LIBERTY
POWDER METALS

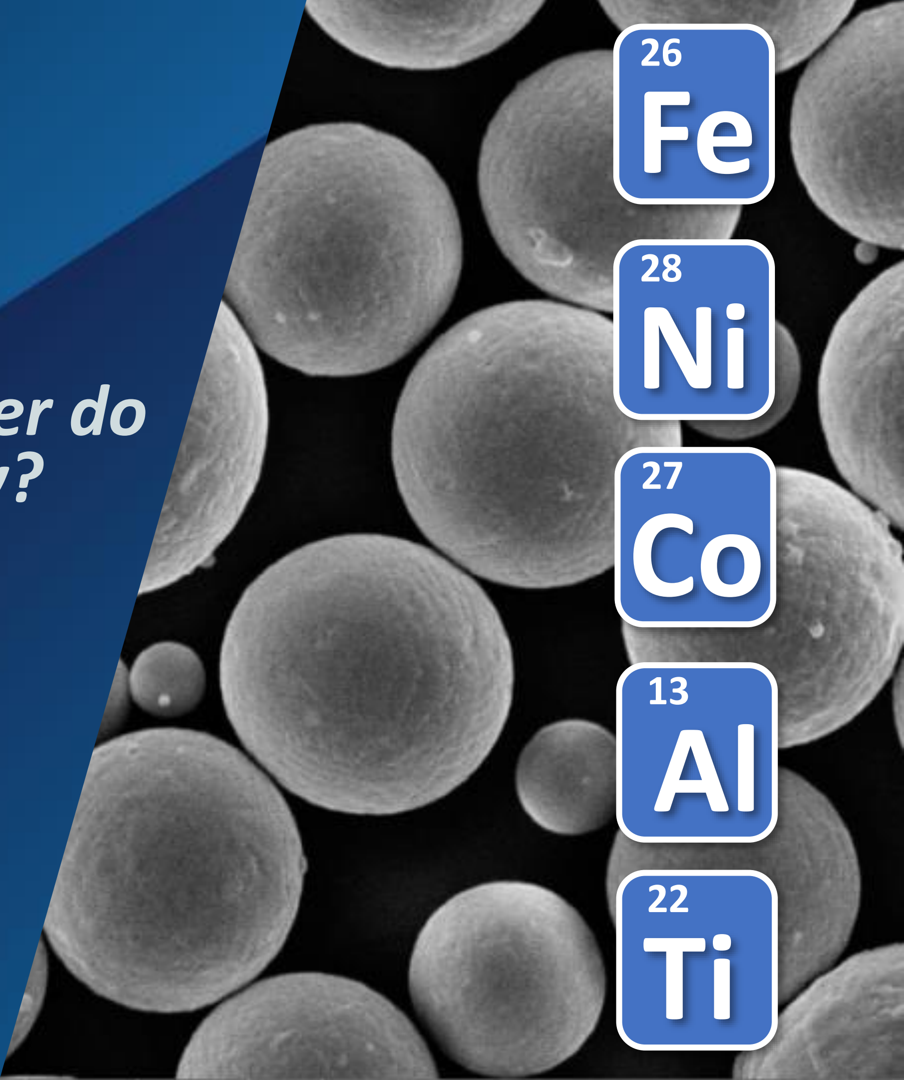
What can powder manufacturer do to improve HIP product quality?

Dr. Xinjiang Hao

EPMA HIP Seminar
8th – 9th March 2022

MEMBER OF
GFG ALLIANCE

LIBERTYSTEELGROUP.COM



Introduction

Liberty Powder Metals is a **UK powder metals producer** and supplier established by **Liberty** who are specialists in metal manufacture, processing and trading along with the production of value-added engineering products from steel, aluminium and other alloys.

Operating as a separate business unit, we are proudly supported by the **Tees Valley Combined Authority**.

We provide a range of vacuum inert gas atomised powders and plasma atomised powders, all of which are optimised for Additive Manufacturing and PM-HIP

Powders engineered for a greener future

Capabilities

State of the art **vacuum inert gas atomiser** which produces **steel**, **nickel** and **cobalt** based **alloys** delivering **highly spherical powders** of **consistent shape,** utilising **anti-satellite technology**

Processing, characterisation and **optimisation** for steel, nickel, cobalt, aluminium and titanium alloys.

Vacuum Induction Melting

250kg batch size

Annual capacity of 400 tonnes

Fe, Ni, Co

25% R&D commitment

Atomisation

State of the art

Close-coupled inert gas (argon) atomization

Anti-satellite technology

Processing

Application specific particle sizing

Additive Manufacturing
PM-HIP

Sieving, blending, and optimisation carried out under inert atmospheres

Characterisation

Suite of standard powder analysis techniques

Particle size distribution
Hall/Carney flow
Density
ONH
Full chemistry

Our powder journey started in 2015

LIBERTY POWDER METALS

2015-2019 CASCDE project :

- £18m government funded R&D project collaborated with partners in the supply chain
- Investigated production of **high quality metal powders** for Net-Shape and Additive Manufacturing (NSAM) in key industrial sectors
- Developed understanding of how powder characteristics affect NSAM processes and part properties

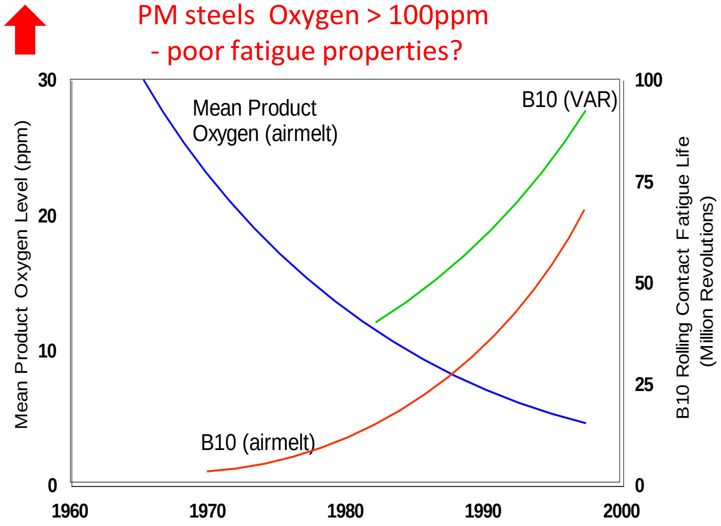
HIP, AM, MIM

Powder → NSAM process → Parts

2019 – 2021 Powder plant installation and commissioning

Zero defects?

- Properties such as fatigue, toughness, and corrosion of PM components are strongly affected by defects:
  - Porosity - Hot Isostatic Pressing (HIP) can produce 100% dense parts, eliminating pores.
  - Inclusions - have a huge impact on HIP part properties. Non-metallic inclusions in steel: oxides, sulphides, silicates, nitrides, etc.



Wrought product (bearing steel)

What have we done?

Inclusion evolution from feedstock to part:

- Inclusions in 316L (17-4PH) feedstock
- Inclusions in **air** and **vacuum** melting gas atomised powders
- Inclusions in HIP samples
- Inclusions in additive manufactured samples
- Inclusion type, size, morphology and distribution

→ **Effect of inclusion on properties**

Feedstock

↓

Powder

↓

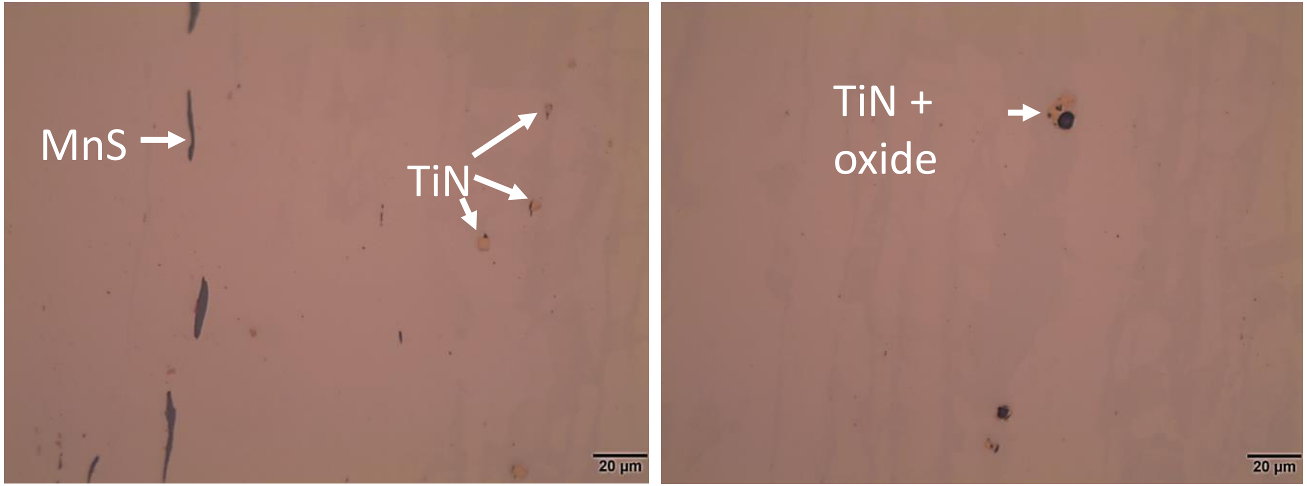
Part

Air or vacuum melting N_2 gas atomisation at sub-contractors

Hot Isostatic Pressing (HIP)

Inclusions in 316L bar feedstock

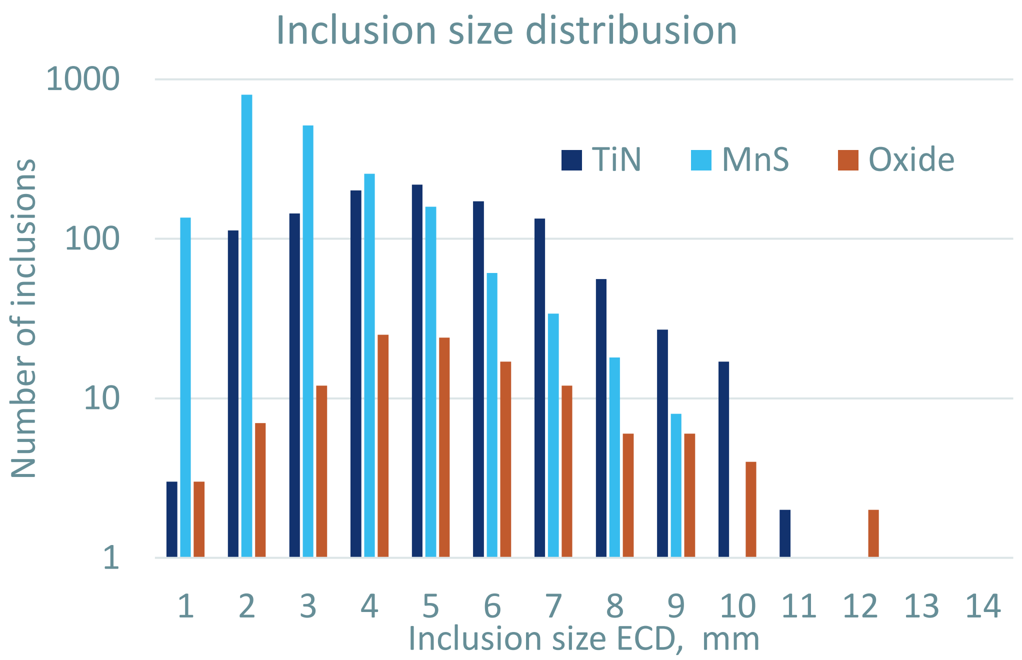
How does feedstock affect powder quality?



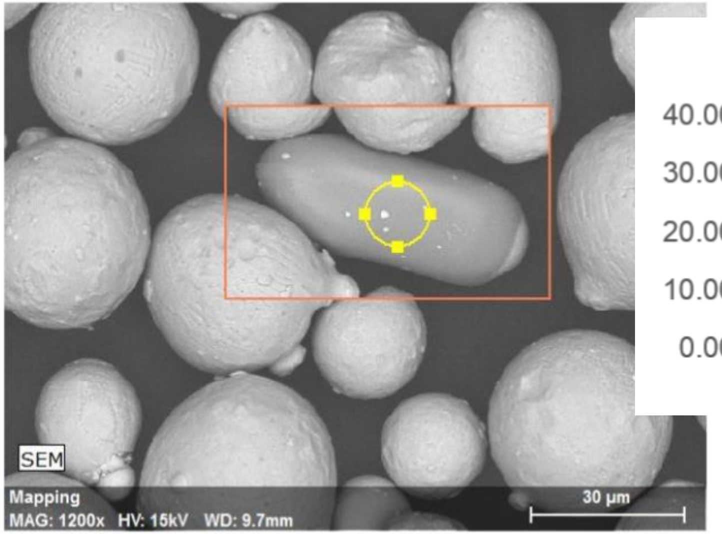
- Elongated MnS along rolling direction (anisotropy)
- Large amount of TiN particles (316L + Ti)
- Oxides co-located with TiN

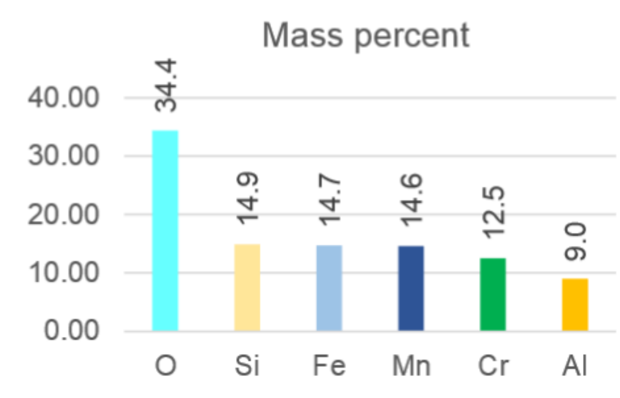
Inclusions in 316L bar feedstock: automated SEM inclusion analysis

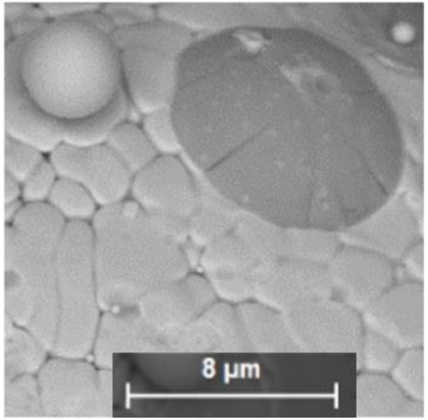
- MnS, oxide, and TiN inclusions
- Most of inclusions are less than 10 µm
- A few large oxide inclusions are larger than 10 µm



Inclusions in air melt powders



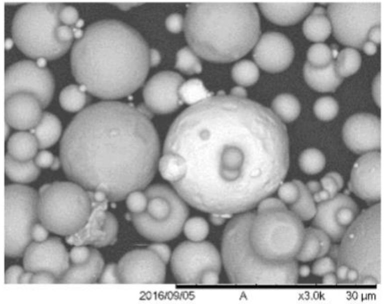


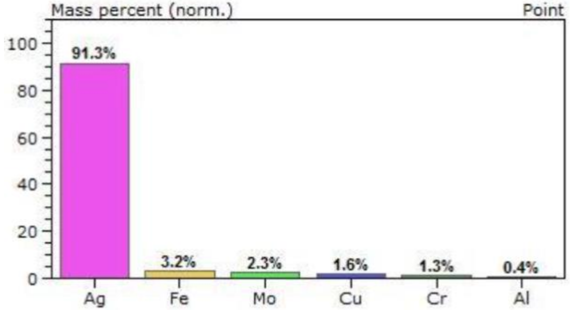


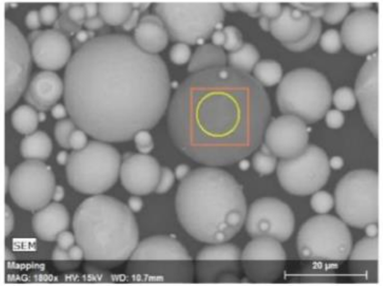
Inclusions from slag, either as separate particle or cap on powder surface

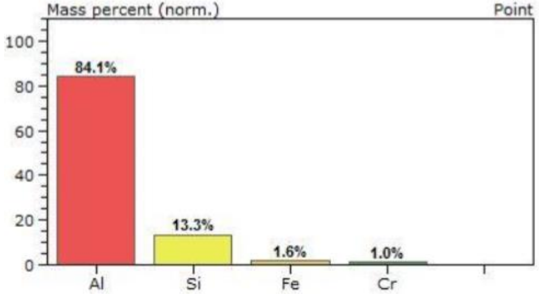
Contaminants in vacuum melt powders

- Contaminants: Al, Ag, Au particles
- How to avoid cross contamination?



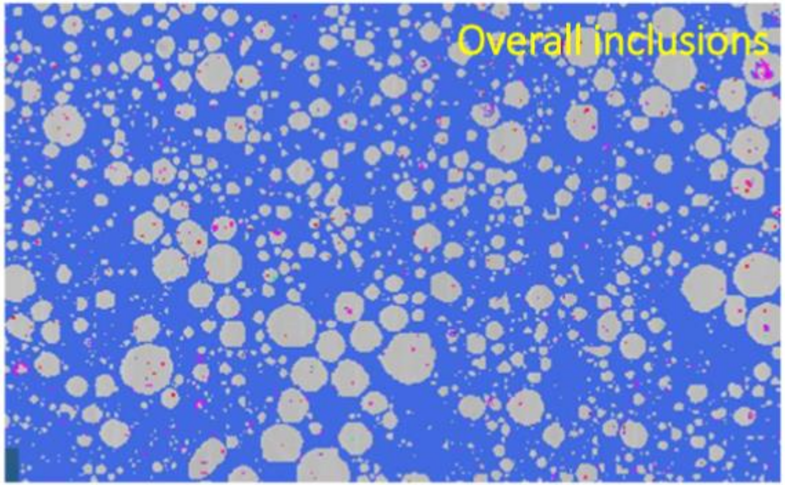




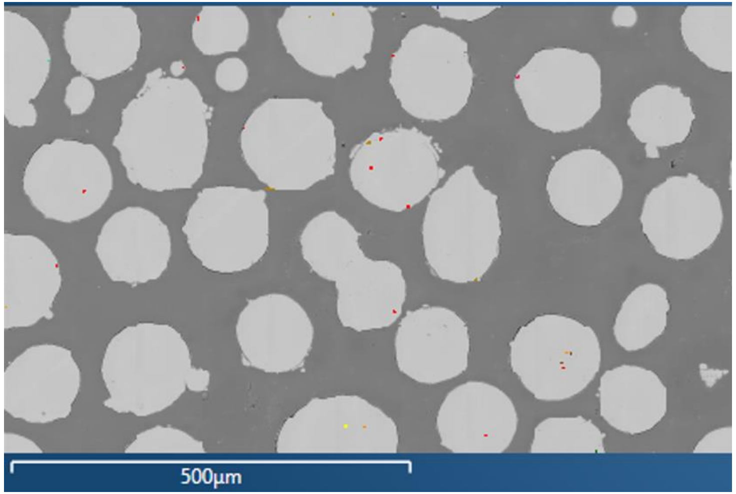


Inclusions inside of powder: air vs vacuum melting

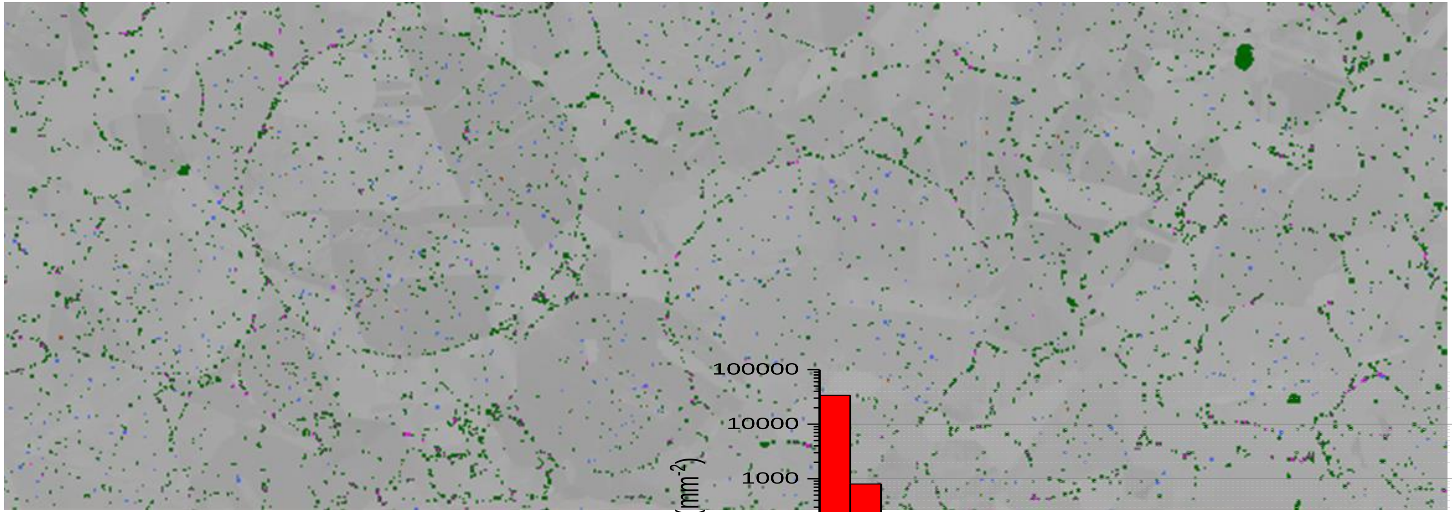
Air melting

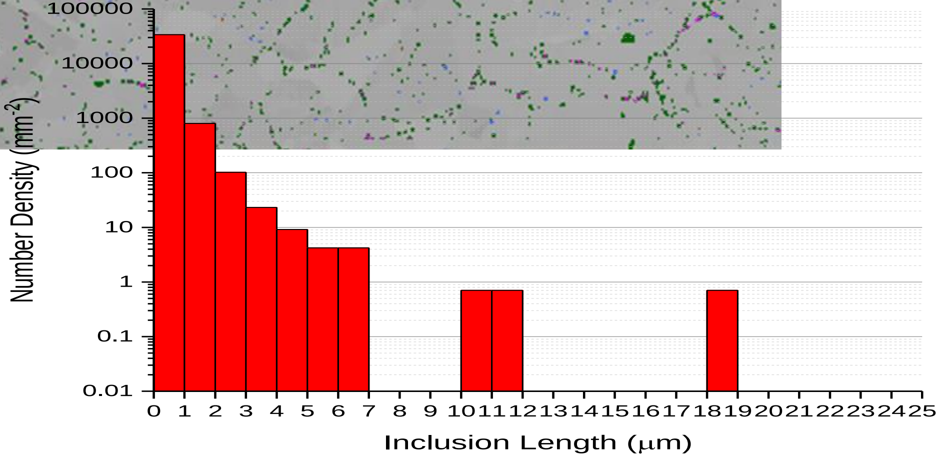


Vacuum melting

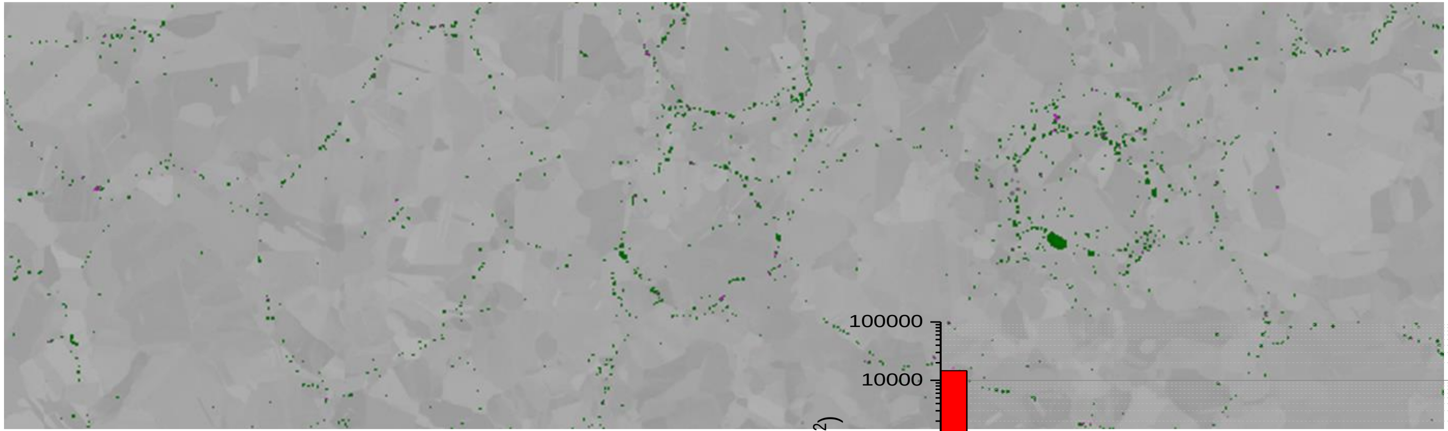


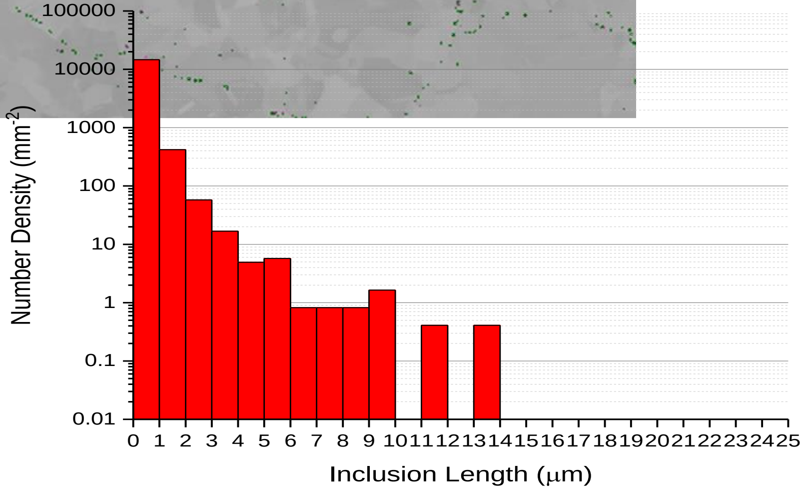
HIP: air-melt 316L



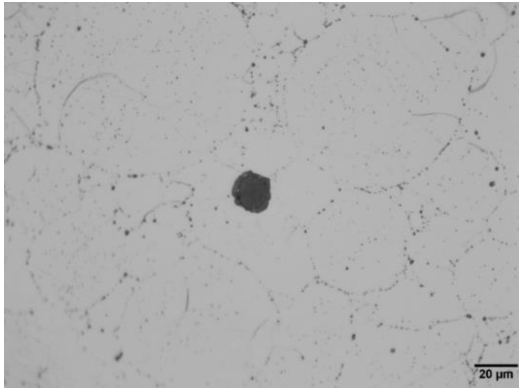


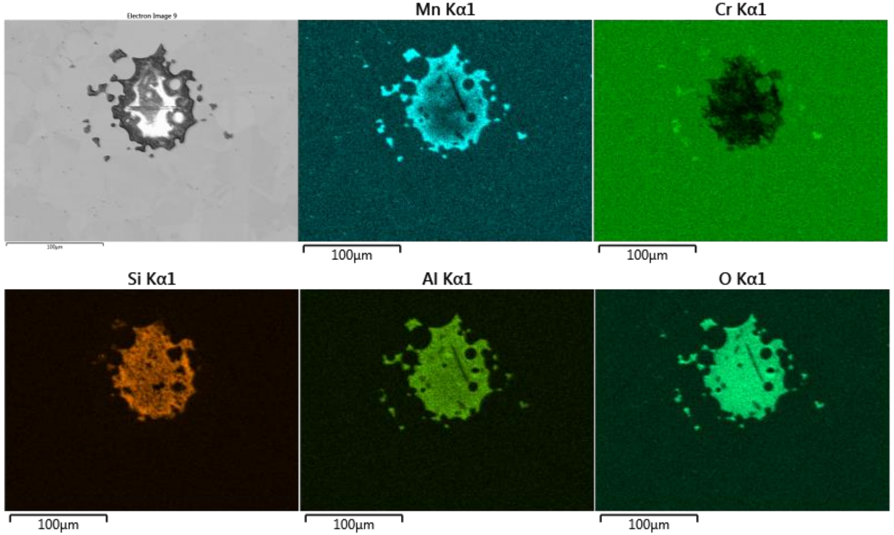
HIP: vacuum-melt 316L



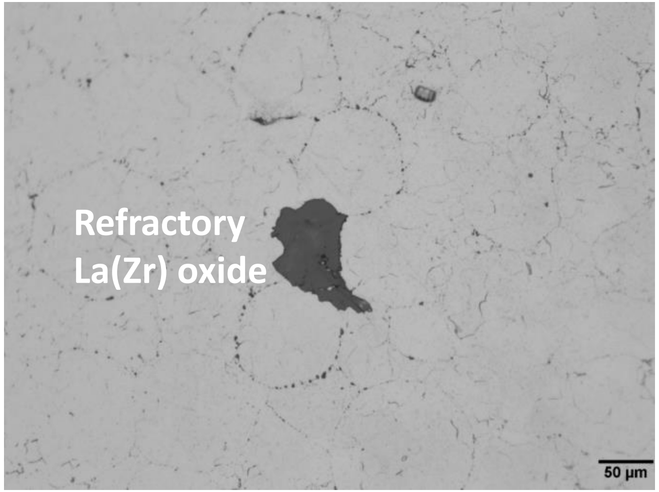


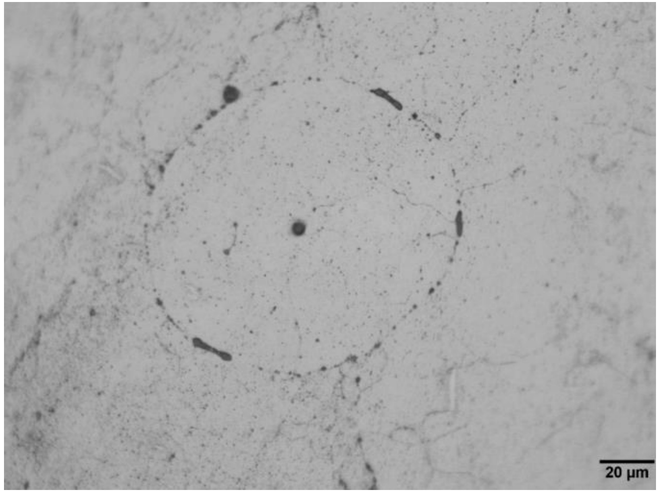
HIP: air-melt 316L



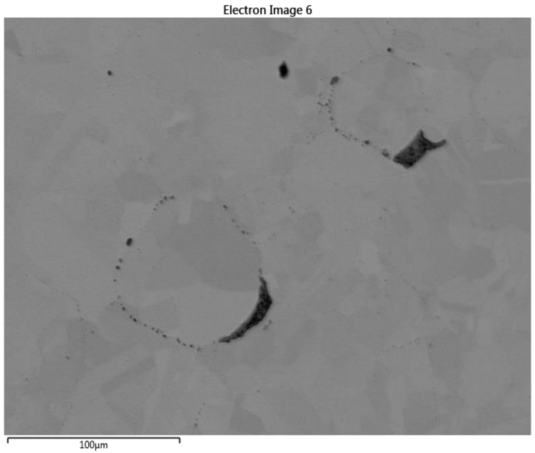


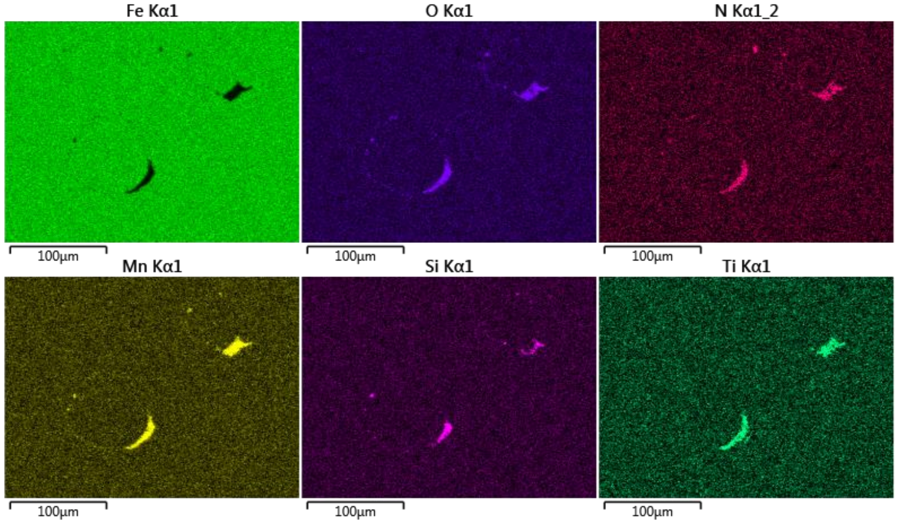
HIP: vacuum-melt 316L





HIP: vacuum-melt 316L (106-150)

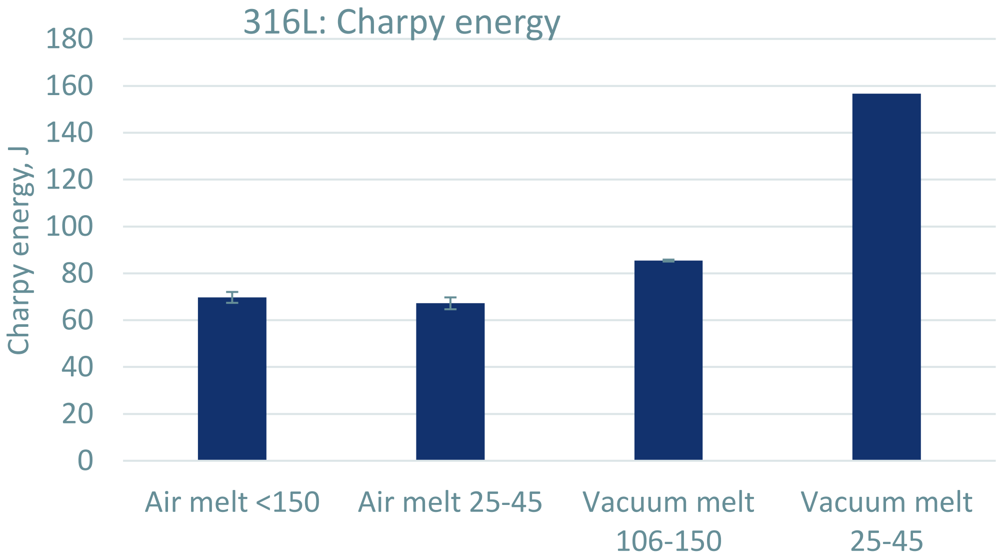




Analysis results of large inclusions (>2 μm)

Sample	Number density (/mm^2)	Number of inclusions			
		TiN	Mn(Ca)S	TiAlMg oxide	MnCrSi oxide
Feedstock	75.4	532	699	39	
Air-melt powder	32.9			24	270
Vacuum-melt powder	9.6			5	28
HIP (air-melt)	31.3		1		68
HIP (vacuum-melt)	21		4		43
AM (air-melt)	9.0			26	153

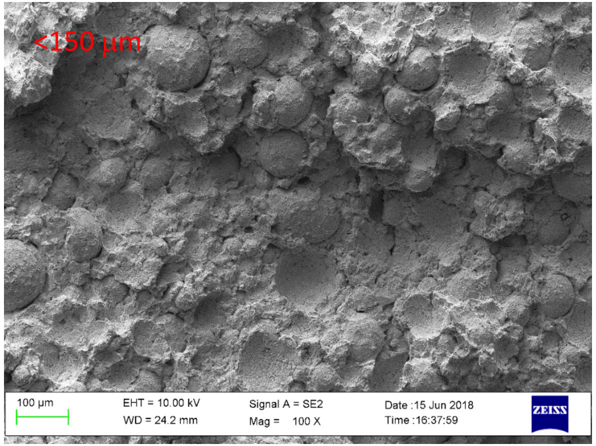
HIP 316L: mechanical property

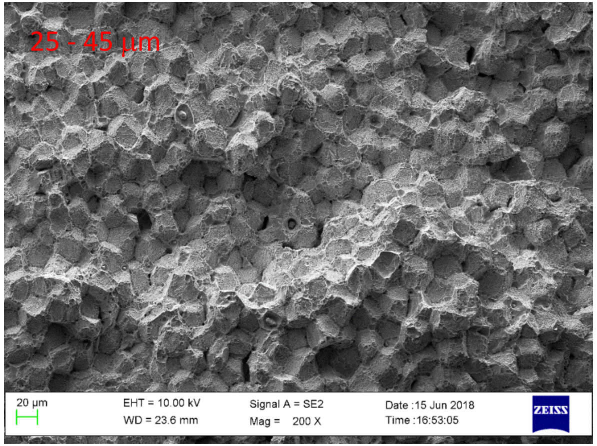


- Poor Charpy impact property compared to wrought 316L (>250 J)
- Vacuum melt powder gives better property
- Role of inclusions

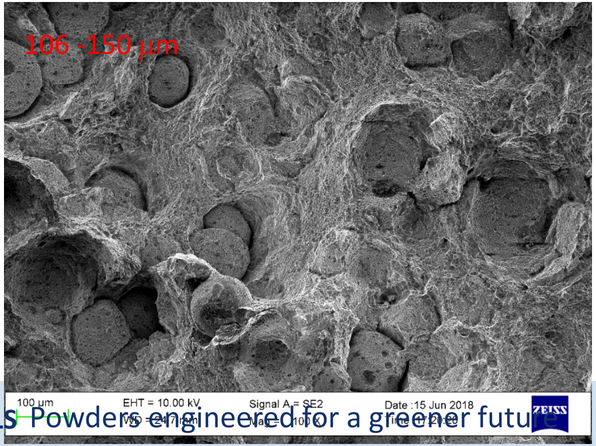
HIP 316L: fracture

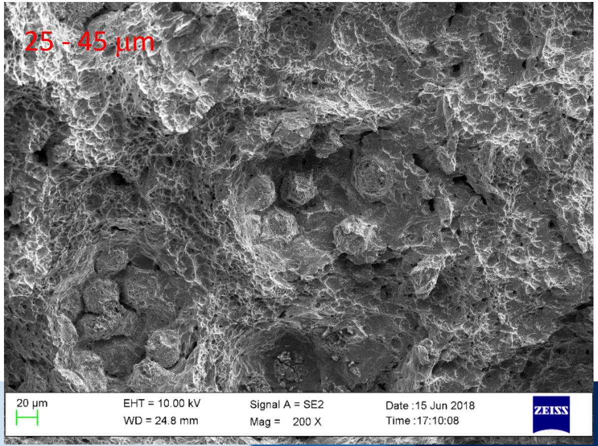
Air melt





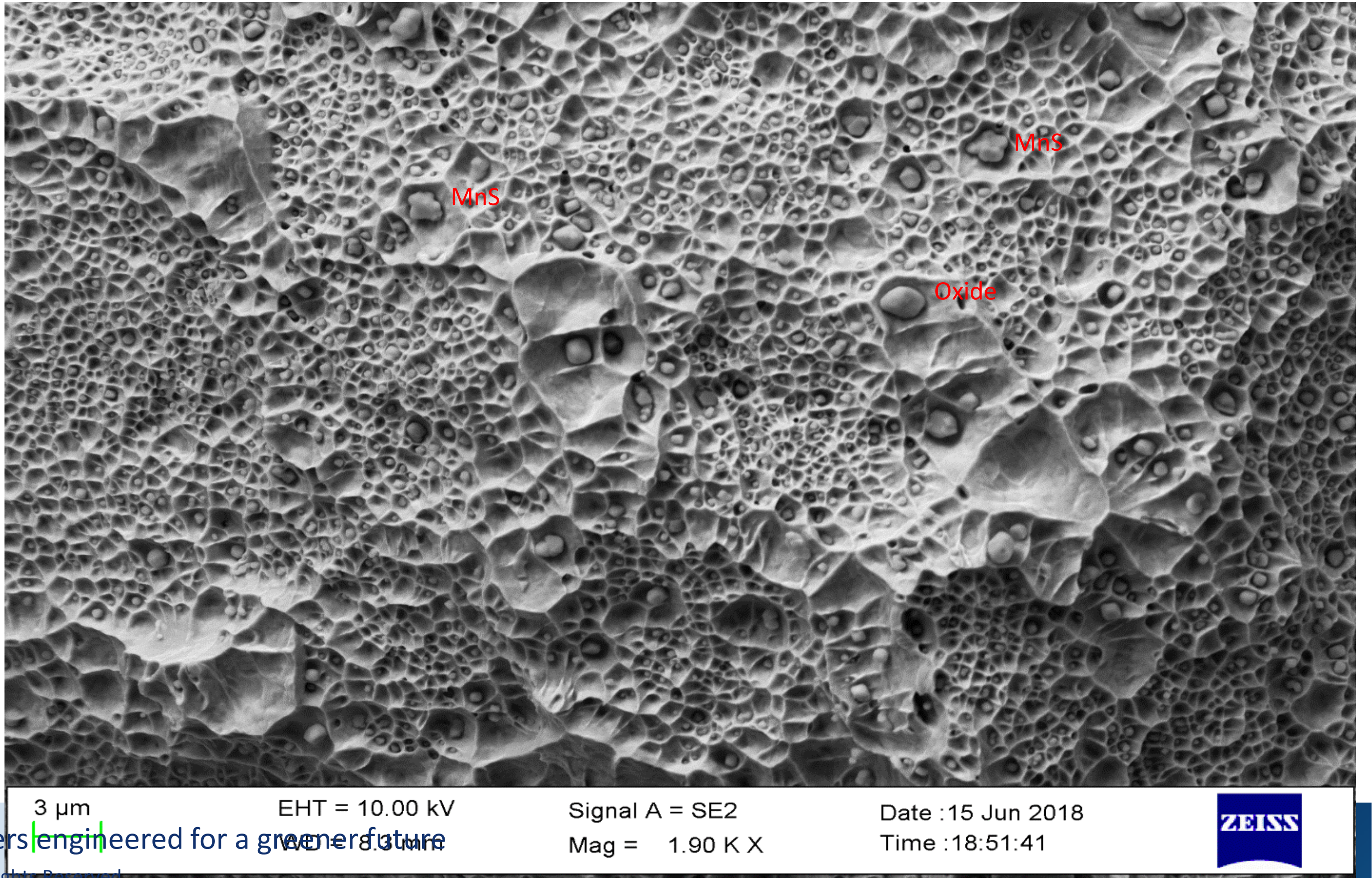
Vacuum melt



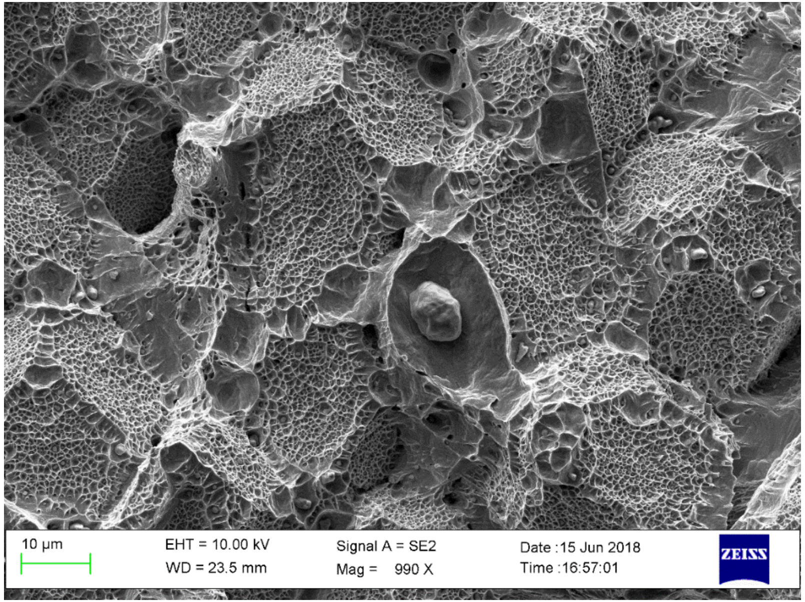


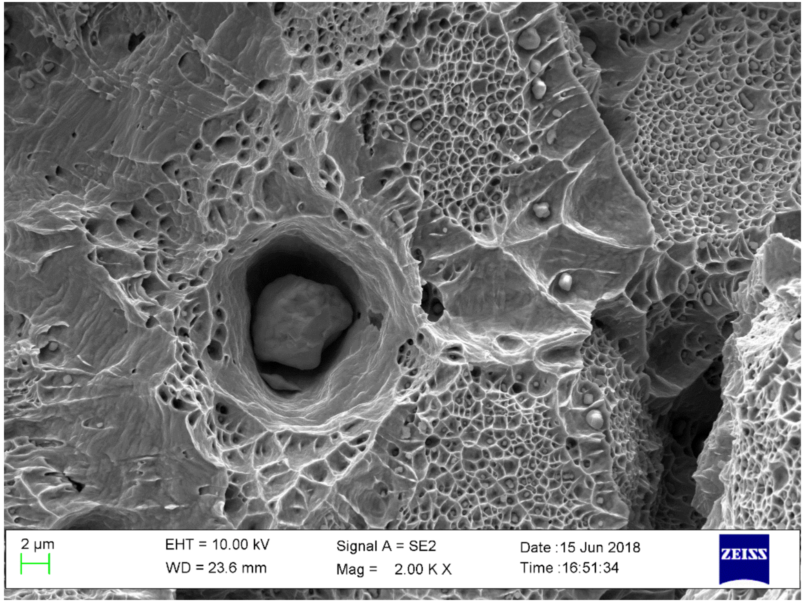
Air melt 316L <150 μm:

- Debonding at prior powder boundaries (PPBs)
- High number density of inclusions (oxides, MnS) at PPBs



Air melt 316L 25 - 45 μm:



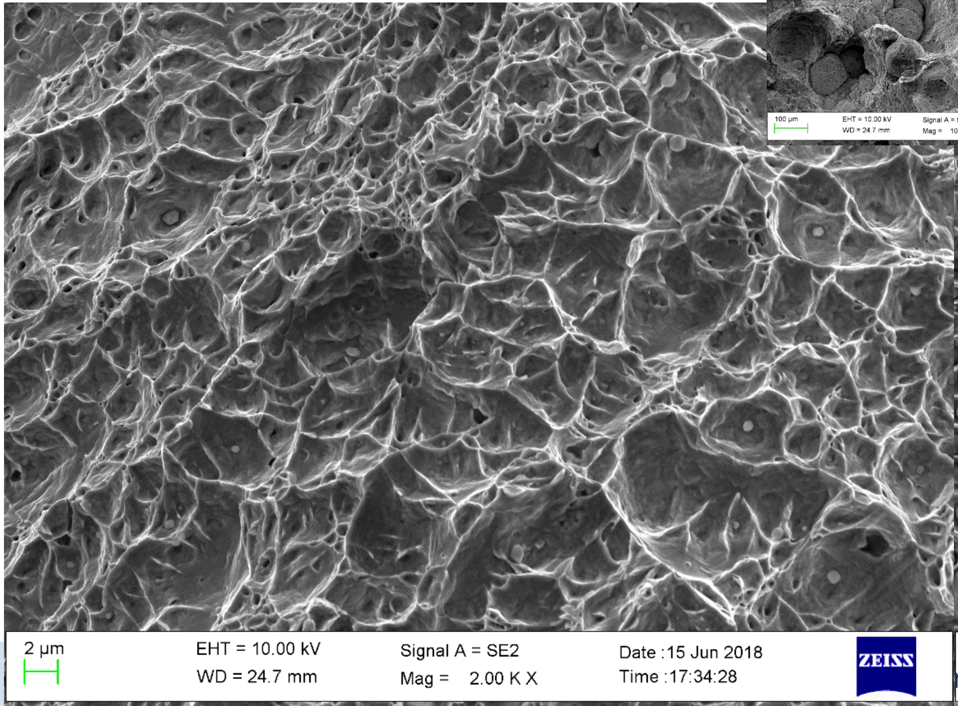


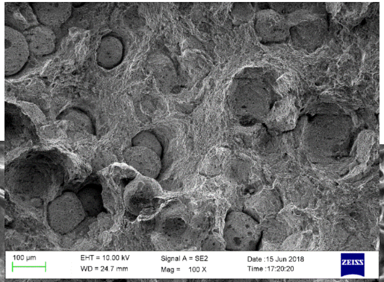
- Debonding at prior powder boundaries (PPBs)
- High number density of inclusions (oxides, MnS) at PPBs

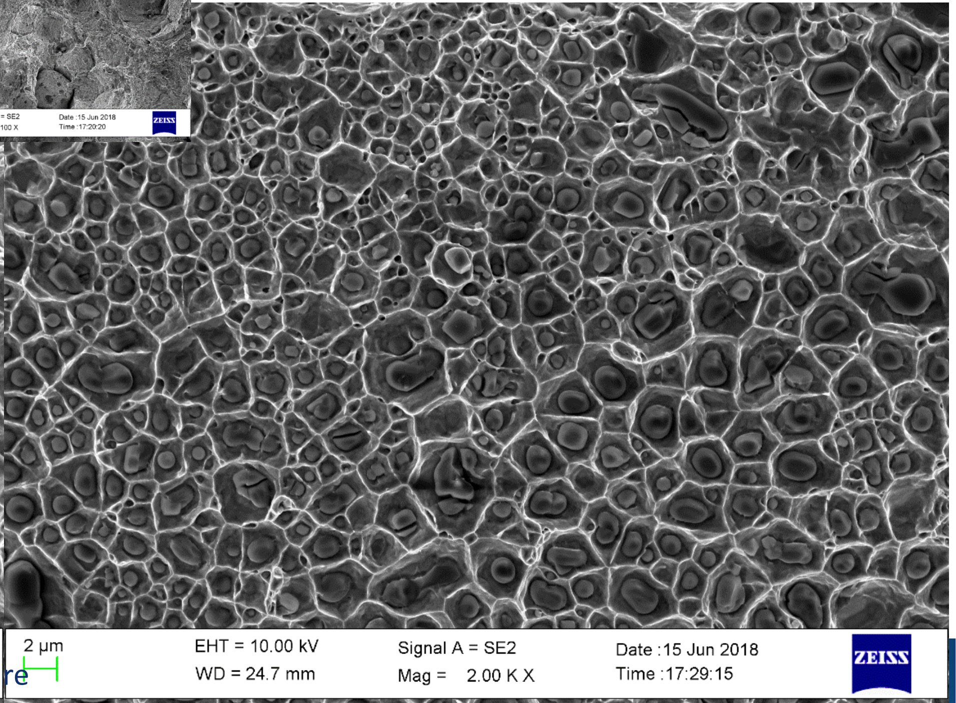
Vacuum melt 316L 106 - 150 µm:

Trans-granular crack

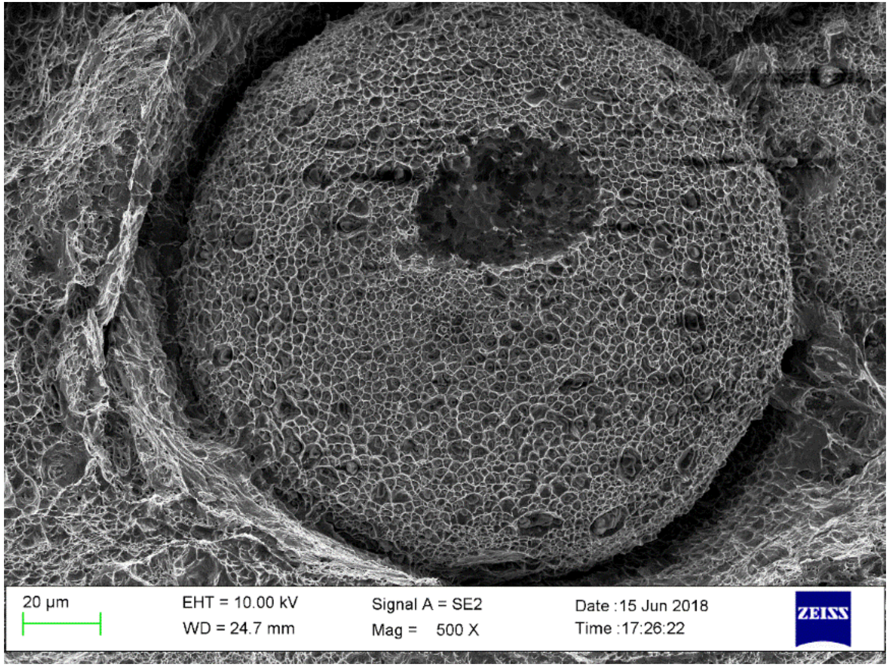
Inter-granular crack

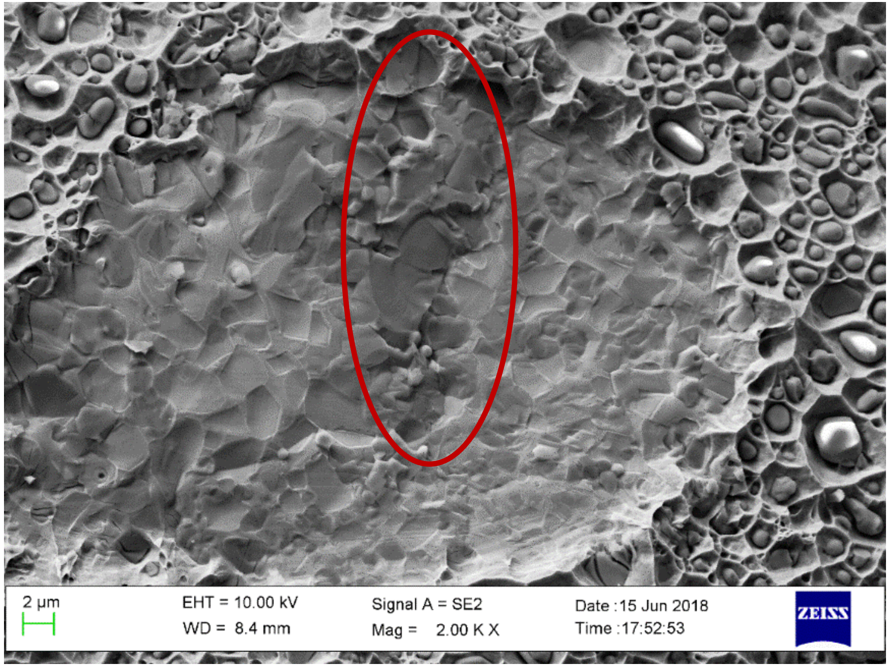






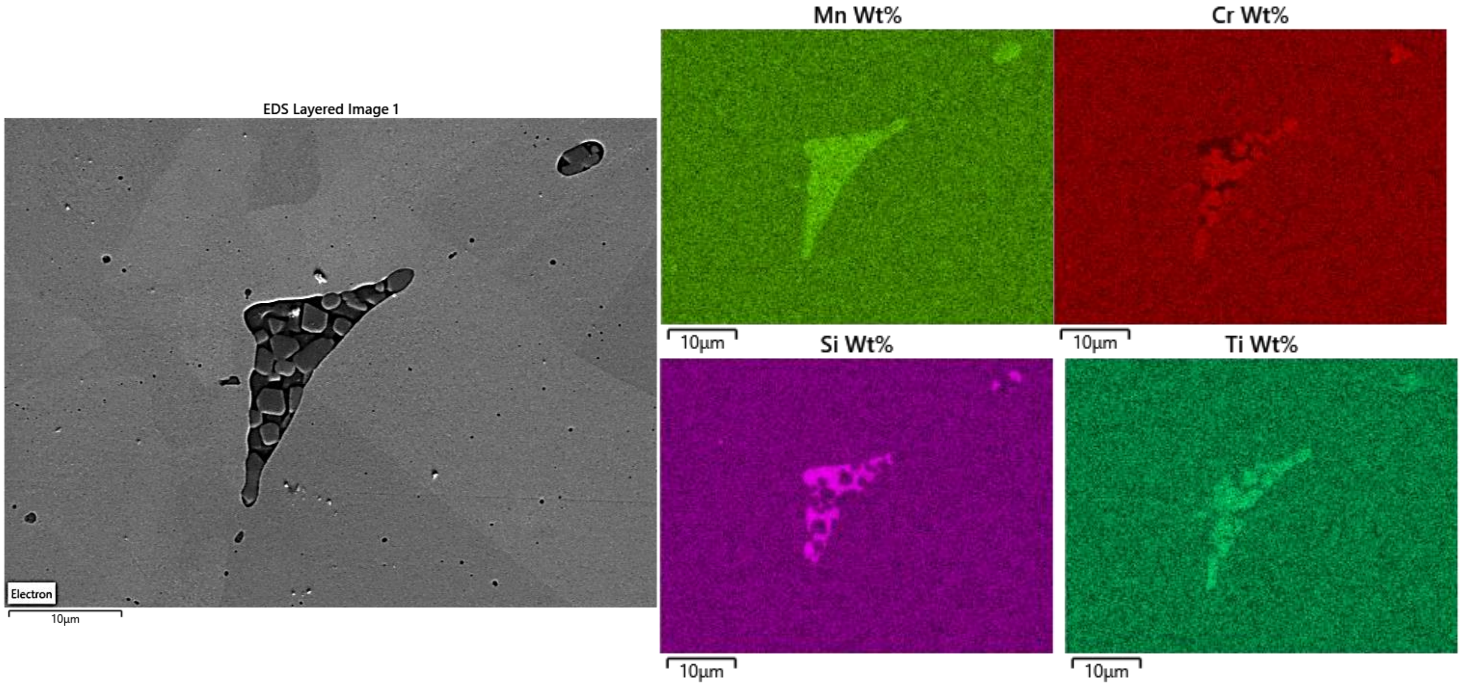
Vacuum melt 316L 106 - 150 μm:





- Fracture initiated at large Inclusions (oxides and MnS)

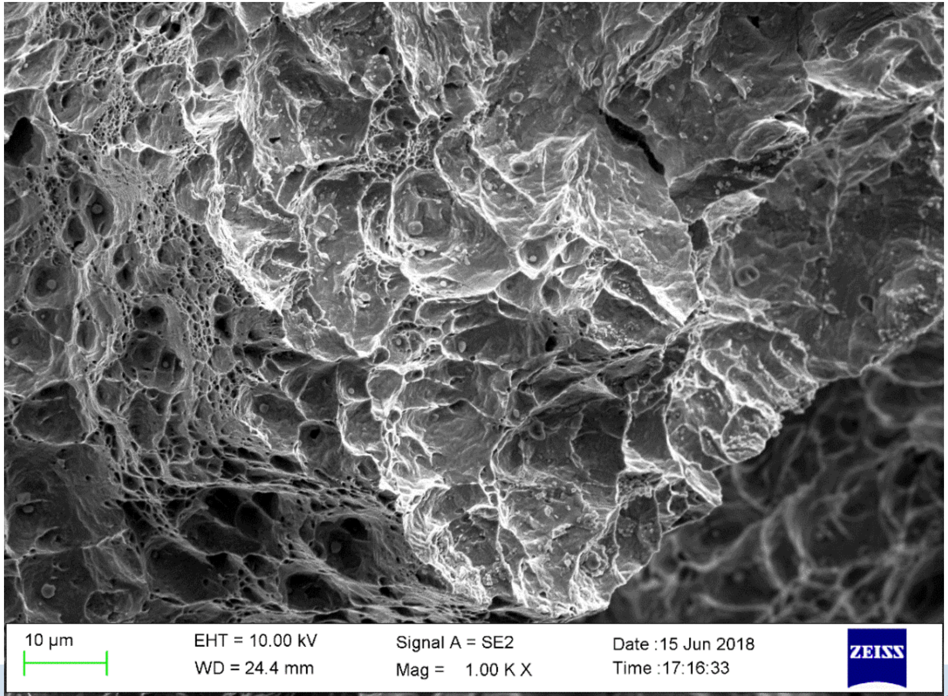
Vacuum melt 316L 106 - 150 µm:

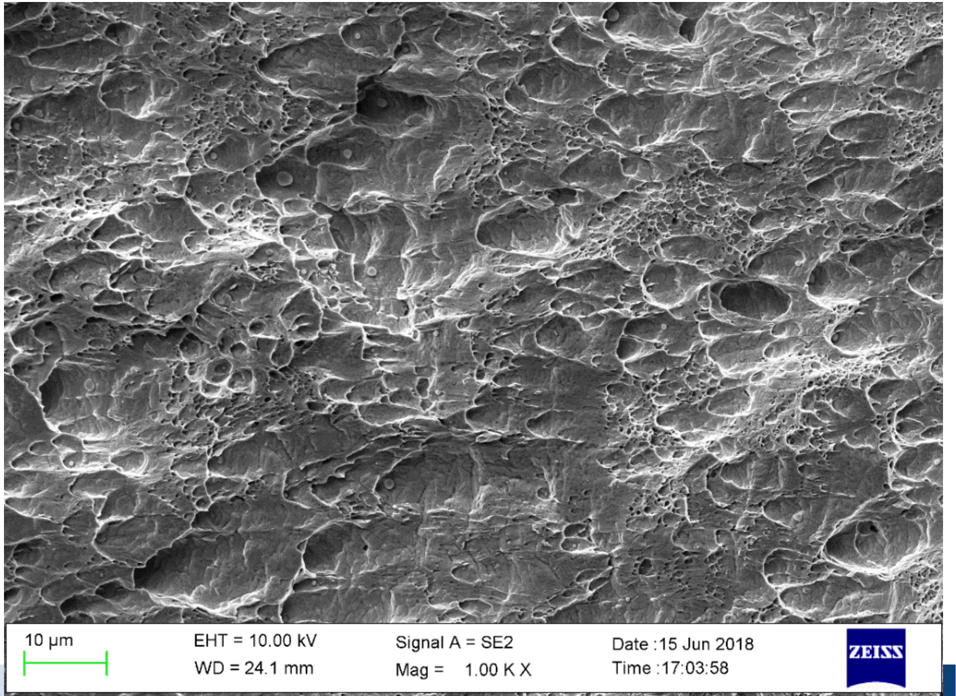


Cr(Mn, Ti) oxide + Mn(Si) oxide

Vacuum melt 316L 25 - 45 µm:

- Trans-granular fracture
- Fewer inclusions on fracture surface





Summary

- ✓ Although MnS is the most common inclusion in wrought steels, MnS was not found in the 316L atomised powders. Sub-micrometer MnS particles precipitate during HIP.
- ✓ Most inclusions in powder and HIP component are Mn(SiCr) oxide
- ✓ Inclusions on powder surface and subsequently PPB cause de-bonding during fracture, which significantly reduces toughness – powder surface cleanliness is a key factor.
- ✓ Cleaner powder produced through vacuum melt gas atomisation gives the best properties after HIP.

What can powder manufacturer do to improve HIP product quality?

Cleaner powder = better properties

- Clean melting process – vacuum induction melting
- Select suitable refractory materials – avoid contamination from refractories
- Thorough cleaning when changing material – avoid cross contamination
- Powder handling (transferring, sieving, packaging) under inert gas – reduce oxidation and moisture pickup
- HIP cannister filling and sealing at powder plant – reduce oxidation, moisture pickup, and contamination

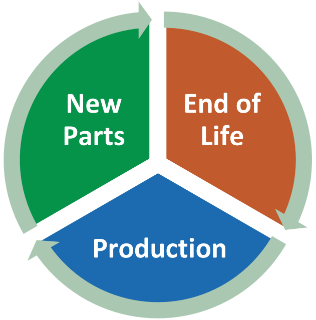
What can powder manufacturer do to improve HIP product quality?

Net shape requirements

- Good flowability
- Higher packing density and lower scatter

Economics and environment

- Recycling of waste (end-of-life, machining swarf, off-cuts, powders etc)
- Alternative PSD – Separate AM cut to reduce both AM and HIP powder costs



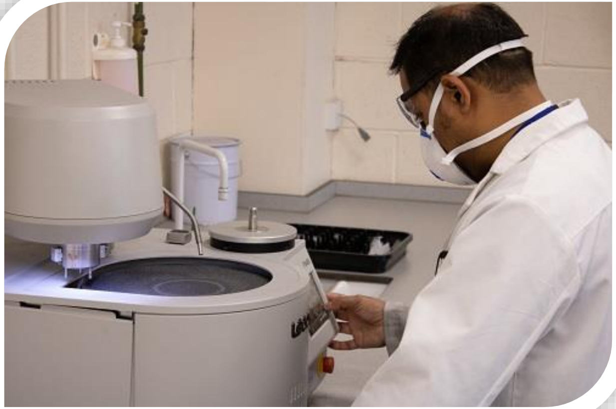
We welcome suggestions from the powder HIP community!

Collaboration

- Liberty Powder Metals is committed to R&D and next generation alloy development
  - 25% of our capacity used for research and development
  - Working in partnership with our customers
  - Creating new and advanced alloy properties
  - Advancing AM and PM HIP technology

LIBERTYPM CUSTOMPLUS+

NEXT GENERATION ALLOY DEVELOPMENT



Acknowledgements

Thanks to UKRI Future Leaders Fellowship and AMSCI-UK for financial support of CASCADE project and to all project partners:

Johnson Matthey

Renishaw

The Manufacturing Technology Centre (MTC)

Atomising Systems Limited (ASL)

Hybrid Manufacturing Technology (HMT)

University of Birmingham

Farleygreene

Advanced Manufacturing Research Centre (AMRC)

University of Warwick

LIBERTY POWDER METALS

Powders engineered for a greener future

26 Fe

28 Ni

27 Co

13 Al

22 Ti

THANK YOU

Dr. Xinjiang Hao

Principal Scientist

UKRI Future Leaders Fellow

e: Xinjiang.Hao@libertysteelgroup.com

MEMBER OF GFG ALLIANCE

LIBERTYSTEELGROUP.COM