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Project No. 601217-EPP-1-2018-1-BE-EPPKA2-SSA-B

SECTOR SKILLS STRATEGY IN ADDITIVE MANUFACTURING

CU 01: DED-ARC

Session 4.4 Feed-stock Quality

Prepared by: Steven Hall and David Wimpenny

FOR SAM PILOT ATTENDEES AND TRAINERS ONLY

MM17,21

Feed stock quality

- Powder (Steven Hall)
- Wire (David Wimpenny)

Powder Feed-Stock Quality

Steven Hall

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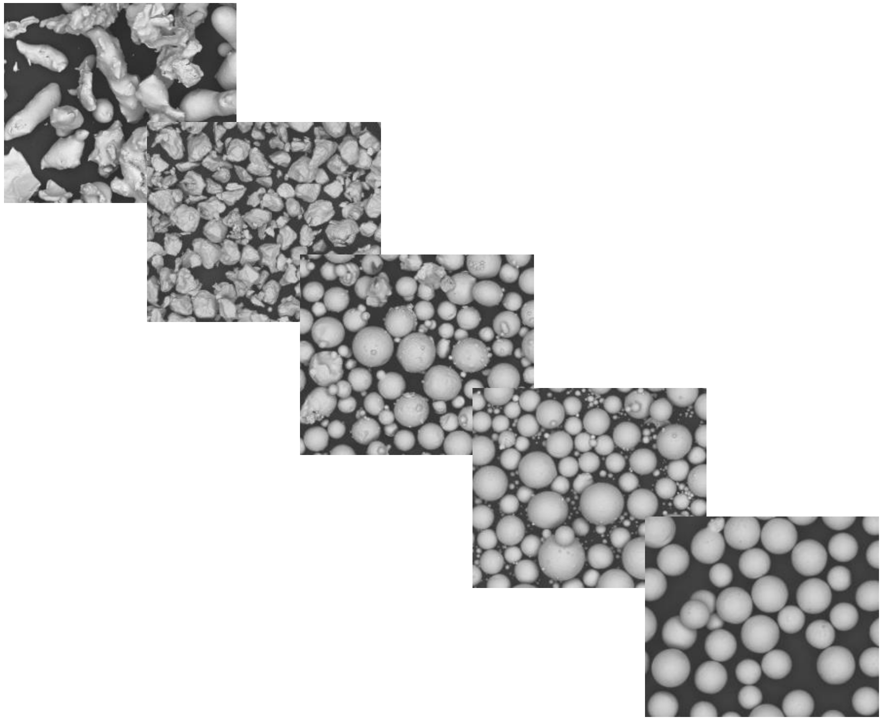
Powder Manufacturing

- Powders come in many shapes and sizes
- The physical properties (size, shape) will affect the way the powder behaves
- The powder properties are usually dictated by the atomisation process



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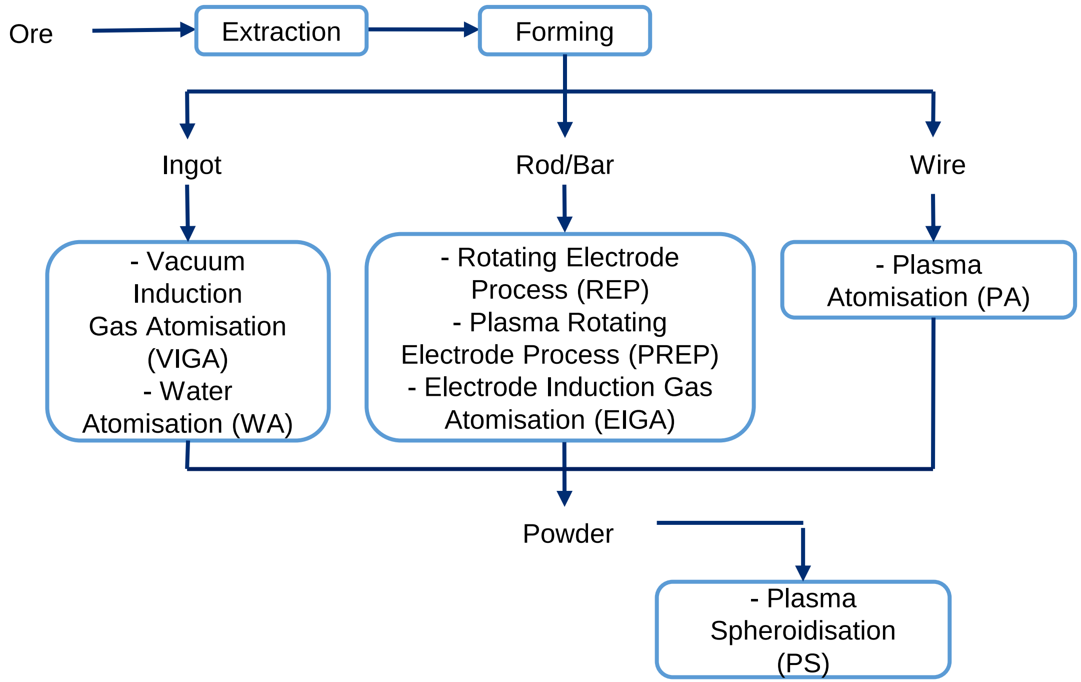
Typical atomisation process flow diagram



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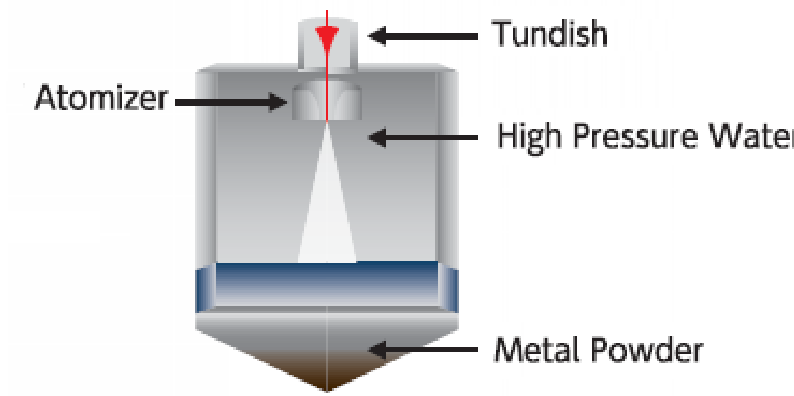
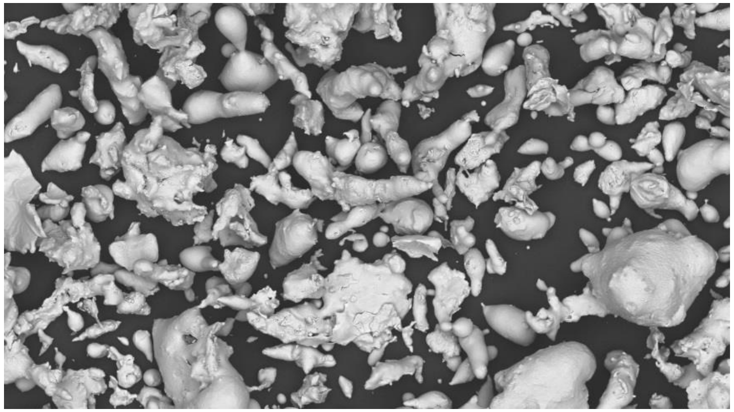
Powder Manufacturing - Water Atomisation

- Uses high pressure water jets to break-up a molten stream of metal into droplets
- Produces highly irregular particles
- Good for press-sintering applications due to the much higher green strength of components
- Very high production rates possible
- Low energy requirements for water pumping however, need to dewater and dry powder after atomisation
- Maximum particle size is much less influenced by atomizer vessel size



http://www.atmix.co.jp/en/e_powder_atomization.html



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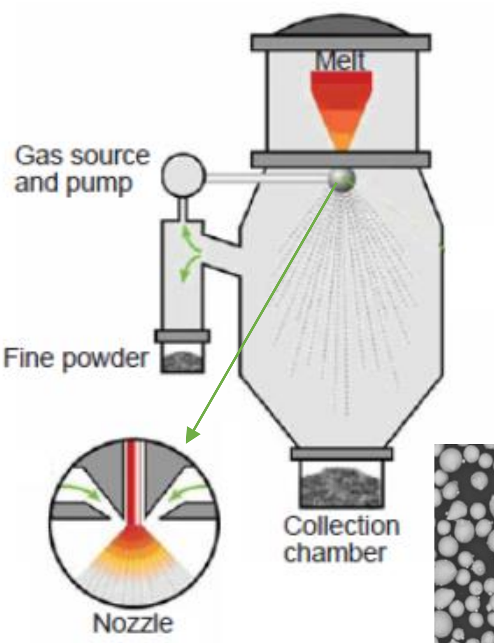
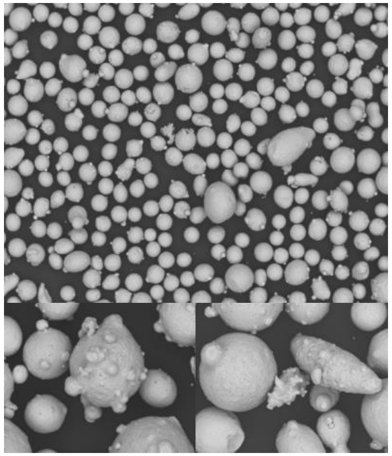
Powder Manufacturing – Gas Atomisation

- Uses a high pressure gas media to break-up a molten stream of metal liquid
- Produces almost all types of metal powder
- Can make highly spherical(ish) particles
- Maximum particle size is limited due to cooling difficulties
- Depends entirely on the atomiser dimensions
- Minimum particle size depends on alloy type but can be as low as sub 10 microns





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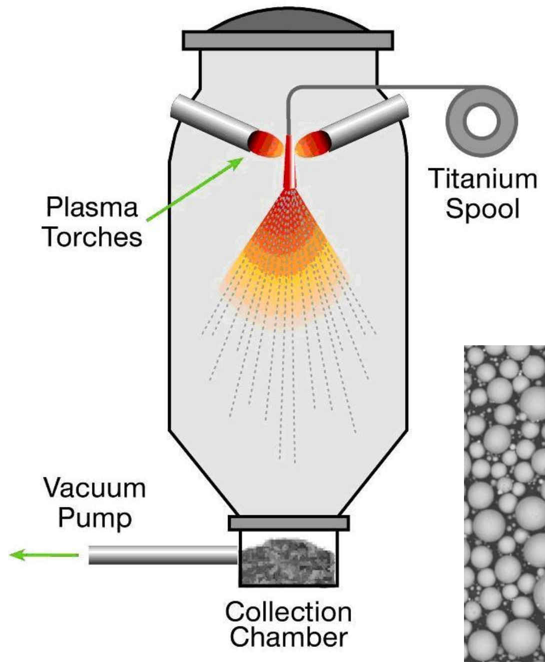
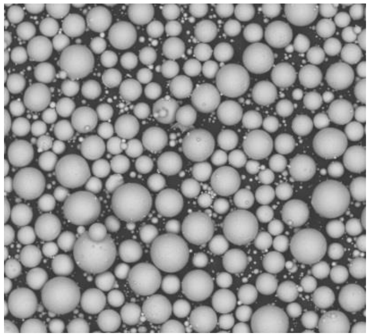
Powder Manufacturing – Plasma Atomisation

- Uses a wire feedstock
- Greater control over material melting
- Argon plasma used as both atomising media and heat source
- Produces highly spherical particles mostly free of satellite particles
- Typical particle size range < 250 µm
- Particle properties controlled by wire feed rate



lpwtechnology.com



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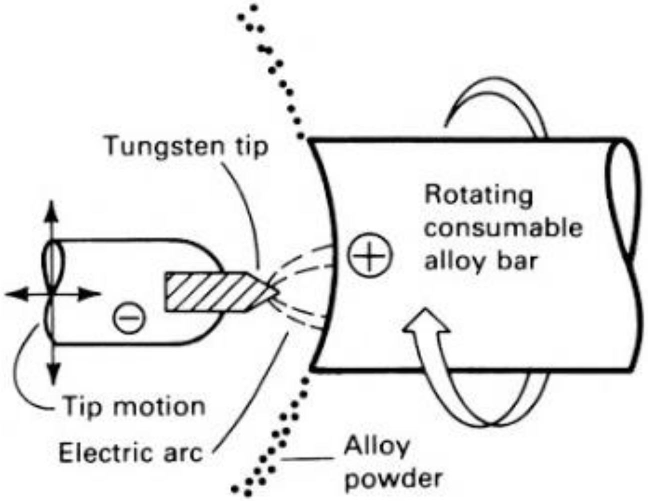
Powder Manufacturing - Plasma Rotation Electrode Process

- Centrifugal atomisation method
- A rotating metal rod melted by a plasma torch
- Vacuum or Argon gas chamber
- Produces highly spherical particles
- Very low occurrence of contamination including oxygen
- Very Expensive!



Superalloys: A technical guide

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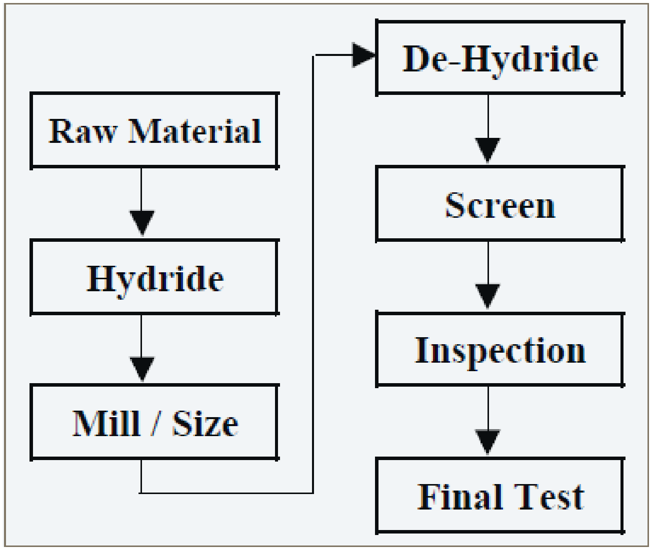
Powder Manufacturing - Hydride-Dehydride Process (HDH)

$Ti\ (solid) + H_2\ (gas) \leftrightarrow TiH_2\ (solid)$

- Mechanical rather than atomisation method
- Relies on the brittle nature of certain metal hydrides
- Metal hydride can be readily crushed and resized
- Can produce high yields in the desired size fraction
- Produces highly irregular particles
- Can have higher levels of oxidation



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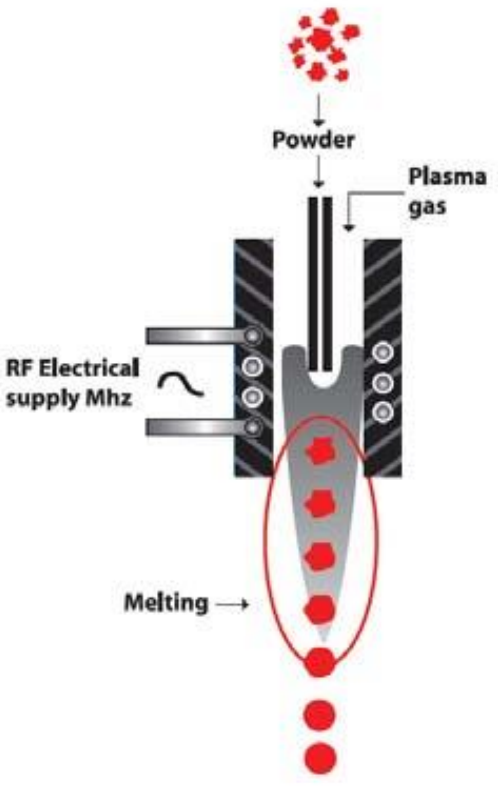
Powder Manufacturing - Plasma Spheroidised

- Uses powder as a raw material feedstock
- A plasma gas stream heats up and melts powder particles
- Increases particle sphericity
- High throughput and yield in desired size fraction
- High levels of oxidation



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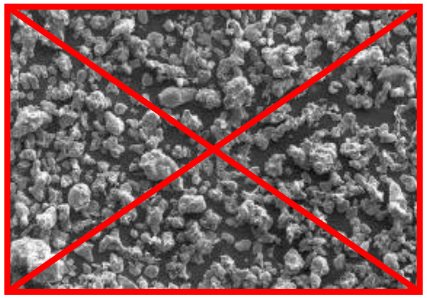
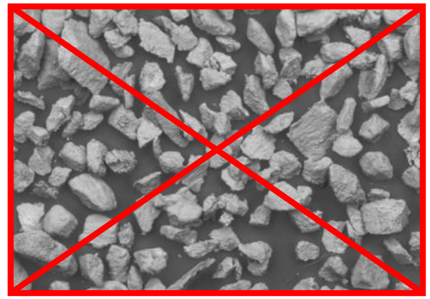
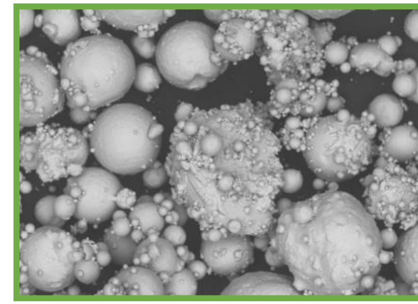
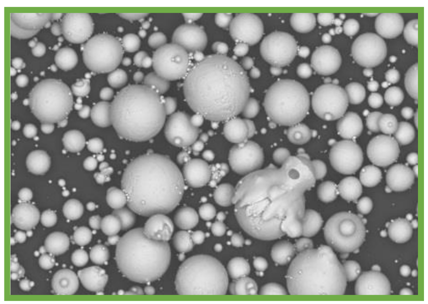
Metal powder production methods – suitability for AM

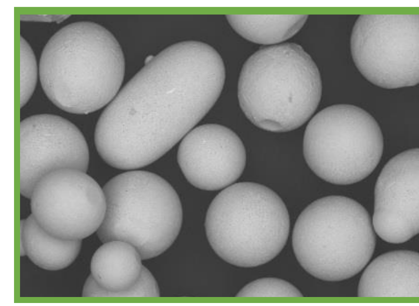

Water atomised


Hydride-de-hydride


Gas atomised


Plasma atomised


Spheridised hydride-dehydride


Plasma rotating electron process

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Powder Manufacturing methods

Manufacturing Process	Particle size (µm)	Advantages	Disadvantages	Common Uses
Water atomisation	0-500	• High throughput • Only requires feedstock in ingot form	• Post processing required to remove water • Satellites present • Low yield < 150 µm	Non-reactive
Gas atomisation (inc. EIGA)	0-500	• Wide range of alloys available • High throughput • Spherical powders	• Satellites present • Low yield < 150 µm	Reactive alloys Ni, Co, Fe, Ti, Al
Plasma Atomisation	0-200	• Extremely spherical	• Requires feedstock in wire or powder form • High cost	Ti
PREP	0-100	• High purity powders • Highly spherical powder	• Low productivity • High cost	Ti, Exotics
HDH	45-500	• Low cost	• Irregular particle morphology • High interstitial content (O, H)	Ti64, limited to metals which form a brittle hydride

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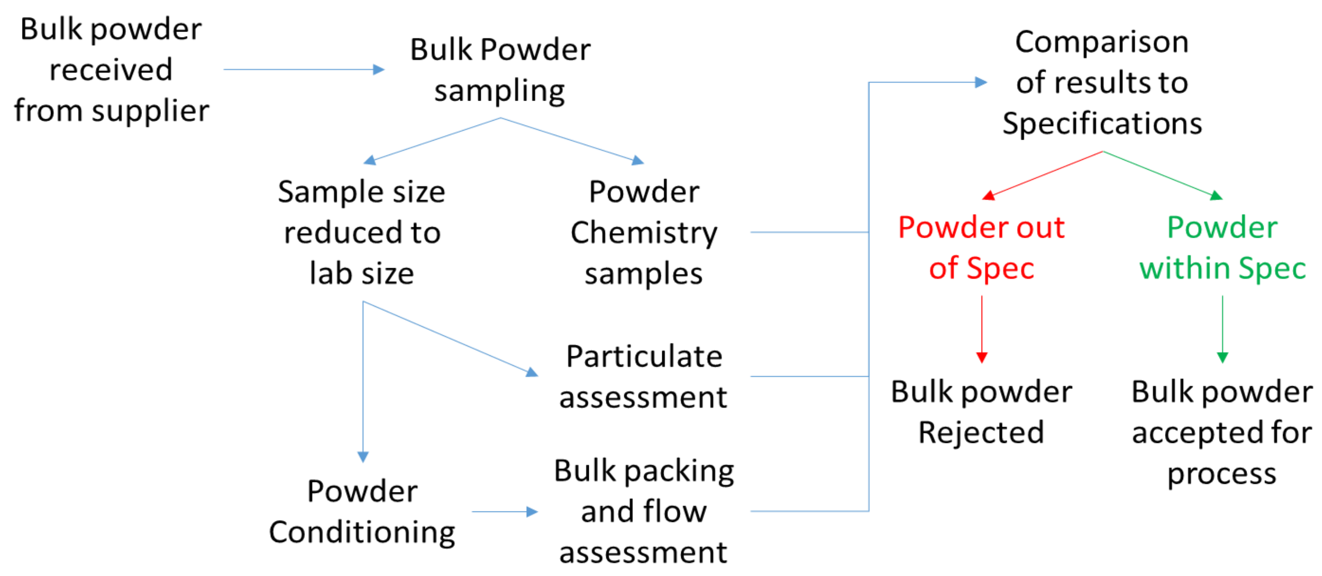
What makes a good DED powder?

- Conformance to chemistry composition/specification
  - Alloying elements
  - Interstitial elements
- Free from foreign particulate contamination
- Correct particle size fraction
  - Powder bed fusion: 15-45 µm
  - DED: 40 - 80 µm
    45 – 106 µm
    50 – 150 µm
- Ability to flow easily through powder feed system
- Spherical morphology

Powder Testing



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What to measure

- Particle size distribution (PSD)
- Flow and bulk density
- Particle shape (morphology) – ideally spherical
- Conformance to chemistry composition/specification
    - Alloying elements
    - Interstitial elements
- Free from foreign body contamination

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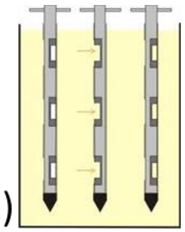
Powder Sampling

(1)

(2)

(3)

(4)


Before testing a sample has to be taken

- AM build can use anywhere from 10 kg up to 200 kg
- It is not practical to measure every particle!
- Typically 100-500g sample taken for analysis
- Need to follow sampling procedure to ensure representative samples are taken

MTC –Powder samp

Detailed set of work instructions which needs to be followed

mtc Manufacturing Technology Centre	MTC Work Instruction for:	Skill Requirement & Level		WI Number	Version
	Powder blending and sampling	Powder blending Powder sampling	Level (3) Level (3)	AM-WI-002-v1.0-Powder blending and sampling	1.0

mtc Manufacturing Technology Centre	MTC Work Instruction for:	Skill Requirement & Level		WI Number	Version
	Powder blending and sampling	Powder blending Powder sampling	Level (3) Level (3)	AM-WI-002-v1.0-Powder blending and sampling	1.0

Throughout this procedure the following PPE and RPE must be worn at all times unless otherwise stated.

PPE/RPE	Quantity	PPE/RPE Description & Part Number (if applicable)
Gloves	As required	Disposable, powder-free nitrile gloves (EN374)
Half mask	1 per user	Face-fitted half mask with P3R filter (EN140; EN143) OR

mtc Manufacturing Technology Centre	MTC Work Instruction for:	Skill Requirement & Level		WI Number	Version
	Powder blending and sampling	Powder blending Powder sampling	Level (3) Level (3)	AM-WI-002-v1.0-Powder blending and sampling	1.0

The following documents and H&S assessments apply throughout this procedure:

Document ID	Issue	Document Description
ASTM B215	10	Standard Practice for Sampling Metal Powders
SSOW 154	1	SSOW for Operation of Powder Preparation Equipment
AM-WI-001	1.0	Work Instruction for Powder Lab sample management

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Powder Characterisation – Test Cell

- Temperature & Humidity Controlled Cell

  - Representative sample from the bulk
  - Same testing temperature
  - Same testing humidity
  - Pre-conditioned powder

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Particle Size Distribution (PSD) by sieving

- Sieves divide particulate materials into size fractions
- Labour intensive
- Can be difficult to achieve consistent results
- Can under/over estimate non-spherical particles
- Not suitable below 35 µm

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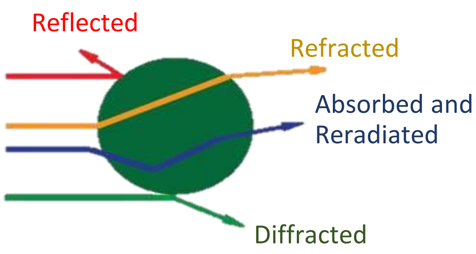
Particle Size Distribution (PSD) by laser diffraction

Measures interaction of powder particles with a laser source

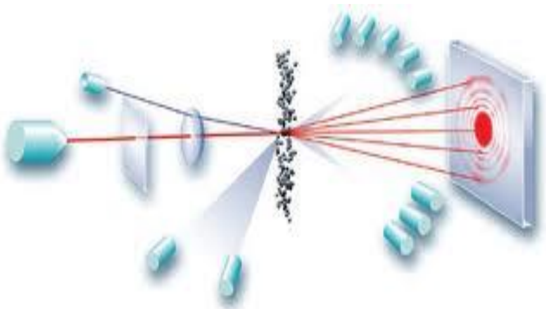


Advantages

- Small sample size
- Measures particles as fine as 1 µm
- Fast measurement
- Highly repeatable

Disadvantages

- Black box technique
- No information on particle shape
- Relies heavily on dispersion



Reported particle size assumes particle is spherical!

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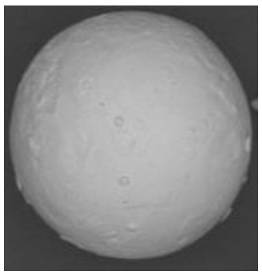
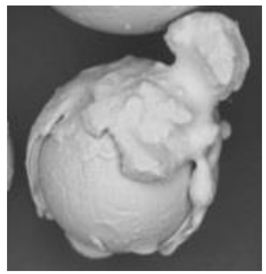
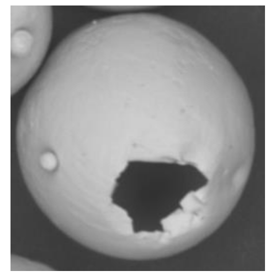
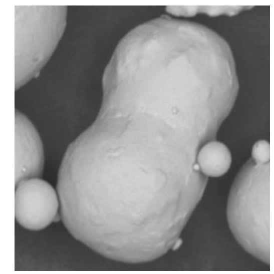
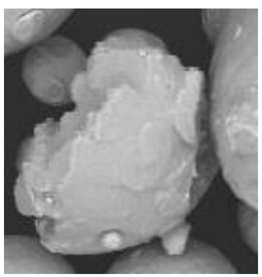
Particle Shape & Defects

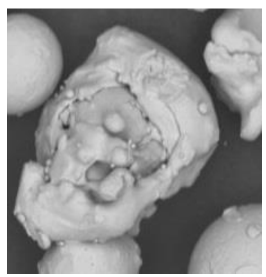

Spherical

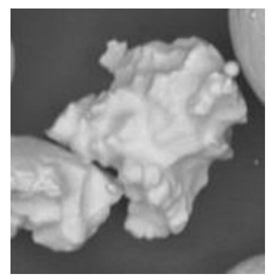

'Splat Cap'

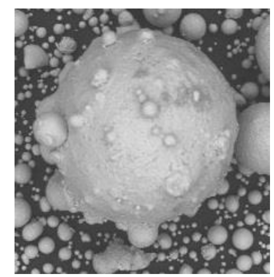

Open Porosity


Elongated


Broken


Agglomerated


Irregular


Satellited

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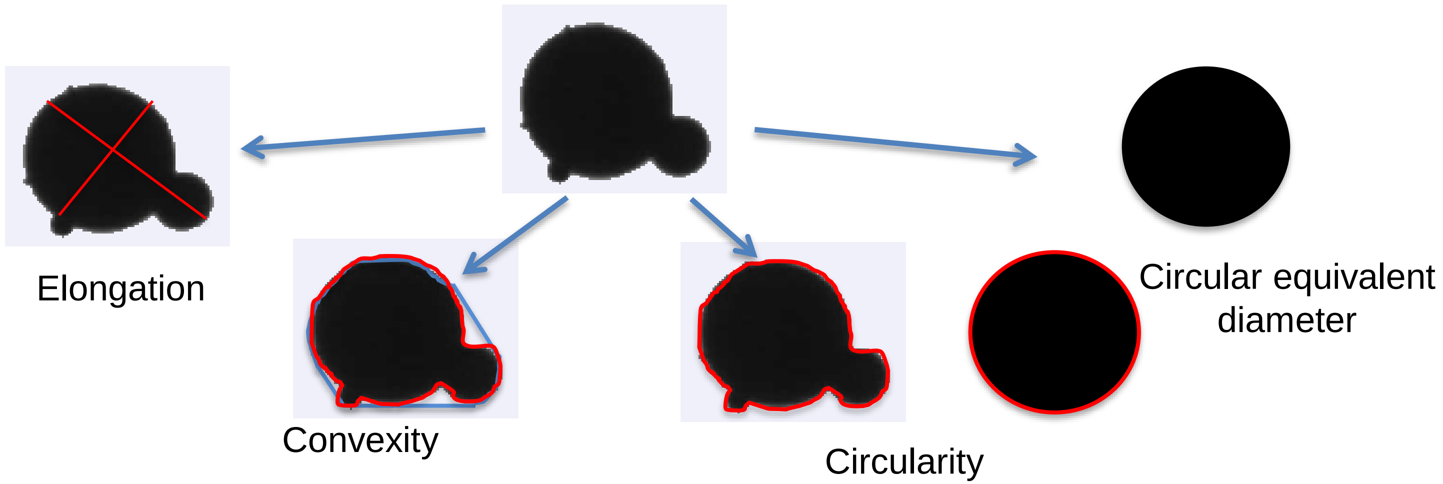
Particle shape (morphology)

Quantifying Shape Using Image Analysis

In image based particle size techniques images of individual particles are capture in real time and can be analysed in terms of their morphological parameters:



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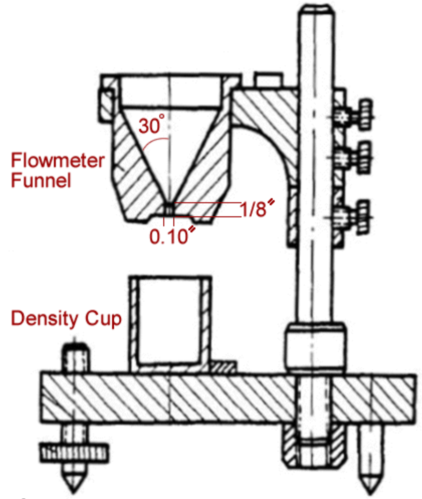
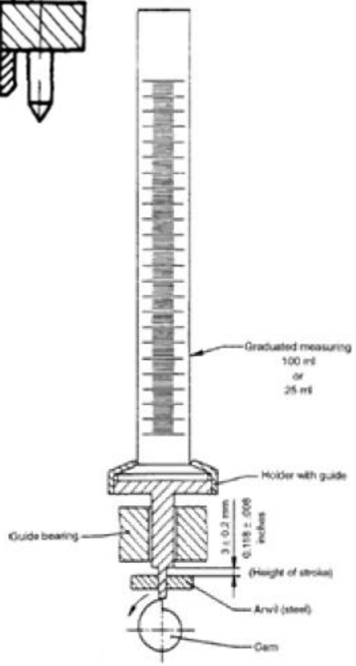
Powder Density

- Can be measured a number of ways!
  - Solid (or true)
  - Apparent (or poured)
  - Tapped
- Different handling methods will effect the level of packing
  - Bulk density is not strictly a material constant
- Difference between poured/tapped density can provide information about the cohesivity of the powder





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Powder Flow

- Hall Flow
  - Time taken for 50g of a metal powder to pass through a hopper
  - Static start to flow or dynamic start to flow

- Carney Flow
  - As above but with a larger diameter outlet orifice

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Chemical composition and Interstitial elements

Alloying Elements

- Inductively coupled plasma – optical emission spectroscopy (ICP-OES)

Interstitial Elements

- Inert Gas Fusion
  - Oxygen, nitrogen and hydrogen
- Combustion Infrared Detection
  - Carbon and sulphur

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Common Aerospace Alloys

- Titanium Alloy - Ti64
- Nickel Alloy - In718
- Aluminium Alloy – Scalmalloy

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Ti-6Al-4V

- **Ti-6Al-4V** (UNS designation **R56400**), **TC4**, **Ti64** ASTM **Grade5**
- Alpha-beta titanium alloy with a high specific strength and excellent corrosion resistance.
- Commonly used for low temperature structural components

	V	Al	Fe	O	C	N	H	Y	Ti	Remainder Each	Remainder Total
Min	3.5	5.5	--	--	--	--	--	--	--	--	--
Max	4.5	6.75	.3	.2	.08	.05	.015	.005	Balance	.1	.3

https://en.wikipedia.org/wiki/Ti-6Al-4V

Other elements are tolerated to very low levels

Iron 0.3%
Oxygen 0.2%
Carbon 0.08%
Nitrogen 0.05%
Hydrogen 0.015%
Yttrium 0.005%

This is referred to as interstitial contamination
Can cause catastrophic embrittlement

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In718 / Inconel 718

- Precipitation-hardening nickel-chromium alloy
- High strength and good ductility up to 704°C (https://www.upmet.com/products/nickel-alloys/)

Inconel	Element, proportion by mass (%)														
	Ni	Cr	Fe	Mo	Nb & Ta	Co	Mn	Cu	Al	Ti	Si	C	S	P	B
718[2]	50.0–55.0	17.0–21.0	Balance	2.8–3.3	4.75–5.5	≤1.0	≤0.35	≤0.3	0.2–0.8	0.65–1.15	≤0.35	≤0.08	≤0.015	≤0.015	≤0.006

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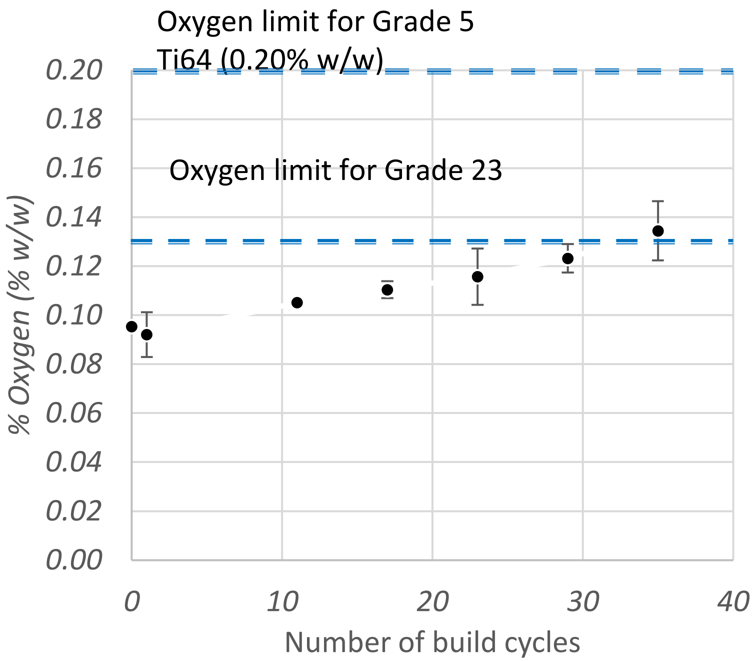
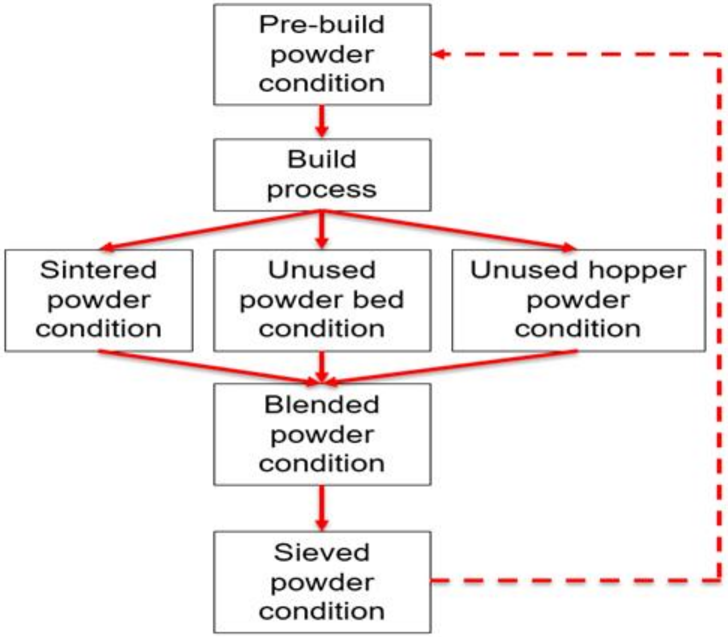
Unlike PBF-LB powder feed-stock is rarely reused in DED (unless it is just the material in the hopper)

Powder Characterisation

Chemical Analysis-Powder Recycling





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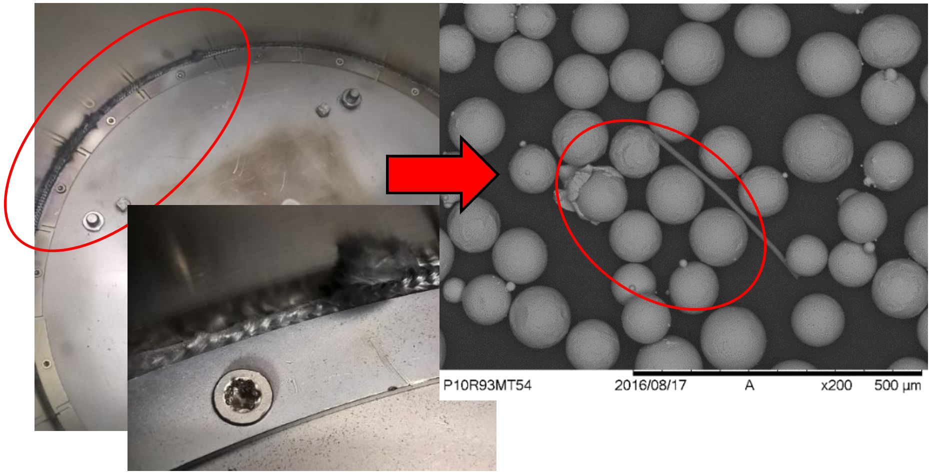
Foreign body contamination

- Sources include:
  - Atomising equipment & process
  - Handling equipment
  - ~~AM equipment & process~~
  - Organic matter
  - Cross-contamination

Unlike PBF-LB powder feed-stock is rarely reused in DED (unless it is just the material in the hopper)



- Degraded ceramic ropeseal used in handling equipment was found in Ti64 powder batches

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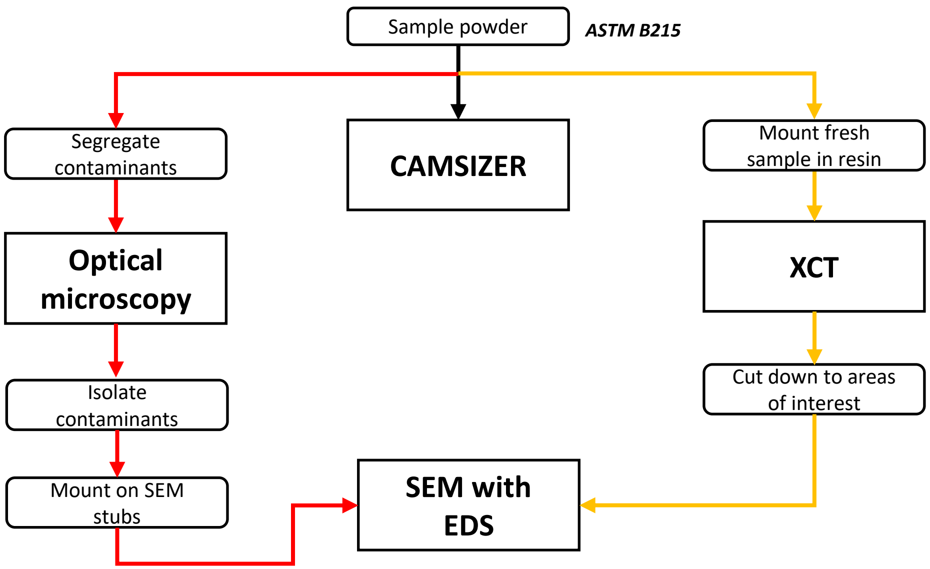
Foreign body contamination - detection



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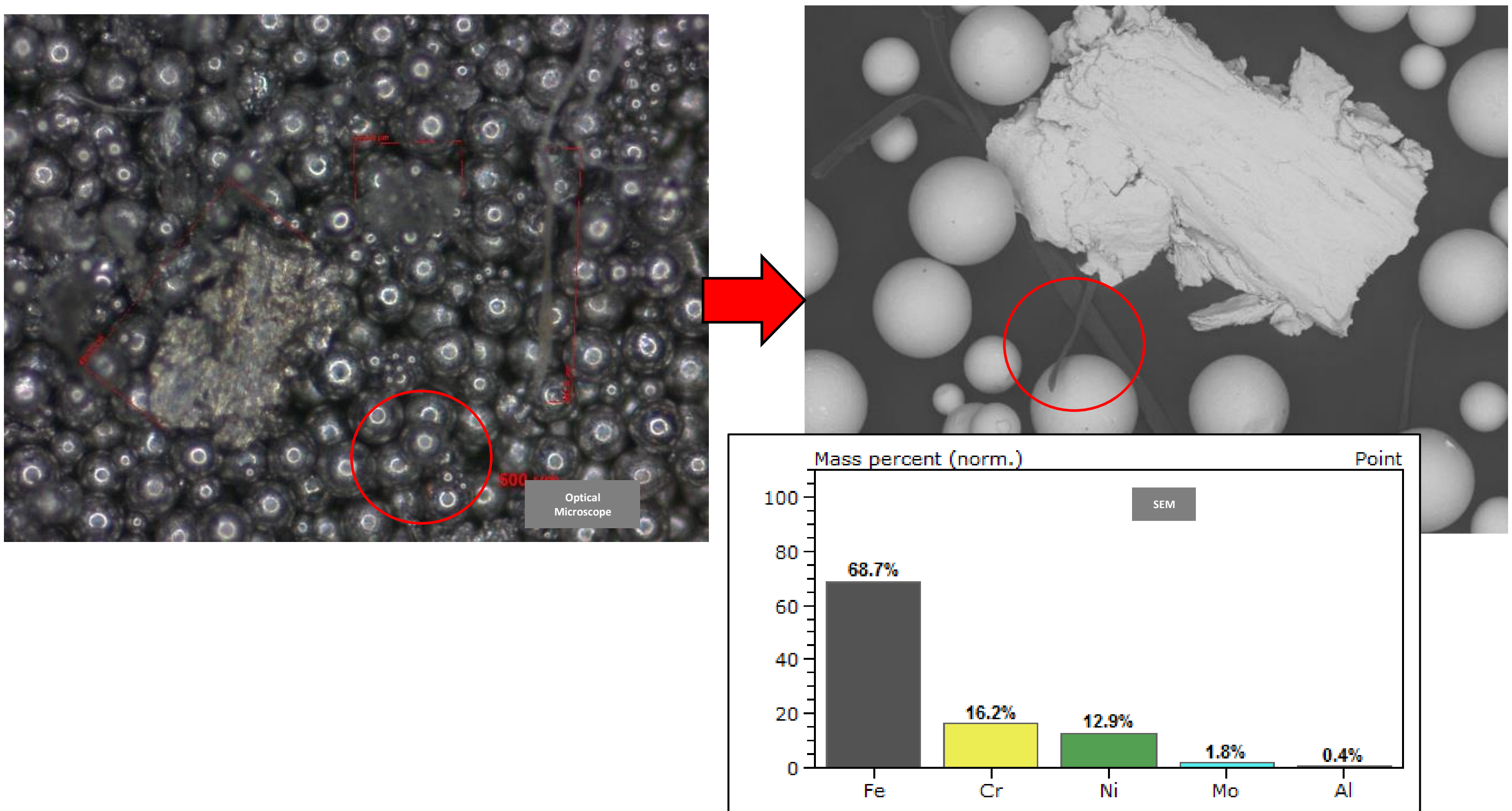
Foreign body contamination -microscopy



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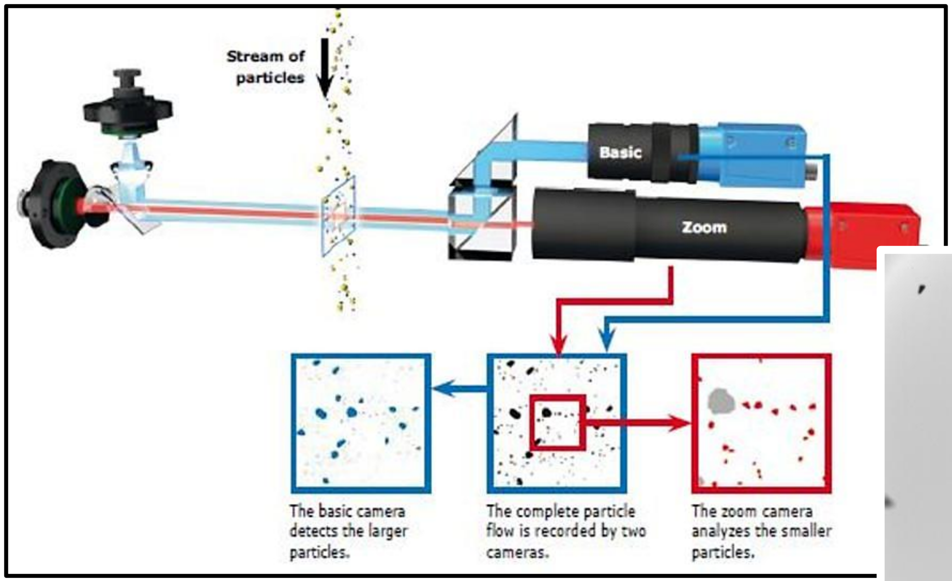
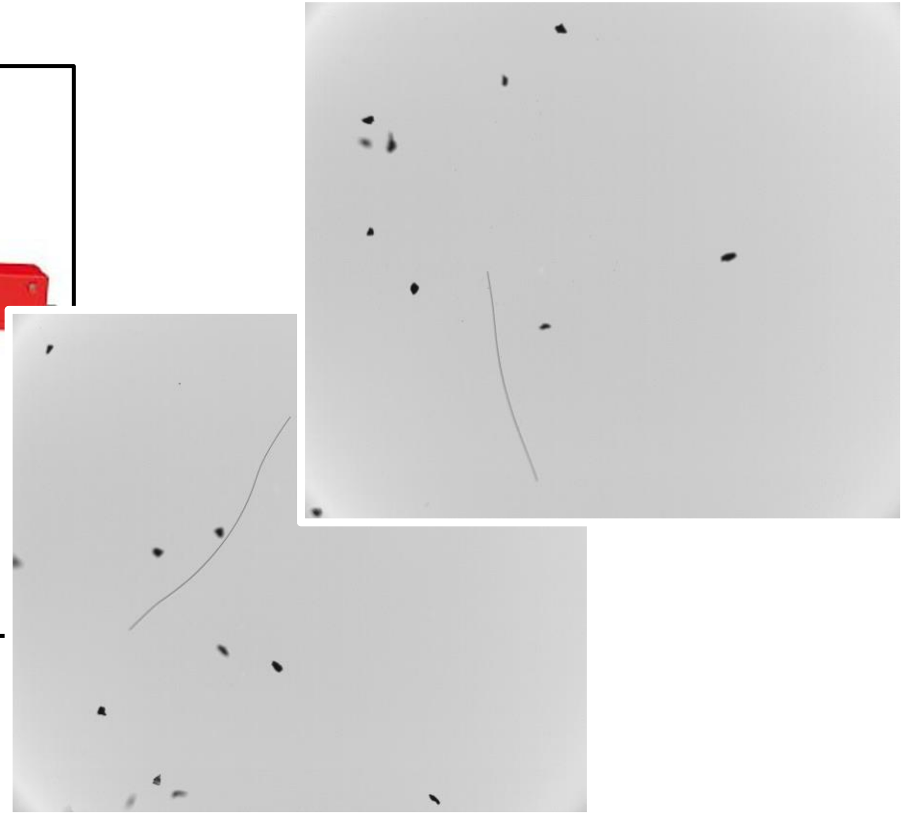
Foreign body contamination - Camsizer





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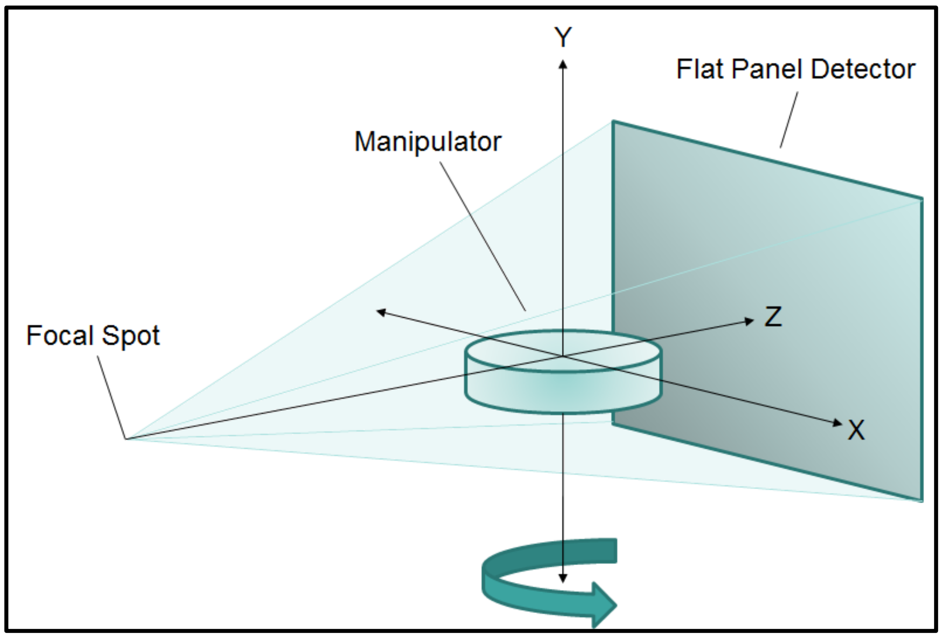
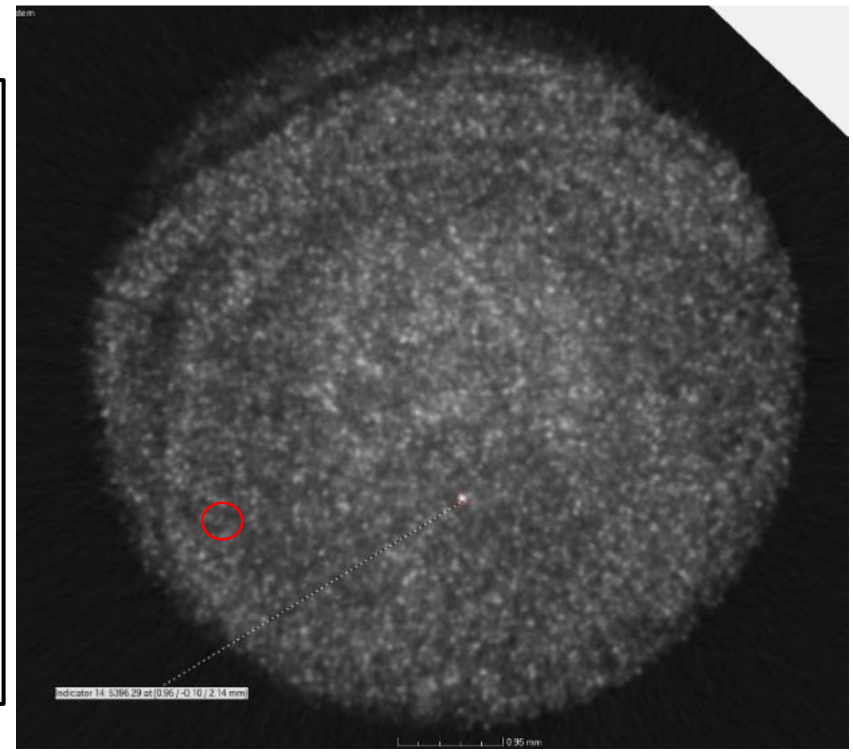
Foreign body contamination - XCT





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Impact of Powder quality

- Difficult to detect low levels of contaminants
- No high throughputs methods available for detection
- No standards on contamination assessment of powder batches
- Mechanical impacts of types of contamination are unknown
- Acceptable limits for containments are unknown

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Wire Feed-Stock Quality

David Wimpenny

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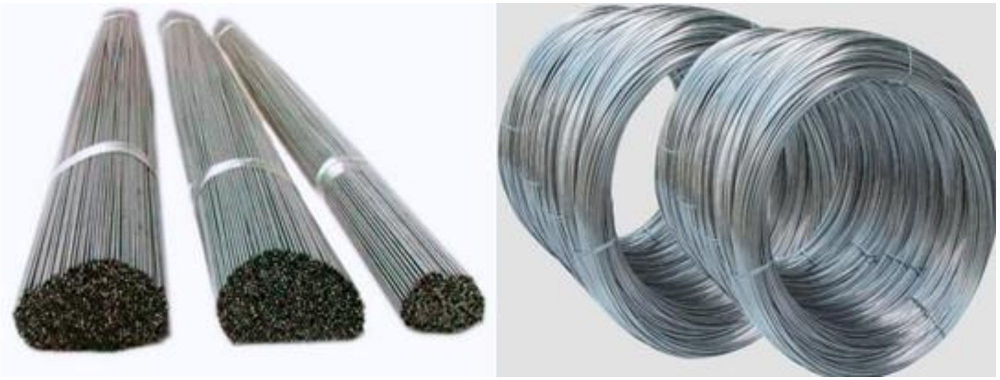
Wire Feedstock Characterisation

- One key advantage of wire feedstock over powder is the simplification of validation
- Unlike powder, the dimensions of wire feedstock can be guaranteed by suppliers to high precision
- Chemistry can also be more easily guaranteed from initial source material



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Materials in DED – Wire quality

- Wire feedstock has minimal defects compared to powder
- This is due to technology transferred from mature welding consumable supply chain

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Materials in DED – Wire quality

- Quality (absorbed moisture and dimeter variance) linked to porosity in weld deposit
- Defects also caused by surface absorption in reactive materials
- Wire stock may have scratches or cracks which can translate directly into porosity
- Gas porosity not normally as issue with wire (unlike powder)

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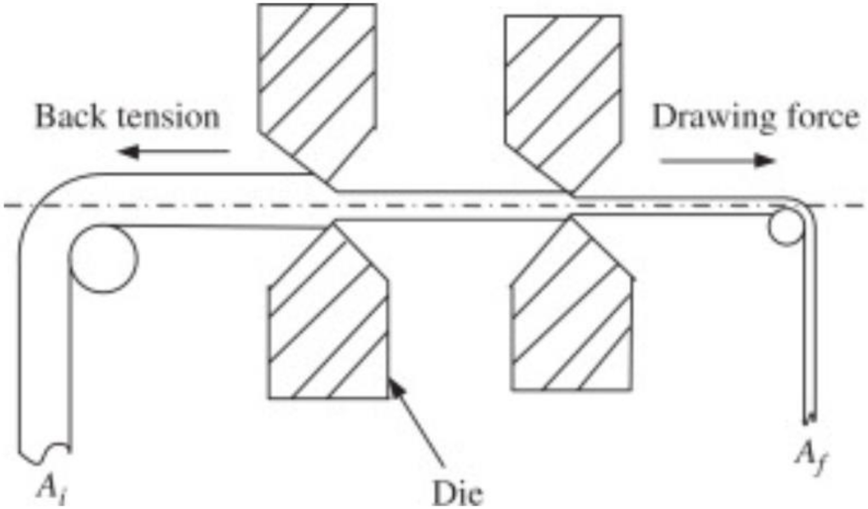
Wire production

- Rods made of steel or non-ferrous metals and alloys are pulled through conical dies having a hole in the centre.
- The included angle of the cone is kept between 8 to 24°.
- As the material is pulled through the cone, it undergoes plastic deformation and gradually undergoes a reduction in its diameter.
- At the same time, the length is increased proportionately.



https://extrudesign.com/what-is-wire-drawing-tube-drawing-and-making/?utm_content=cmp-true

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Hot rolled rod

Pickling, descaling

Remove scale -causing surface defects.

Lubricating

• Cu and Sn are used as lubricants for high strength materials. Or conversion coating such as sulphates or oxalates.

• Oils and greases for wire drawing
• Mulsifiable oils for wet wire drawing
• Soap for dry drawing.

drawing\

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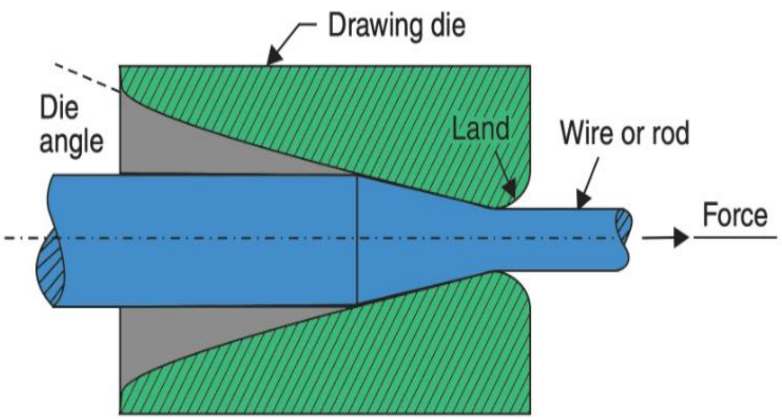
Wire Drawing



Key variables in wire drawing process are;

1. Reduction ratio
2. Die angle
3. Friction

Improper control of these parameters will cause defects in the drawn material.

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Key issues in wire drawing

- Dies tend to wear quickly
- Usually made from hard materials (alloy steel, tungsten carbide or even diamond)
- Reduction in the cross-sectional area in one pass is 25–30%.
- Wire has to pass through several dies to reach the desired diameter .
- To overcome strain hardening periodic heat treatment is required.
- Metal rods to be drawn into wire must be clean
- Sometime pickling in an acid bath is required to remove oxide layer
- Wire is drawn using several power-driven spools or rotating drums.
- Significant heat is generated during drawing process
- To reduce friction, dry soap or a synthetic lubricant is used.
- But despite reducing friction, the dies and drums may have to be water-cooled.

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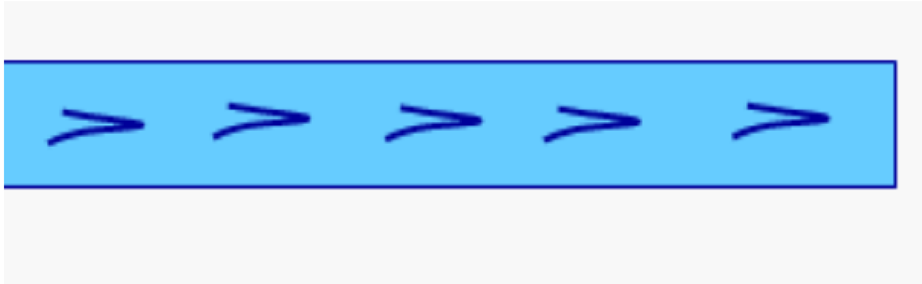
Defects in rod and wiredrawing

- Defects in the starting rod (seams, slivers and pipe).
- Defects from the deformation process, i.e., center burst or cracking (cupping).



- This defect will occur for low die angles at low reductions.
- For a given reduction and die angle, the critical reduction to prevent fracture increases with the friction.

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Defects in Wire

- Include centre cracking (as in extrusion and for the same reasons) and the formation of longitudinal scratches or folds in the material
- Poor surface finish
- Out of round
- Surface contamination (from die material, lubricants)

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Publicly Available Specification (PAS) from BSI

These documents are free to down load once you have registered

Go to

https://pages.bsigroup.com/l/35972/2020-10-06/2lp4rpg

PAS 6010:2020 - PAS 6011:2020 - PAS 6012:2020 Additive Manufacturing

Innovate UK, a part of BEIS, asked BSI to help promote the use of directed energy deposition (DED) additive manufacturing (AM) in the UK. To that end, and working in partnership with UK Research and Innovation (UKRI) and ASTM, we've now published a free series of PASs:

- **PAS 6010:2020** ***Additive Manufacturing - Wire for directed energy deposition (DED) processes in additive manufacturing - Specification*** provides requirements for wire to be used specifically in wire-fed DED AM. This PAS is intended for use by wire suppliers, wire material developers and manufacturers, DED machinery developers and manufacturers, DED users and DED component end-users.
- **PAS 6011:2020** ***Additive manufacturing – Non-destructive testing for use in directed energy deposition – Guide***. This PAS is for anyone wanting to identify suitable methods of inspection of parts manufactured using a DED AM process, as well as for regulatory bodies, material suppliers, certification bodes, inspectors and researchers.
- **PAS 6012:2020** ***Additive Manufacturing — Wire arc — Guide*** outlines the characteristics, benefits, and limitations of Wire Arc Additive Manufacturing (WAAM), a directed energy deposition (DED) additive manufacturing technology that's broadening the applicability of AM.

These documents are available free of charge in order to encourage greater confidence in, and use of, DED AM by high-value UK manufacturing companies.

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Critical issues with wire quality

- (DED) additive manufacturing (AM) for high-value manufacturing.
- It covers:
- Material, including:
  - composition;
  - dimensions and tolerances;
  - cast/helix;
  - surface condition;
  - internal condition;
- Marking;
- Packaging;
- Materials handling;
- Storage;
- Testing methods.

PAS 6010:2020 ***Additive Manufacturing - Wire for directed energy deposition (DED) processes in additive manufacturing - Specification*** provides requirements for wire to be used specifically in wire-fed DED AM. This PAS is intended for use by wire suppliers, wire material developers and manufacturers, DED machinery developers and manufacturers, DED users and DED component end-users.

https://knowledge.bsigroup.com/products/additive-manufacturing-wire-for-directed-energy-deposition-ded-processes-in-additive-manufacturing-specification/standard

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Composition

- As with powder the composition, including interstitial contamination needs to be checked
- Important to takes representative samples

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Dimensions

- Wire diameter needs to be measured;
- Around the circumference of the wire to check for ovality
- Several places along the length of the wire to check for consistency

Tolerances for wire diameter is given the in PAS 6010 (except shown below)

<table>
<tr><th rowspan="2">Nominal diameter</th><th colspan="2">Diameter tolerance</th></tr>
<tr><th>Solid wire and solid wire electrodes</th><th>Cored wires and cored wire electrodes</th></tr>
<tr><td>0,5</td><td rowspan="2">+ 0,01
- 0,03</td><td>-</td></tr>
<tr><td>0,6</td><td rowspan="5">+ 0,02
- 0,05</td></tr>
<tr><td>0,8</td><td rowspan="4">+ 0,01
- 0,04</td></tr>
<tr><td>0,9</td></tr>
<tr><td>1,0</td></tr>
<tr><td>1,2</td></tr>
</table>

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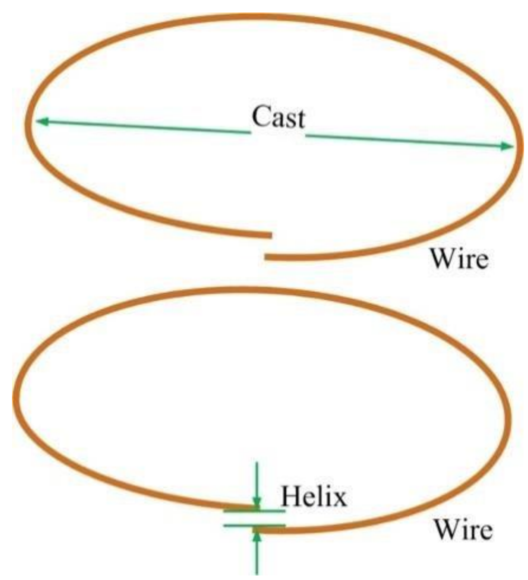
Cast & Helix

- Cast - diameter of loops formed when wire is placed onto a flat surface unrestrained
- Helix - vertical separation between any part of one loop of wire placed on a flat surface and the flat surface

Need to be controlled to prevent problems with wire feeding




metals
MDPI

Article

Study in Wire Feedability-Related Properties of Al-5Mg Solid Wire Electrodes Bearing Zr for High-Speed Train

Bo Wang [1], Songbai Xue [1,*], Chaoli Ma [1], Jianxin Wang [2] and Zhongqiang Lin [3]

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Received: 24 September 2017; Accepted: 20 November 2017; Published: 23 November 2017

Surface condition

Wire needs to be free from surface defects, oxide and scale which could prevent it from be feed

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Internal condition

Wire should be free from internal defects, including porosity and cracks

Figure 1: Treatment: Quenched and tempered. Magnification: 2x. Sampling/Specification: Wire surface. Testing result: Longitudinal defects on the surface.

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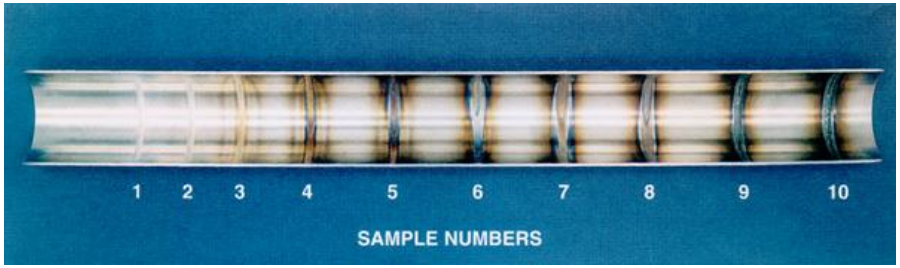
Impact of wire defects

Wire feed problems – inconsistent or interrupted feed

- Reduced mechanical properties – due to wire contamination

Interstitial contamination

- Oxygen
- Nitrogen
- Carbon
- Hydrogen



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Impact of wire contamination on mechanical properties of DED-arc samples fatigue properties

- Significant work undertaken to assess the impact of wire contamination on mechanical properties

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International Journal of Fatigue

Volume 122, May 2019, Pages 208-217

Criticality of porosity defects on the fatigue performance of wire + arc additive manufactured titanium alloy

Romali Biswal [a], Xiang Zhang [a], Abdul Khadar Syed [a], Mustafa Awd [b], Jialuo Ding [c], Frank Walther [b], Stewart Williams [c]

- Work undertaken by Coventry and Cranfield University
- Unlike PBF-LB parts produced by DED-Arc seldom contains gas pores
- However, there is a risk that wire feed-stock can become contaminated
- Trials performed to measure level of porosity introduced
- Impact on fatigue properties
- Two types of specimens were tested: (1) control group without porosity referred to as reference specimens; (2) designed porosity group using contaminated wires to build the specimen gauge section, referred to as porosity specimens.

- Test results have shown that static strength of the two groups was comparable, but the elongation in porosity group was reduced by 60% and its fatigue strength was 33% lower than the control group

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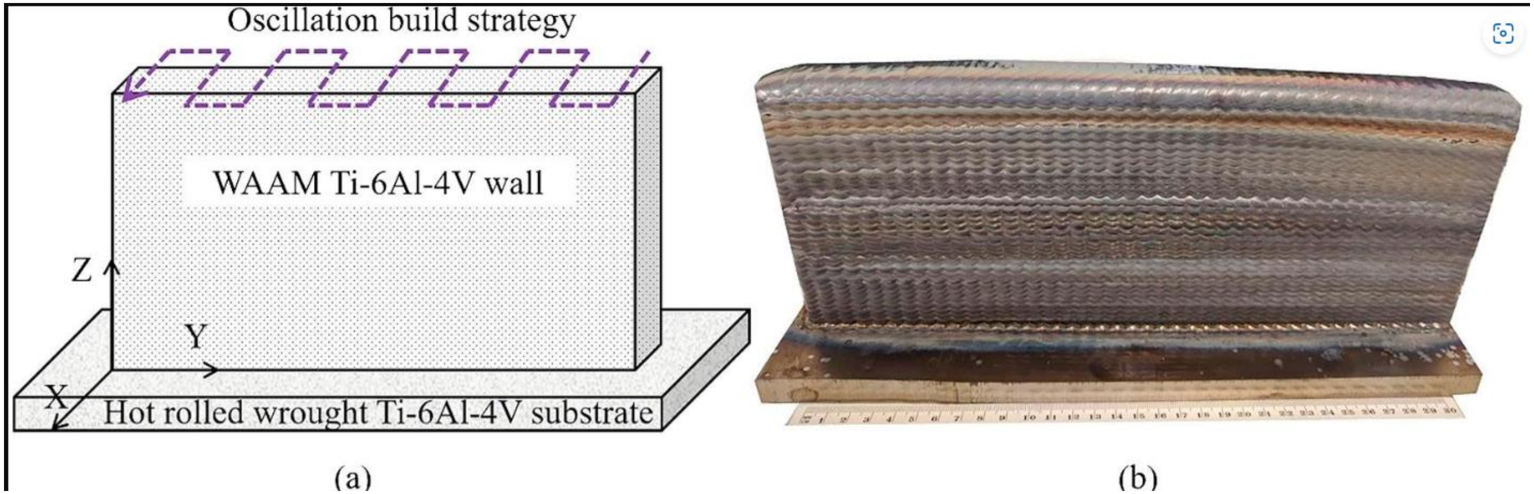
WAAM Specimens produced;

(1) Control group without porosity (reference specimens)

(1) Higher porosity samples produced with contaminated wire (porosity specimens)

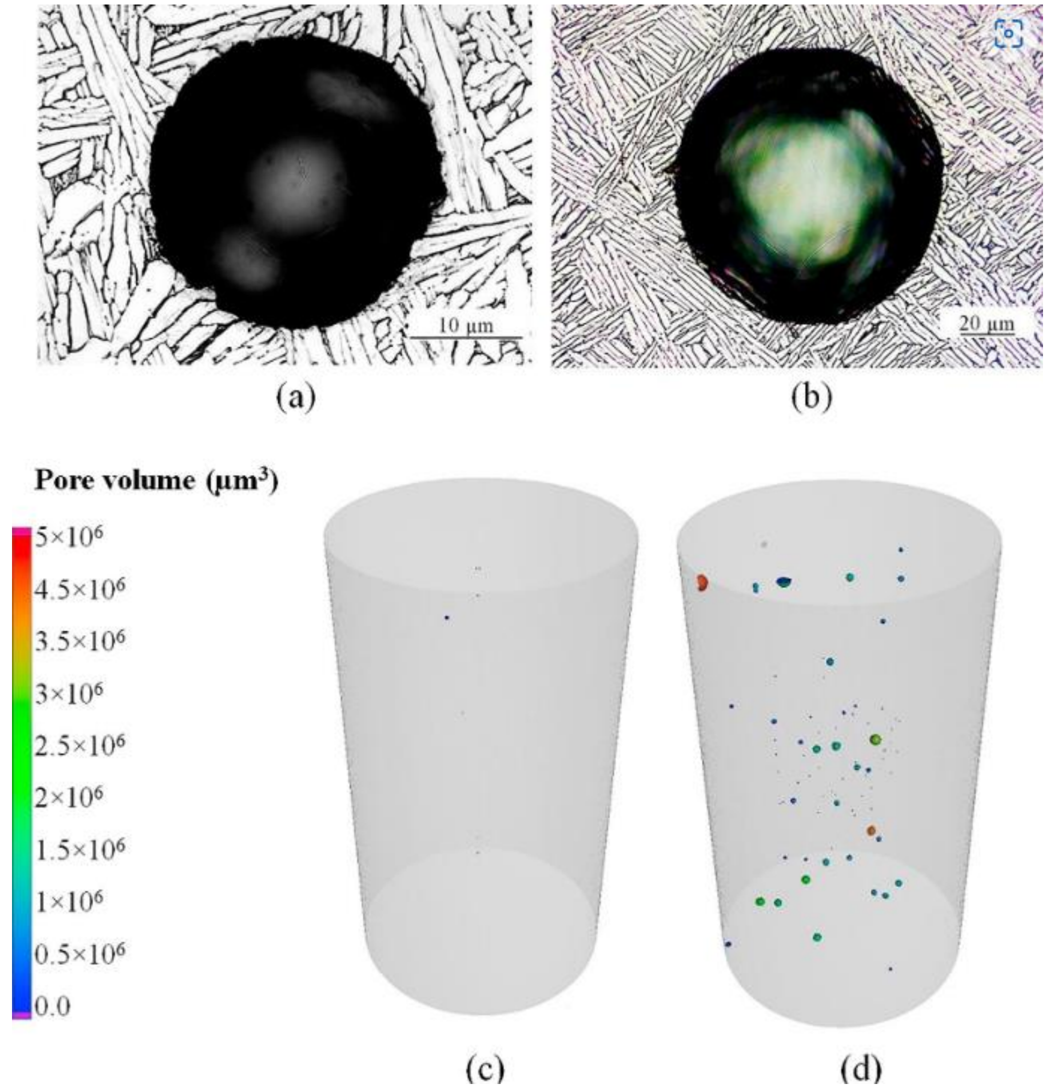
	Al	V	Fe	O	C	N	H	Residual	Ti
Wire	6.24	4.18	0.131	0.15	0.013	0.009	0.005	<0.4	Bala
Reference wall	5.61	3.8	NA	0.18	NA	0.008	0.007	NA	NA
Porosity wall	5.89	3.8	NA	0.17	NA	0.017	0.006	NA	NA
Allowable [42] limits or max	5.5–6.75	3.5–4.5	0.3	0.2	0.08	0.05	0.015	<0.4	Bala



Porosity formed by contamination

Optical image of representative porosity in (a) reference wall, (b) porosity wall, (noting different magnification)

XCT of specimen gauge section of reference specimen, density is 99.99%
porosity specimen density is 99.96%



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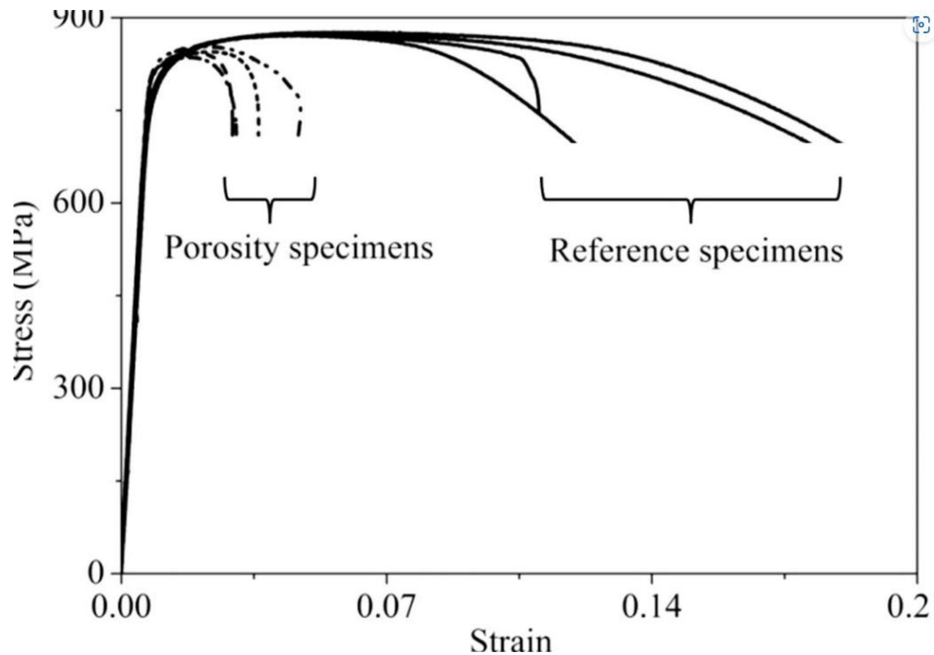
Tensile Properties

Significant drop in elongation to failure for porosity samples

Specimen	0.2% offset strength (MPa)	Ultimate tensile strength (MPa)	Uniform Elongation (%)
Reference	802±7	859±4	10±2
Porosity	825±5	842±8	4±1



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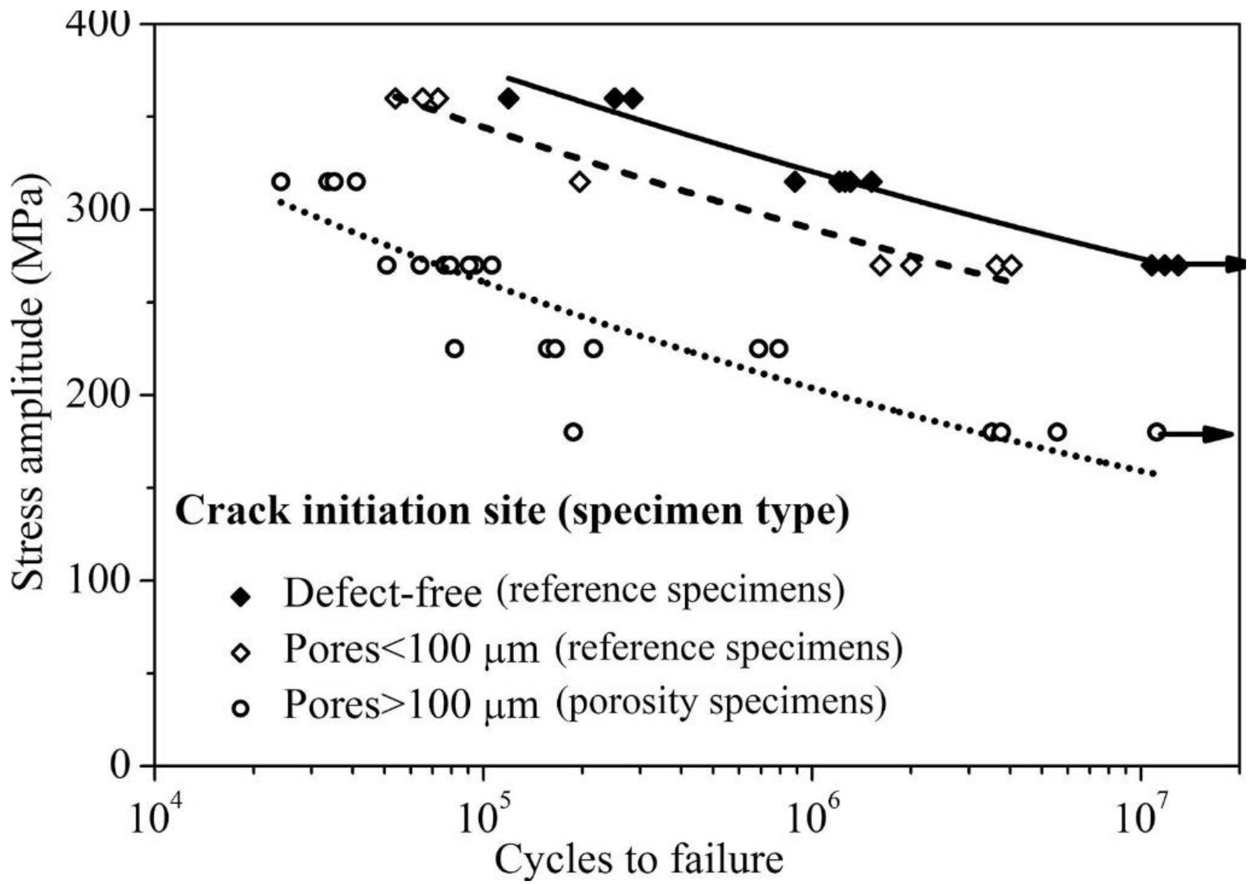
Fatigue strength

Compared to reference specimens fatigue strength of porosity specimens was reduced by a factor of 1.5 (at 107 cycles).



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How to reduce risk of wire contamination

- Careful selection of suppliers
- Close monitoring of incoming wire
- Appropriate storage
- Careful selection of wire feed equipment (some comes with wire straightening and cleaning capability)

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www.skills4am.eu

Thank you & Questions ?

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