Progress of Cast Superalloys at NETL

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NATIONAL ENERGY TECHNOLOGY LABORATORY

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NATIONAL ENERGY TECHNOLOGY LABORATORY

FUNDING AND SUPPORT

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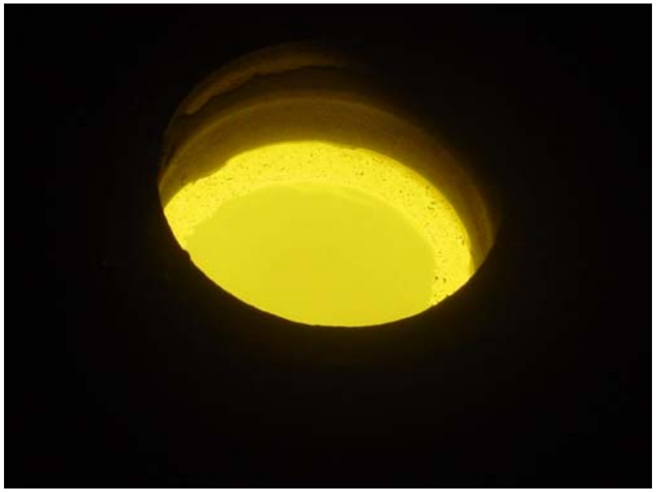
Materials focus and some processing capabilities

- R&D high temperature structural materials for power generation
  - Focus on Steels and Ni-superalloys
  - Identify and select materials capable of withstanding creep, fatigue and corrosion at 760°C (1400°F) at 35 MPa (5000 PSI)
- Melt facility capable of
  - 300 lb VIM and Air melting
  - 20, 30, 50 lb VIM
  - 3, 4, 6 and 8” VAR/ESR



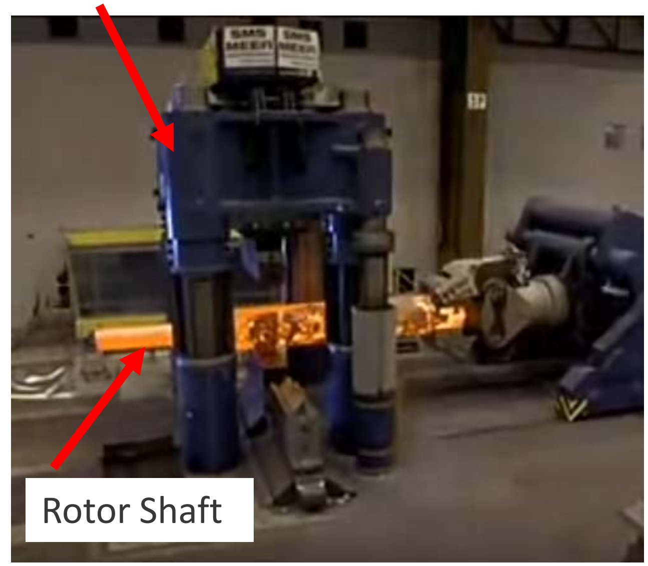
Manufacturing

Some background on steam plant components

- Steam turbine rotors are typically cast into an ingot form then forged and machined to final dimension
  - The window of hot working some superalloys is only 100°C, preventing the forging process
- If we could eliminate the forging process manufacturing costs could be lowered
- Rotors typically aren't cast to size because of segregation and grain size considerations.
- Billets are forged and extruded for pipe and tubing.
- Other components such as turbine casings and valve chests are cast due to their complex geometry.

50,000 lb Casting

Forging



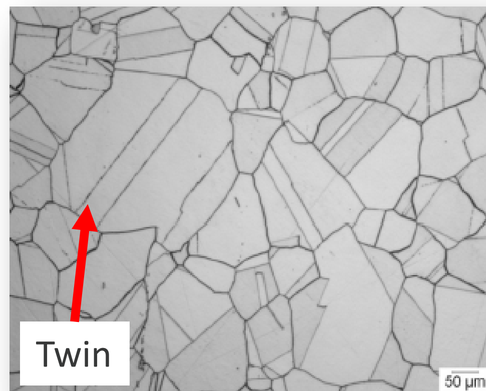
Forging of power transmission shaft,
Screenshot of youtube video, copyright is by Saarschmiede GmbH Freiformschmiede in Germany

Inconel 740H

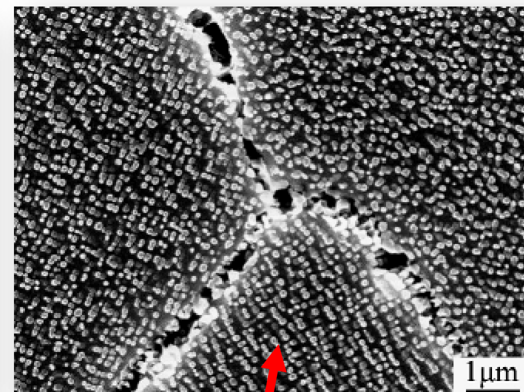
Work on cast H282 has been successful; Update on a cast version of IN 740H

	% Wt												Strengthener		PPM					
Element	Mn	Si	Cr	Ni	Co	Mo	Nb	Ti	Al	Fe	Cu	P	Ti+Al+Nb	Ti:Al	C	N	O	S	B	La
Min	0	0	23.5	bal	15	0	0.5	0.5	0.2	0	0	0	1.20	2.5	50	-	-	0	6	-
Nominal	0	0.15	24.5	bal	20	0.1	1.5	1.35	1.35	0	-	-	4.20	1	300	-	-	-	-	-
Max	1	1	25.5	bal	22	2	2.5	2.5	2	3	0.5	0.03	7.00	1.25	800	-	-	300	60	-

- IN740H is a derivative of Nimonic 263 intended for structural use in high temperature applications/boiler side
  - Gamma prime strengthened
  - Minimal Eta formation
- Boiler certified
  - UNS # N07740
  - Approved by Code Case 2702 under ASME B31.1 by Code Case 190
  - Max temperature 800°C



ASTM #3 grain size

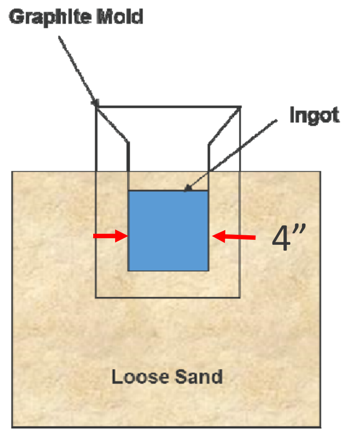


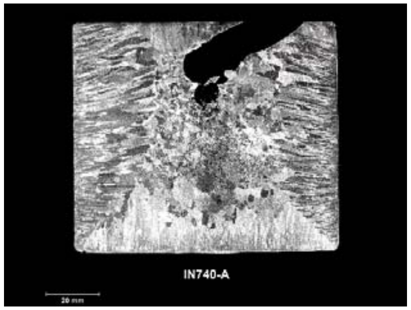
γ'

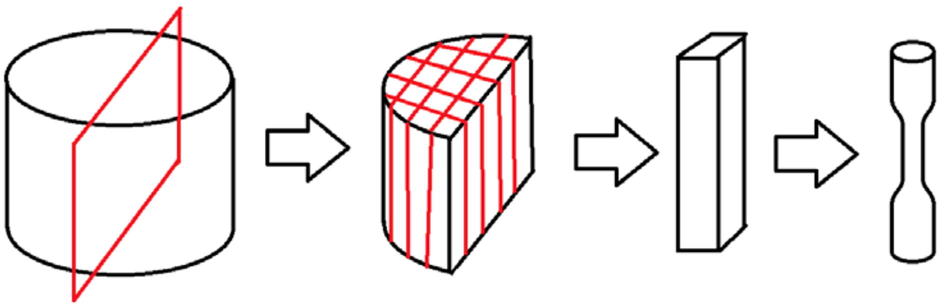
Mechanical Testing

Results from the 4” round test

- Commercially pure feed stocks used
- Chemistry of pre-alloyed materials tightly controlled
  - NiCrLa, NiB, Ni-50Cr, Ni-30Co-30Cr
- Vacuum / Ar atmosphere melt and pour
- Cast IN740H into 4” graphite mold with slow cooling
- Computationally based homogenization and aged
  - 1120°C/ 1hr /AC / 850°C/16hr
  - Simulates thick wall sections
  - **Not forged**—cast structure
- Extracted tensile and creep samples from 4” round
- Test mechanical properties
  - Followed ASTM standards
- Tracked columnar and equiax location of samples



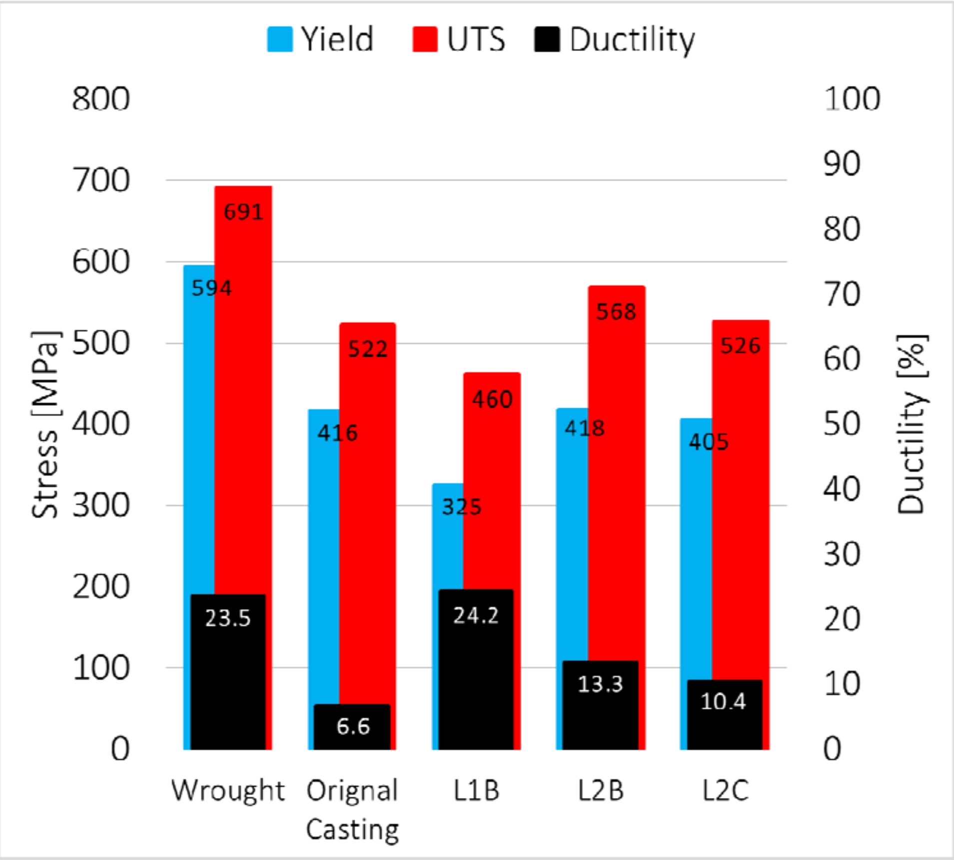




Hot Tensile Results

From the homogenized 4" casting, as per ASTM E7

- All tensile tests from 800°C
- "First Heat casting" shows good strength but poor ductility
- "L1B" shows good ductility but reduced UTS and yield
- "L2B" and "L2C" show intermediate ductility and improved strength
- Columnar grained samples showed slightly higher ductility than Equiaxed samples
  - Plotted values are averaged from both orientations

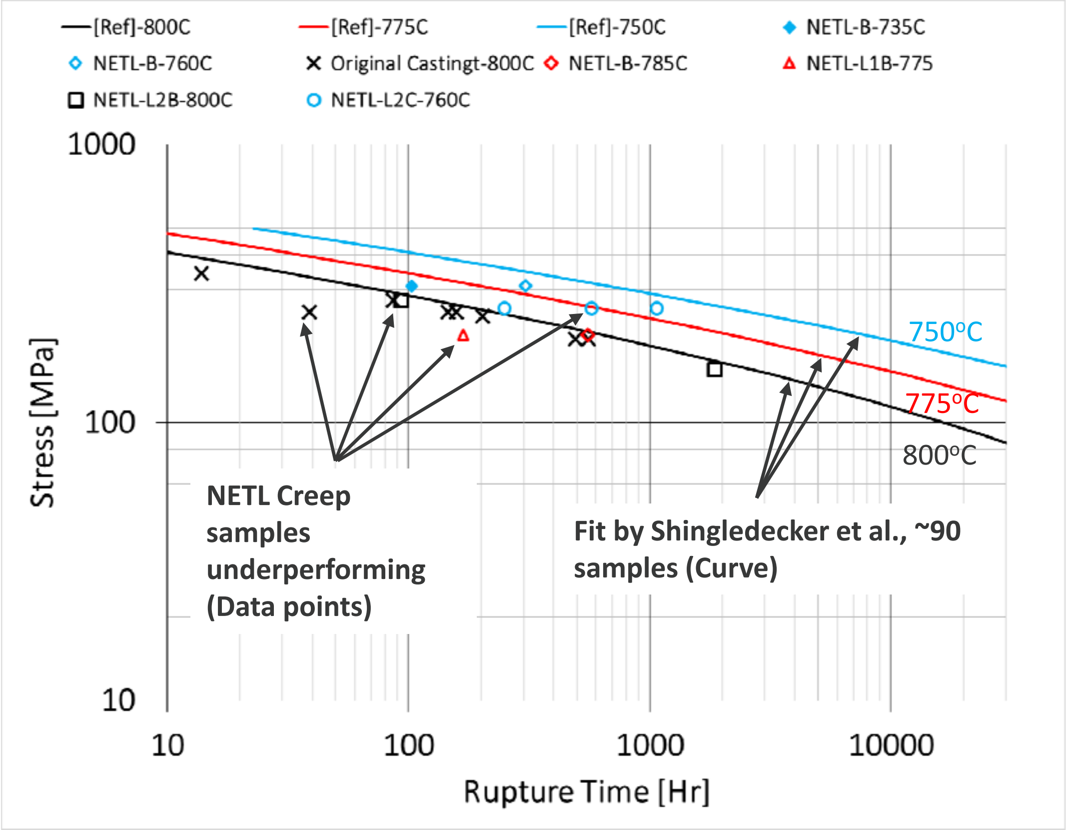


Creep Results

From the homogenized 4” casting, as per ASTM E139

- Cast creep samples significantly underperforming compared to wrought alloy
- All temperatures, stresses and heats

- BLACK – 800°C
- RED – 775°C
- BLUE – 750°C

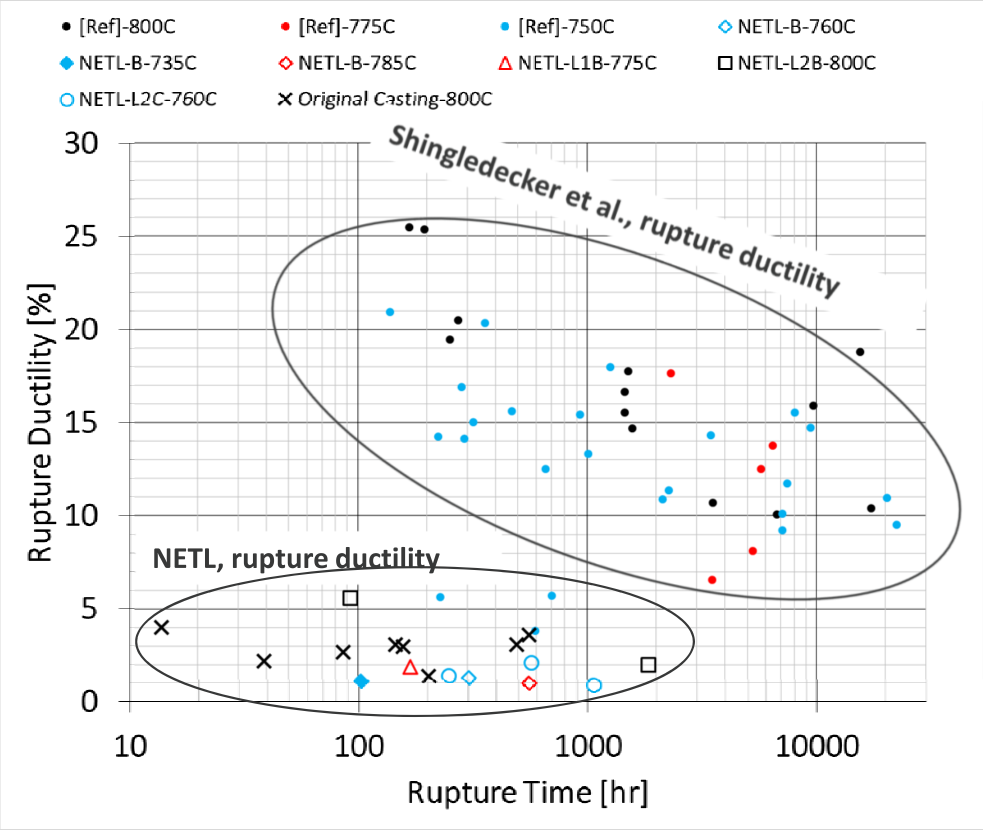


Creep Results

From the homogenized 4” casting, as per ASTM E139

- **Cast creep samples have significantly less rupture ductility than wrought samples**
- **All temperatures, stresses and “heats”**
- **Ductility <5%**
- **Unacceptable for structural materials**

- **BLACK – 800°C**
- **RED – 775°C**
- **BLUE – 750°C**

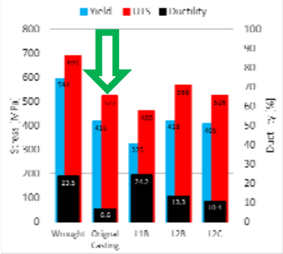


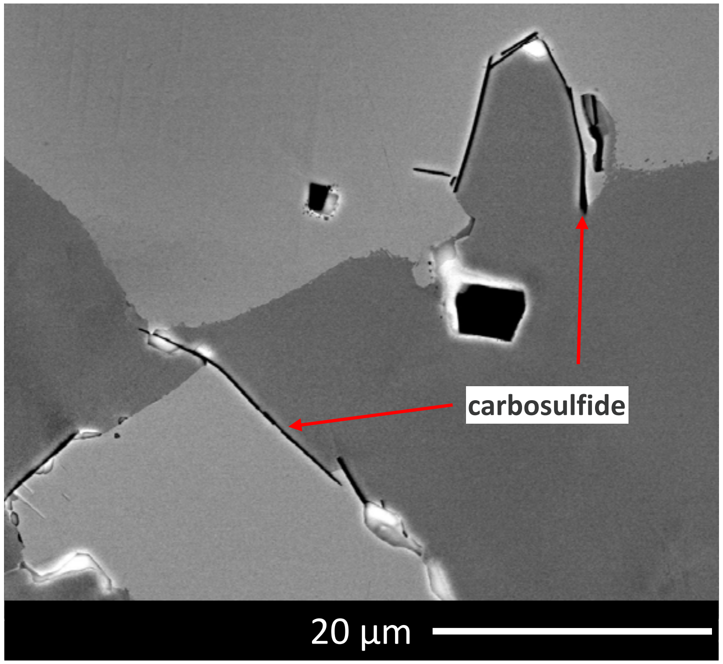
Shingledecker, J.P. et al., Mat. Sci. Engr. A 578 (2013) 277-286

Analysis

Original casting “A”

- Chemistry within spec, although high sulfur (100 PPM)
- Heat A showed poor ductility
- Analysis of the original casting showed angular particles rich in Nb, C and S, likely Carbosulfides
- Could it be that sulfur was causing the low ductility?
  - Lanthanum was subsequently added to strip the sulfur from input materials
  - Results indicate sulfur levels below 10 ppm

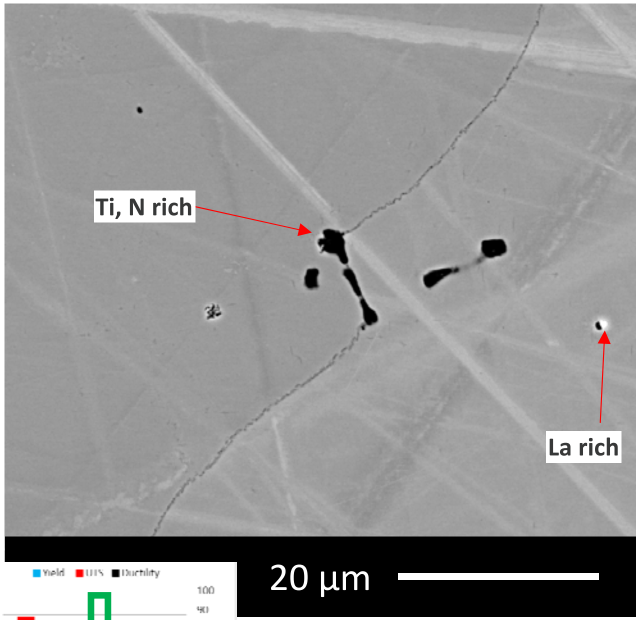


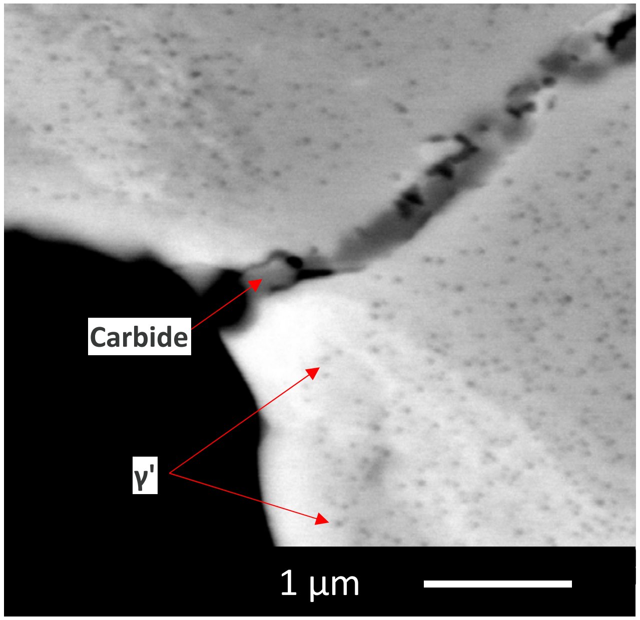


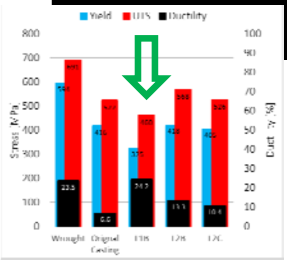
Microstructure

As observed from "L1B" heat

- L1B showed good ductility
- La added to reduce sulfur content
  - Reduction in sulfur to <10 PPM
- Mo added to modify the carbides
- ~250-500 nm carbides observed
  - Carbon measured ~300-400 PPM
- Low density of gamma prime
  - Slight decrease near TiN and carbides (GB's)
- No eta phase detected
- Ti, N and La rich phases in matrix and grain boundaries



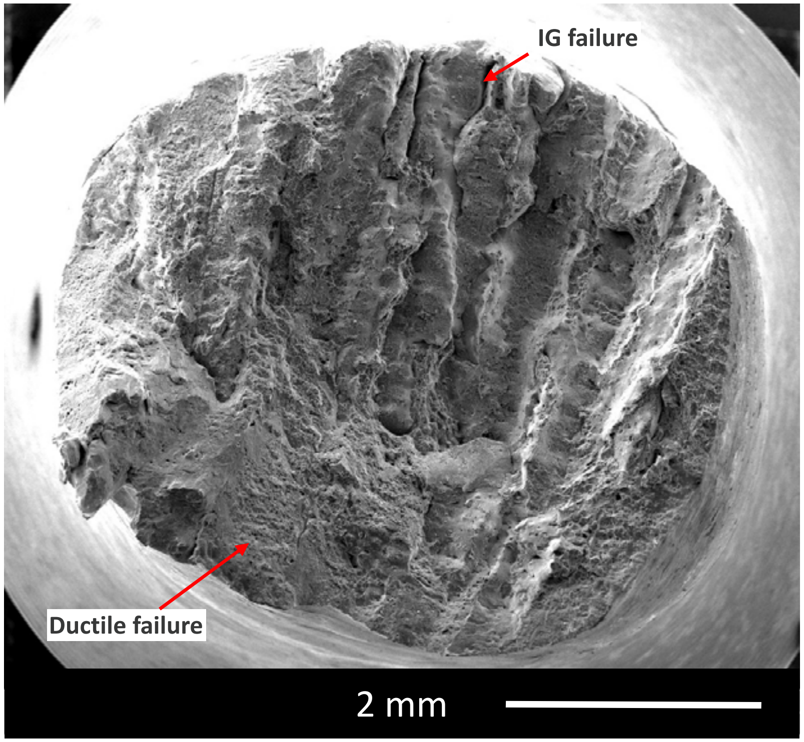


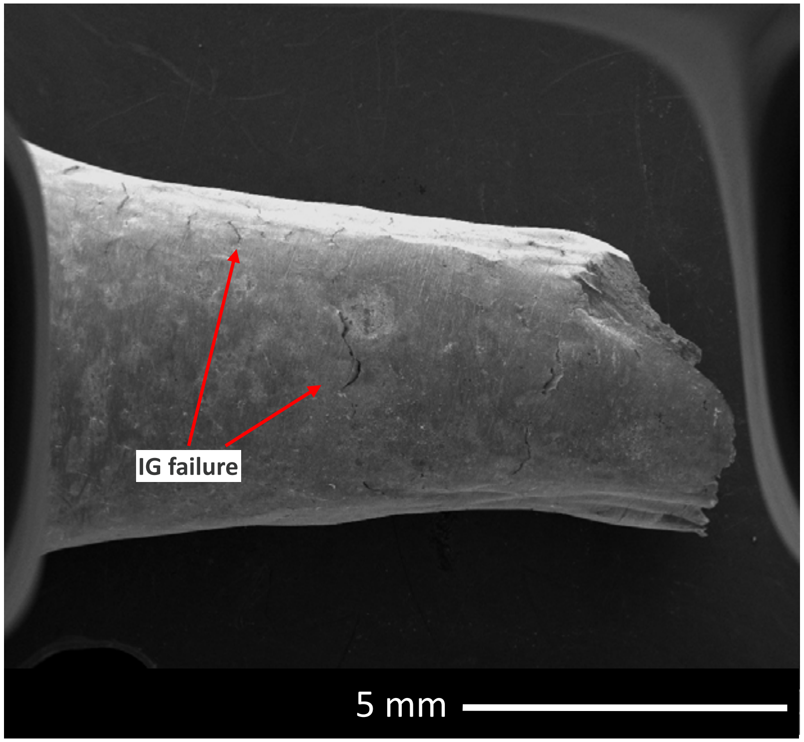


Failure Mode

As observed from “L1B” heat

- Fracture morphology appeared inter-dendritic / intergranular
- Significant secondary cracking, not limited to fracture plane
- Observed both in tensile and creep
- ~50% of sample shows ductile fracture
  - Consistent with measured ductility





Failure Mode

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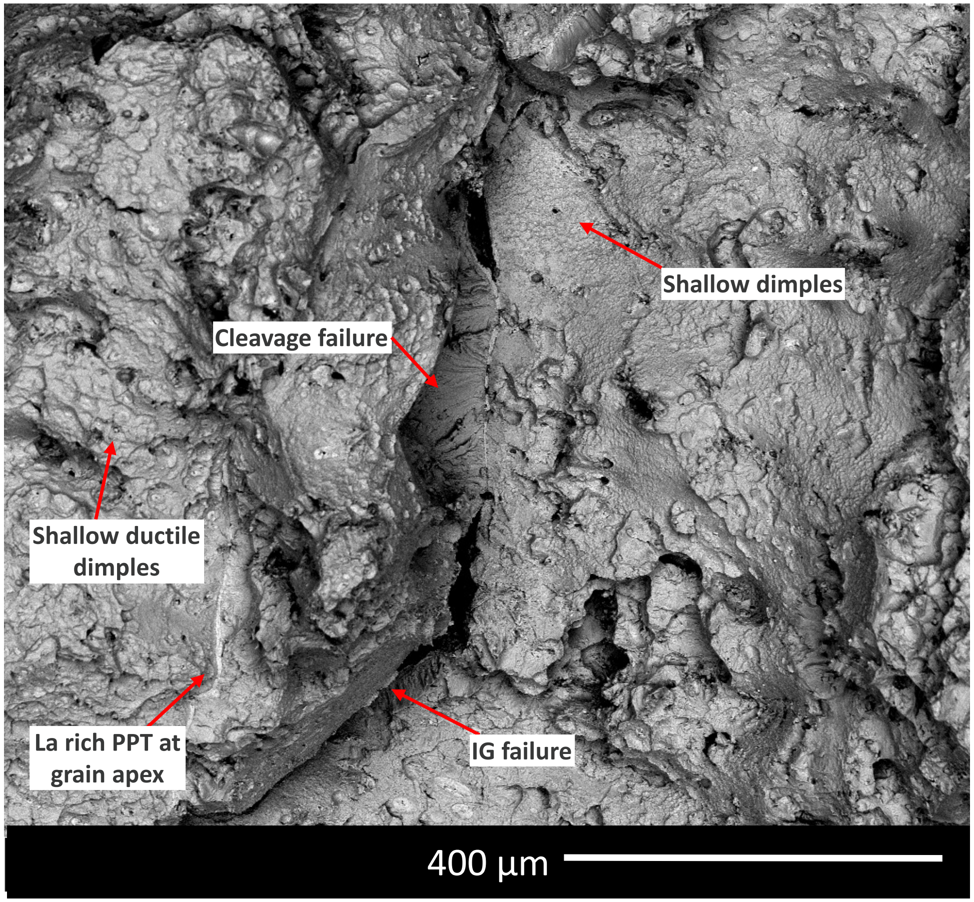
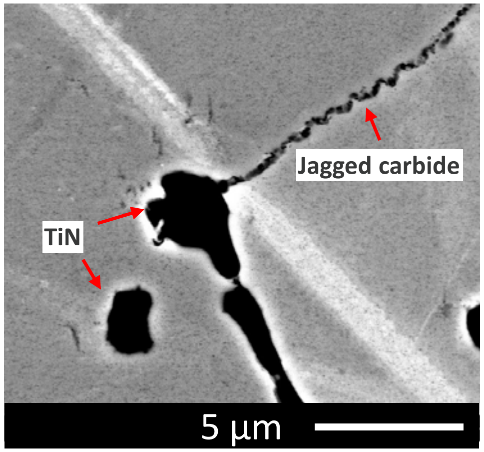
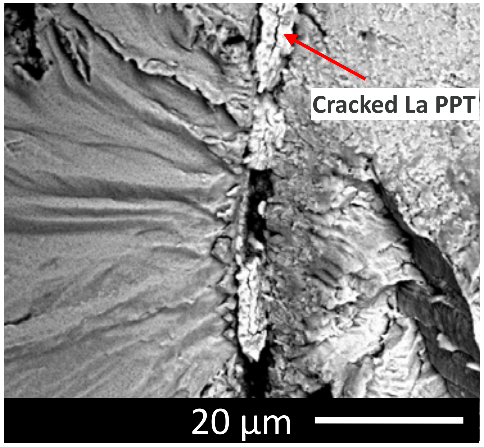
As observed from “L1B” heat

- Carbide morphology appears thick and continues, although jagged
- Not detecting TiN phases from fracture surface
- Ductile dimples are wide and shallow
    - Sometimes bright (La) phase at bottom
    - Suggesting no influence
- La rich grain boundary precipitates appear near intergranular fractures
    - Evidence of IG failure at La rich PPT
    - Cleavage failure suggests La rich PPT held GB together until final fracture
    - Conclude La rich phases do not influence fracture process



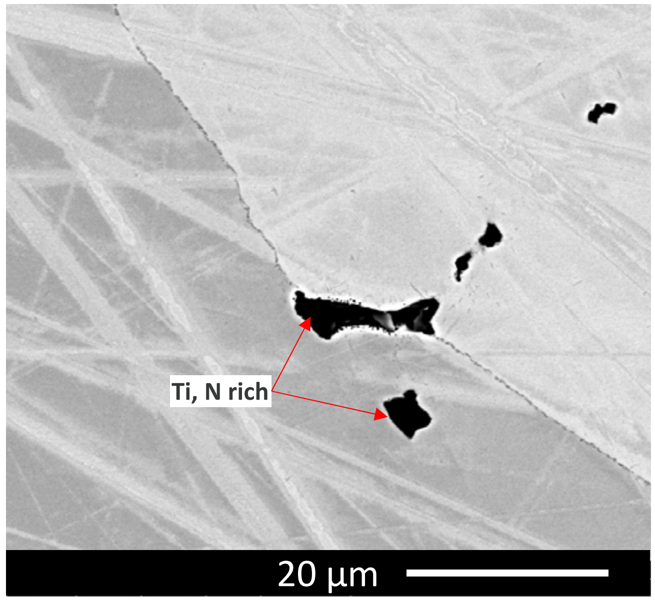


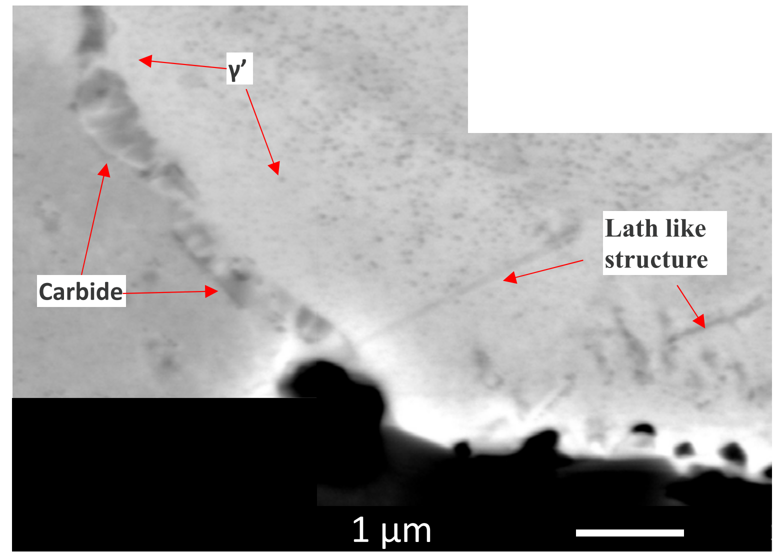


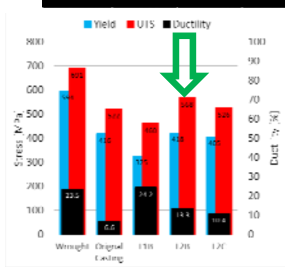
Microstructure

As observed from "L2B" heat

- L2B increased Ti and Al to yield more gamma prime
- Large 250-750 nm carbides again observed
  - Carbon measured ~300-400 PPM
- Ti-N phases on GB and in matrix
- Lath like structure detected
  - EDS shows Cr rich
- La rich phases detected but not shown here
- L2B showed lower ductility than L1B



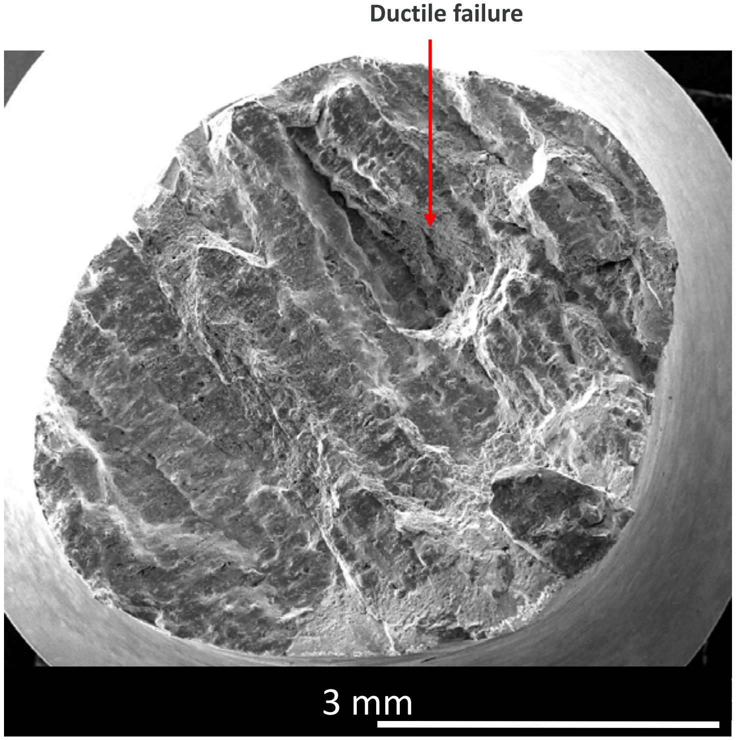


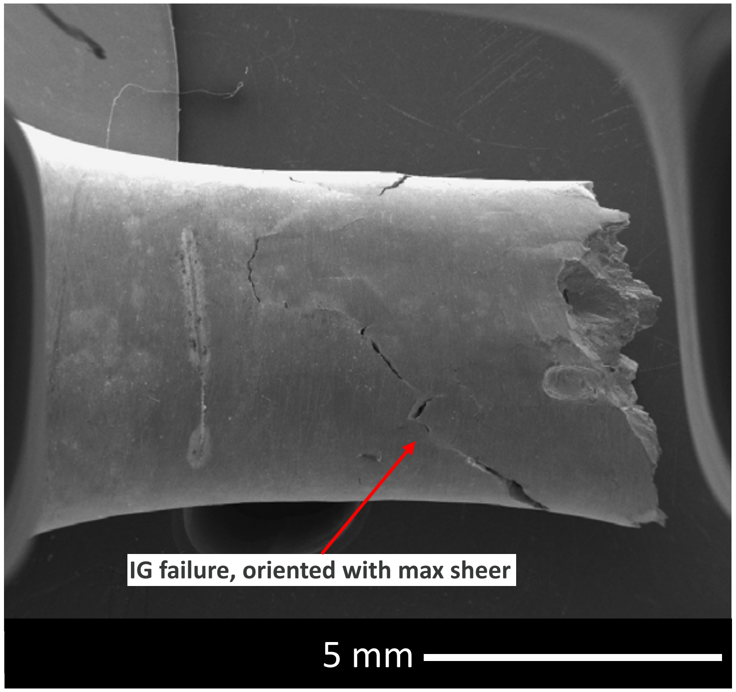


Failure mode

As observed from “L2B” heat

- Mostly intergranular / inter-dendritic failure
- Very limited ductility
  - When observed associated with max shear plane
- Similar features observed in both creep and tensile

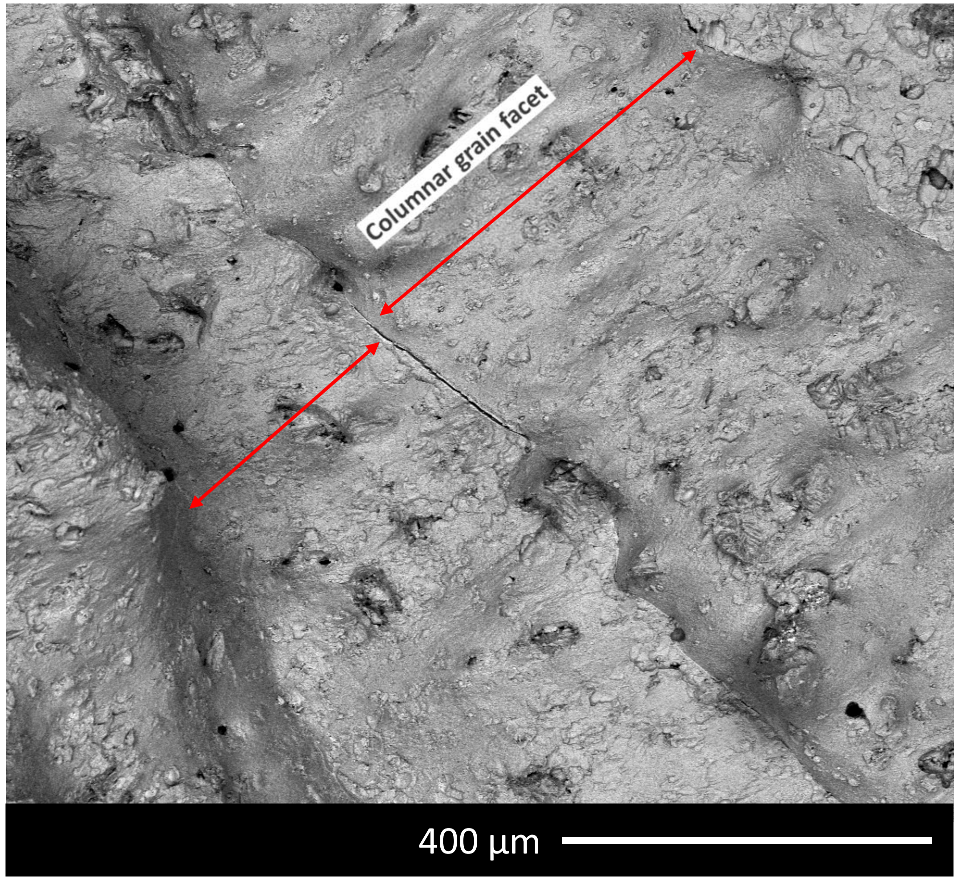


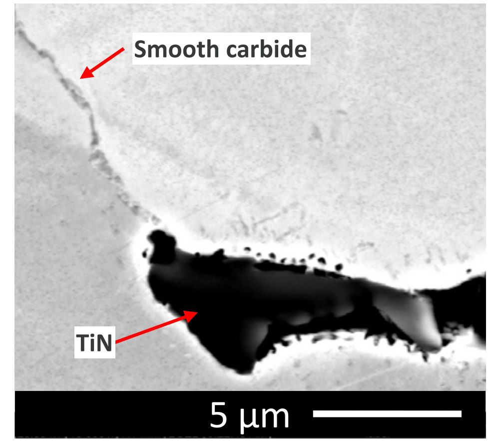


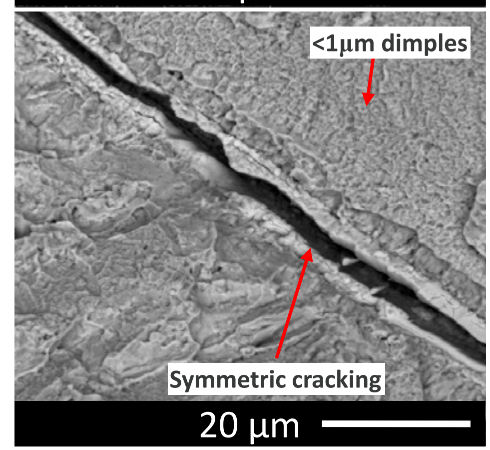
Failure mode

As observed from “L2B” heat

- Ductility extremely limited
  - Sub micron size dimples
- Carbide morphology thick and smooth
- Nearly symmetric cracking of La GB PPT
  - Further suggests La PPT are not root of failure
- TiN phases detected in centers of grains, with ductile dimple surrounding PPT
- Hypothesis:
  - The morphology and location of carbides lead to IG failure with limited ductility as stress is unable to transverse carbides



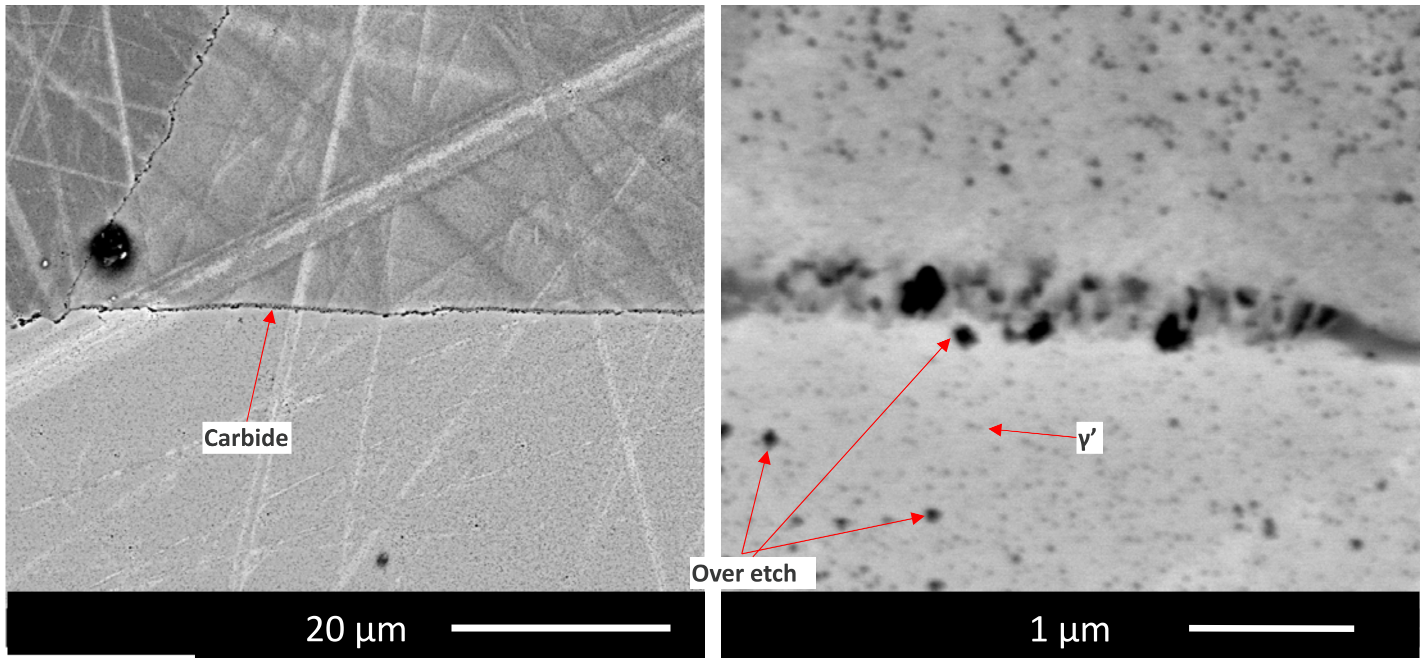


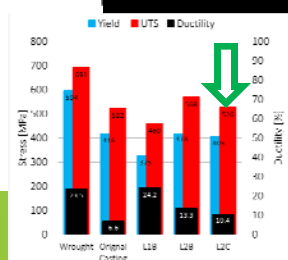


Microstructure

As observed from “L2C” heat

- Boron was added to strengthen grain boundaries
- Used master alloys absent of La, N
  - Absence of TiN and La PPT from previous “heats”
- “Smooth” carbides coat the grain boundaries
  - ~250-500 nm in thickness
  - Carbon measured ~300-400 PPM
- Note: Sample may be over etched, rather than bi-modal γ

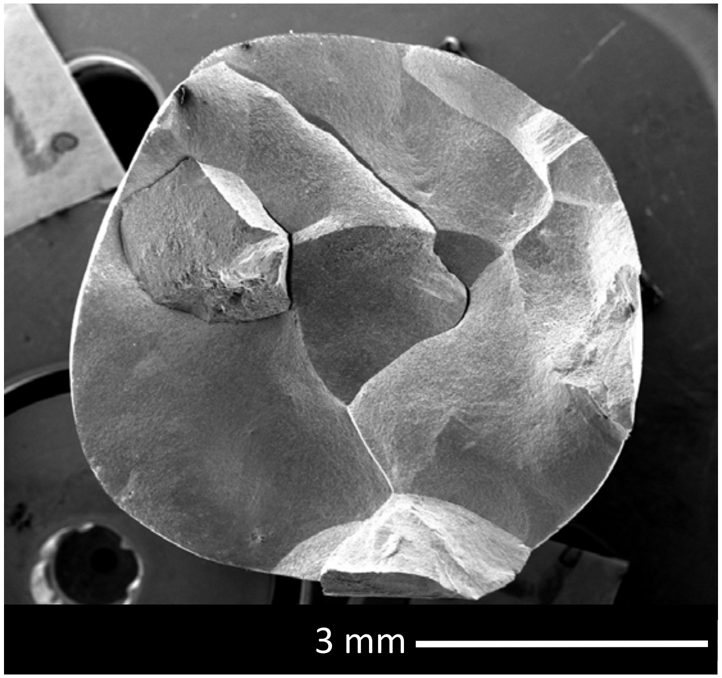


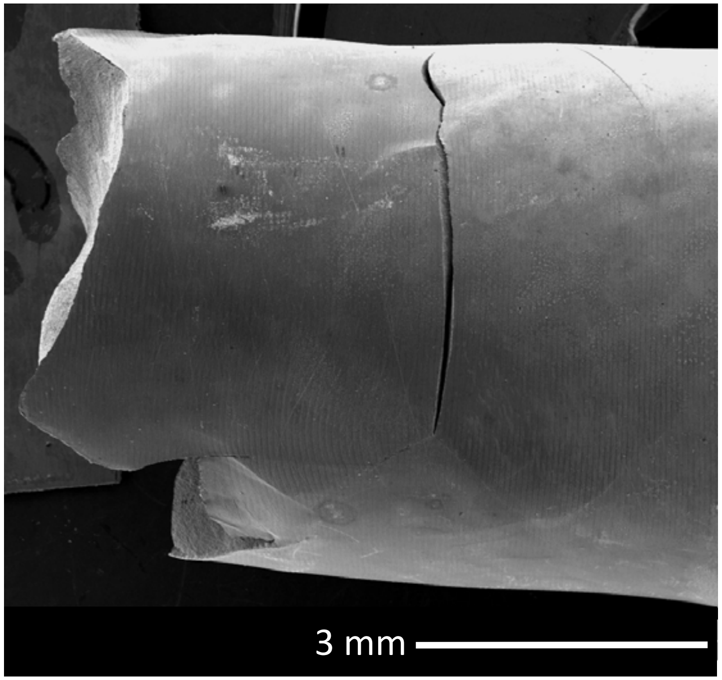


Failure Mode

As observed from “L2C” heat

- Complete intergranular failure observed
- Both creep and tensile samples
- Significant secondary cracking along grain boundaries

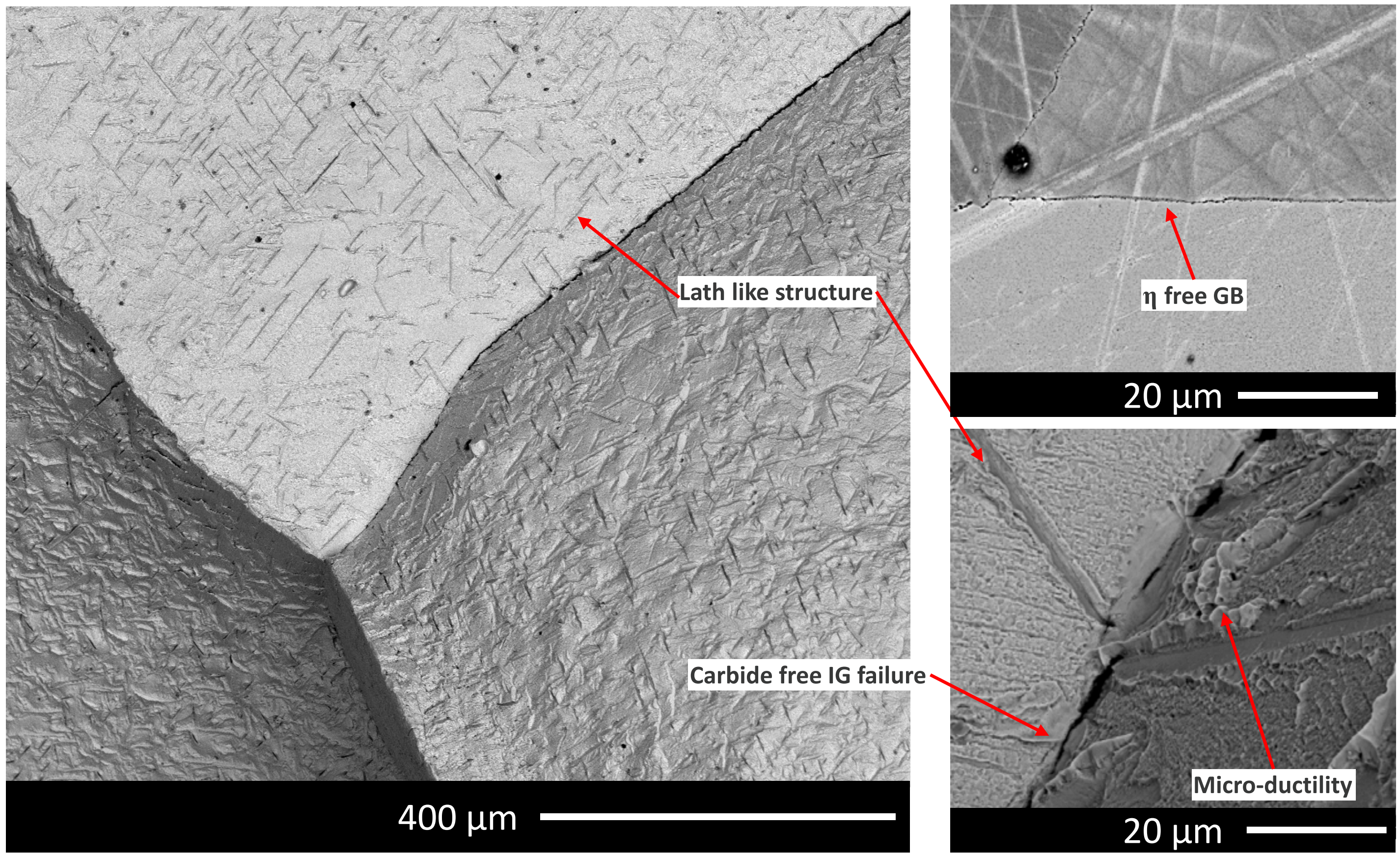




Failure Mode

As observed from “L2C” heat

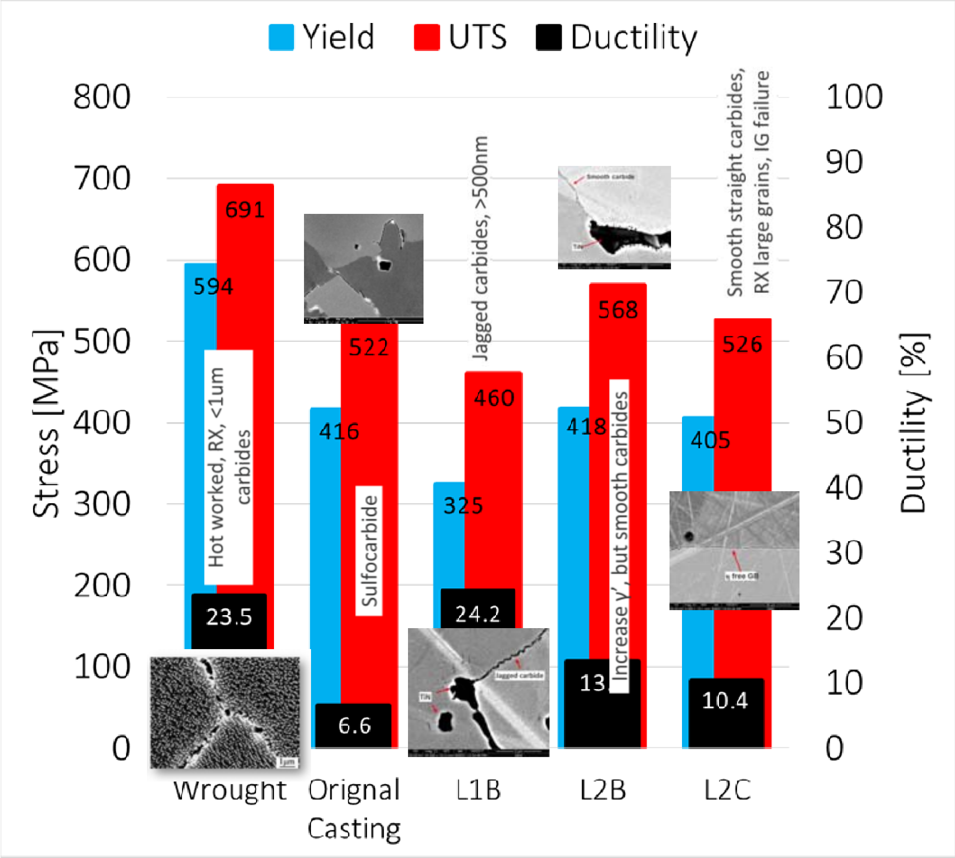
- Lath like structure observed on grain facets
  - Likely η phase or $M_{23}C_6$ (Cr)
  - No η detected from xrd
- No detection of carbides at IG failure



Summary

Observations from casting IN740H

- Work continues to optimize mechanical properties of cast versions of IN740 to improve:
  - Low tensile strength
  - Low creep ductility
- Niobium rich carbosulfides were detected and eliminated with lanthanum additions
  - Poor mechanical properties were measured
- Increase in molybdenum content to eliminate cellular carbide formation
  - Somewhat successful in avoiding cellular carbides, however mechanical performance remained poor
- Increase in boron content to enhance grain boundary ductility
  - Marginal change in mechanical properties
- Smooth thick carbides tended to reduce mechanical properties while the jagged morphology tended to increase ductility



Future work

Possible ways to improve performance

	% Wt												Strengthener		PPM					
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Nominal	0	0.15	24.5	bal	20	0.1	1.5	1.35	1.35	0	-	-	4.20	1	300	-	-	-	-	-
Max	1	1	25.5	bal	22	2	2.5	2.5	2	3	0.5	0.03	7.00	1.25	800	-	-	300	60	-

- Decrease grain size to increase the surface area of grain facets / limited by heat conduction
  - Would reduce GB carbide thickness—but is difficult to achieve in large castings
- Modify carbides via heat treatment
  - Thick carbides appear to be contributing to poor mechanical properties (ductility).
  - However, carbides may continue to grow under creep so they need to be stabilized (lower ductility seen in creep)
  - Chemistry changes: Fairly tight chemical ranges—the cast version may need to expand beyond the wrought range
  - Reduce carbon to < 100 PPM to reduce GB carbide thickness
    - (however, higher C generally is better for castability)
  - Increase carbide formers; Mo, Nb, Cr, Ti
  - Increase boron to 0.01% wt (slightly above specifications, 100 PPM) , or add zirconium (150 PPM) to increase grain boundary ductility
  - Increase Ti content, to favor eta plates; expected to act to span grains
  - Experiments continue

Thank you

Questions?

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