Feedstock for Metal Additive Manufacturing

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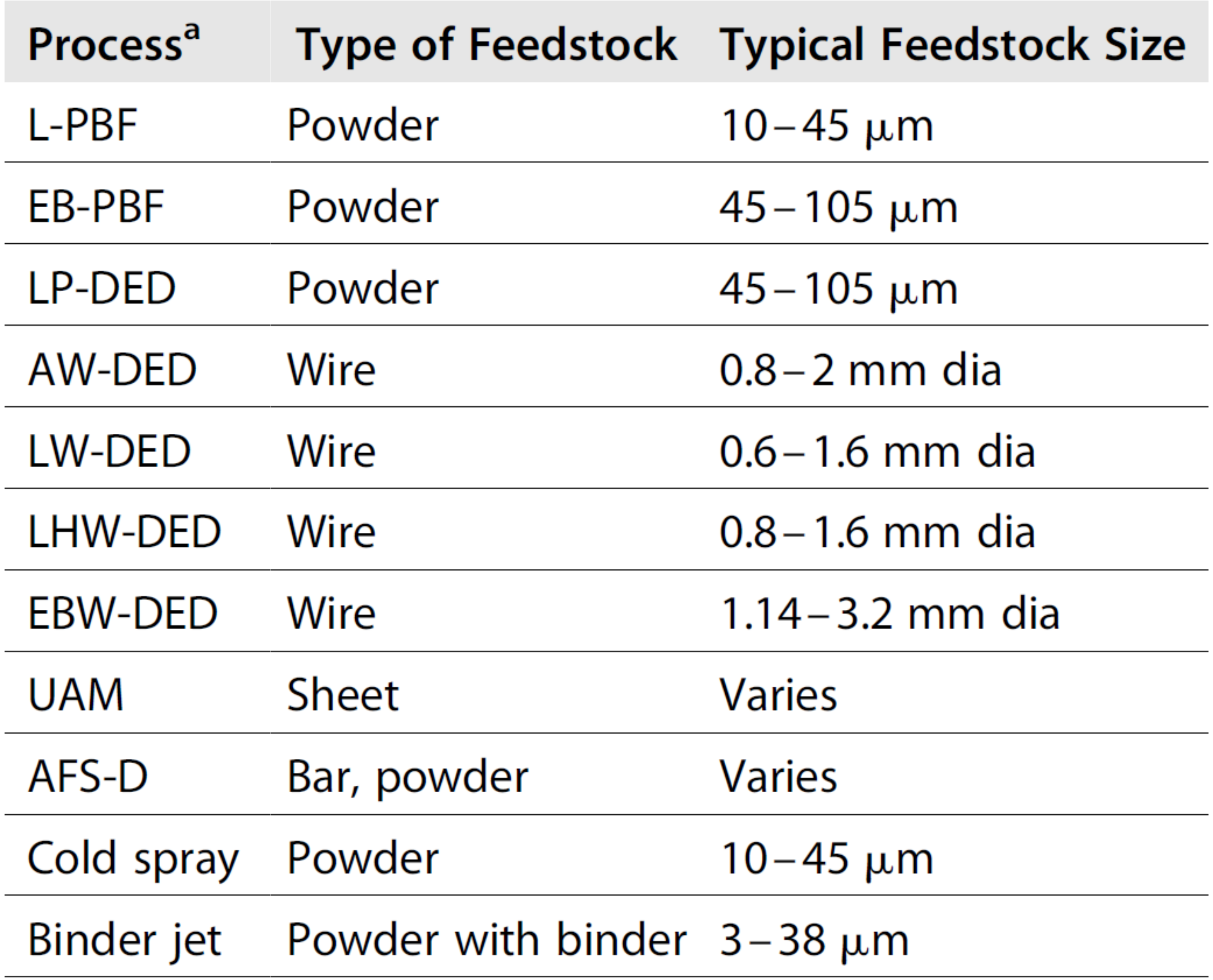
Introduction

- Feedstock enables AM
- Powder, Wire, and others
- Responsibilities of the "mill" are passed onto the user
    - Chemistry
    - Cleanliness
    - Pedigree
    - Conformance to other standards



Process[a]	Type of Feedstock	Typical Feedstock Size
L-PBF	Powder	10–45 μm
EB-PBF	Powder	45–105 μm
LP-DED	Powder	45–105 μm
AW-DED	Wire	0.8–2 mm dia
LW-DED	Wire	0.6–1.6 mm dia
LHW-DED	Wire	0.8–1.6 mm dia
EBW-DED	Wire	1.14–3.2 mm dia
UAM	Sheet	Varies
AFS-D	Bar, powder	Varies
Cold spray	Powder	10–45 μm
Binder jet	Powder with binder	3–38 μm

Supply Chain – Standard Feedstock

- Powder supplied by AM machine manufacturers
- Common Alloys (Ti-6Al-4V, Al-Si-10Mg, Inconel 625, Inconel 718, CoCr, 316L Stainless Steel)
- Always developing parameters for new alloys (e.g. NASA's GRCop42)
- Flowability, chemistry, and even material properties are solved by AM machine manufacturers, given parts are printed with standard material set.
- Typically bottled, relabeled powder from powder manufacturing OEMs, with company specific requirements and often price markups.

EOS CoCr powder

Supply Chain – Non-Standard Feedstock

- Go to powder OEMs directly
- Large number of alloys available off-the-shelf, many for AM specifically.
- PSD and flowability often not ideal for specific AM machines
  - EOS M100 flowability struggles
- Ambiguous specifications require focused requirements and back-and-forth with OEM to ensure powder quality
- Often cheaper if purchased at scale

Inconel 718 off-the-shelf

Supply Chain – Custom Feedstock

- Large powder OEMs or boutique feedstock manufacturers.
- User provides comprehensive powder spec.
    - Manufacturing Method
    - Chemistry
    - PSD
    - Flowability Requirements
- Small batches can be quite expensive, often up to 3x the cost of a commercial lot.
- Lead times can extend notably (months)
- Feasible batch size very vendor specific
    - 30lb atomizers, vs 500lb atomizers
- Non-standard manufacturing methods:
    - ball milling, chemical reduction, etc.

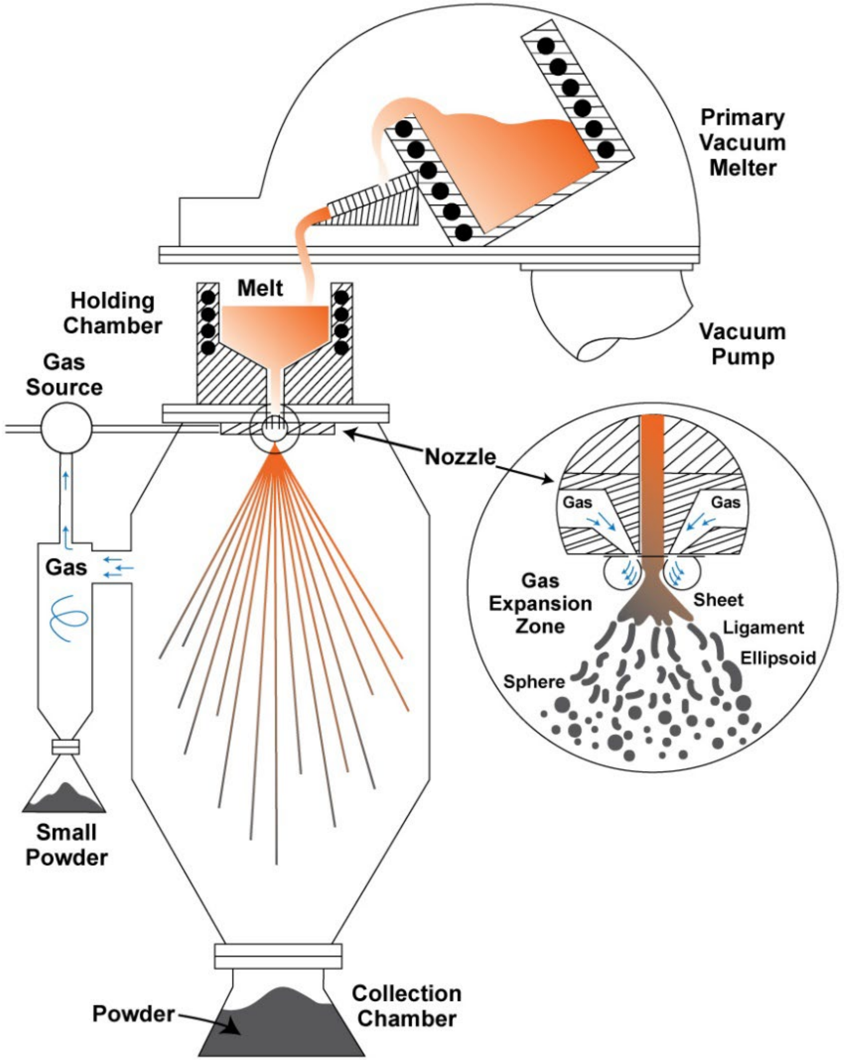
“custom” NiCr powder

Powder

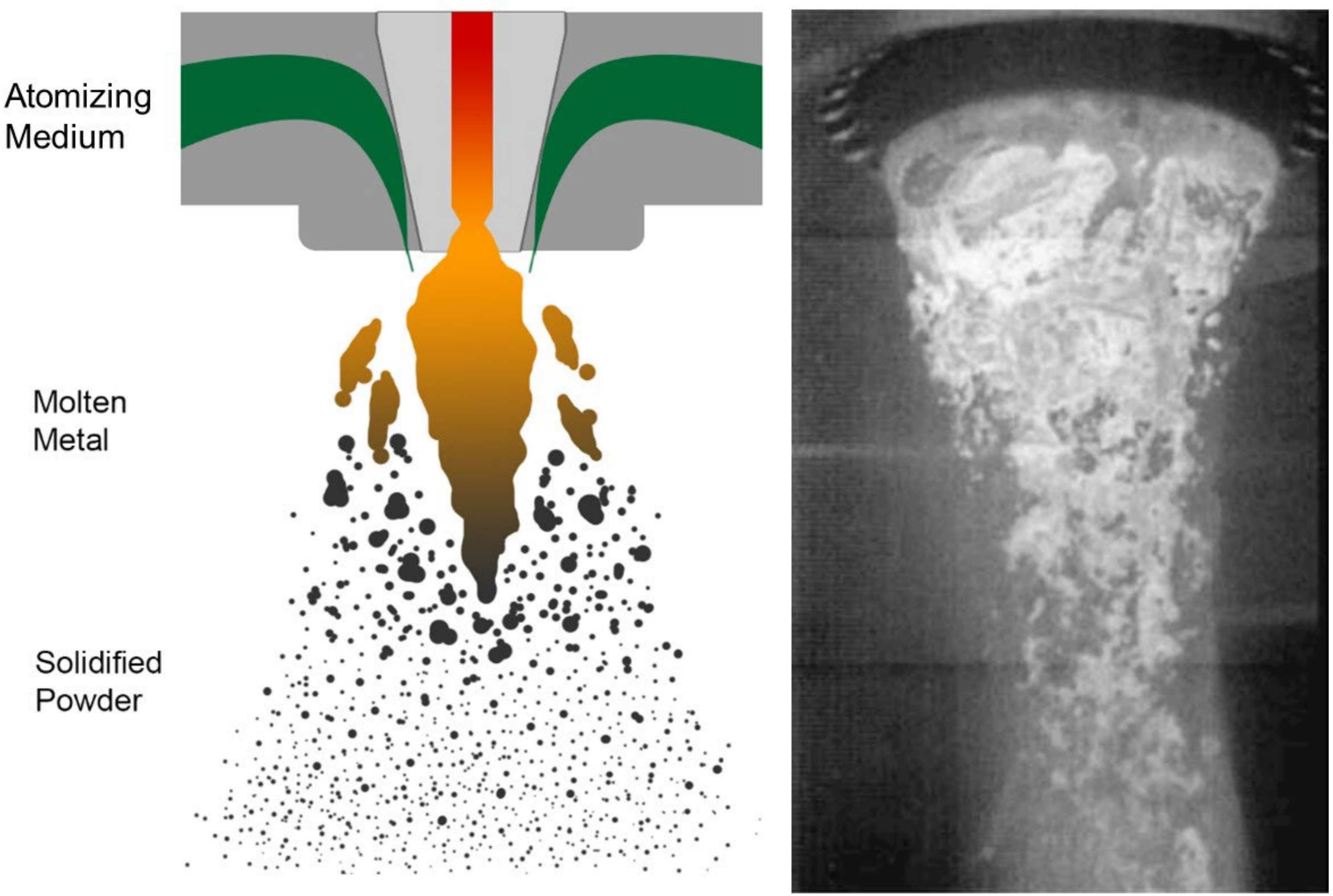
Powder

- Most popular feedstock for metal AM
  - Laser Powder Bed Fusion
  - Electron Beam Powder Bed Fusion
  - Laser Powder Directed Energy Deposition
  - Cold Spray
  - Binder Jet
- Powder Production
- Powder Specifications
- Characterization

Powder Manufacturing: Inert Gas Atomization

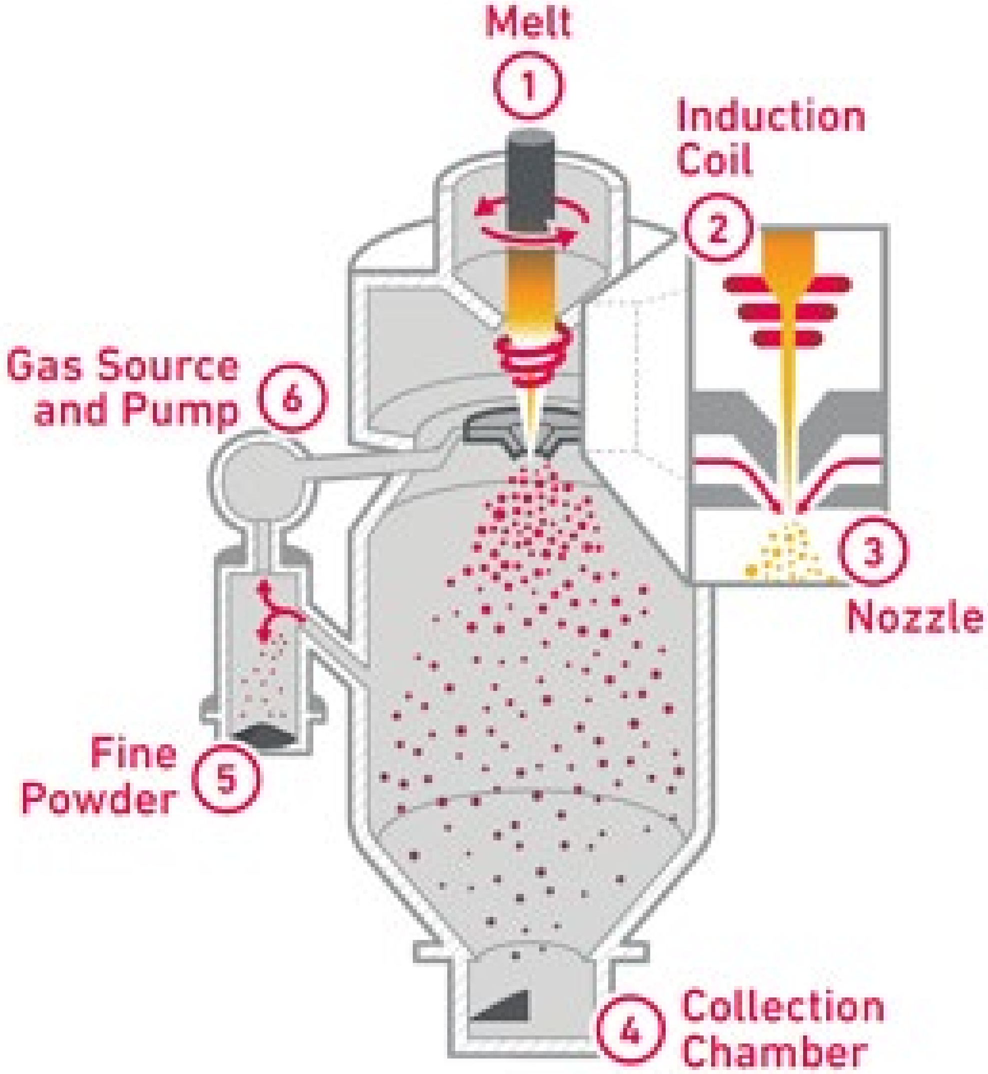


Crucible Gas Atomization [9]

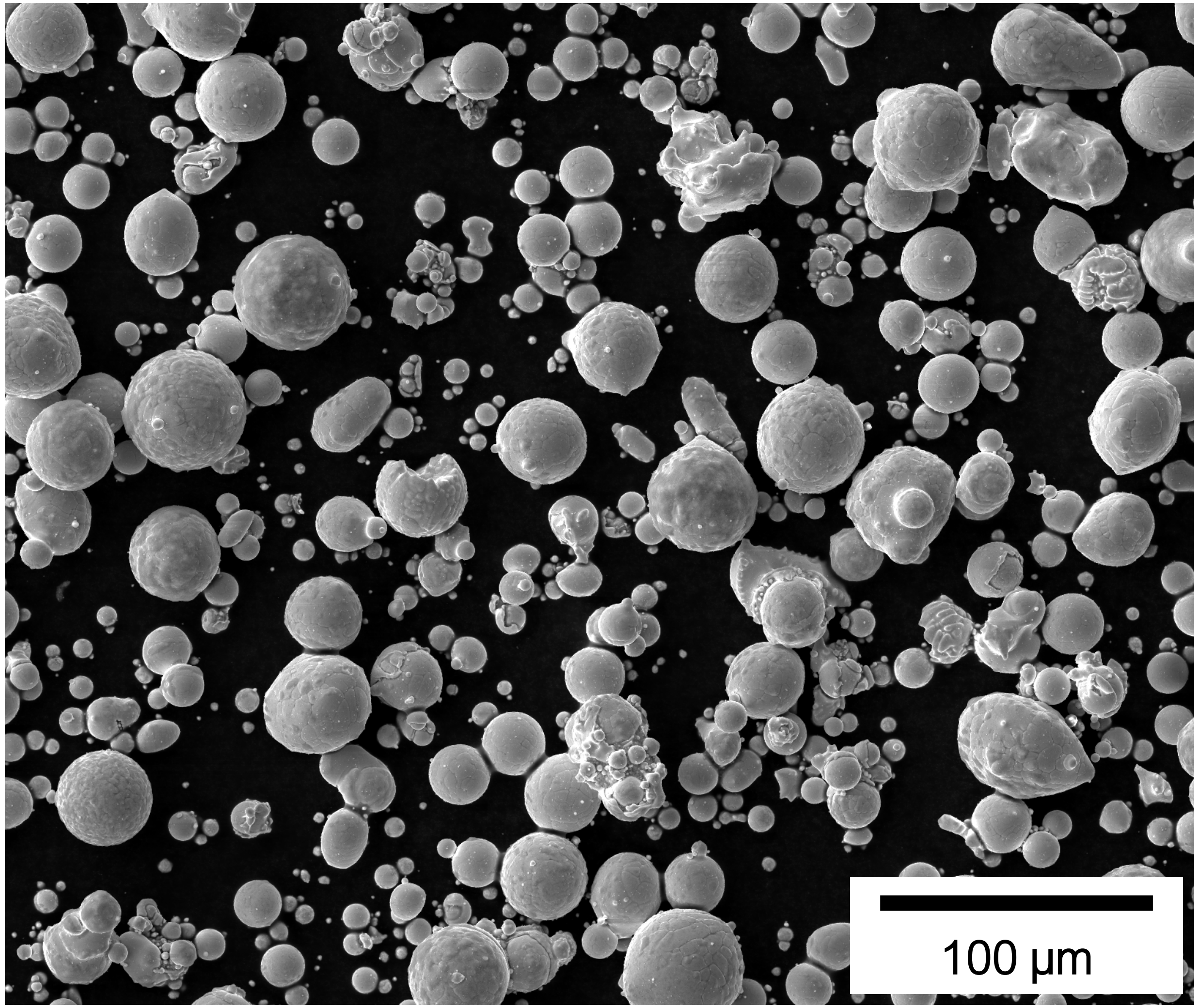


Nozzle Schematic [10]

Powder Manufacturing: Inert Gas Atomization



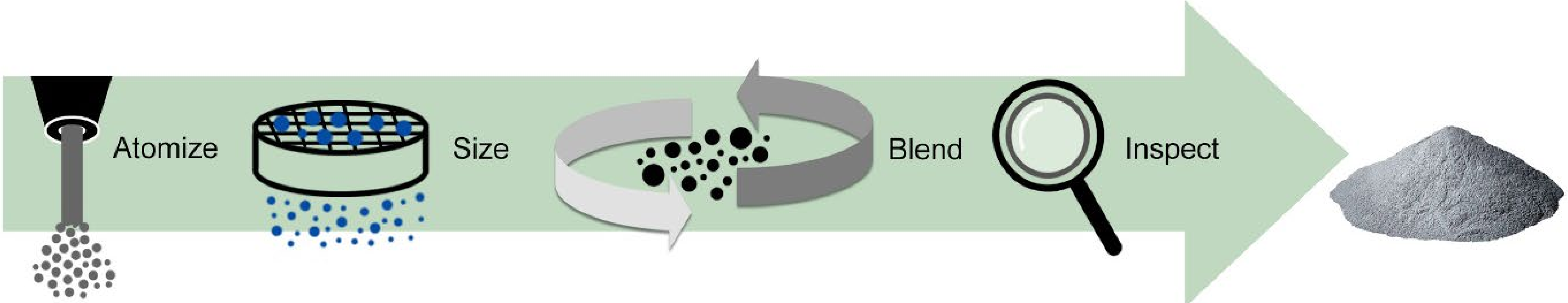
Electrode Induction Gas Atomization (EIGA) [11]



Gas Atomized Powder

Powder Manufacturing: Inert Gas Atomization

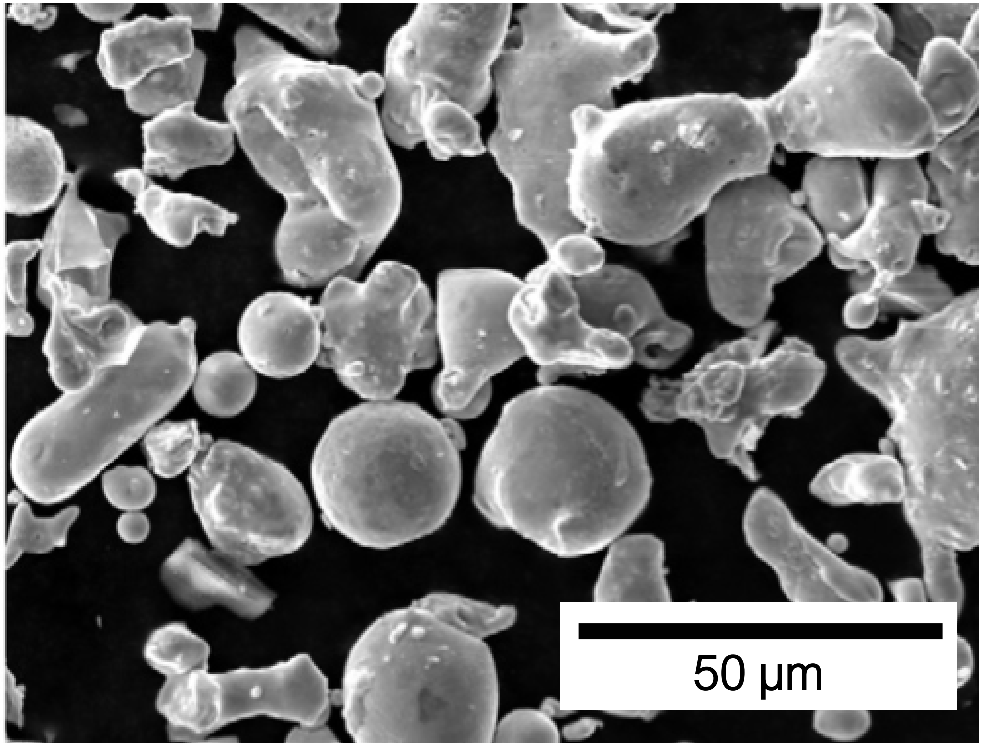
- Used for nearly every standard alloy (e.g. Fe, Ni, Cu, Ti, Al, etc.)
- Nozzle design essential to yield
  - Amount of powder atomized in e.g. LPBF size range (10-45 μm) is proprietary, but often <50%
- Spherical powder is formed due to the surface tension of the liquid drops
  - Improves flowability but can also lead to trapped gas porosity
- Powder is sieved and blended with other heats to form a lot



Powder Manufacturing: Other Atomization Types

Water Atomization:

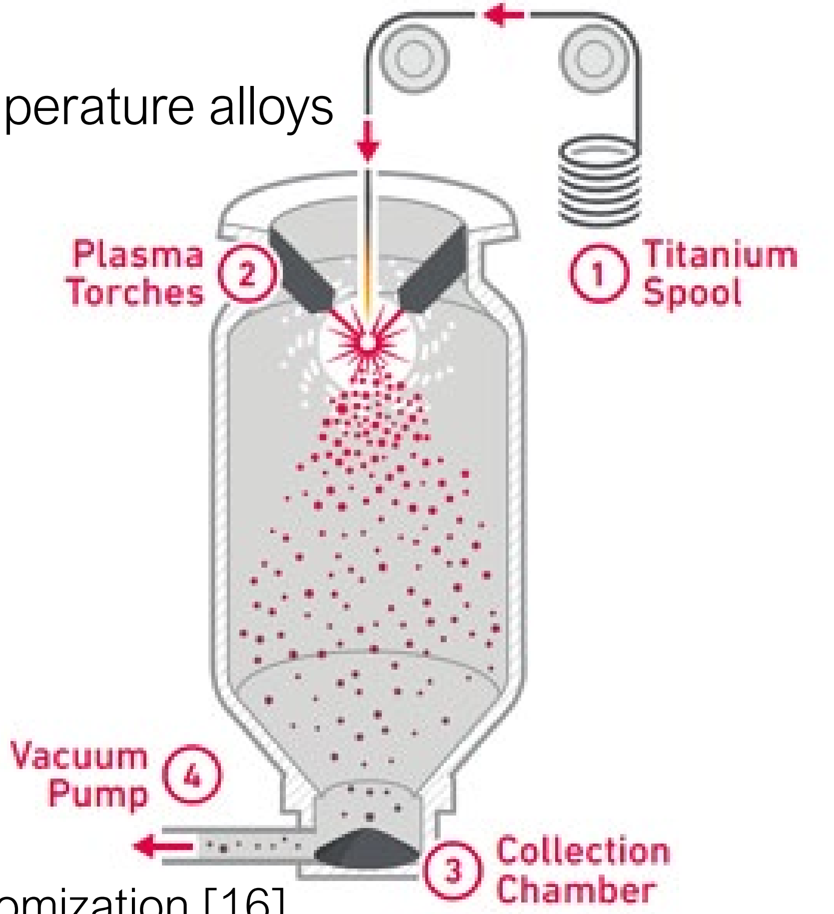
- Less expensive, but less spherical powder and moisture considerations.
- More common for ferrous alloys in traditional PM



Water Atomized Powder [29]

Plasma Atomization:

- Starting stock wire/bar.
- Plasma melts and atomizes powder
- Highly spherical
- Higher melting temperature alloys



Wire Plasma Atomization [16]

Powder Manufacturing: Other

Plasma Spheroidization:

- Non-spherical (e.g. milled) powder poured through a plasma to melt and spheroidize.
- Thermal arc, radio-frequency, microwave plasmas
- Powder rejuvenation, and flowability improvements.

High Temperature Remelting Solidification (HRS):

- Powder falls through vertical furnace and melts spheroidizes.
- Lower processing temperature and reducing atmospheres possible.

Rotary Atomization:

- Liquid stream impacts a rotating disk and spreads out forming droplets.
- Lower melting point alloys

Plasma Rotating Electrode Process (PREP):

- Bar stock used to form a plasma arc, which is rotated to release liquid droplets that solidify.
- Highly Spherical

Powder Manufacturing: Other cont.

Not necessarily for AM, but can be used in combination with a spheroidization or remelting process

Ball Milling:

- Mechanical deformation of the powder leads to flattening, shattering, and welding to form new particles. Contamination, oxygen, and flowability major concerns.

Hydride-dehydride:

- Metal is reacted with hydrogen to form a brittle hydride which can be milled
- Dehydridization returns the metal to pure state
- Has been used in metal AM without spheroidization

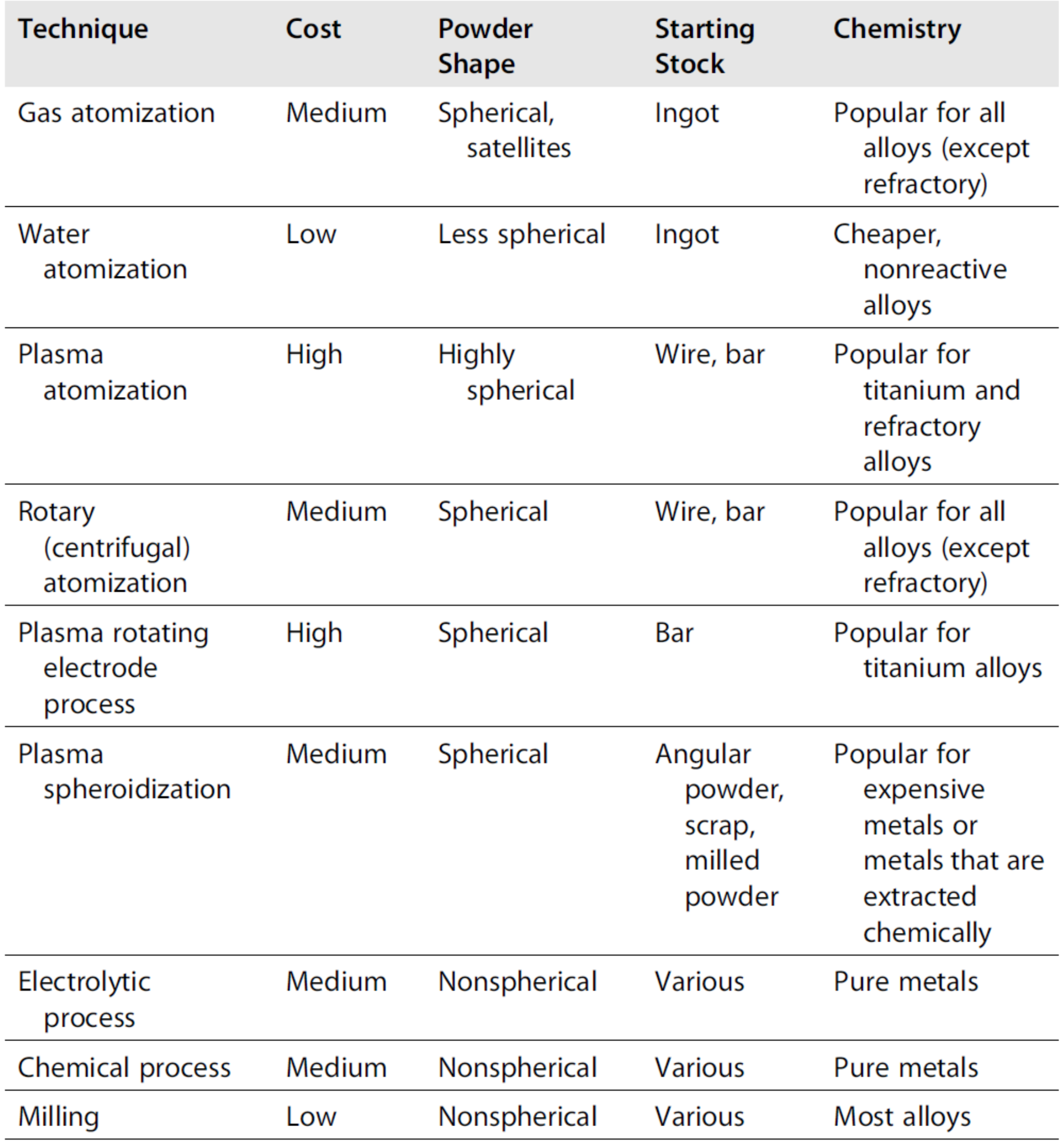
Electrolytic Methods:

- Galvanic process reduces metal ions into powder/sponge

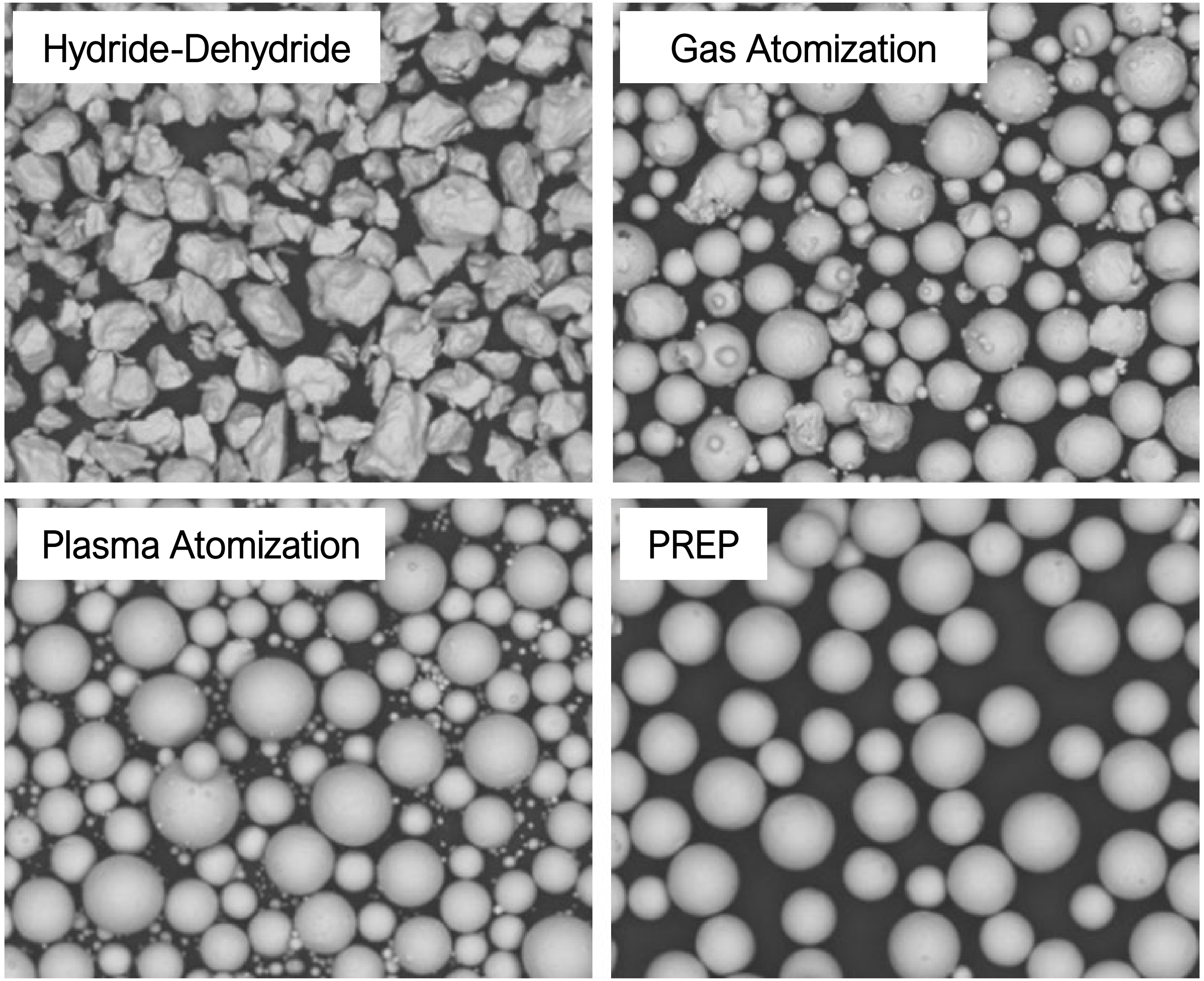
Chemical Methods:

- Oxide reduction, precipitation, and thermal decomposition

Powder Manufacturing: Summary



Technique	Cost	Powder Shape	Starting Stock	Chemistry
Gas atomization	Medium	Spherical, satellites	Ingot	Popular for all alloys (except refractory)
Water atomization	Low	Less spherical	Ingot	Cheaper, nonreactive alloys
Plasma atomization	High	Highly spherical	Wire, bar	Popular for titanium and refractory alloys
Rotary (centrifugal) atomization	Medium	Spherical	Wire, bar	Popular for all alloys (except refractory)
Plasma rotating electrode process	High	Spherical	Bar	Popular for titanium alloys
Plasma spheroidization	Medium	Spherical	Angular powder, scrap, milled powder	Popular for expensive metals or metals that are extracted chemically
Electrolytic process	Medium	Nonspherical	Various	Pure metals
Chemical process	Medium	Nonspherical	Various	Pure metals
Milling	Low	Nonspherical	Various	Most alloys

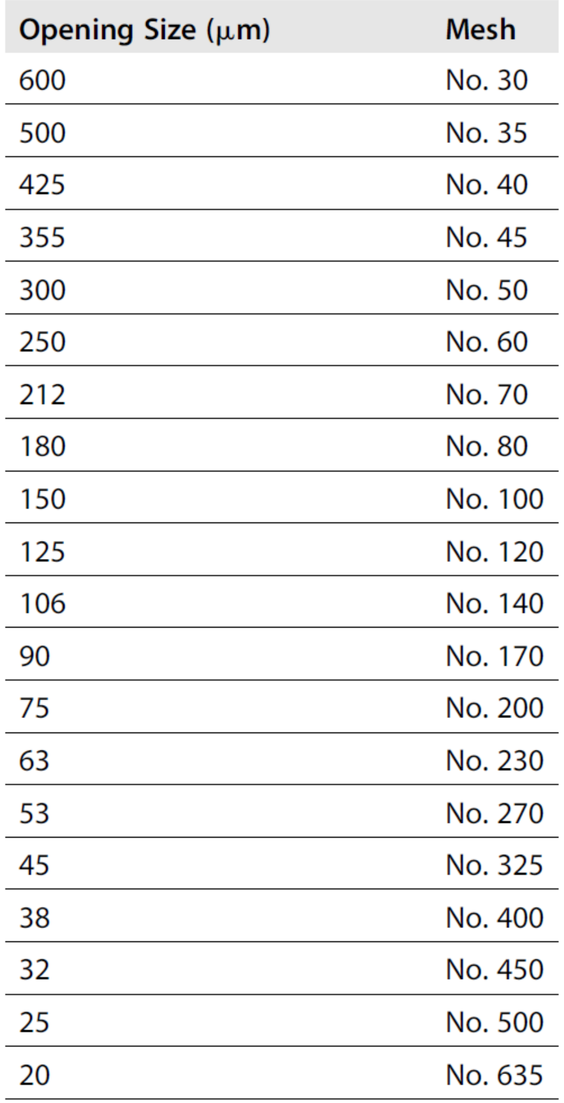


Representative Images of Powder [4]

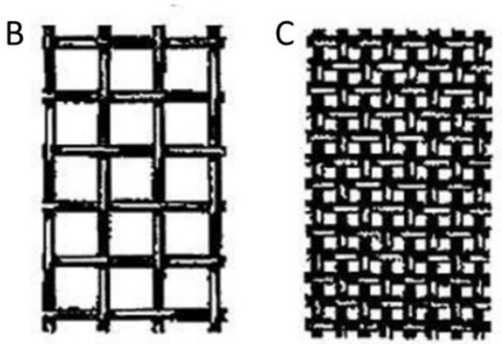
Powder Requirements: Powder Size Distribution

AM machines have ideal size range for powder. Can be specified in a few ways:

- “cut” based on mesh sizes is the simplest way but lacks some specificity
  - 10-45 μm
  - -53 μm (-270 mesh)
  - -270/+500 mesh (25-63 μm)
- d values require actual knowledge of the PSD and report standard percentiles, specified by e.g. ASTM B822
  - e.g. d_{10} = 20μm, d_{50} = 30μm, d_{90} = 50μm

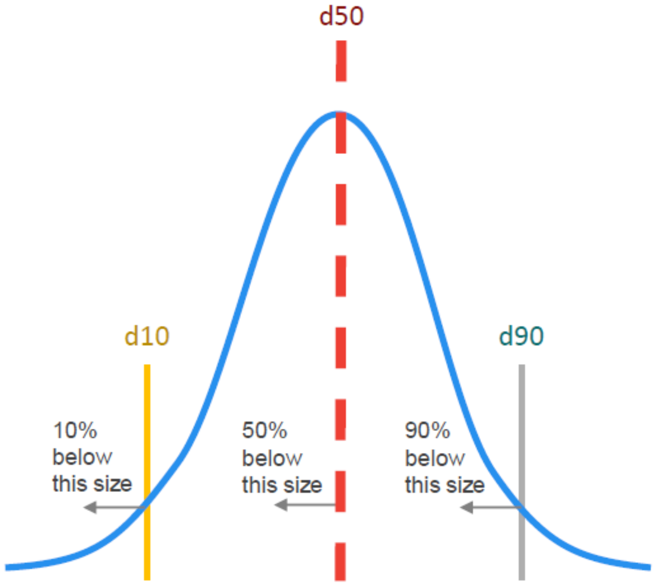


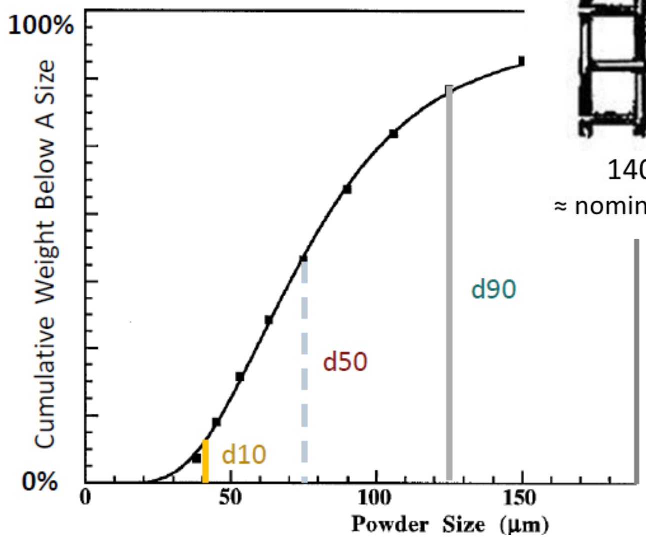
Opening Size (μm)	Mesh
600	No. 30
500	No. 35
425	No. 40
355	No. 45
300	No. 50
250	No. 60
212	No. 70
180	No. 80
150	No. 100
125	No. 120
106	No. 140
90	No. 170
75	No. 200
63	No. 230
53	No. 270
45	No. 325
38	No. 400
32	No. 450
25	No. 500
20	No. 635



140 mesh
≈ nominally 106 μm

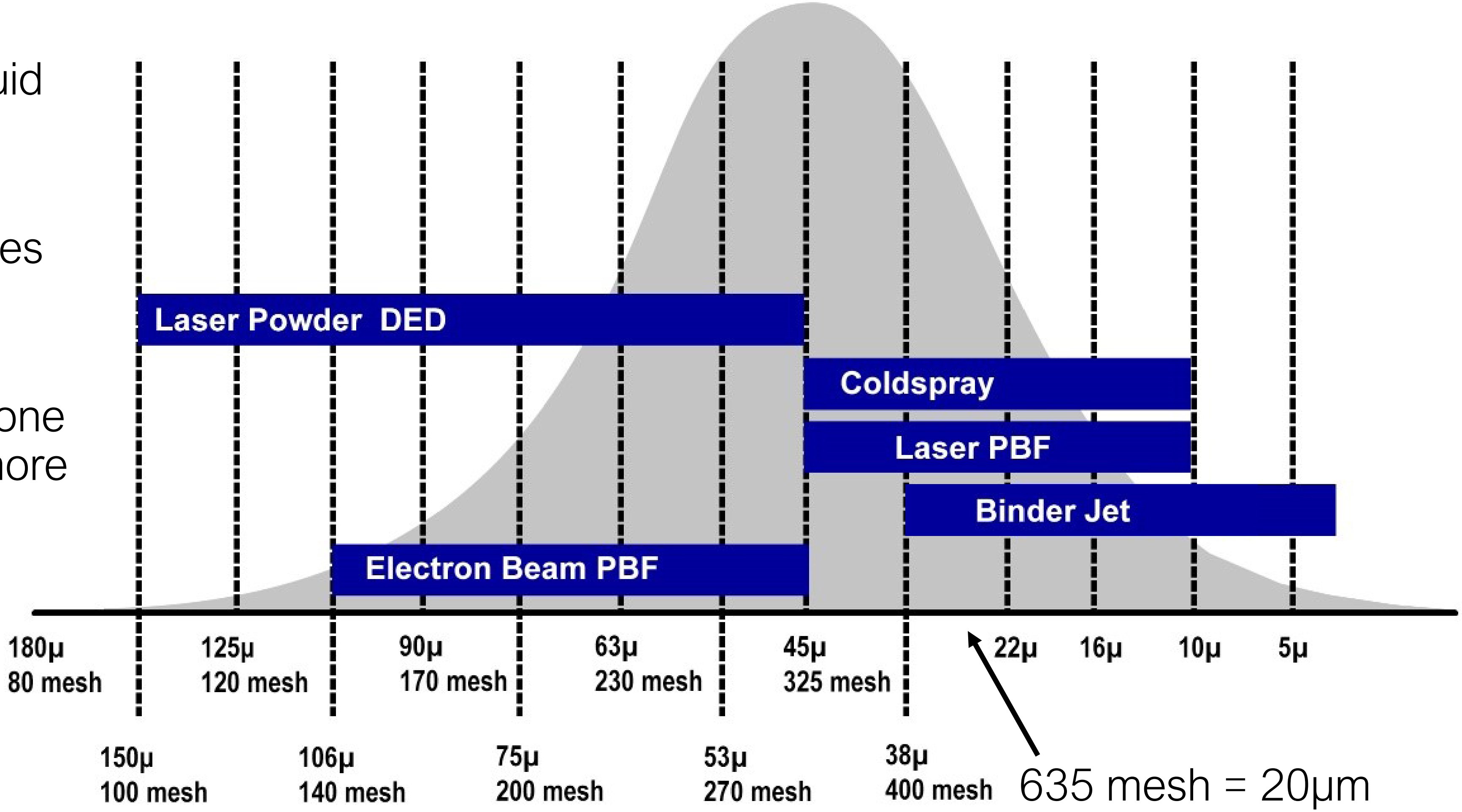
325 mesh
≈ nominally 45 μm





Powder Requirements: PSD

- Binder jet powder carried in liquid so flowability a lower concern
- Fines in LPBF cuts can greatly impact flowability. Removing fines (e.g. 635 mesh) is borderline impossible
- Air-based techniques (e.g. cyclone separation) can remove fines more efficiently.



635 mesh = 20µm

Powder Requirements: Other

Chemistry

- Target elements and amounts
- Min/Max limits on elements
- Specific impurity elements (e.g. S, P)

Cleanliness

- Oxygen and Moisture control
- Sealing powder in Ar gas
- Limited interaction with plastic
- Inclusions from powder manufacturing (e.g. crucible liner, foreign contamination)

Safety

- Respirators and fume hoods often necessary for powder handling
- PPE, ESD boots/mats
- Waste management
- Condensate (especially for Al, Ti, Nb)

Powder Characterization: PSD

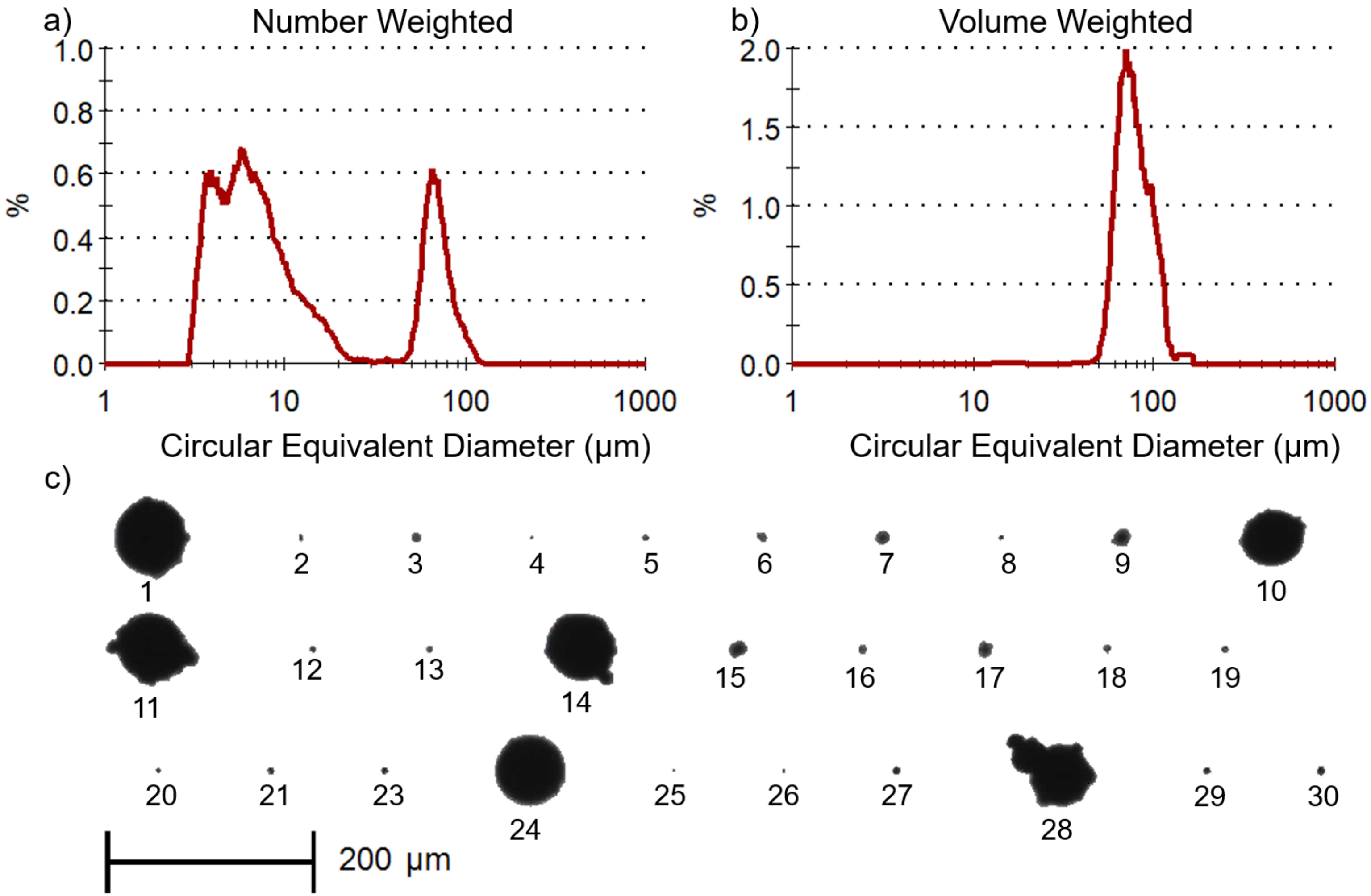
For d_{10}, d_{50}, and d_{90}, the actual PSD must be measured.

- Laser scattering particle analysis:
  - powder falling through laser causes scattering
  - angle of scattering depending on size of particle
  - Powder size distribution can be calculated without measuring individual particles
  - On the order of minutes
- Direct powder measurement:
  - Using an automated optical/electron microscope hundreds/thousands of individual particles are imaged (slow)
  - Wider range of distributions available (shape features)
- Silhouette measurement:
  - Light source casts a shadow of particles which can be imaged.
  - Balance of speed and descriptiveness

Powder Characterization: PSD

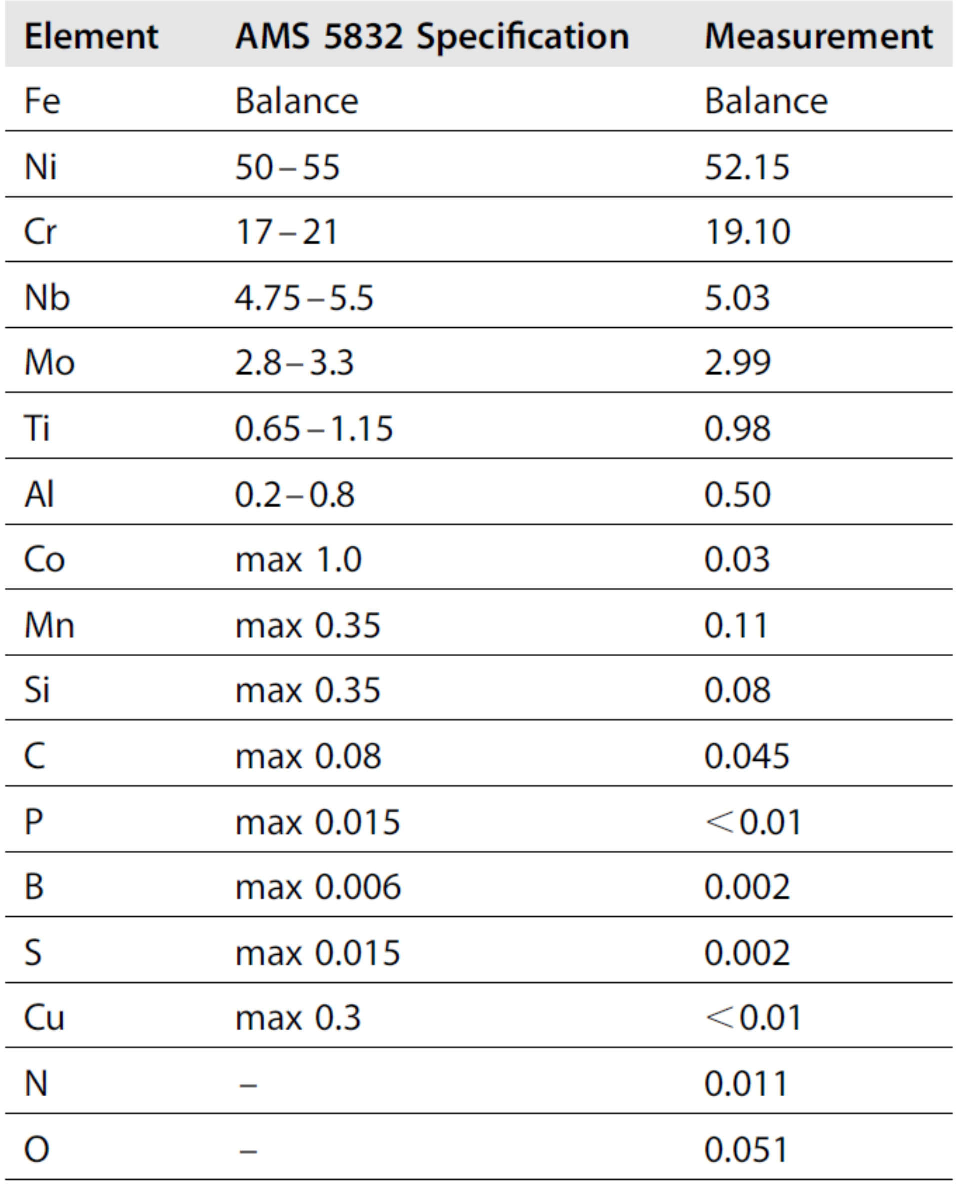
Number vs Volume (Mass) weighted

- Most physical measurements (e.g. sieving) are dependent on the mass of powder, but certain properties (e.g. flowability) rely on the number of particles.
- Specification of fines (d10) is based on weight/volume



Powder Characterization: Chemistry

- Chemistry must be specified, e.g. for IN718 in AMS 5832
- Atomized powder and printed parts can have different compositions due to preferential evaporation of certain elements (e.g. Ti-6Al-4V)
- Inductively-Coupled Plasma (ICP) ionizes metal ions which can be measured using atomic emission spectroscopy (AES) or mass spectroscopy (MS)
  - Accuracy can vary based on element and amount, but often accurate within 1%.
- Combustion-based techniques used for measuring O, N, S, and C, and are accurate in the ppm-ppb range.
- XRD can measure phases and phase amounts, which can inform composition
- Chemical extraction can be used to dissolve certain phases, to measure undissolved phases
- Moisture can affect oxidation and flowability; measured separately

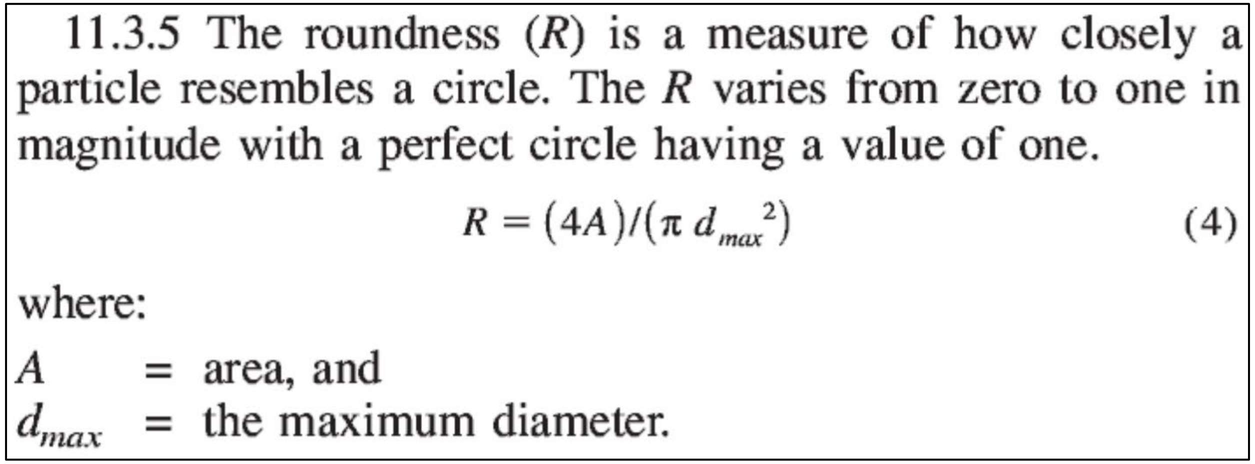


Element	AMS 5832 Specification	Measurement
Fe	Balance	Balance
Ni	50–55	52.15
Cr	17–21	19.10
Nb	4.75–5.5	5.03
Mo	2.8–3.3	2.99
Ti	0.65–1.15	0.98
Al	0.2–0.8	0.50
Co	max 1.0	0.03
Mn	max 0.35	0.11
Si	max 0.35	0.08
C	max 0.08	0.045
P	max 0.015	<0.01
B	max 0.006	0.002
S	max 0.015	0.002
Cu	max 0.3	<0.01
N	–	0.011
O	–	0.051

Powder Characterization: Morphology

Particle shape descriptors are not necessarily part of the powder spec. or purchasing process, as they are mostly based on manufacturing method (e.g. ASTM E1877).

- Aspect Ratio
- Elongation
- Equivalent Circular Diameter
- Feret Diameter
- Form Factor
- Particle Breadth
- Particle Length
- Roundness



11.3.5 The roundness (*R*) is a measure of how closely a particle resembles a circle. The *R* varies from zero to one in magnitude with a perfect circle having a value of one.

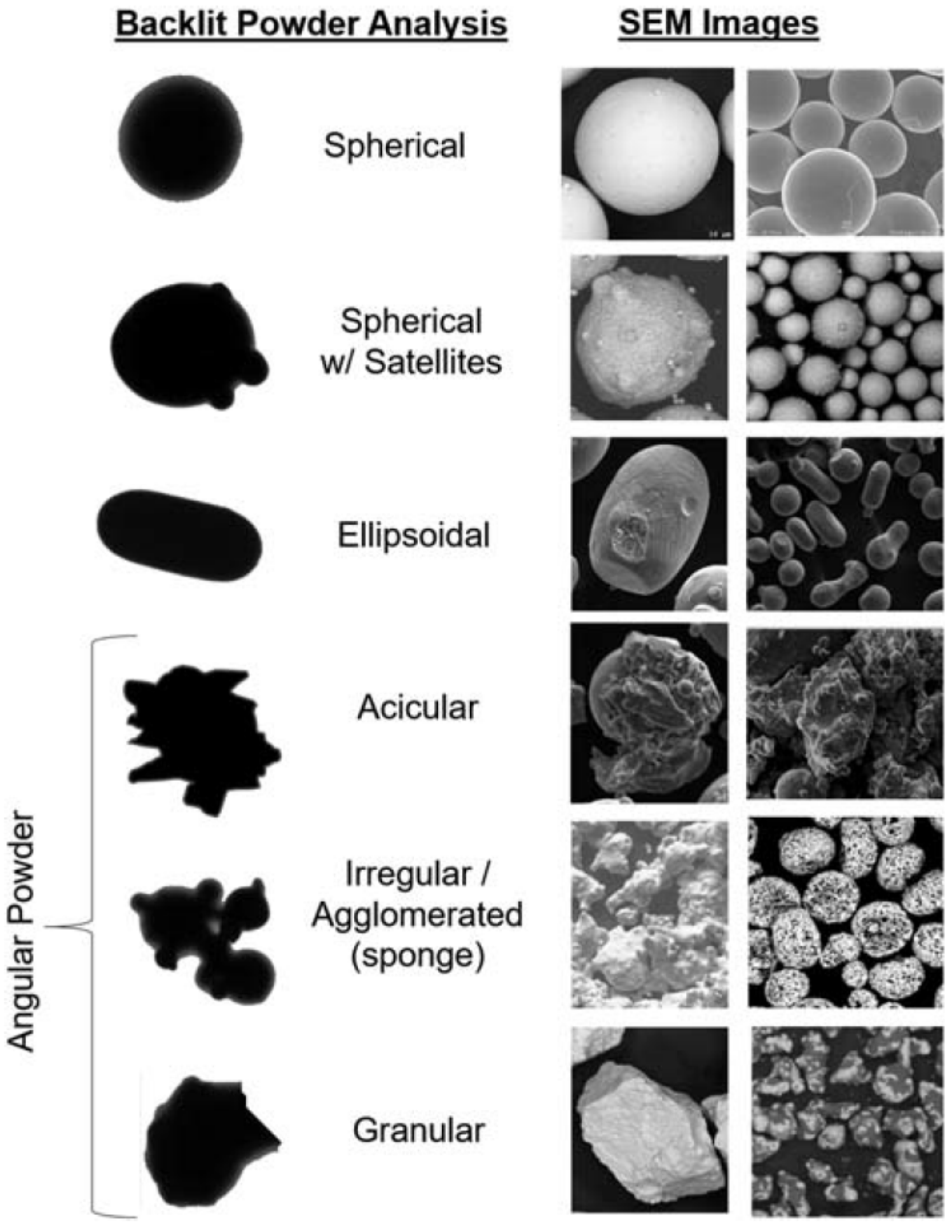
$$R = (4A)/(\pi\, d_{max}{}^2) \qquad (4)$$

where:

A = area, and

d_{max} = the maximum diameter.

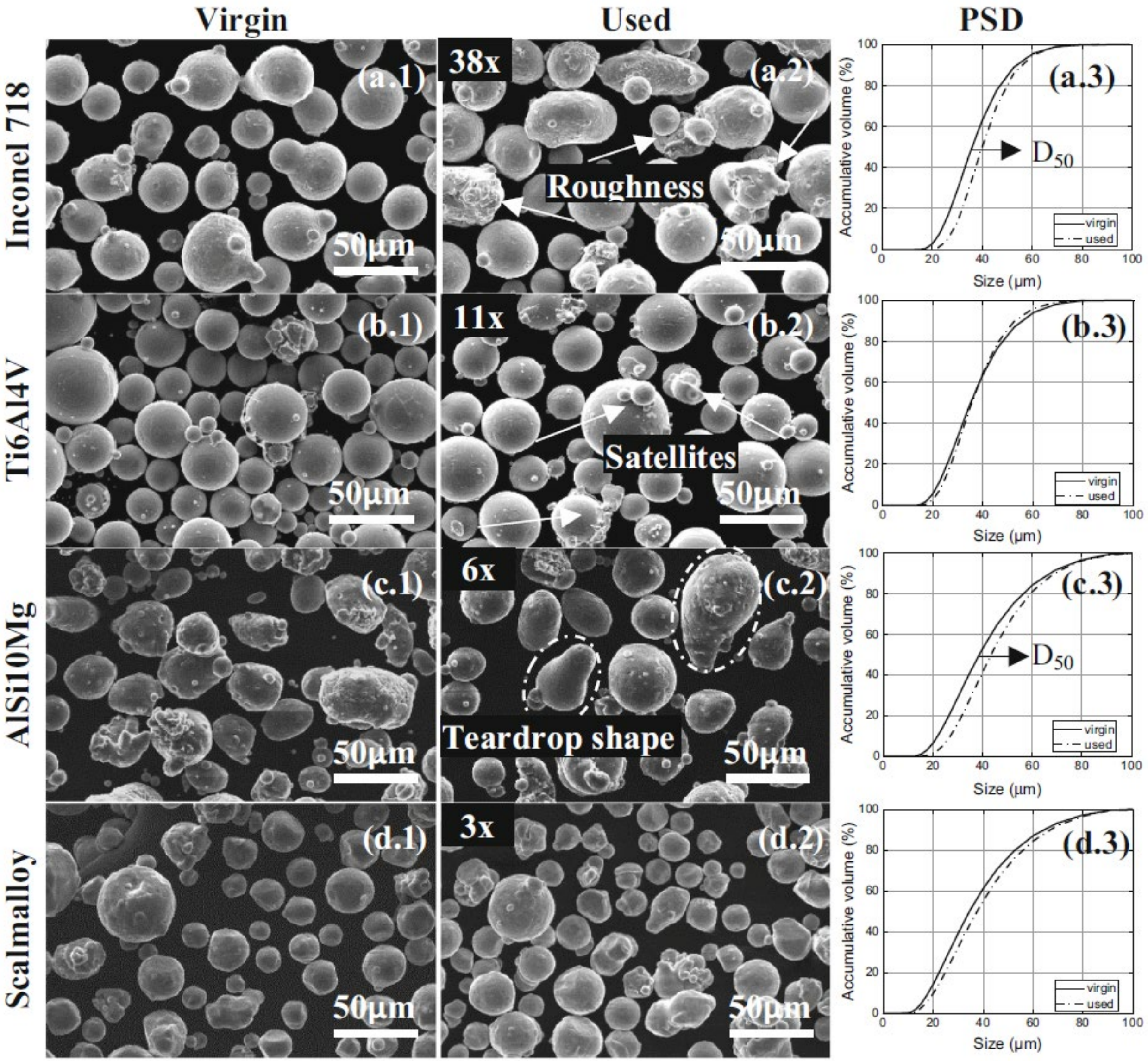
ASTM E1877



Courtesy of PAC

Powder Characterization: Morphology

- Satellites more common in gas atomized particles
- Reuse can lead to the accumulation of non-spherical powder



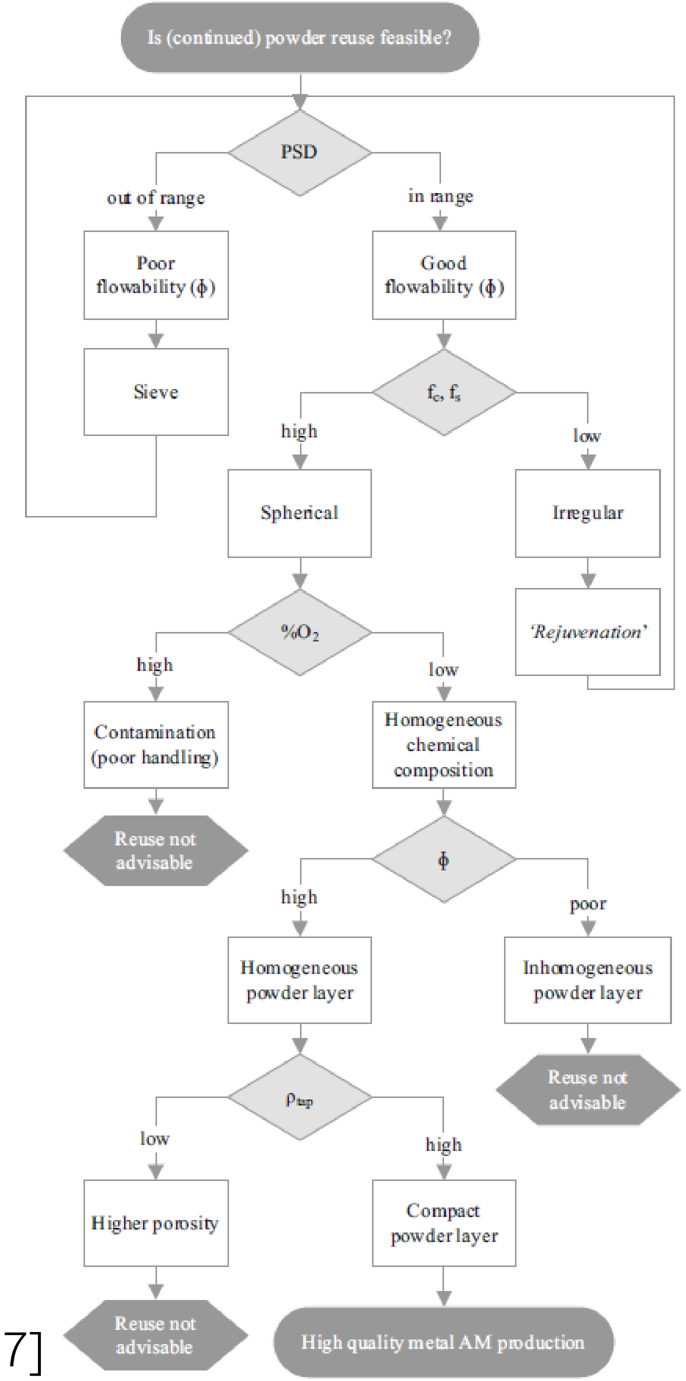
Images of reused powder for various alloys [47]

Powder Characterization: Rheology

- Powder flowability is an essential characteristic for AM
- Each machine has different flow requirements (e.g. hopper vs cylinder)
- Hall Flow ASTM B213 or Carney Flow (ASTM F1877) is the simplest method
- Rotating drum techniques produce many flow properties.
- Packing density is mostly a heritage PM property
- Powder spreading and spread density are not easily measured

Powder Characterization: Reuse

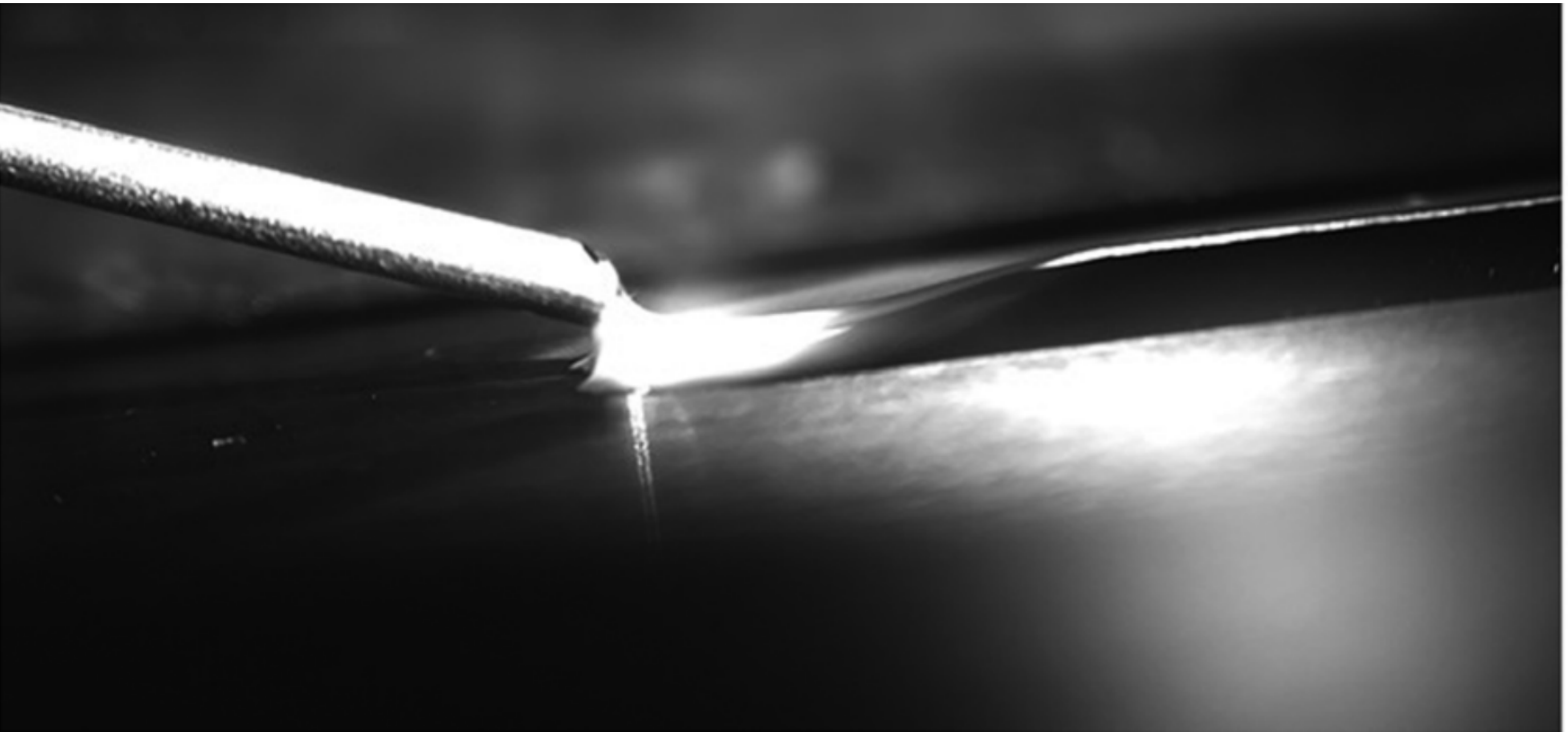
- Powder recycling is an essential aspect to the sustainability and material savings promised by AM
- Most protocols involved blending sieved reused powder with virgin powder.
- Oxygen pickup is the most observed change in reused powder
- Flowability can often be improved with reuse

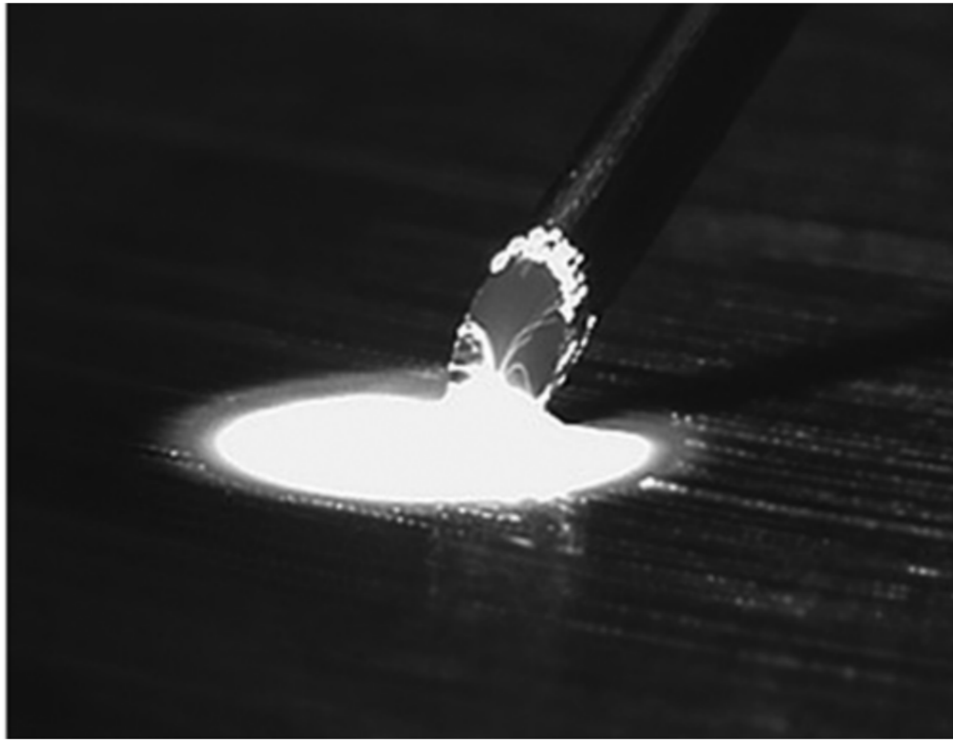


Recycling Flow Chart [47]

Powder Characterization: Other

- Powder coating: Coating one powder with an oxide/metal/carbide. Allows for micro editions and alloy development.
  - Chemical deposition
  - Plasma Spray
  - Mechanical Mixing
- Elemental mixing: Useful for alloying, but inhomogeneous. Especially common in DED where a large melt pool allows for more mixing.
- Printing non-standard powder: milled, HDH powder

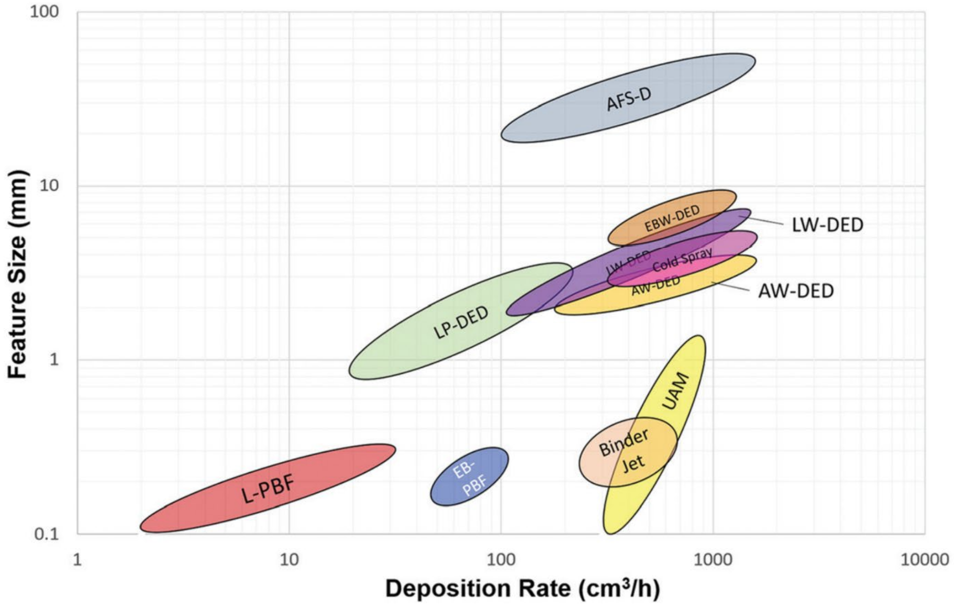


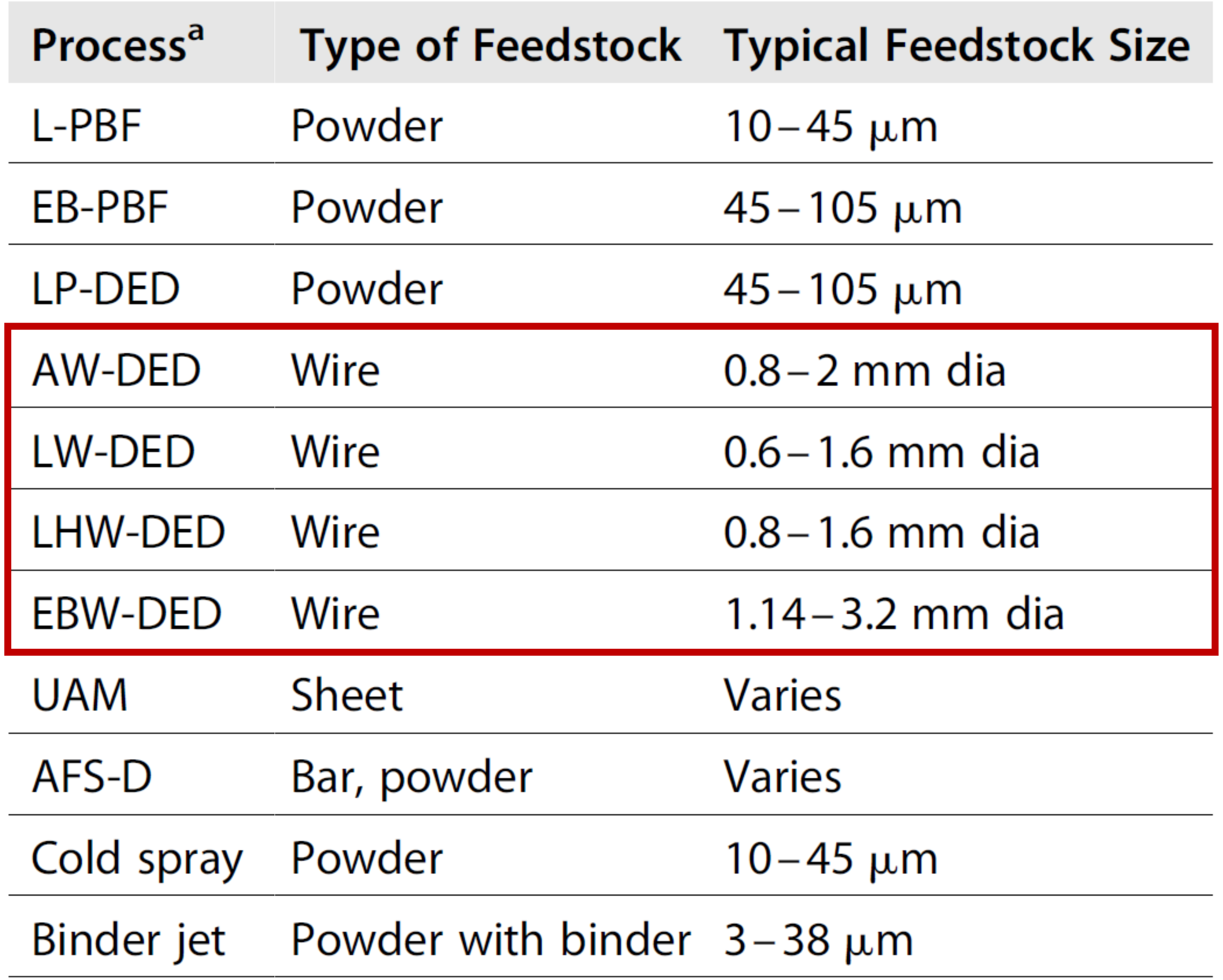


Wire

Wire Feedstock

- High Deposition Rates





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UAM	Sheet	Varies
AFS-D	Bar, powder	Varies
Cold spray	Powder	10–45 μm
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Wire Supply Chain And Safety

Supply Chain:

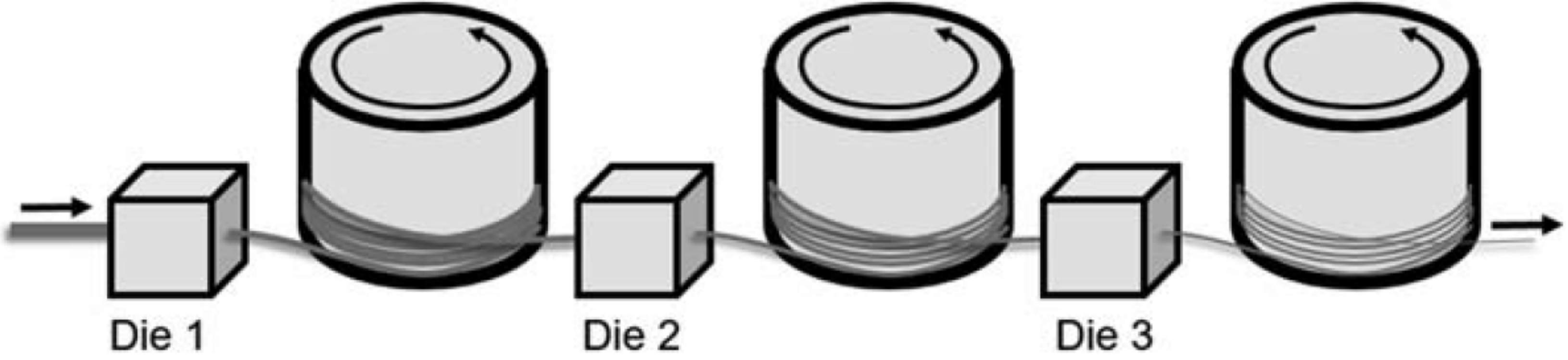
- Ti, Ni, Fe alloys all commonly exist in wire form
- Cost/weight can be significantly lower than powder
- Custom batches of wire can be more expensive
- Solid or cored wire possible

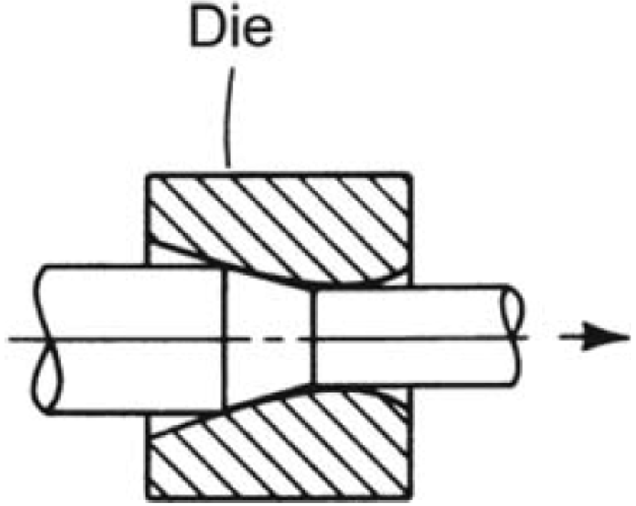
Safety:

- Not an inhalation risk
- Simplified handling
- Ideal for in-space or repair
- Reactive metals still require some caution

Wire Manufacturing

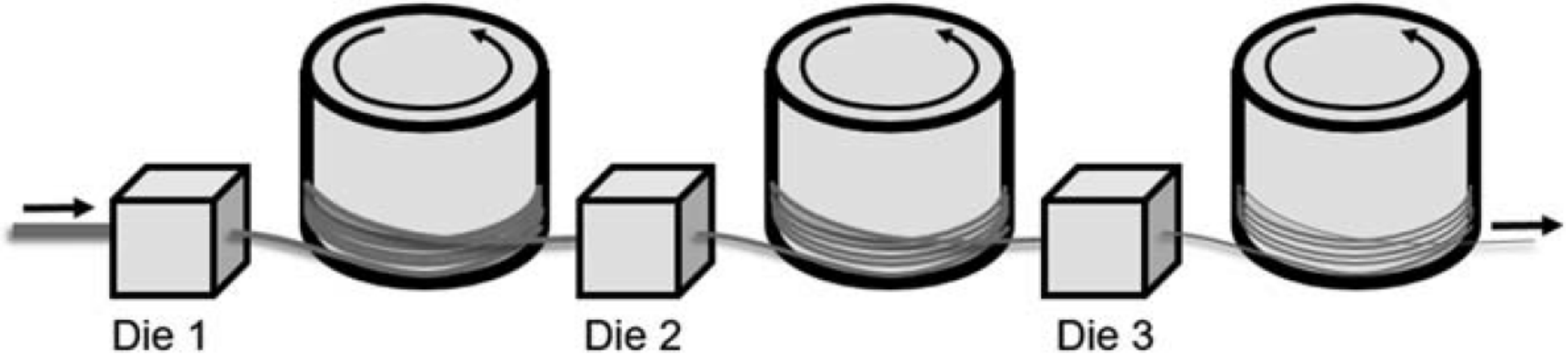
- Wire drawing uses progressively smaller dies to reach the desired diameter.
- Descaling: Often accomplished by high-angle bends that release scale
- Annealing often necessary to optimize the hardness of the wire at different steps, or to avoid breaking
- Dies often made from hard materials (carbides, tool steels), and reduce the material by ~20%
- Solid Lubricants can be used to preserve die life

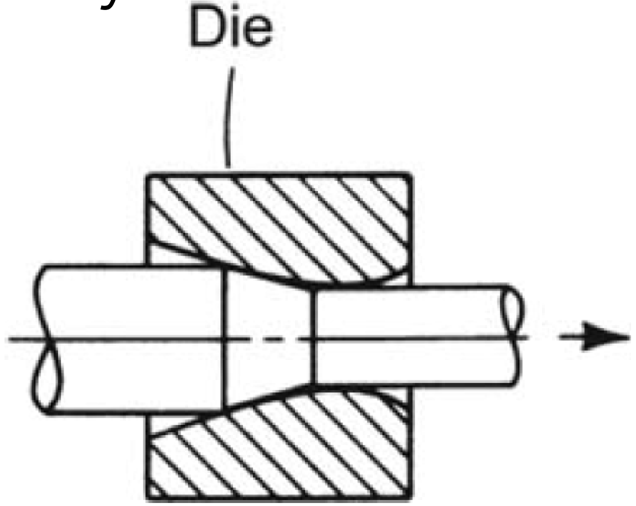




Wire Specifications

- American Welding Society (AWS) sets standards for wire (and rod)
- Designations, e.g. ER316L
  - ER = “electrode or rod”
  - 316 = composition (300 series stainless)
  - L = “low carbon (max 0.03 wt.%)
- Diameter, Length, Spool Size, etc.
- For welded materials, properties of deposited material may be supplied.





Other Feedstocks

- Ultrasonic Additive Manufacturing
  - Foil feedstock highly custom
  - Typically rolled into foil using traditional methods
  - Al, Cu, Mg, Ni, Ti are used
- Additive Friction Stir Deposition
  - Bar/Rod Stock
  - Manufactured using traditional methods.
  - Most common for Al alloys

Summary

- Powder feedstock specifications and essential characteristics are necessary for the proper operation of the AM process
  - Chemistry
  - Size Distribution
  - Flowability
  - Morphology
  - Safety
  - Reuse
- Atomization plays the primary role in powder manufacturing, but has many variations and many competitors
- Wire has fewer critical characteristics but still emerging
- Non-standard (foil, bar) and recycled feedstock of growing interest.

Thank You!

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With significant contributions from:

- Dave Ellis (NASA GRC)
- Paul Gradl (NASA MSFC)
- Kevin Luo (SLM Solutions)
- Rich Meklus (PAC)
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- Will Evans (NASA MSFC)
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- Tyler Gibson (NASA MSFC)
- Andy Hardin (NASA MSFC)
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- Sam Cordner (NASA MSFC)

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