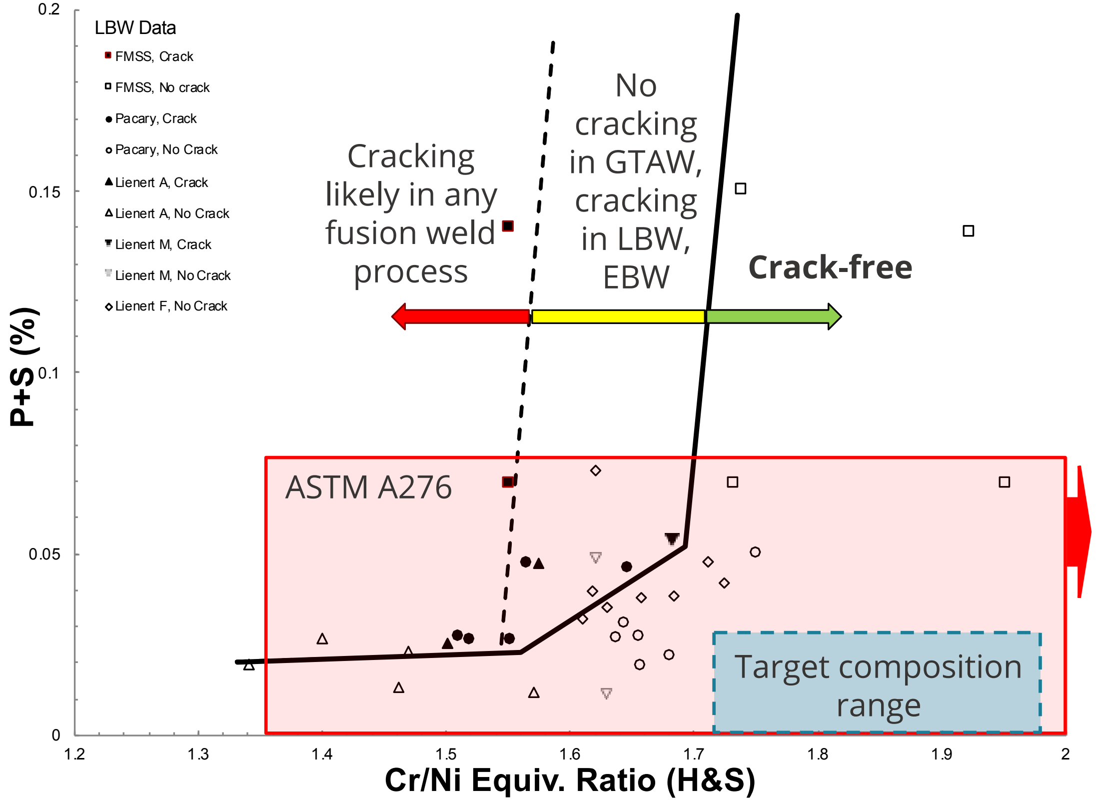
304L Stainless Steel

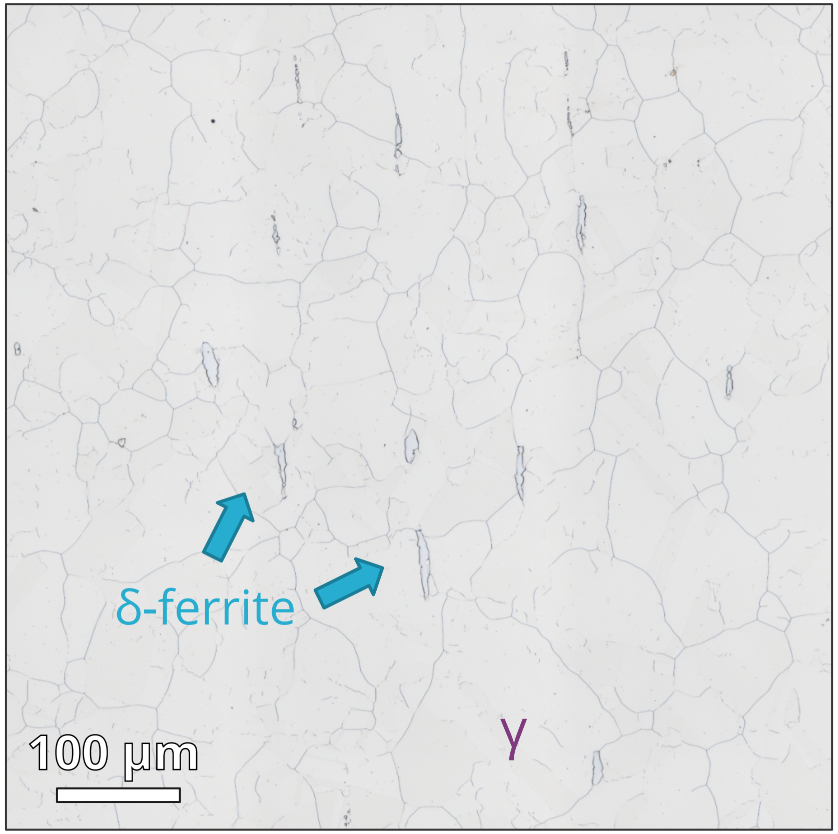
- Most commonly selected austenitic stainless steel

- Concern with laser welding – **solidification cracking**
  - –Tight restriction for impurity elements
  - –Highly controlled $(Cr/Ni)_{eq}$
  - –Secondary remelting (vacuum arc remelting, VAR)

Modified Suutala Weldability Diagram

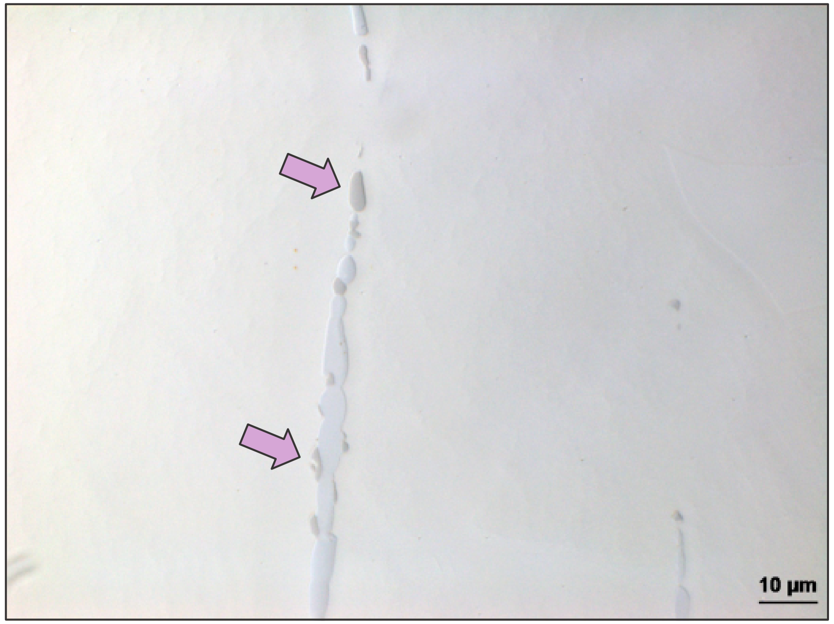


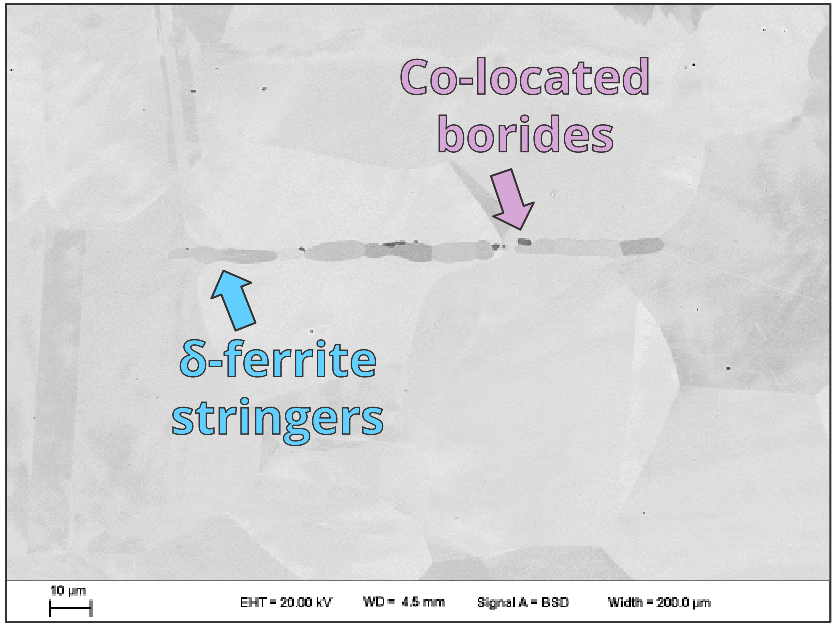
Typical 304L Microstructure



Borides identified in microstructure; raises liquation cracking concern

Inconel 718 HAZ liquation cracks; 43 ppm B



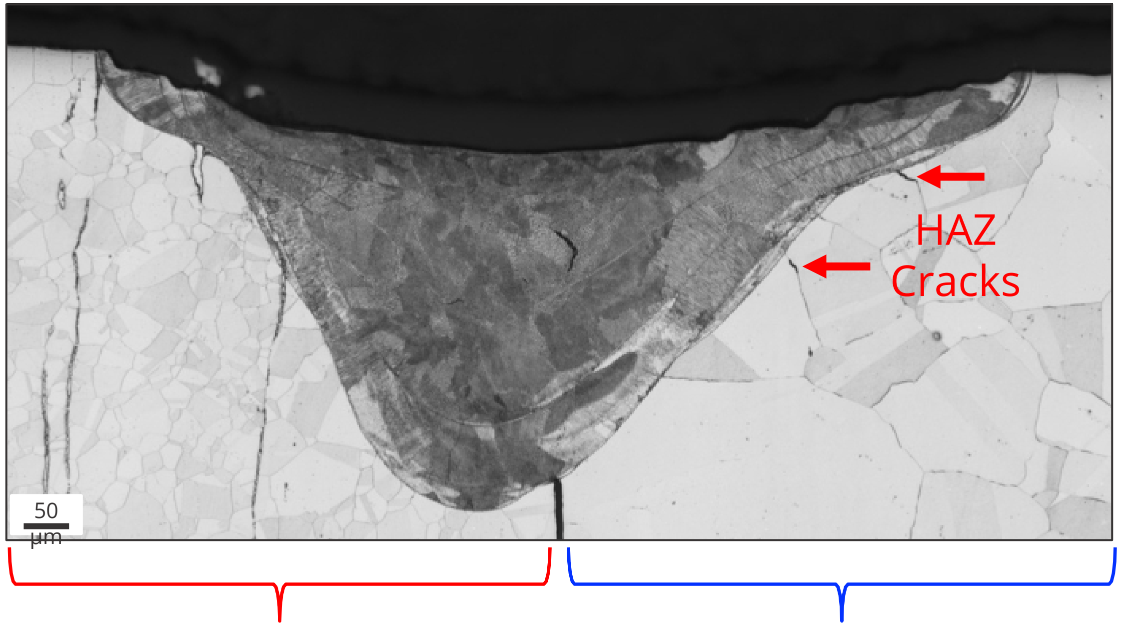


Cr-rich borides observed along δ-ferrite stringers for boron concentrations as low as 10-20 wt ppm!

Chen, W., et al. Met Mat Trans: A, Volume 32A, April 2001, 931-939.

Liquation cracking identified in heat-treated B-containing 304L

Laser welds on 304L with ~20 wt.ppm B

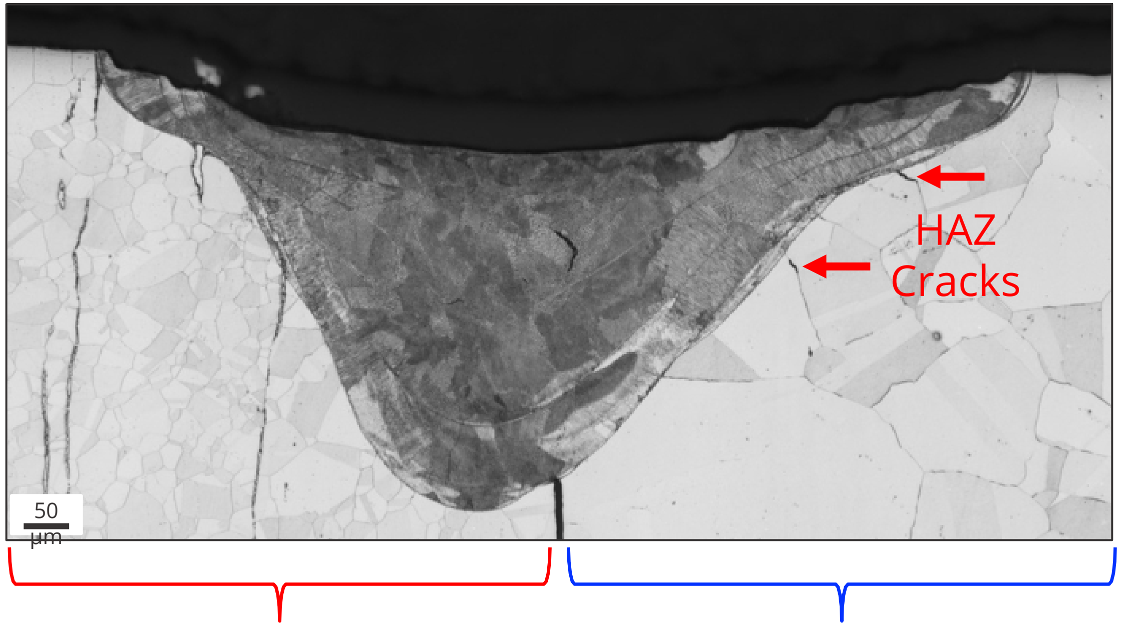


304L part (not brazed)

Brazed 304L part

Liquation cracking identified in heat-treated B-containing 304L

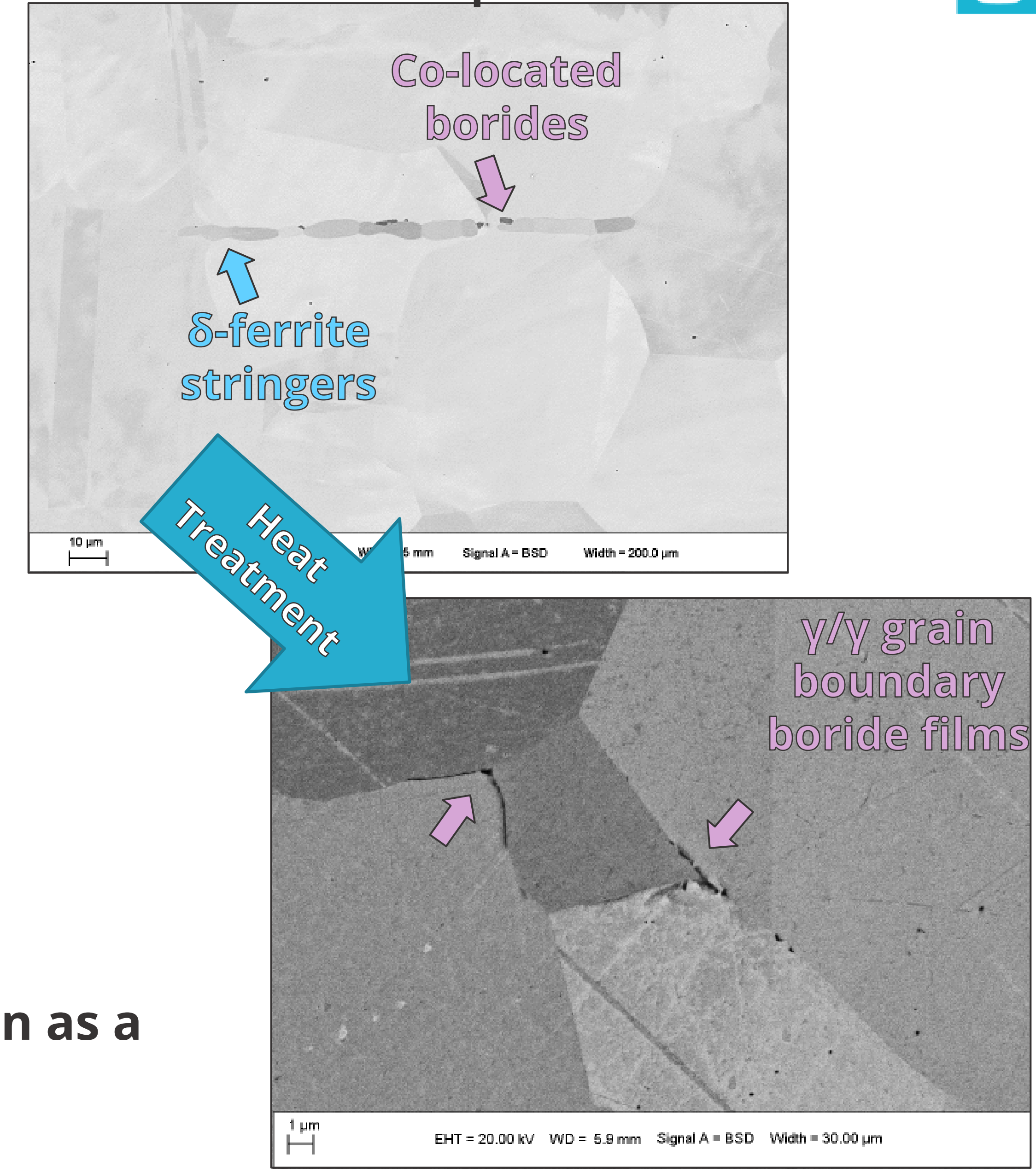
Laser welds on 304L with ~20 wt.ppm B



304L part (not brazed)

Brazed 304L part

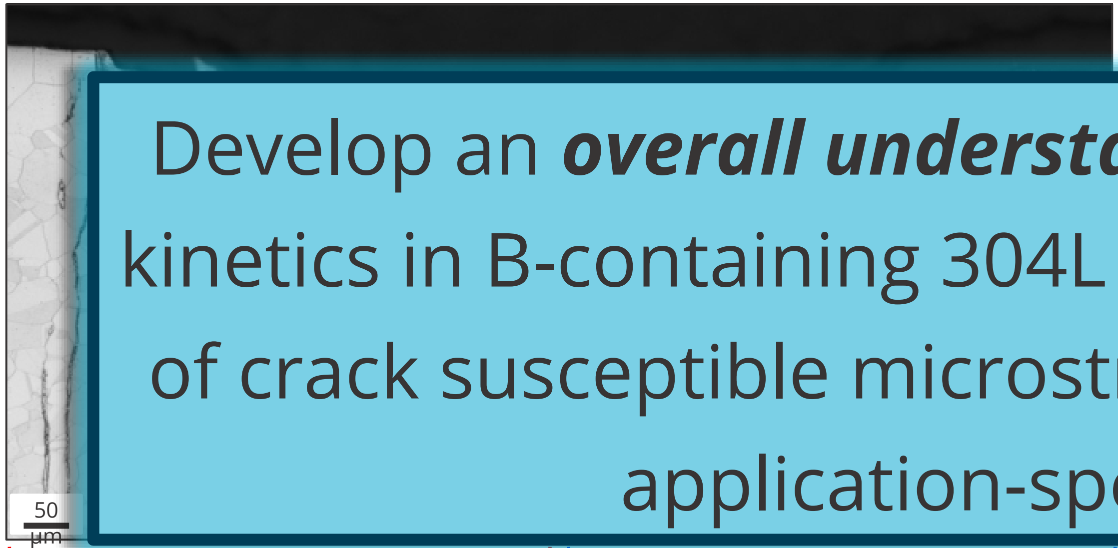
As-received microstructure – not crack susceptible



Fundamental kinetics of microstructural evolution as a function of heat treatment not understood

Liquation cracking identified in heat-treated B-containing 304L

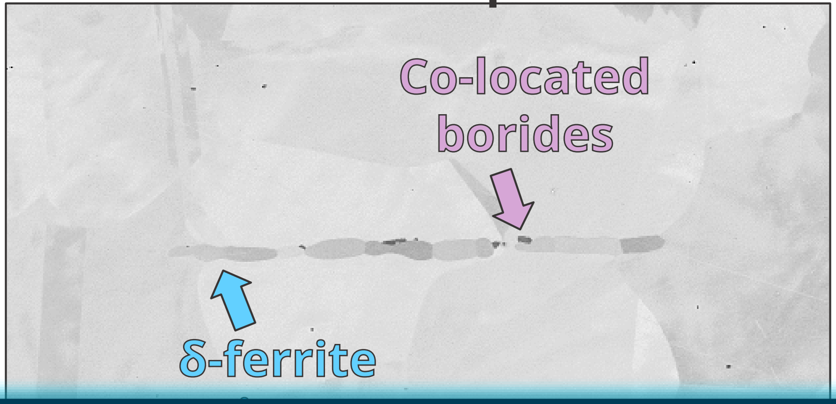
Laser welds on 304L with ~20 wt.ppm B

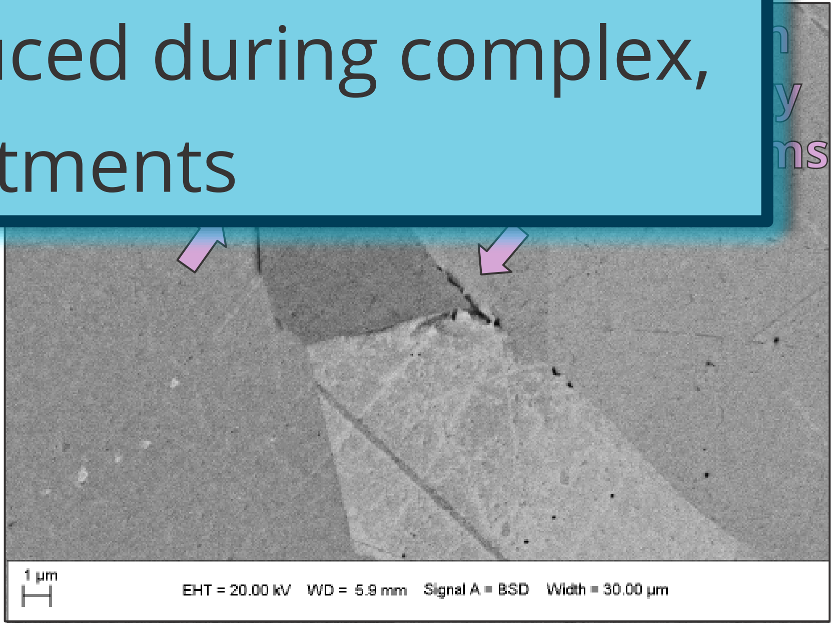


304L part (not brazed)

Brazed 304L part

As-received microstructure – not crack susceptible

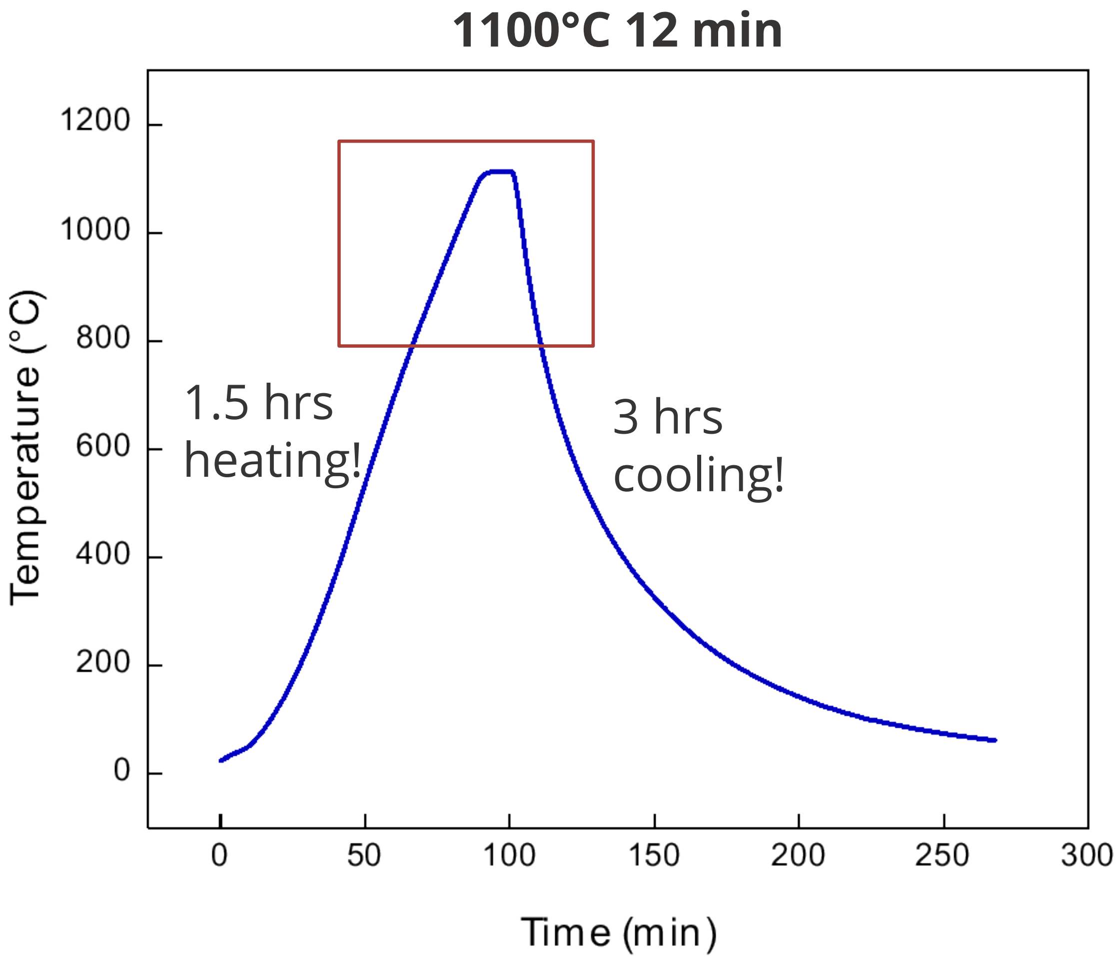


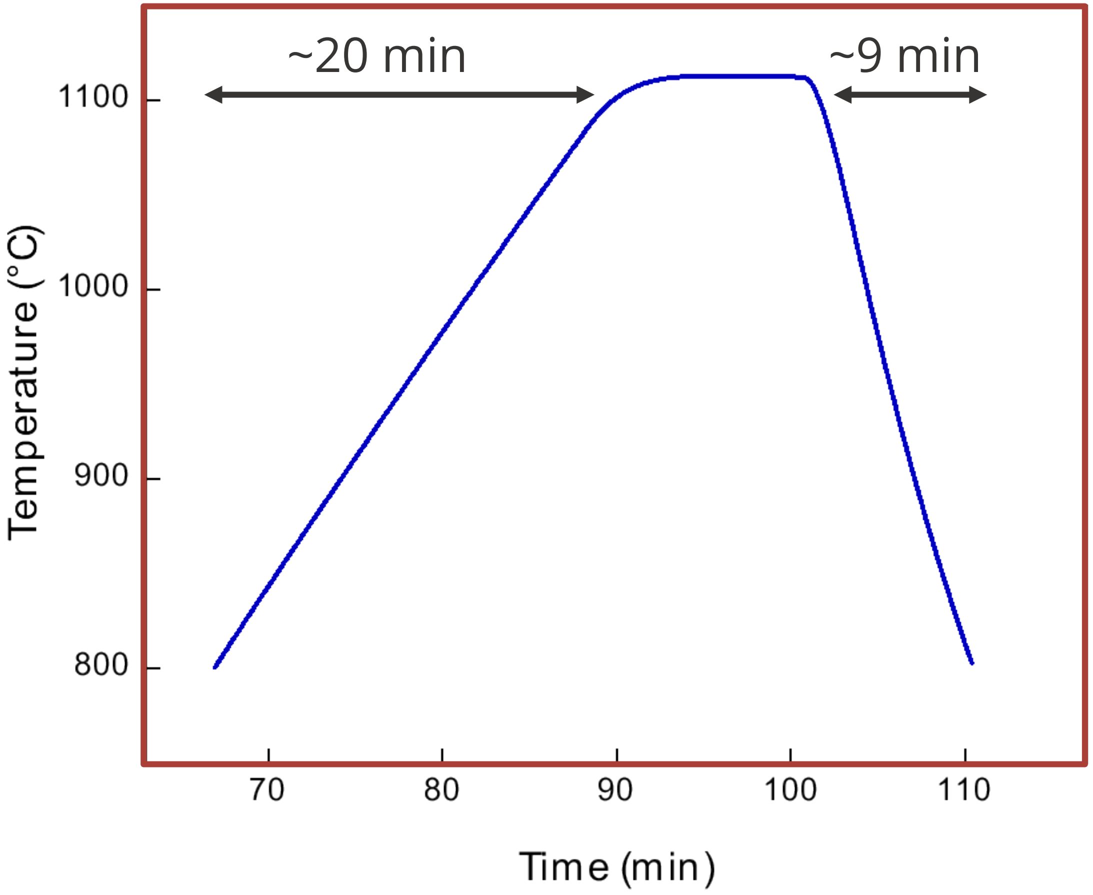


Develop an ***overall understanding*** of the phase transformation kinetics in B-containing 304L stainless steel to enable predictions of crack susceptible microstructures produced during complex, application-specific heat treatments

Fundamental kinetics of microstructural evolution as a function of heat treatment not understood

Problem: Previous weldability trials were conducted with furnace heat treatments



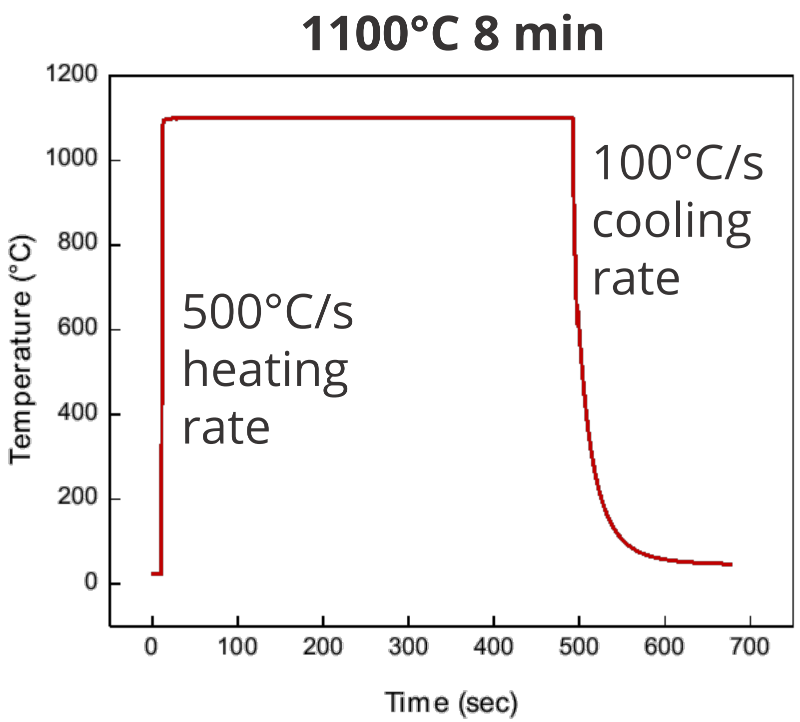


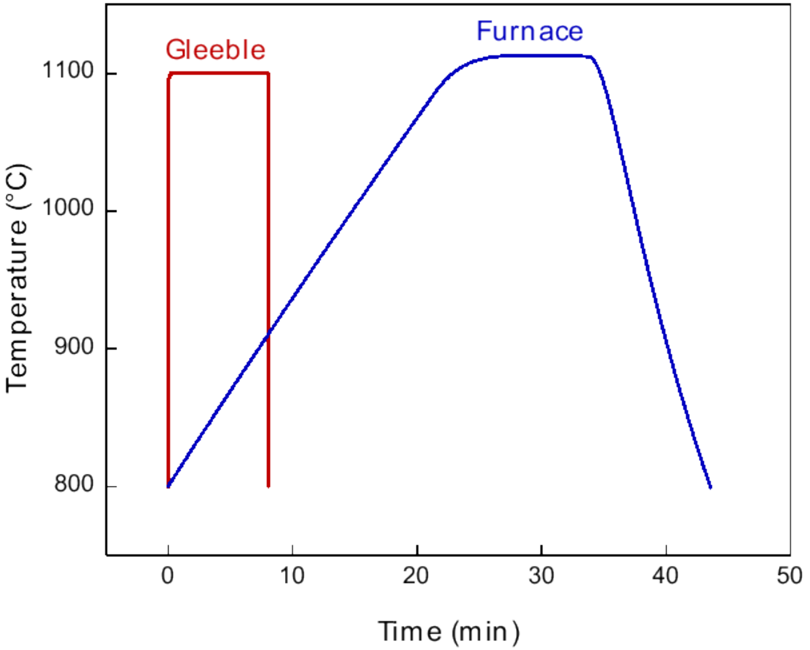
Furnace profiles were selected to replicate part-specific heat treatments

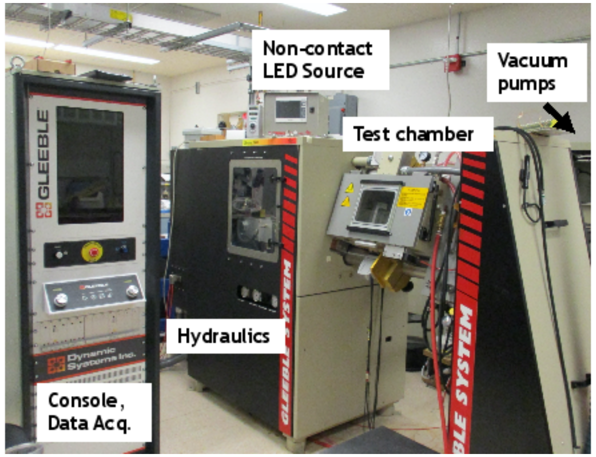
Solution: Utilize Gleeble for Isothermal Heat Treatments

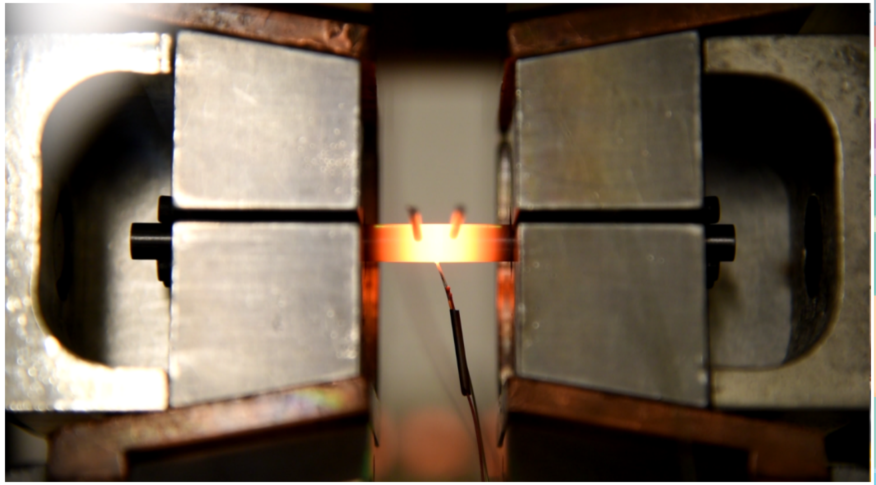
Rapid heating and cooling rates to restrict phase transformations to a single temperature

Temperatures: 1000°C, 1100°C, 1200°C, 1300°C
Hold Times: 1 min, 8 min, 32 min, 64 min
Utilized 304L composition with **~20 wt ppm B**

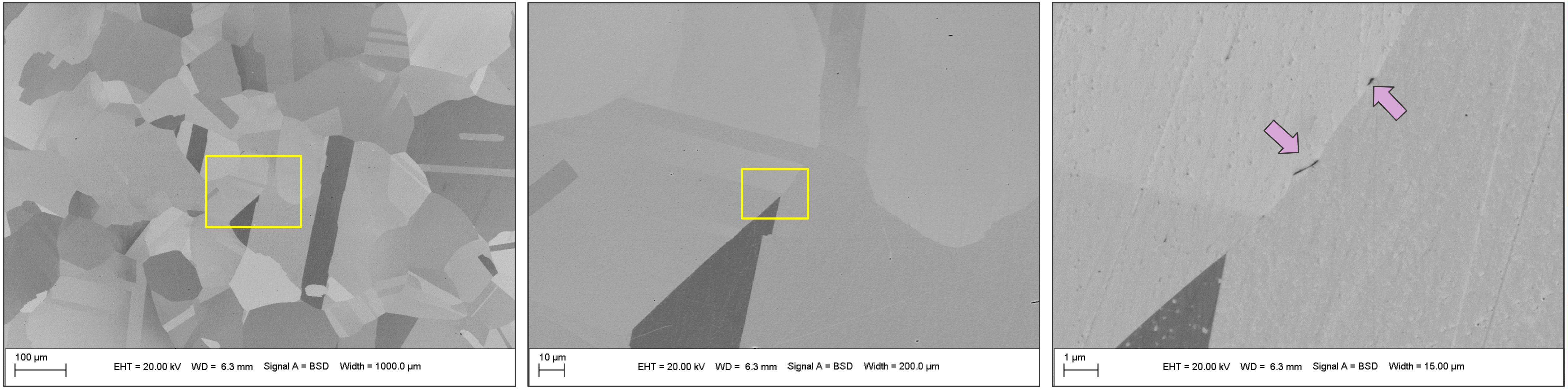






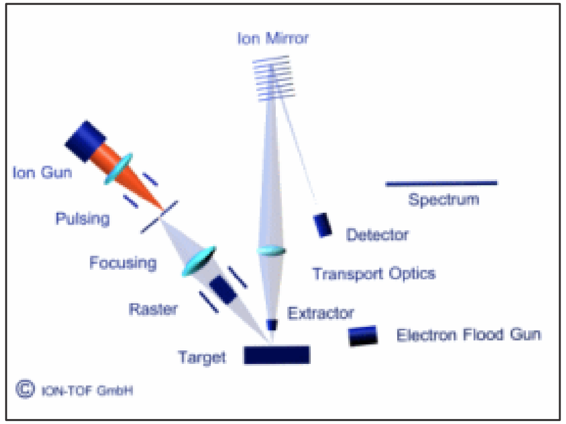


Identification of borides on γ/γ grain boundaries is challenging

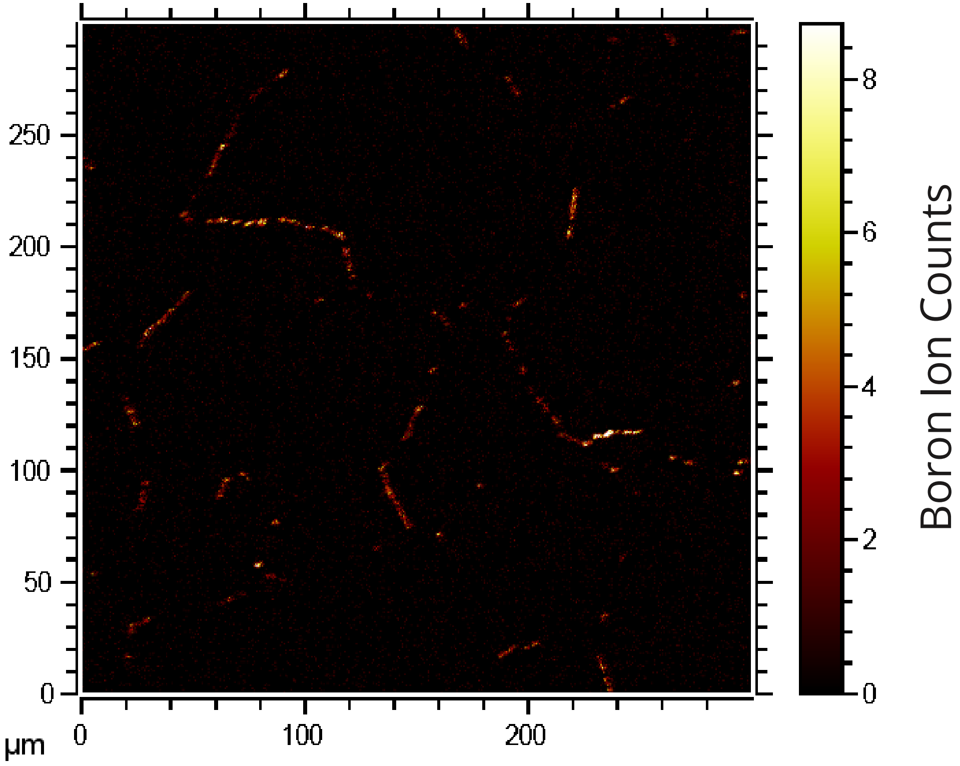


Other characterization techniques present similar challenges (e.g. WDS, TEM, etc.)

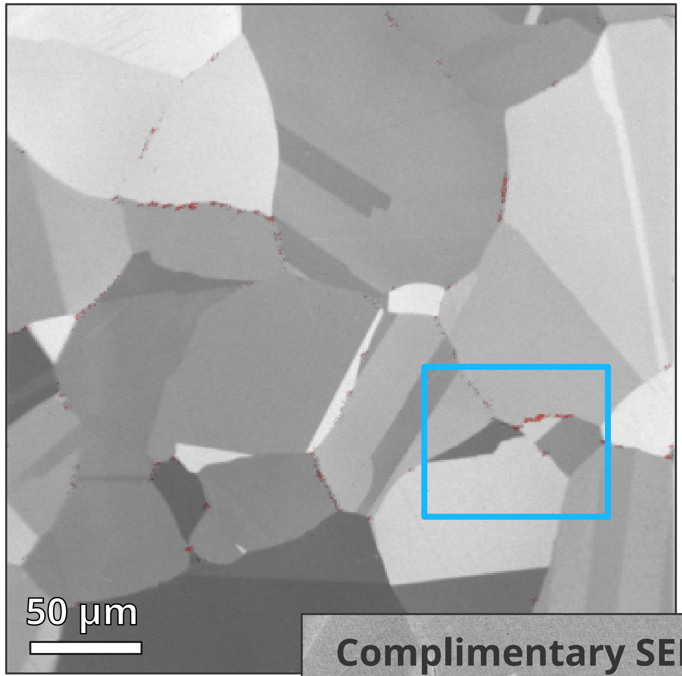
ToF-SIMS enables boron location identification

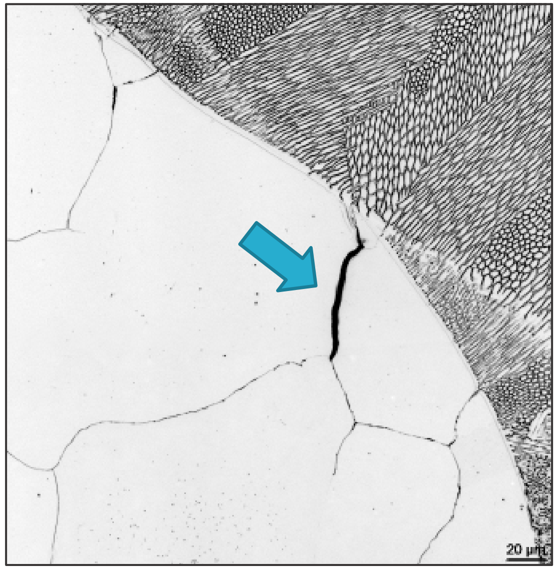


ToF-SIMS Boron Map

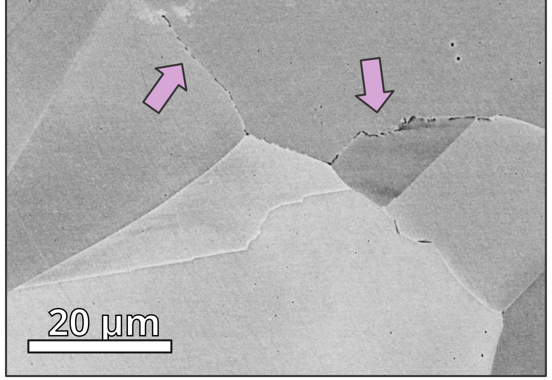


Overlaid B Map and SIMS Secondary Electron (SE) Image





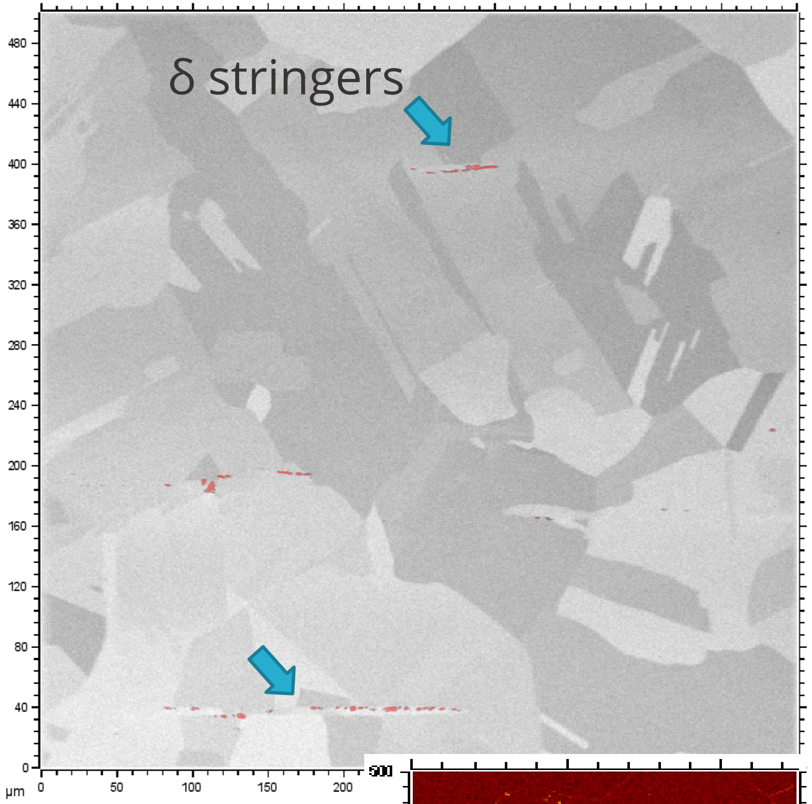
Complimentary SEM BSE

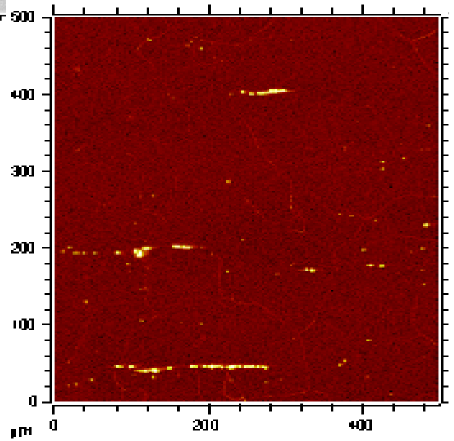


Evaluated microstructure of a crack-susceptible furnace heat treatment condition

Boride dissolution occurs between 1000°C and 1100°C

As-Received





Boride dissolution occurs between 1000°C and 1100°C

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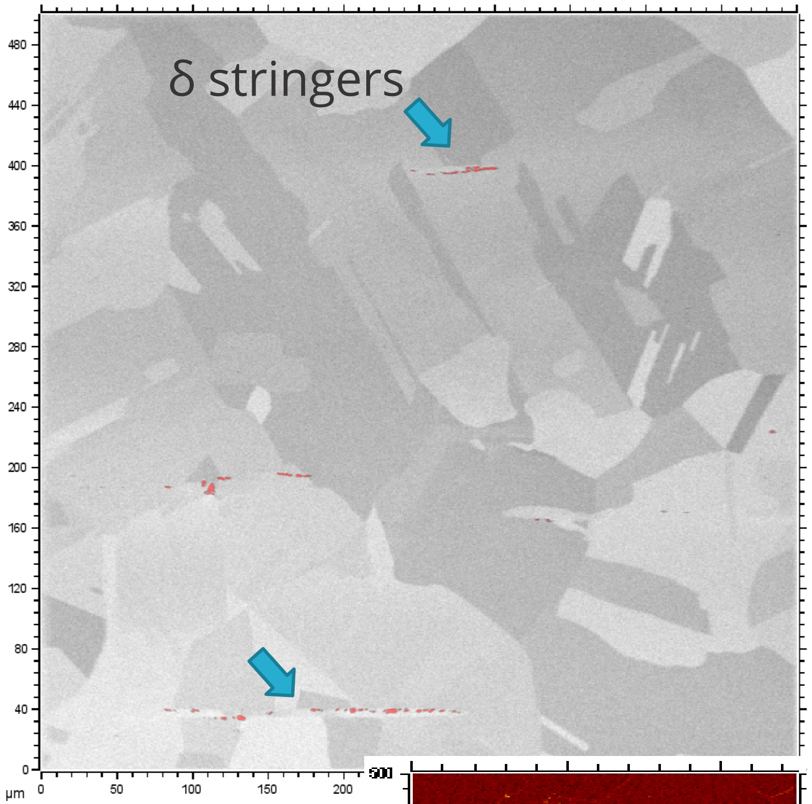
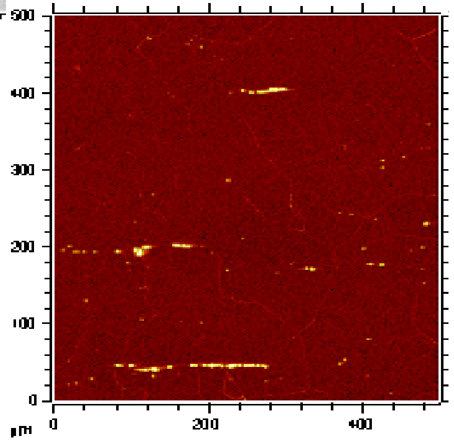
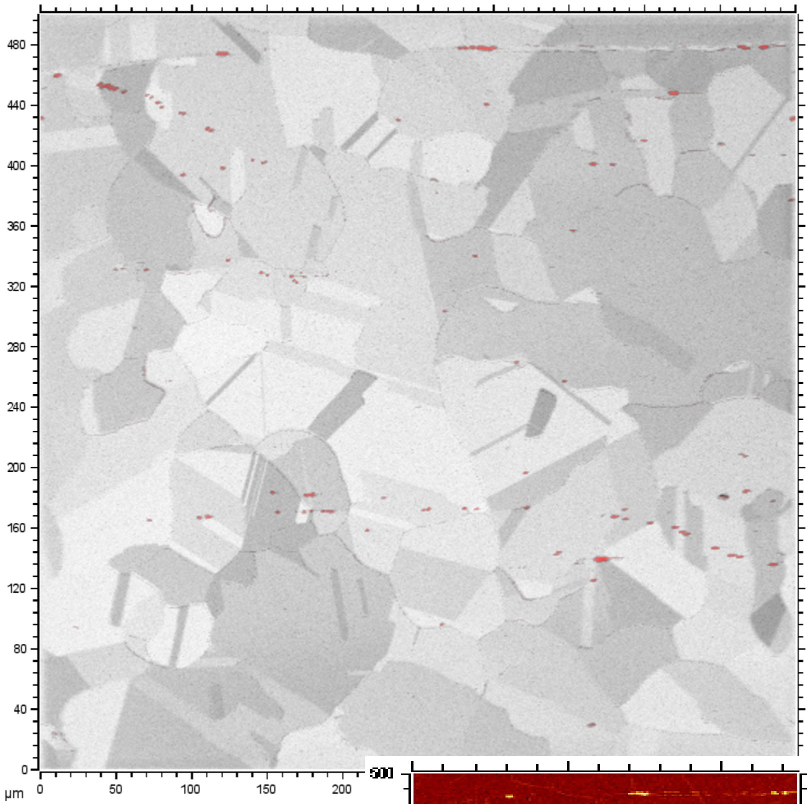
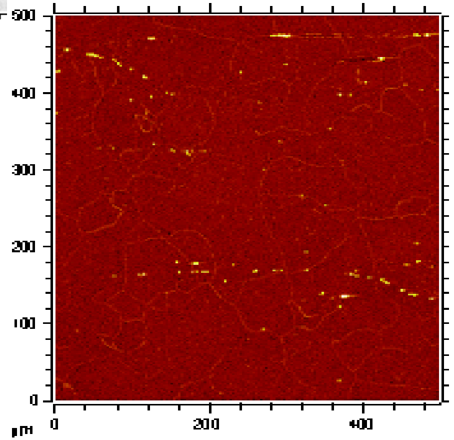




1000°C 32 min

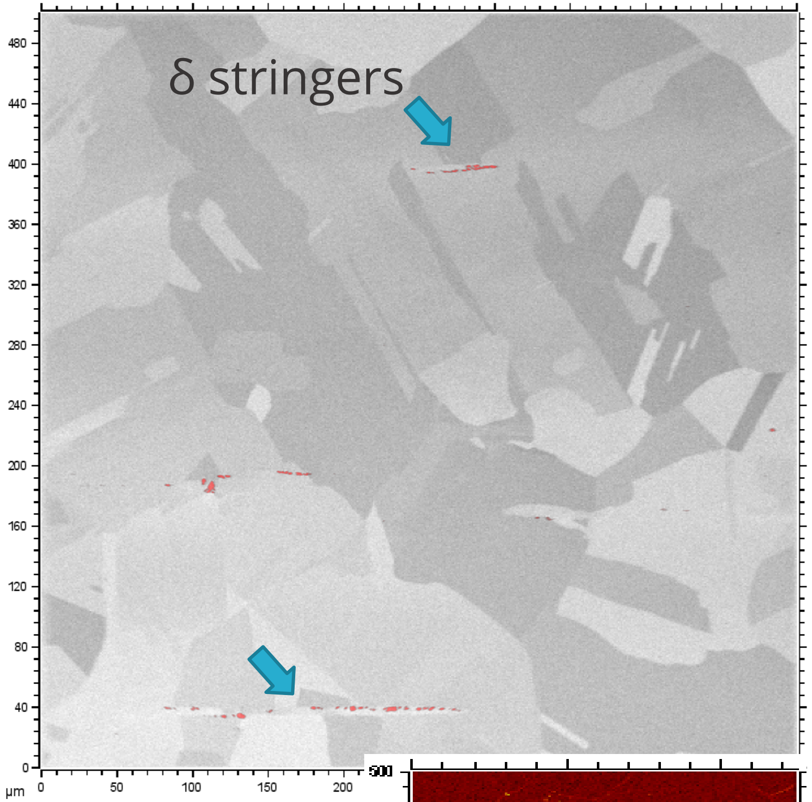


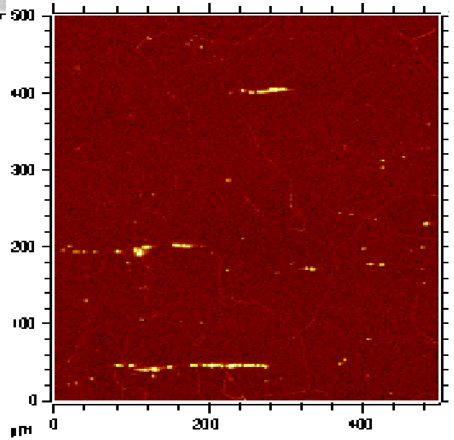
Little (if any) observable changes between as-received and 1000°C 32 min



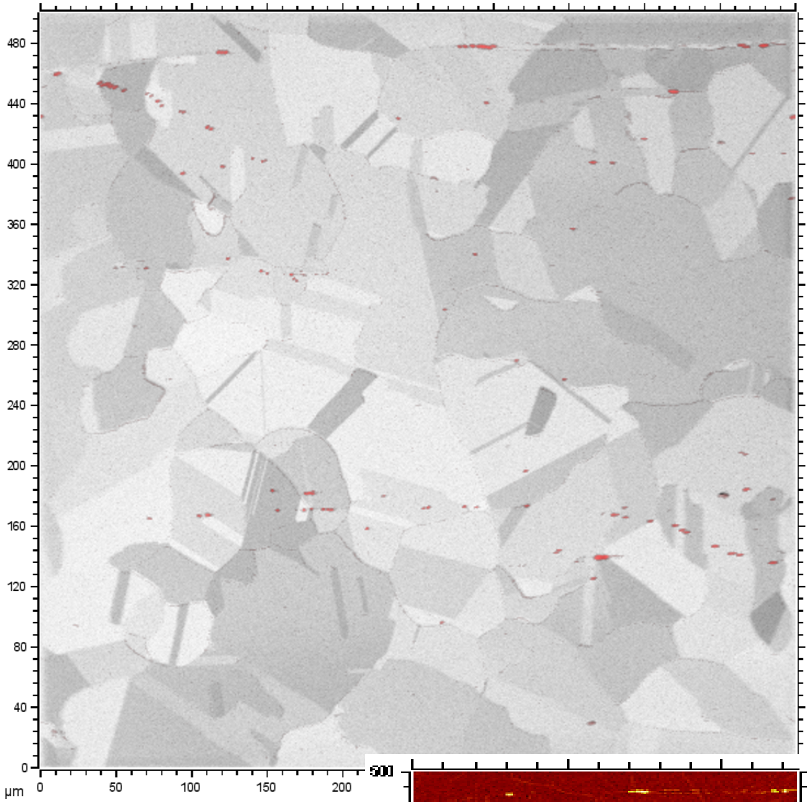
Boride dissolution occurs between 1000°C and 1100°C

As-Received





1000°C 32 min



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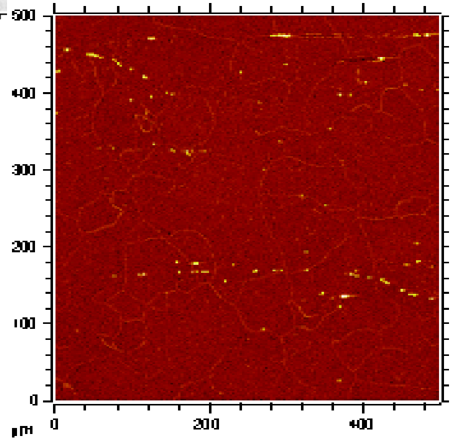
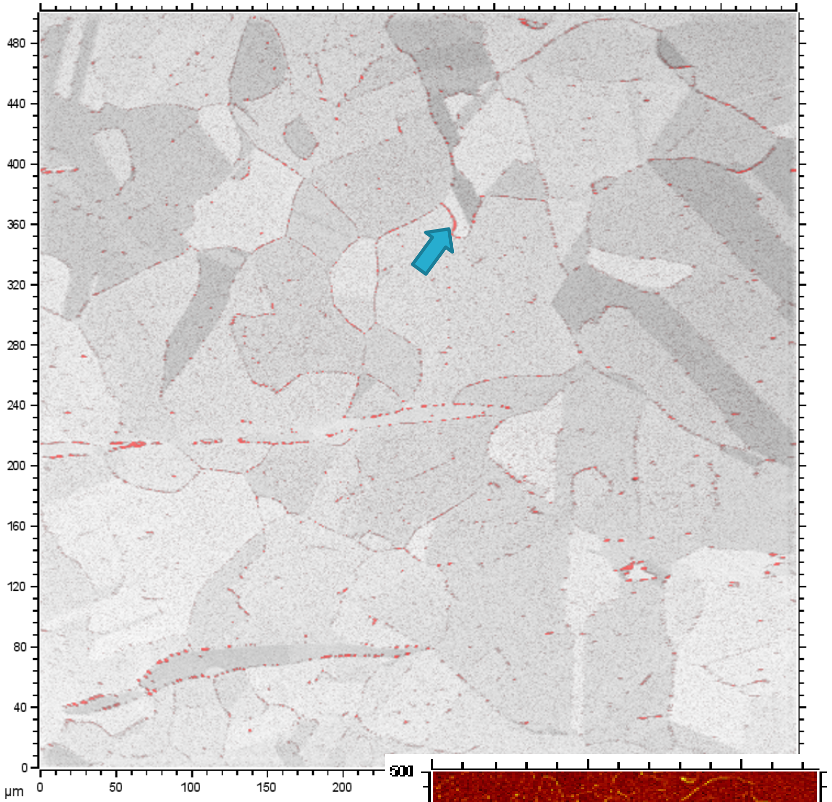
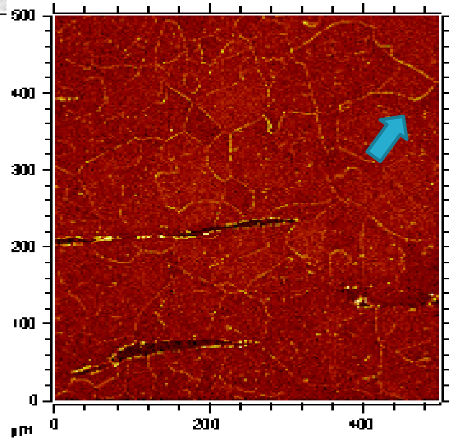


1100°C 1 min

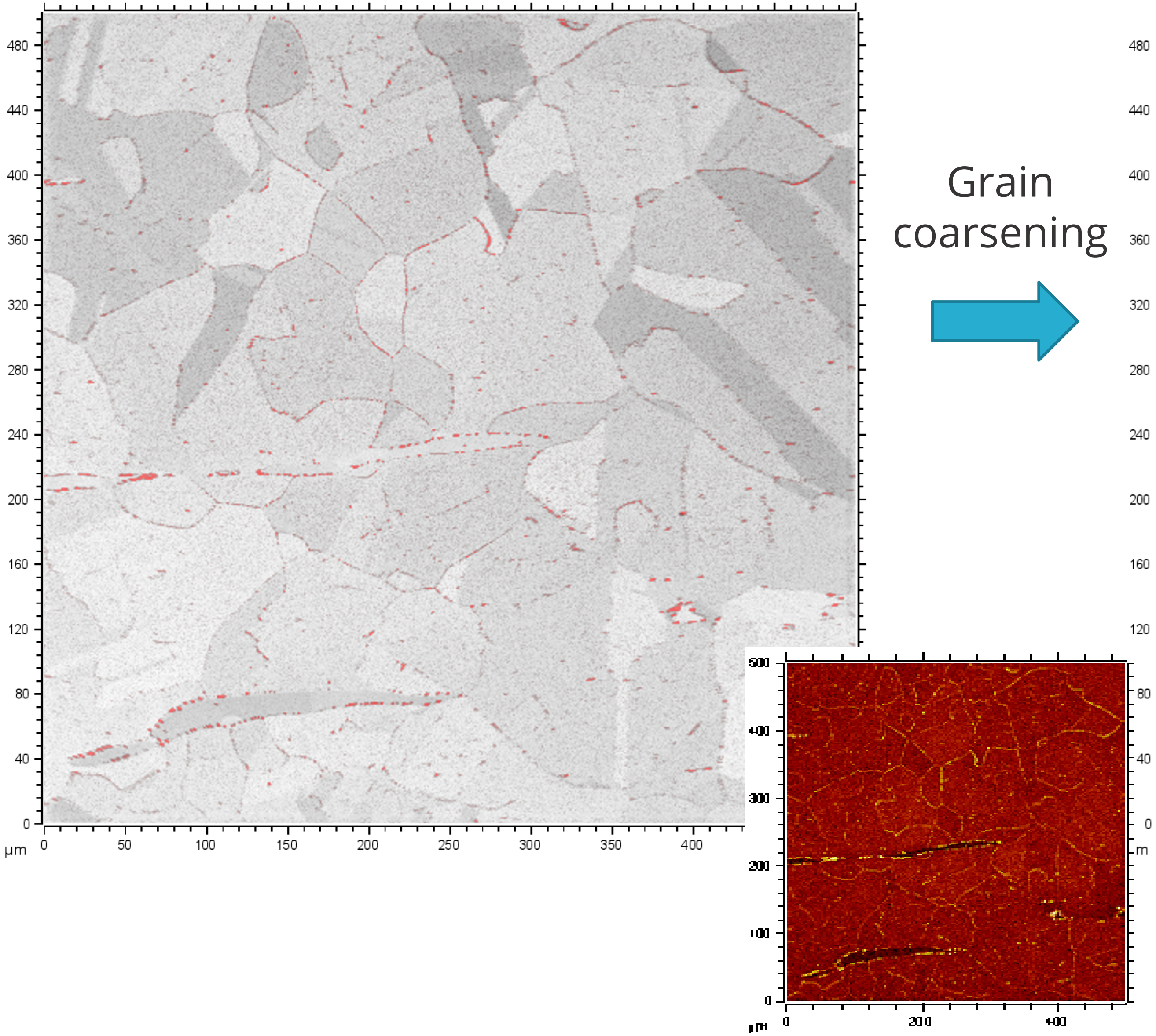


Some boron migration to γ grain boundaries; some remains on δ



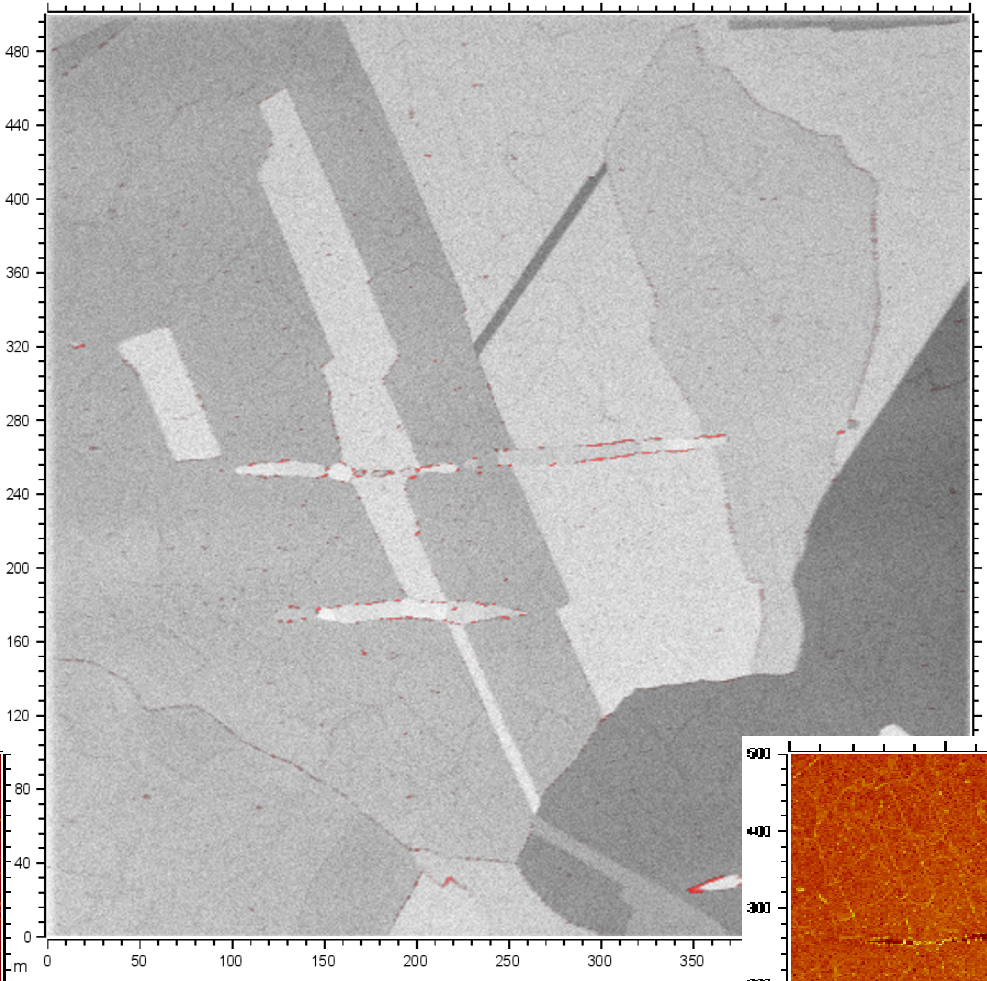
Boron diffusion to γ/γ grain boundaries is rapid

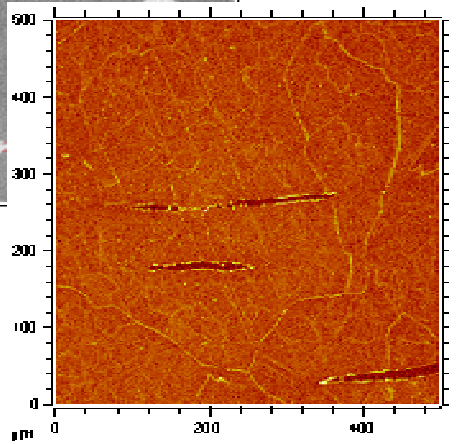
1100°C 1 min



Grain coarsening

1100°C 32 min

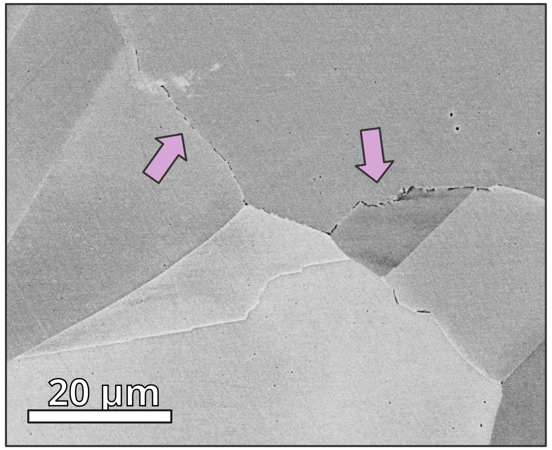


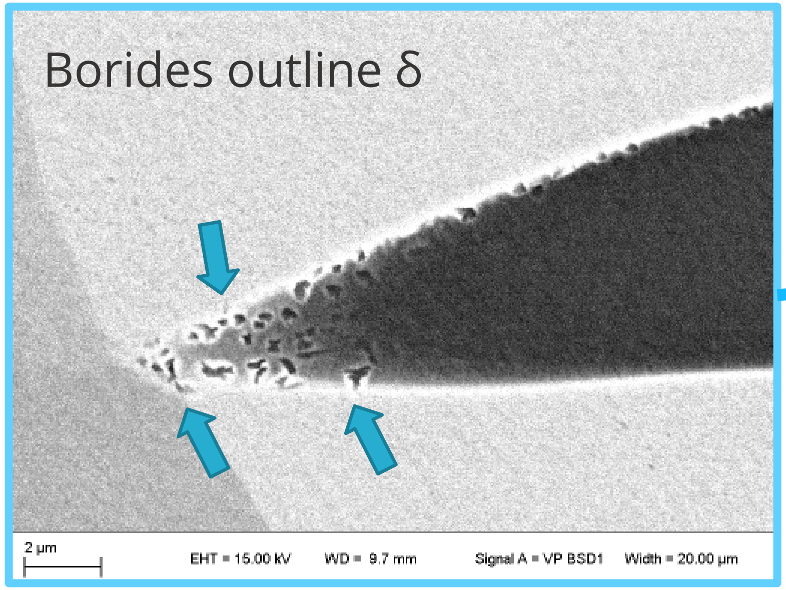


Little (if any) observable changes with additional hold time at 1100°C

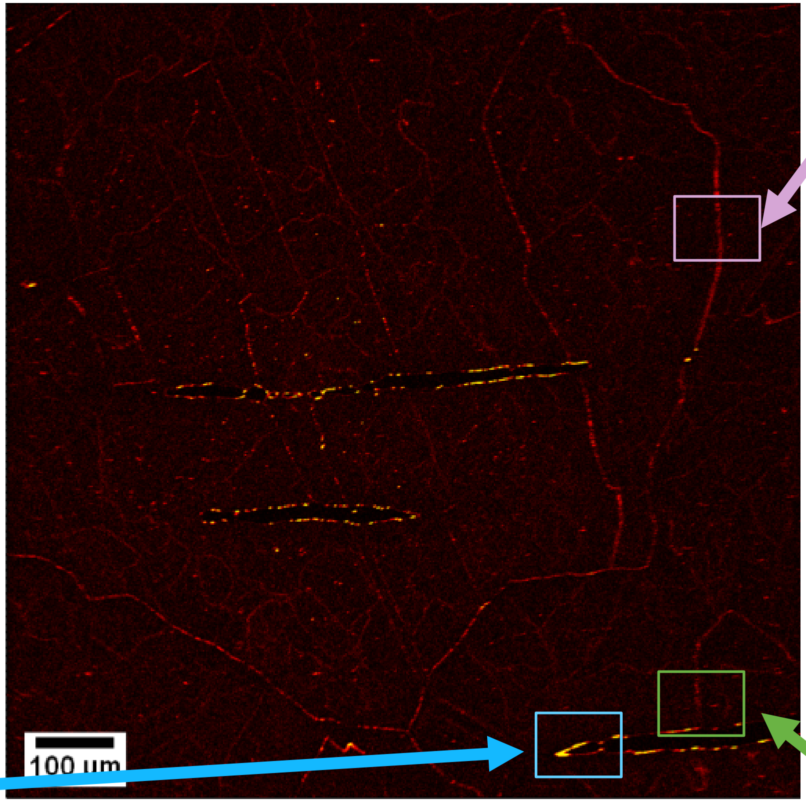
Borides re-precipitate on δ/γ boundaries but not on γ/γ boundaries

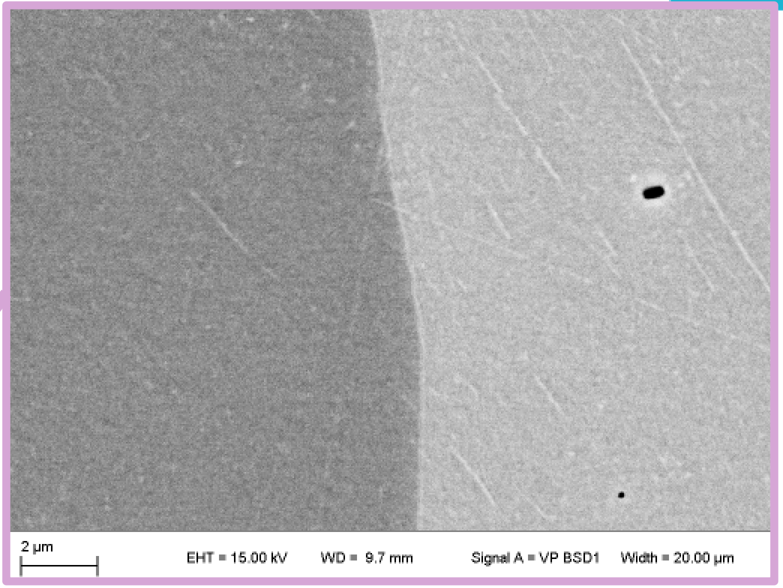
Recall: ability to see borides in SEM



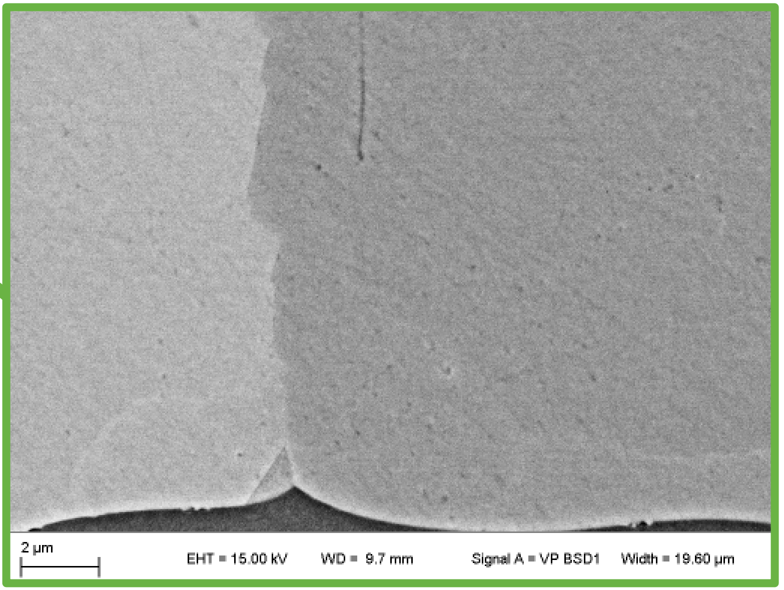


1100°C 32 min





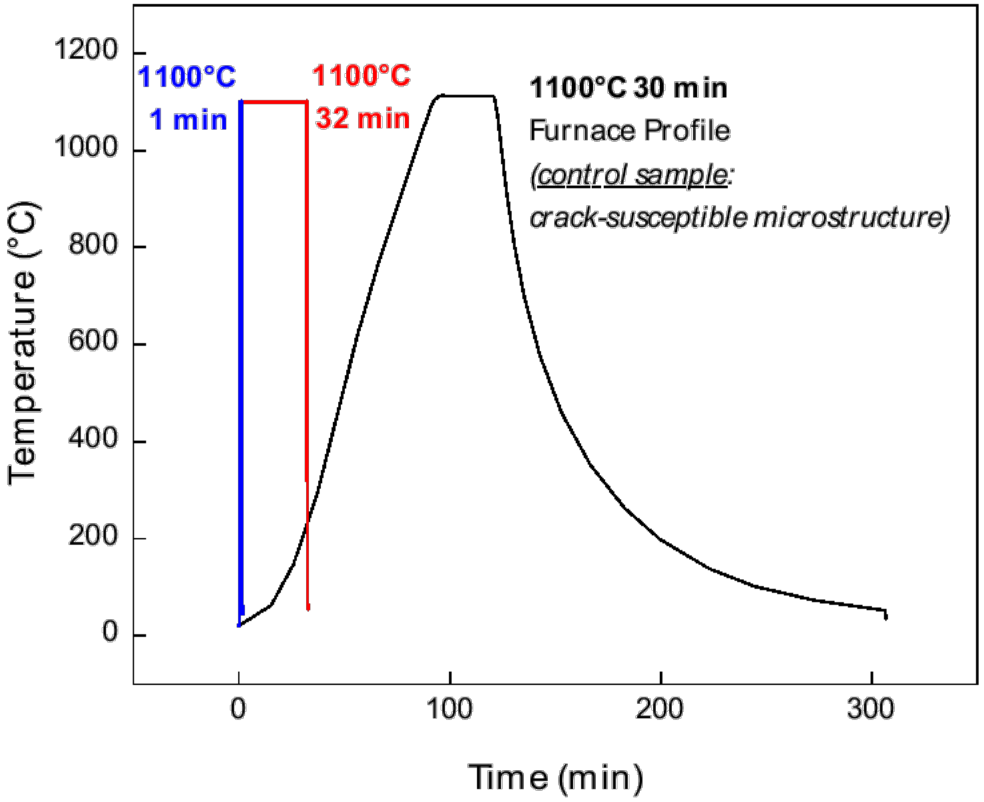
Elemental boron or borides below SEM resolution limit?



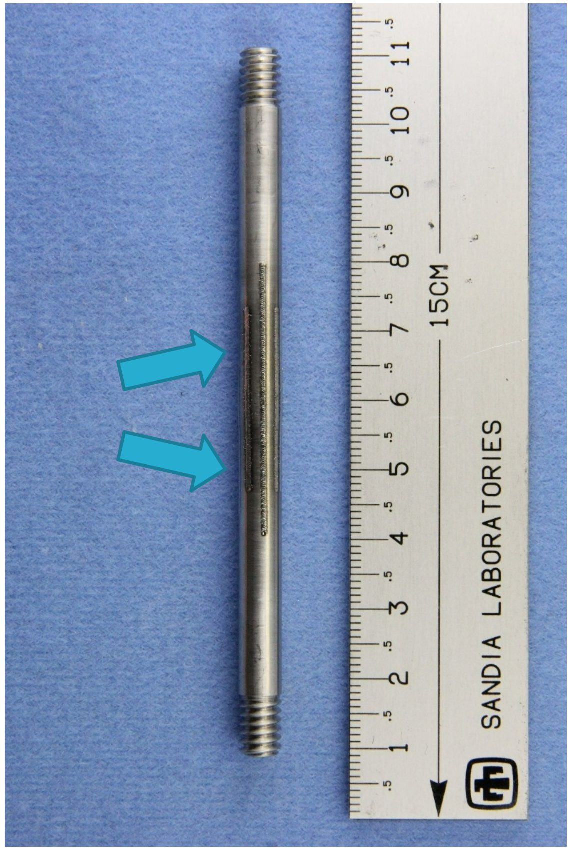
Is this a crack susceptible condition?

Developed test method to correlate phase transformation kinetics to crack-susceptibility

Step 1: Gleeble heat treatments



Step 2: Laser welds directly on Gleeble bars

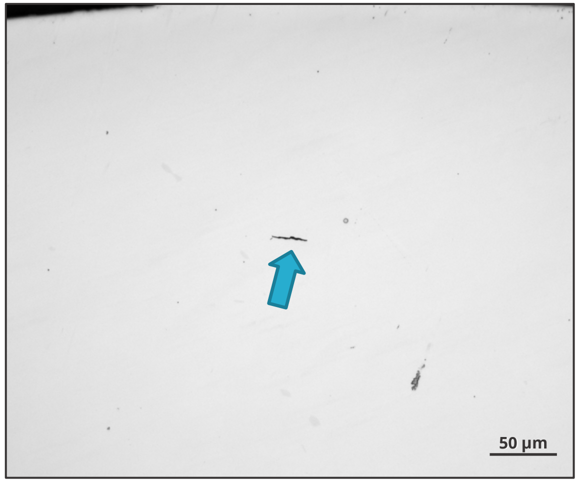


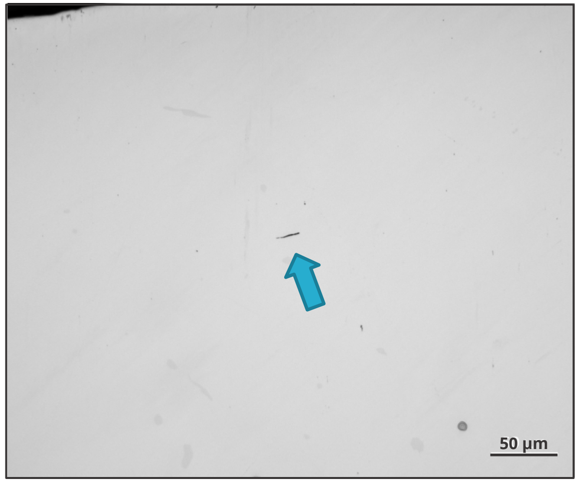
Step 3: Section welds in transverse, scan HAZ for cracks

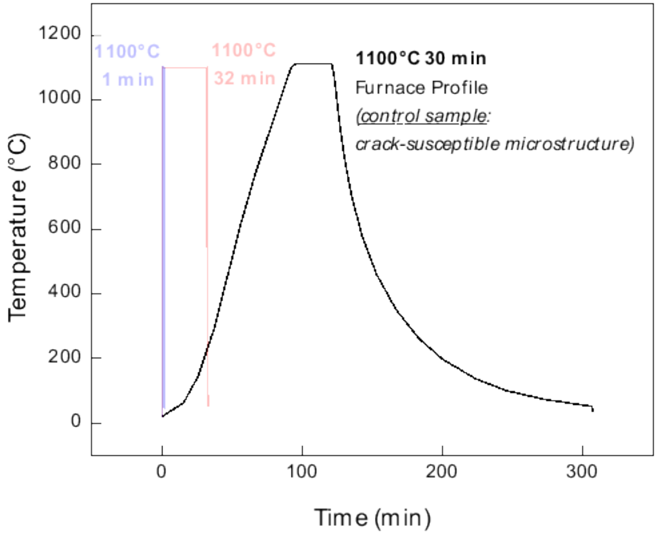
60 weld cross-sections surveyed per Gleeble condition

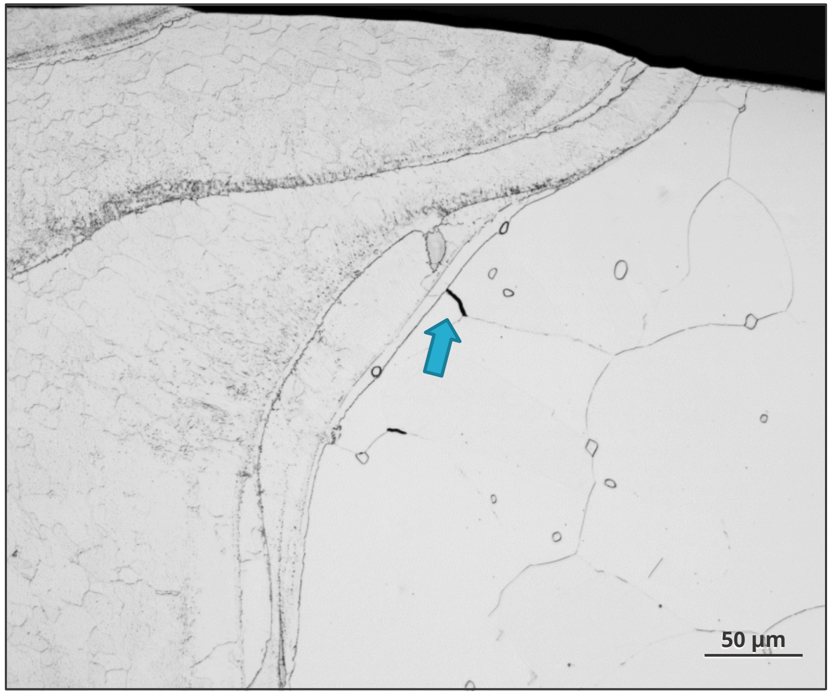
Furnace profile condition is crack-susceptible

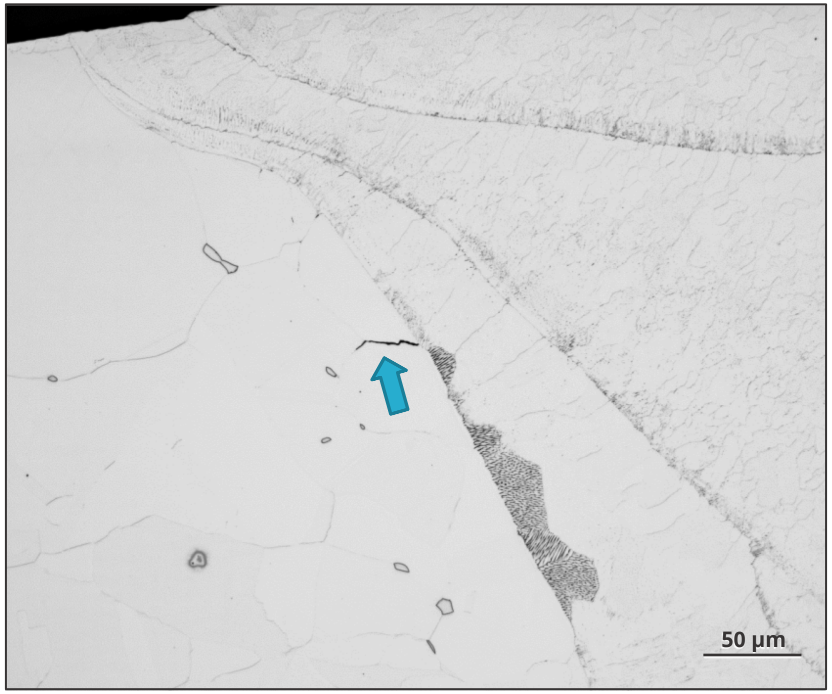
Unambiguous crack determination in as-polished condition

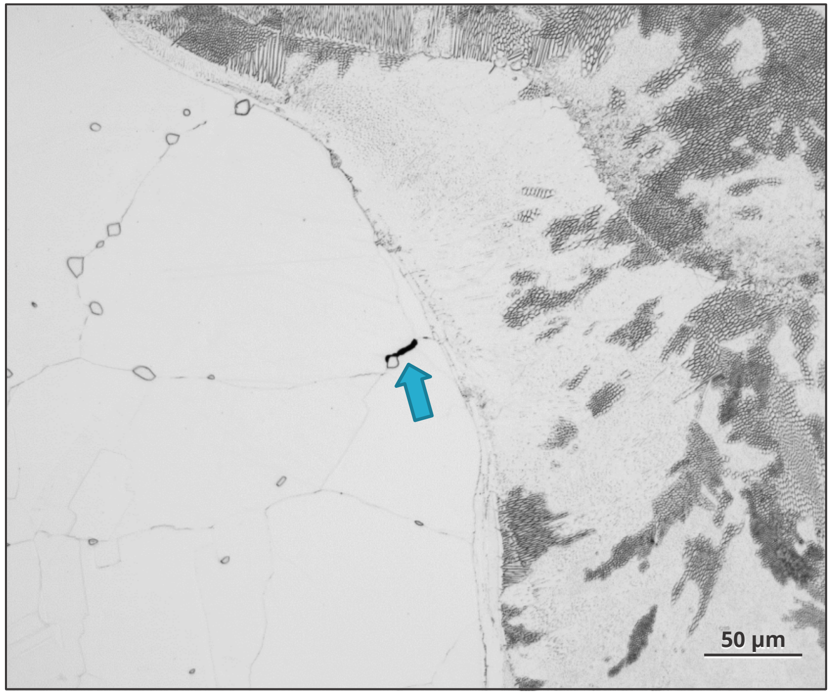




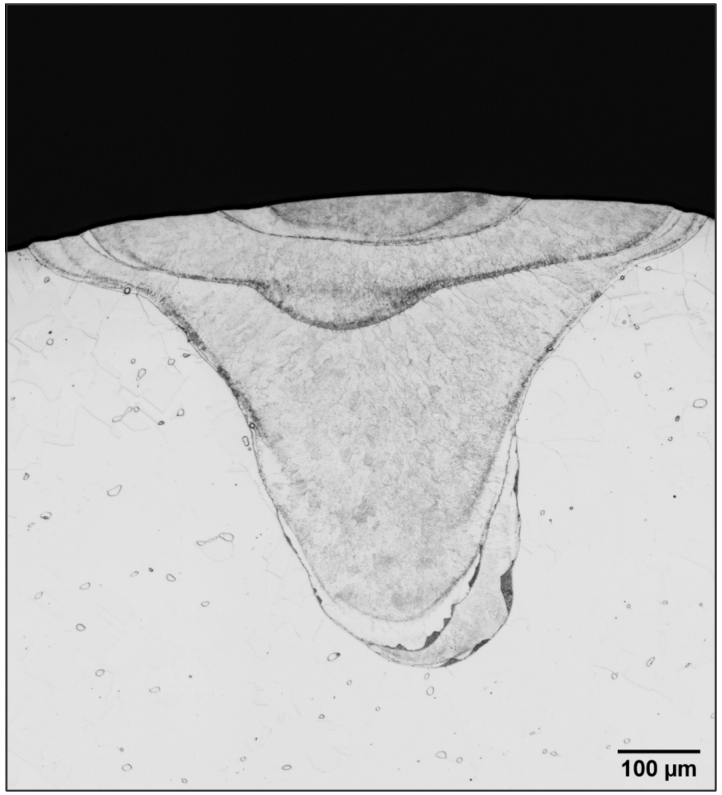


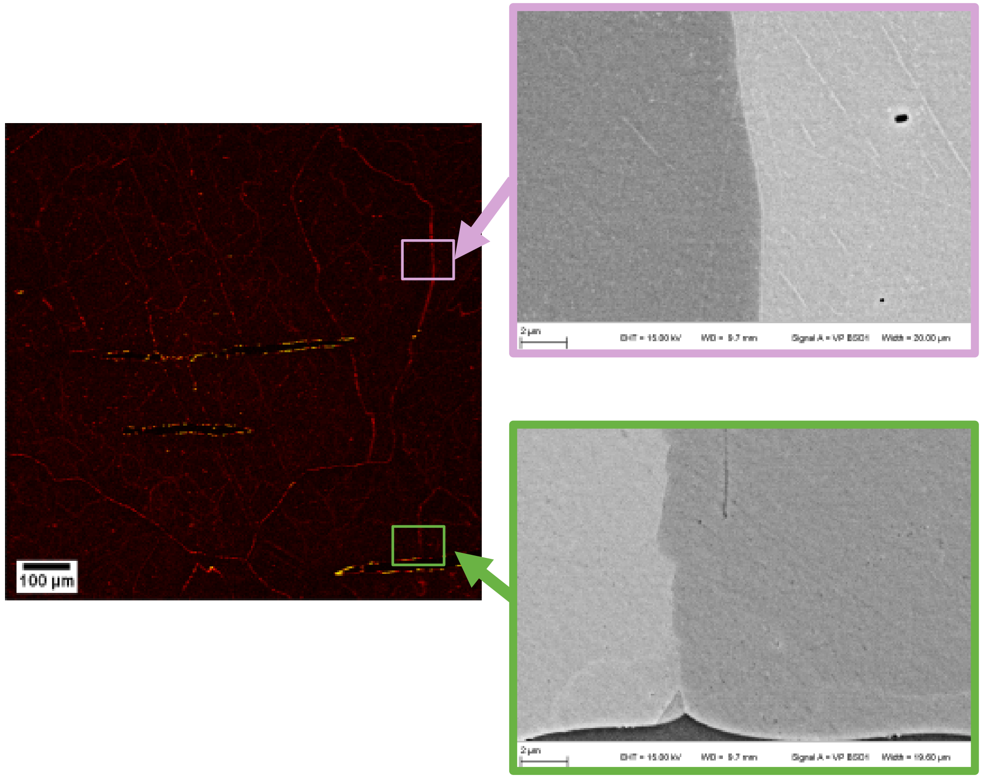


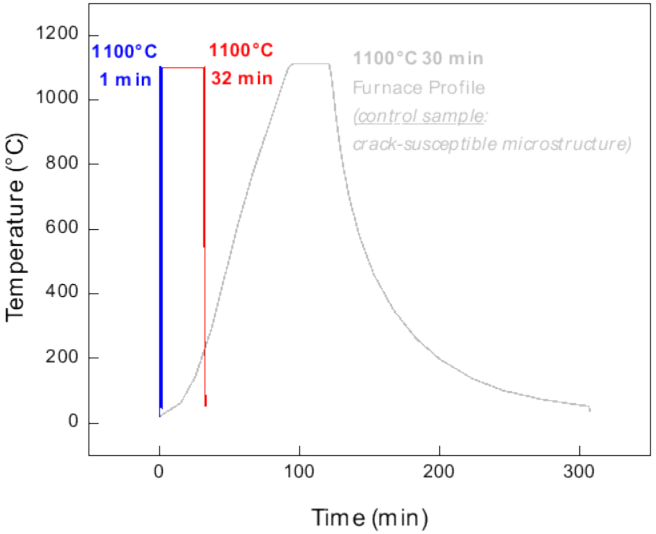




Crack-susceptible microstructure is related to heating/cooling kinetics







Either borides aren't present (elemental boron) or are below resolution limit of SEM (and are small enough to not present a cracking risk)

No cracks observed in any welds for 1100°C 1 min or 32 min!

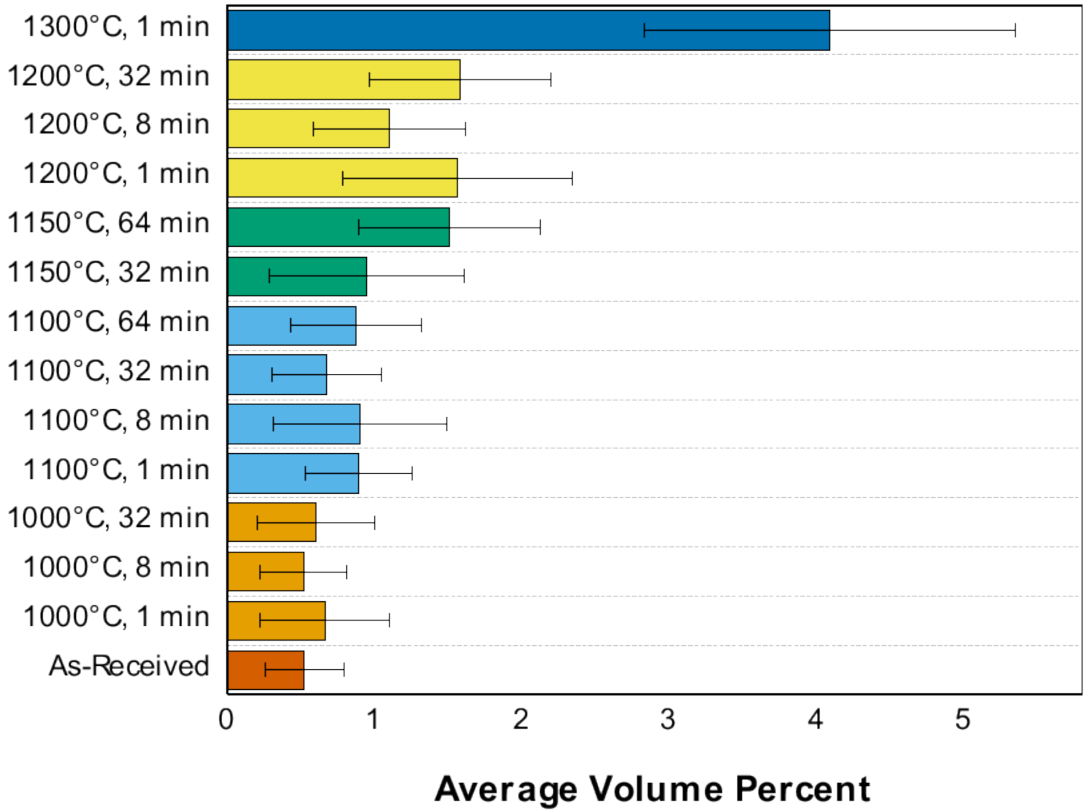
Experiments are in progress to elucidate cooling rate effects on crack-susceptibility

- Critical cooling rate must be between 100°C/s and 0.5 C/s (furnace profile cooling rate)

Quantification of δ-ferrite reveals stability of δ

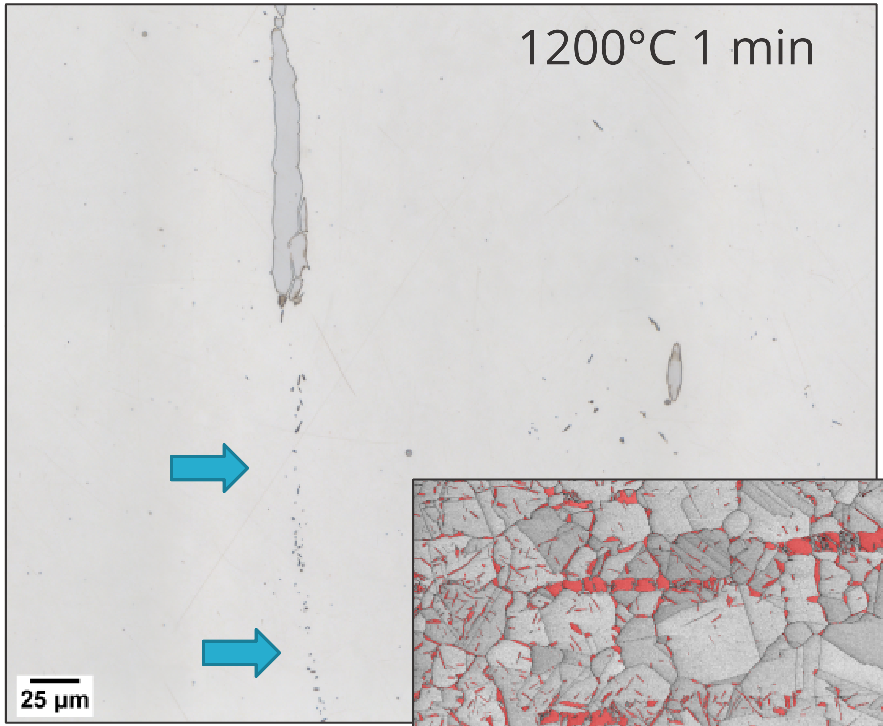
Selective ferrite etchant: 10% NaOH

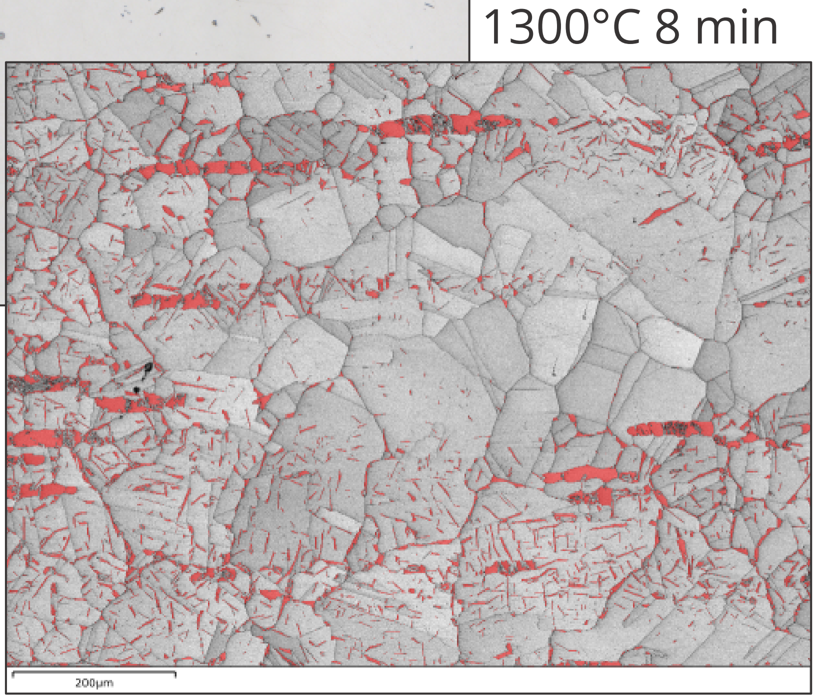
~4.5 mm^2 were surveyed per sample

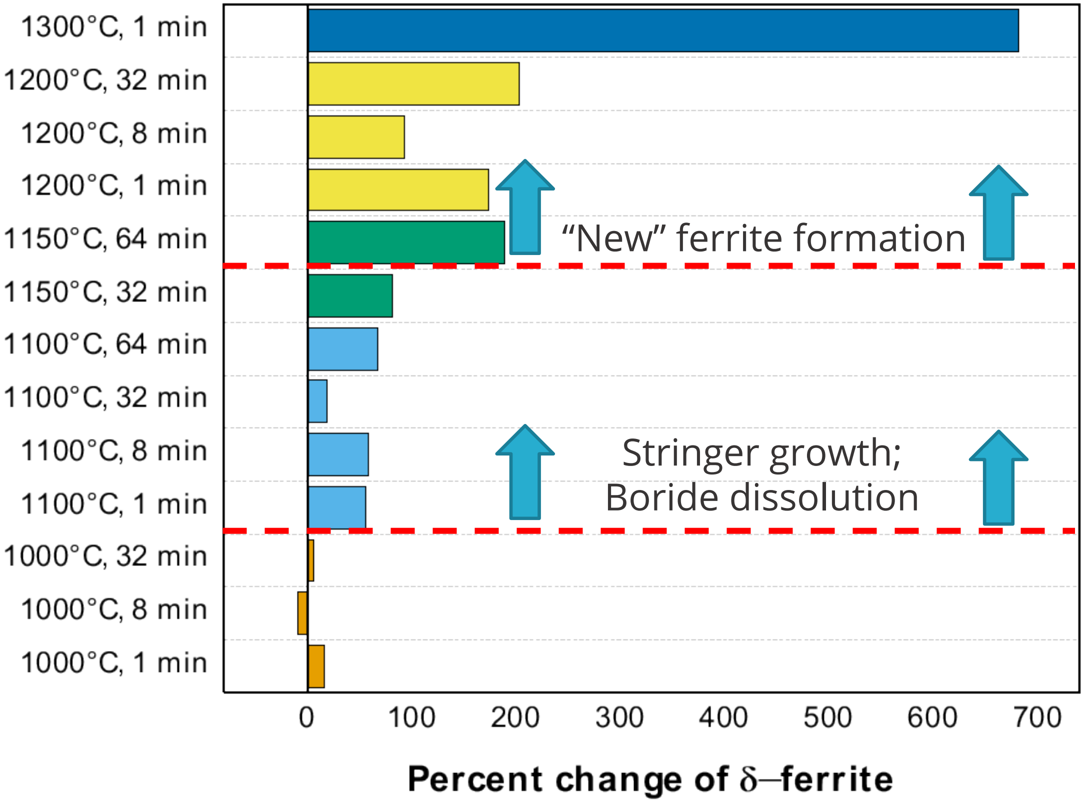


All heat treated conditions have equal to or more ferrite than starting condition

Increase in amount of δ-ferrite associated with stringer growth and new δ formation

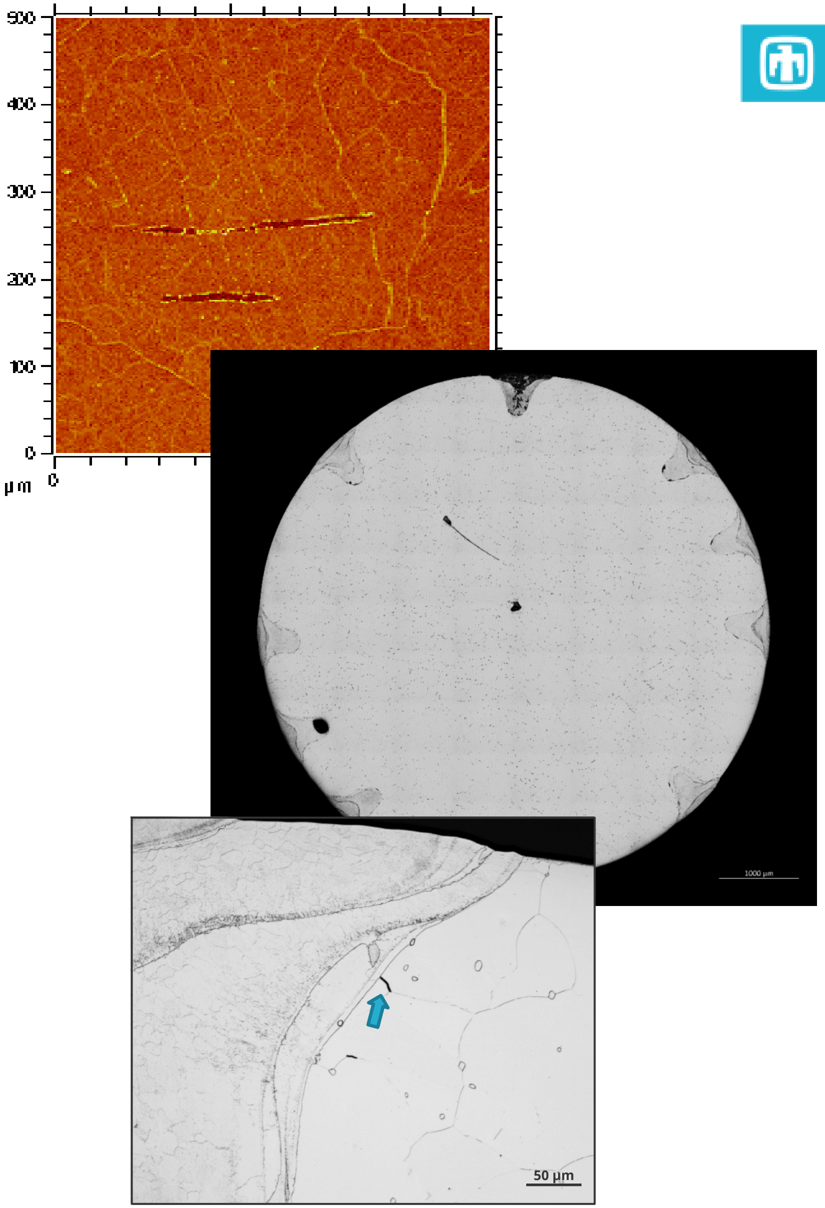






Summary to date & continuing work

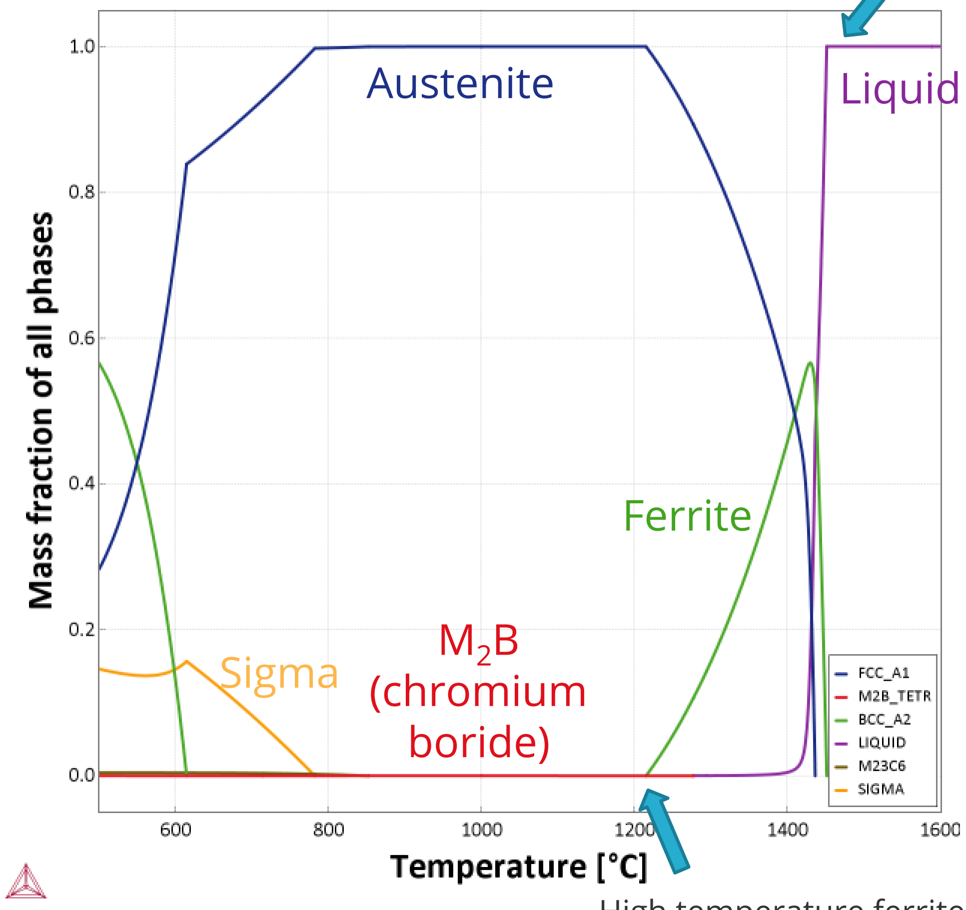
- Boride solvus temperature is between 1000°C and 1100°C
  - Additional experiments in progress at 25°C incremements to narrow in on solvus temperature
- Boron migration to γ grain boundaries is rapid (1 min at 1100°C is sufficient)
- Heat treatments with rapid heating/cooling rates are not crack-susceptible despite evidence of boron diffusion to grain boundaries
  - Cooling rate is significant for generating crack-susceptible microstructures
  - Cooling rate experiments in progress: 10, 1, 0.5, 0.1 °C/s to determine critical cooling rate
- δ-ferrite growth occurs at temperatures above boride solvus (link between ferrite and borides?)
- These results begin to form the kinetic framework which will enable predictions of the crack susceptibility of B-containing 304L stainless when subjected to complex, part-specific heat treatments
- Additional work in progress:
  - In-situ characterization
  - Effect of Cr/Ni_{eq} on B kinetics



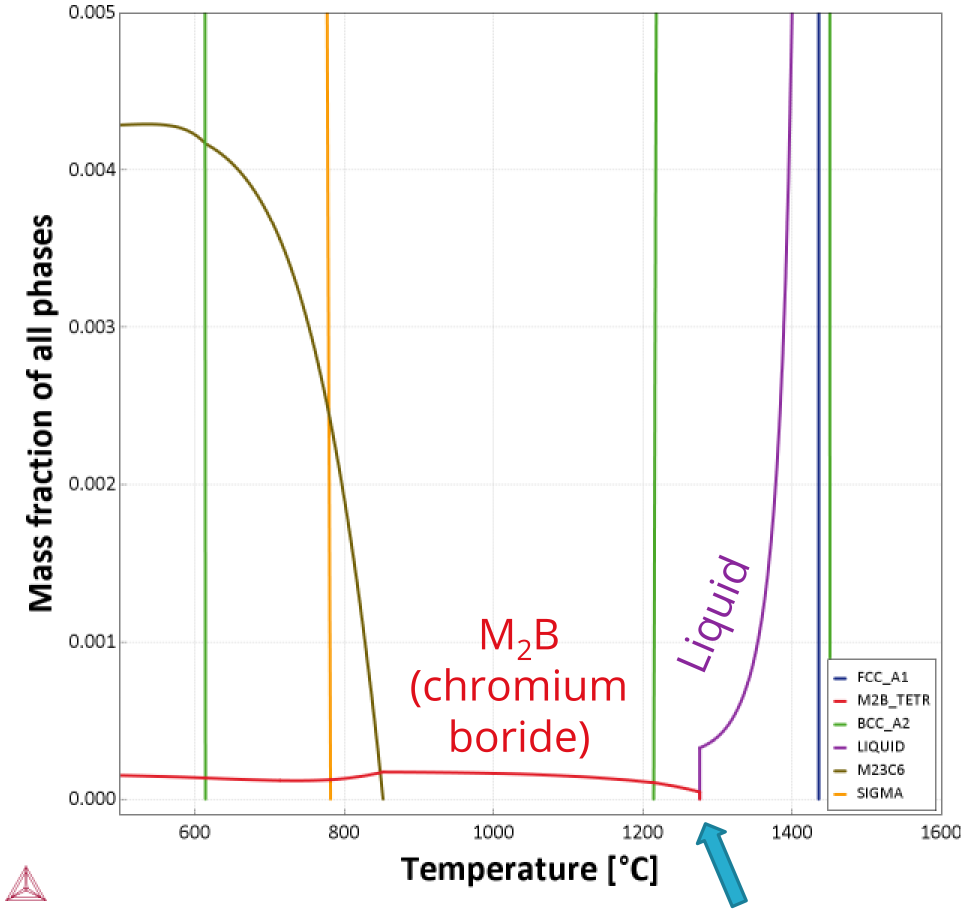
Back-up slides

Thermo-Calc phase fraction predictions for 20 wt ppm B 304L composition

Sample completely liquid above ~1450°C



High temperature ferrite (δ) predicted to be stable above ~1220°C



Boride predicted to be stable up until 1277°C, at which point sample begins melting

Equilibrium phase fraction