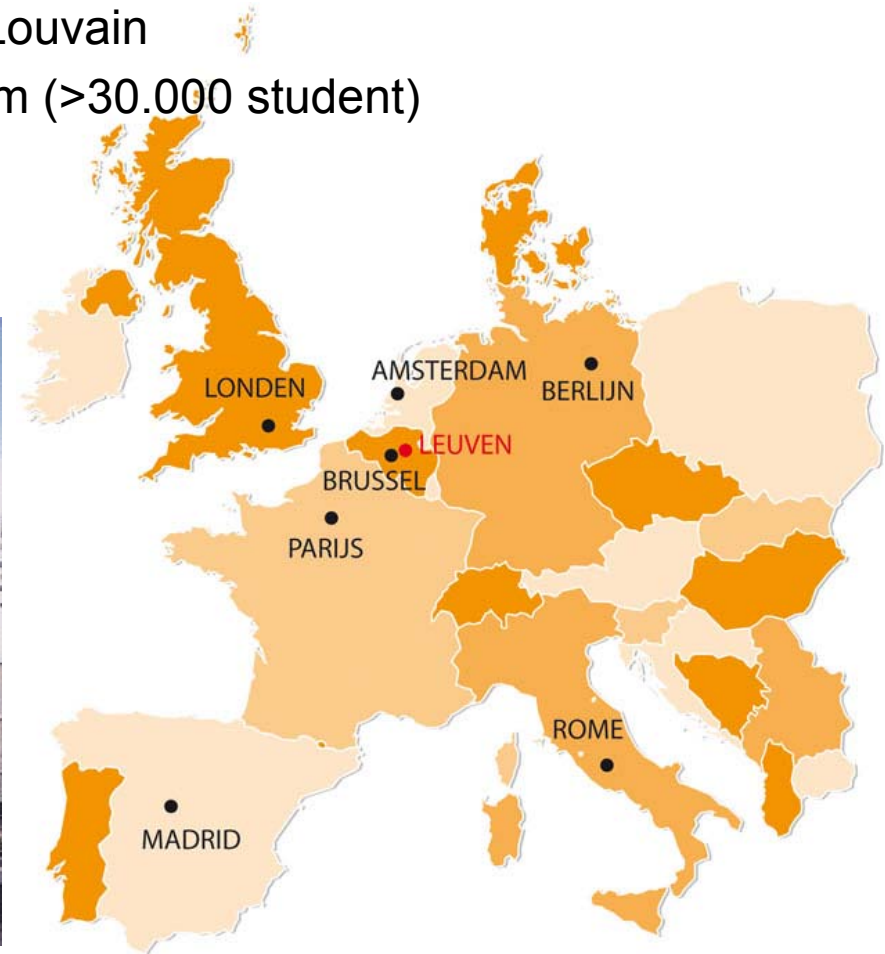


Powder Bed Fusion Additive Manufacturing

Prof. Dr. Ir. Jean-Pierre Kruth
KU Leuven university, Belgium

Introducing KU Leuven university

- Located 20 km East of Brussels, Belgium
- Founded anno 1425 as oldest catholic university
- 1970: split between KU Leuven and UCLouvain
- KU Leuven = largest university in Belgium (>30.000 student)
- Long tradition in manufacturing
 - CIRP Intern. Academy for Production Eng.
 - SME Univ. LEAD award (1998)



Introducing AM activities of KU Leuven university

- **Company:** **Materialise N.V., Leuven**
- Initial activity: producing prototypes in **plastics** by layerwise Rapid Prototyping techniques
- Spin-off of KU Leuven (Division PMA)
- Start: 1990 (Founder: W. Vancraen)
- Today:
 - Largest RP or AM service bureau
(>85 RP/AM machines in one location; >300.000 parts/year in 2011)
 - Largest RP/AM software developer a world
- From 2 to 1000 persons
(2011: Materialise Dental splits off)

1995:
*Mammoth Stereolithography
machine (build capacity
2200 x 840 x 800mm)*



Introducing AM activities of KU Leuven university

MATERIALISE SOFTWARE



Softwares

Magics
(RP, RM, supports, ERP, e-software,...)

3-matic
(facetted CAD)

Mimics
(medical)

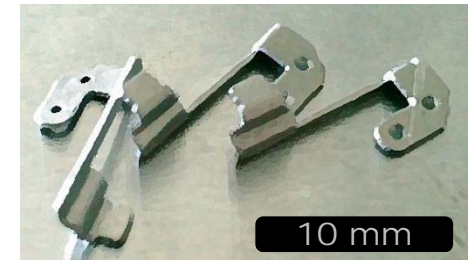
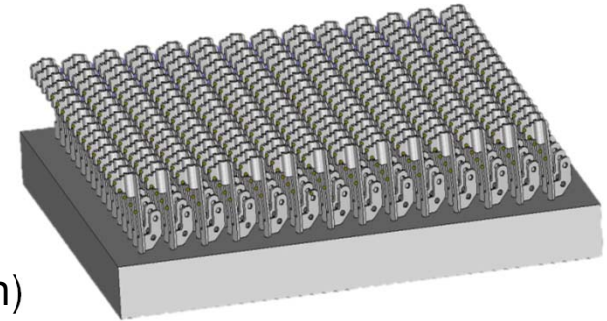
SurgiCase
(surgery planning)

RSM
(hearing aids)

Etc.

Introducing AM activities of KU Leuven university

- **Company:** LayerWise N.V., Leuven
- **Field:** RP & AM of **metallic** products
- Spin-off of KU Leuven (Division PMA)
- **Start:** 2008 (Founders: P. Mercelis, J. Van Vaerenbergh)
- **Today:** 45 persons
- **Production:** > 20,000 metallic parts/year (2011)
- **Activities:**
 - Industrial, medical & dental applications (also artwork)
 - Several patents (dental and others)

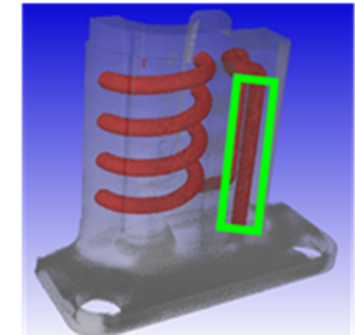
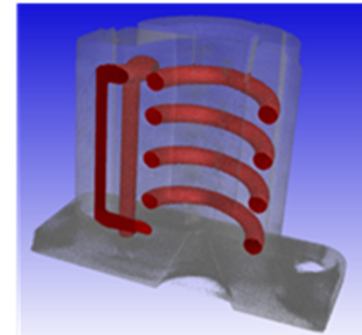
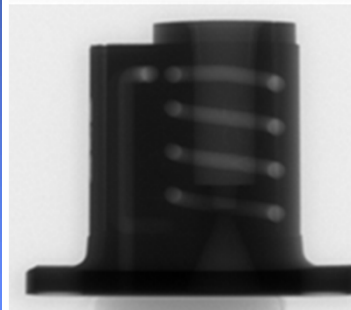
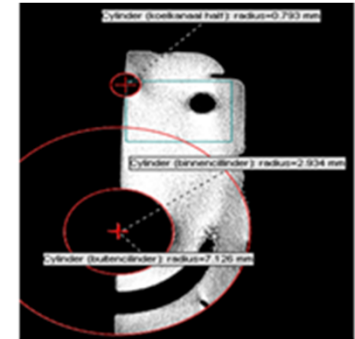
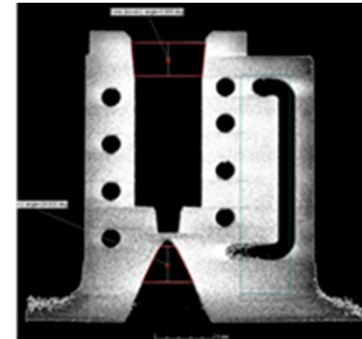
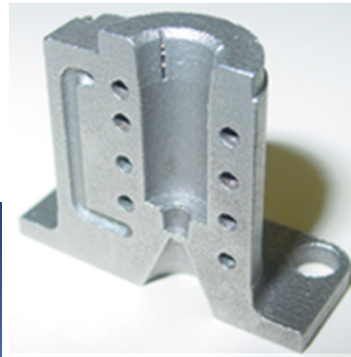


Introducing AM activities of KU Leuven university

- **Company:** **Metris N.V., Leuven** (since 2009 Nikon Metrology Europe N.V.)
- Spin-off of KU Leuven (Division PMA)
- Start: 1995 (Founders: B. Van Coppenolle, L. De Jonge)
- Today: 1000 persons
- **Activities:** (Reverse engineering), **3D coordinate metrology & quality control**
 - 3D CMM, laser scanning probes
 - **X-ray CT for measuring internal & external geometry** (tolerances) and **material quality** (e.g. porosity)



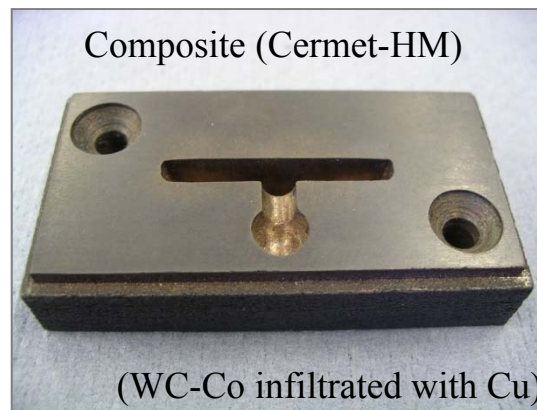
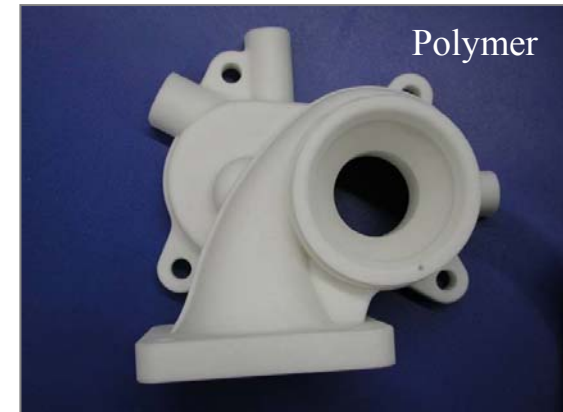
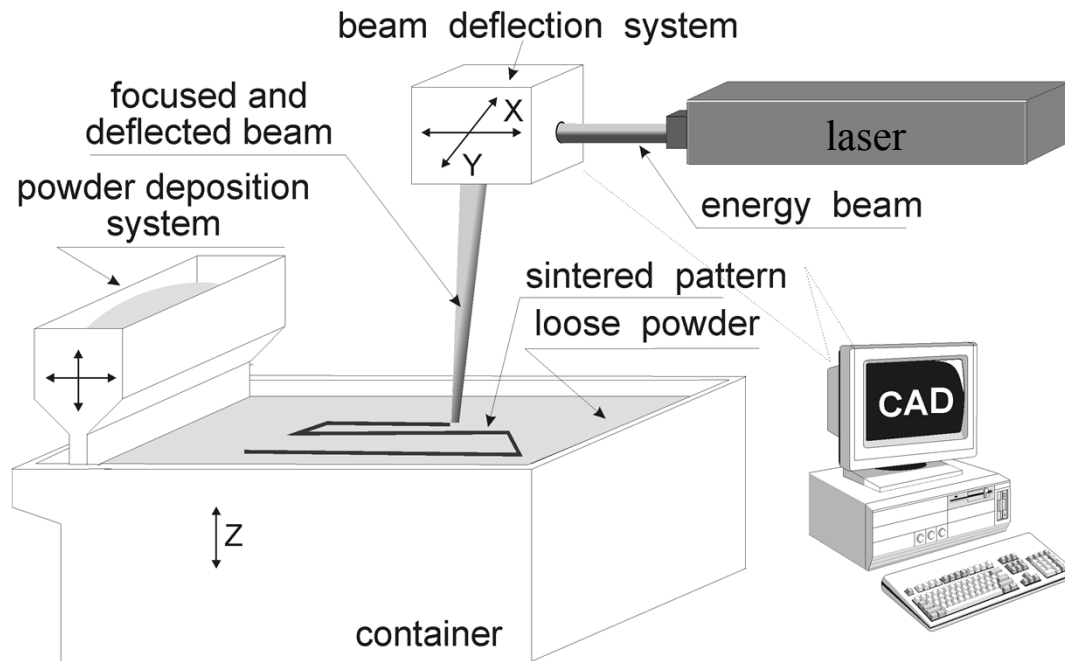
Industrial CT scanner (450 kV)



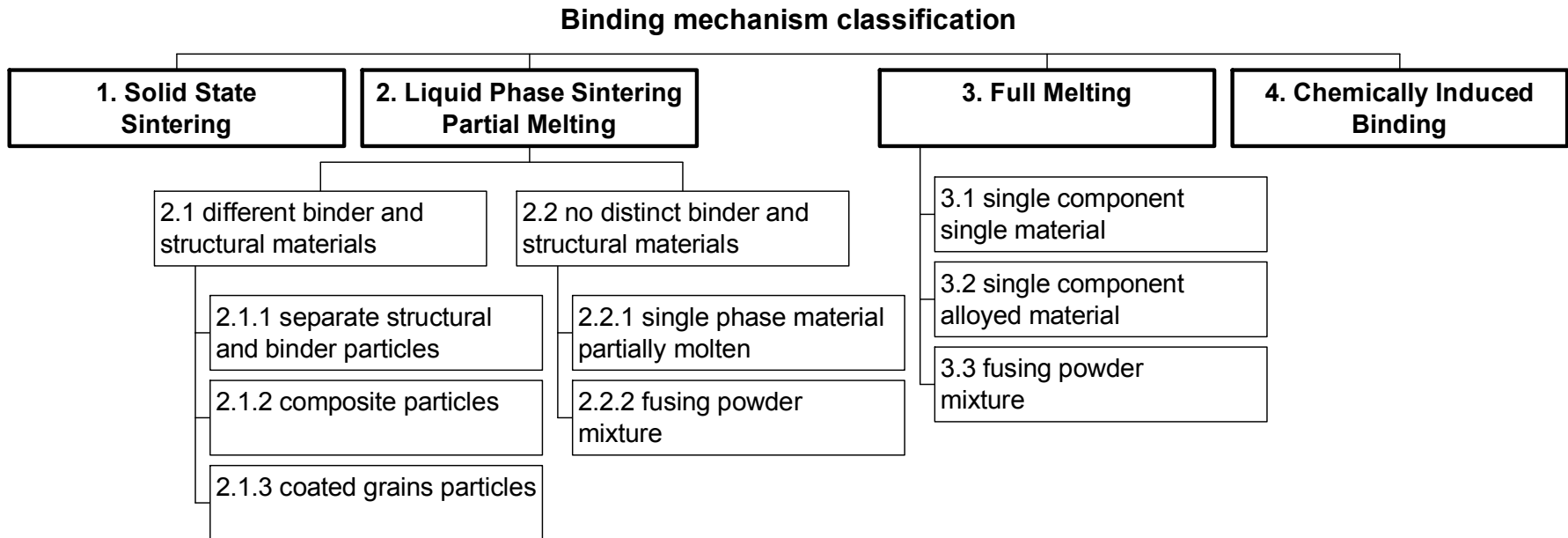
Materials and Processing Issues in Powder Bed Fusion Additive Manufacturing

Prof. Dr. Ir. Jean-Pierre Kruth
KU Leuven university, Belgium

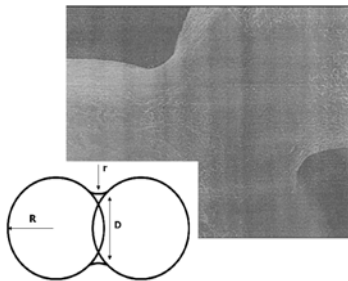
Laser powder-bed fusion AM: SLS, SLM, ...



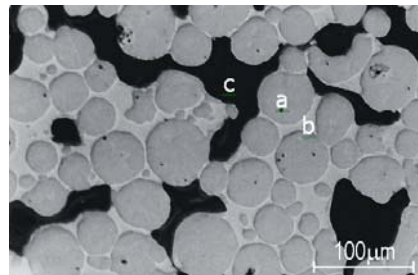
Classification of binding mechanisms



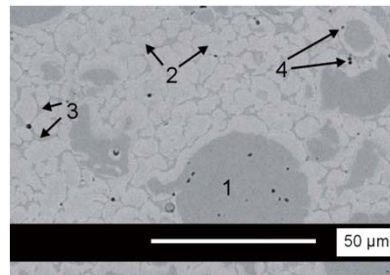
Solid State Sintering



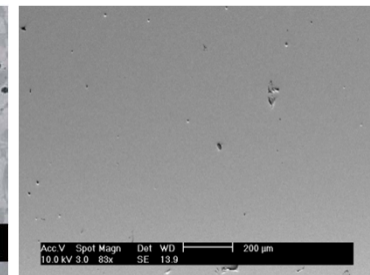
Liquid Phase Sintering



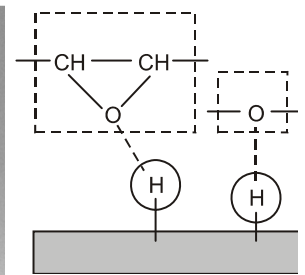
Partial Melting



Full Melting



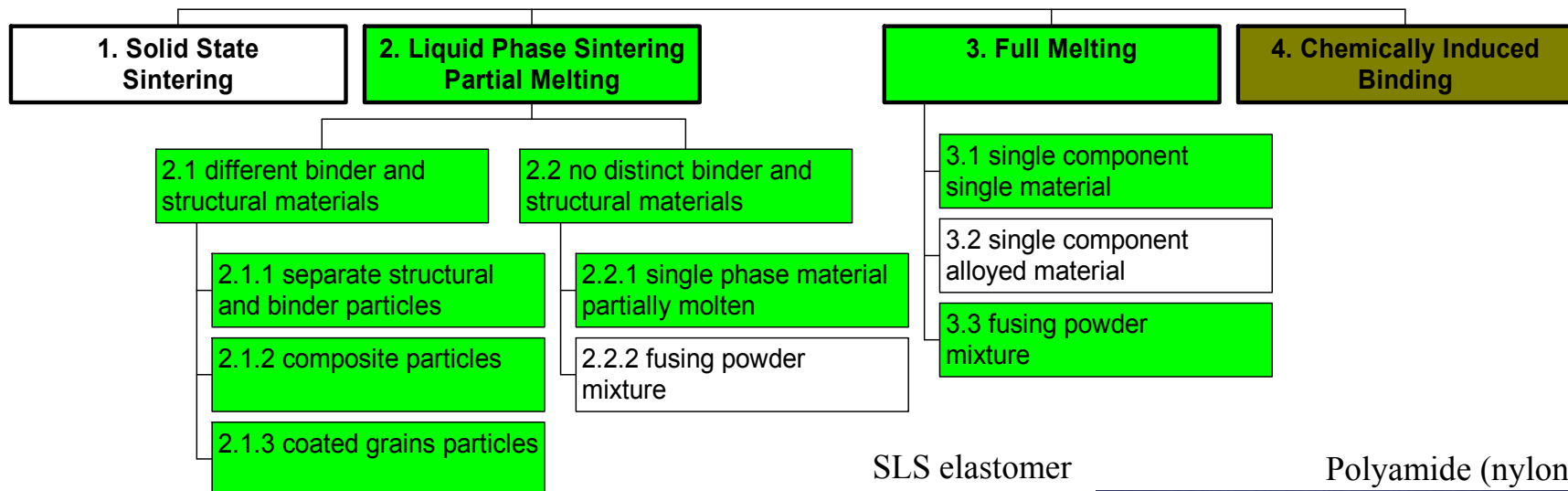
Chemical binding



Main binding mechanisms for polymers

Polymers

Binding mechanism classification



SLS elastomer



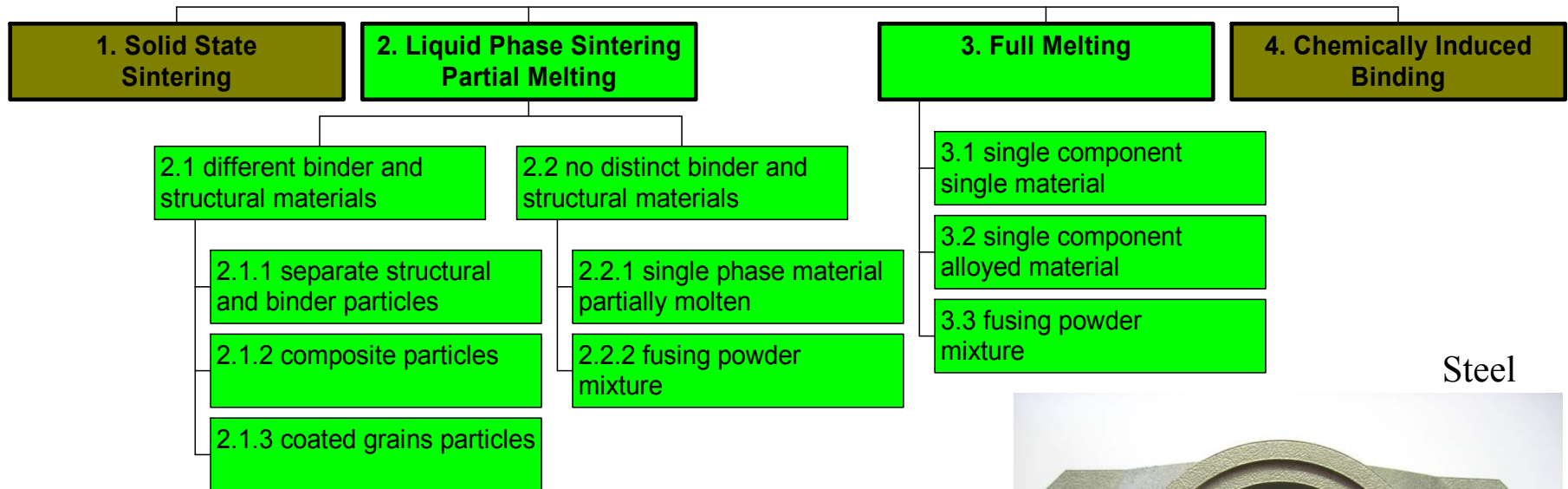
Polyamide (nylon)



Main binding mechanisms for metals

Metals

Binding mechanism classification



Titanium



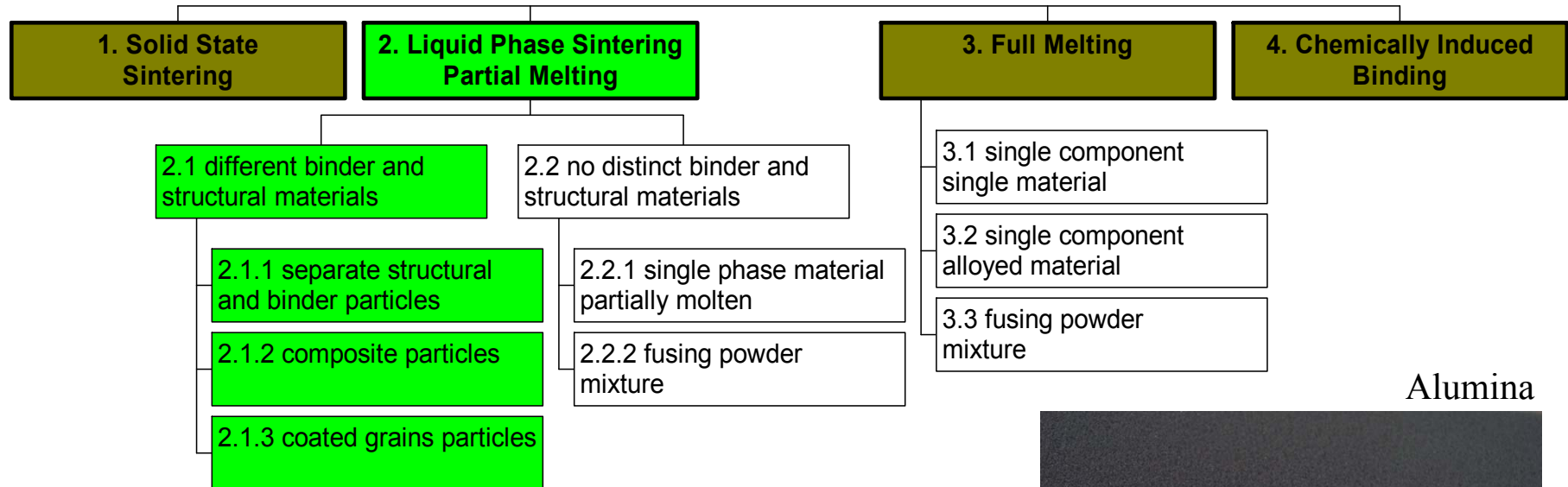
Steel



Main binding mechanisms for ceramics

Ceramics

Binding mechanism classification



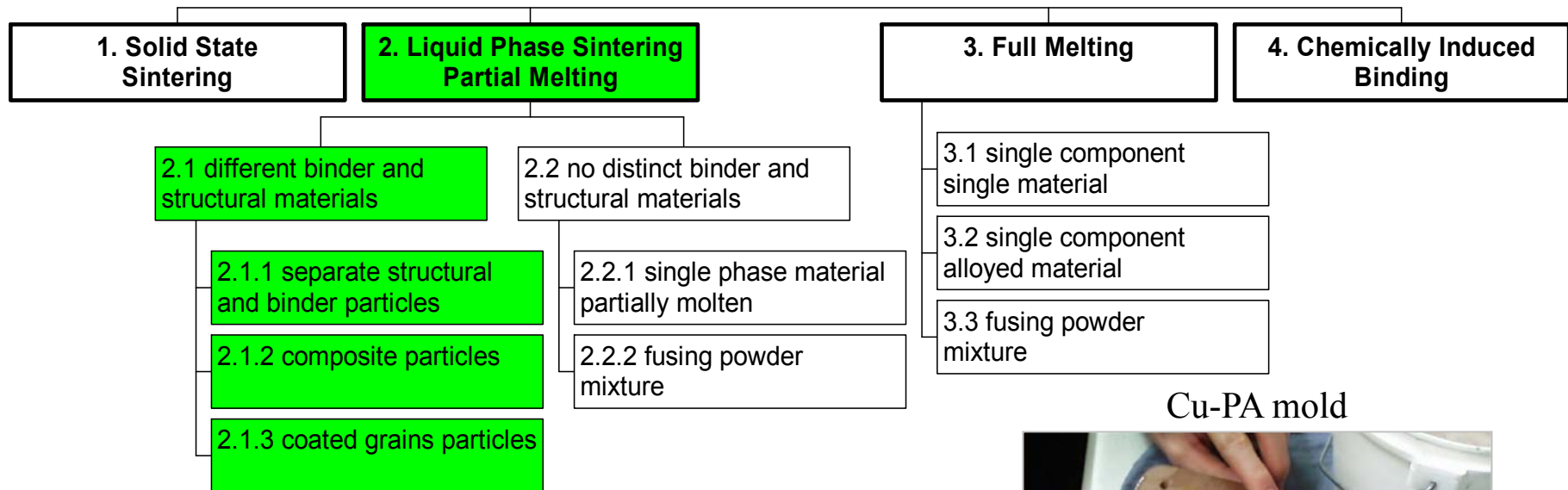
Alumina



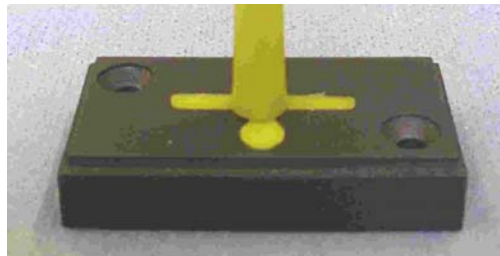
Main binding mechanisms for composites

Composites (cermets and others)

Binding mechanism classification



WC-Co (+ Cu) Cermet/HM



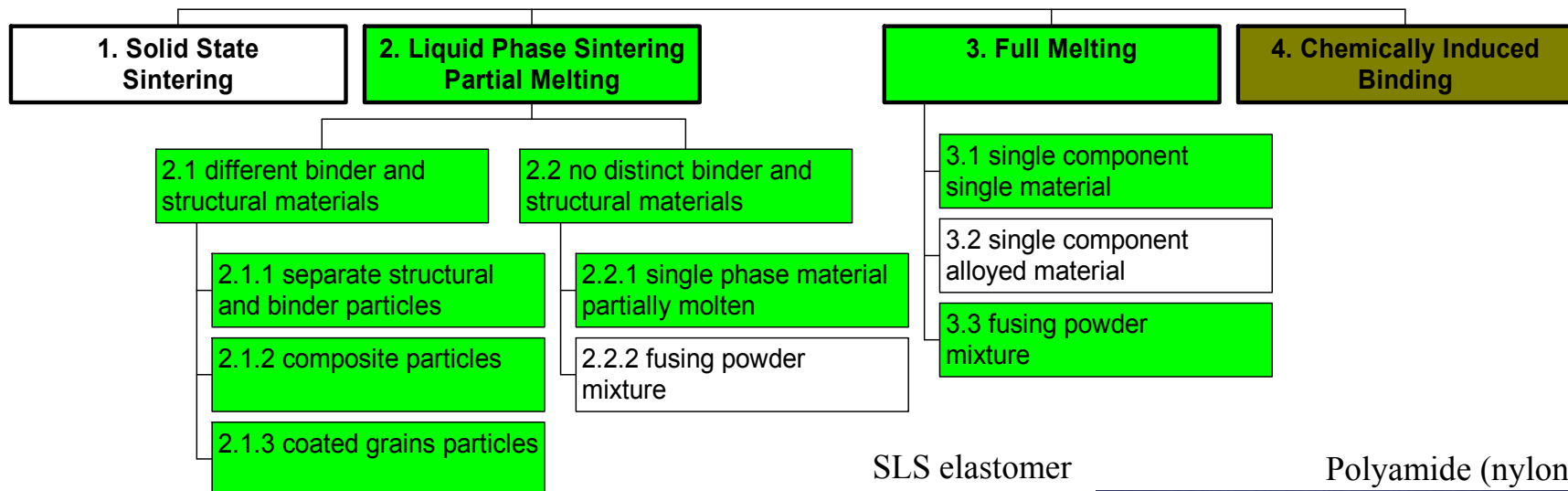
Cu-PA mold



Main binding mechanisms for polymers

Polymers

Binding mechanism classification



SLS elastomer



Polyamide (nylon)



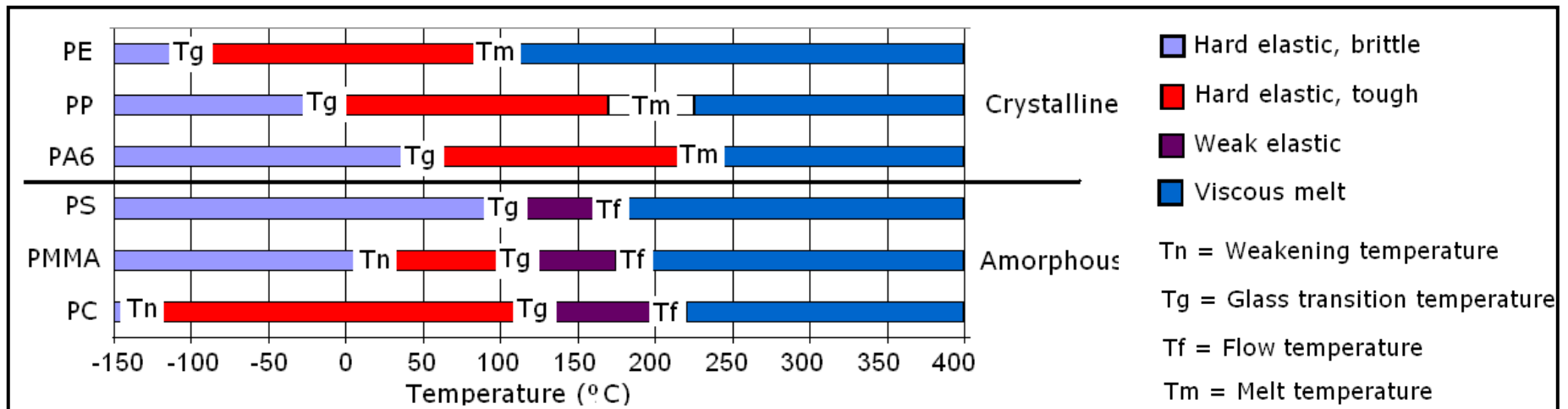
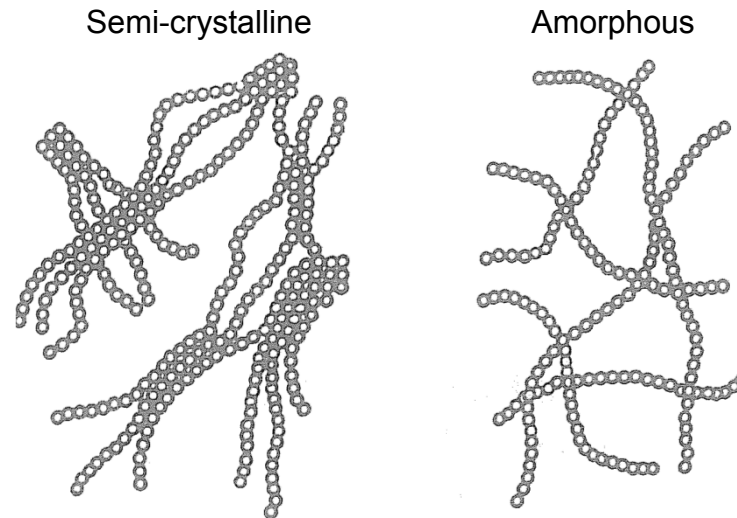
Main distinction in SLS of polymers

Mainly thermoplastics:

- (Semi-)crystalline
- Amorphous

Main SLS consolidation:

Partial or full melting



Main distinction in SLS of polymers

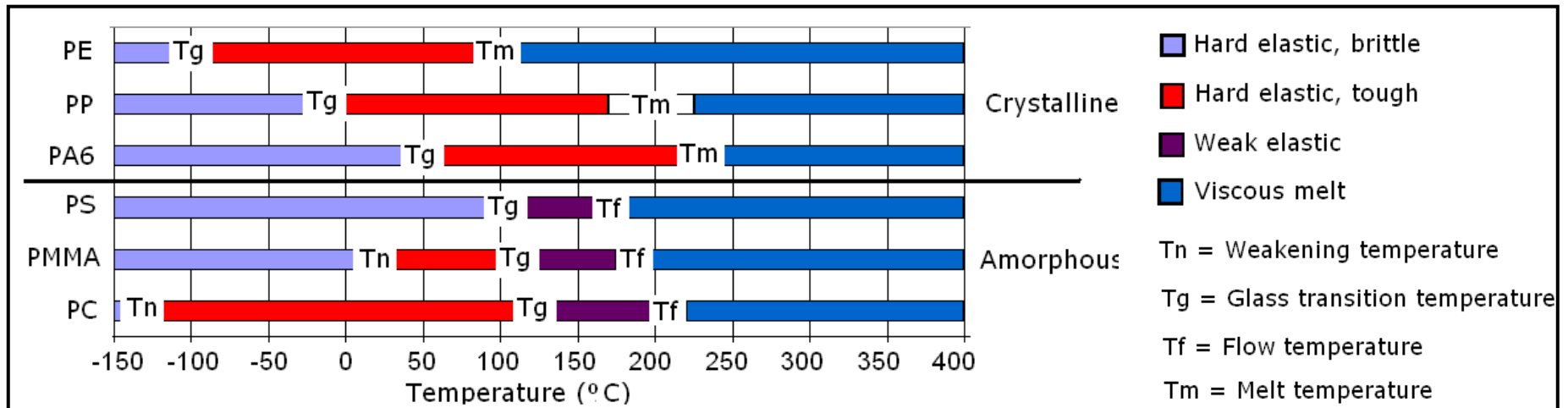
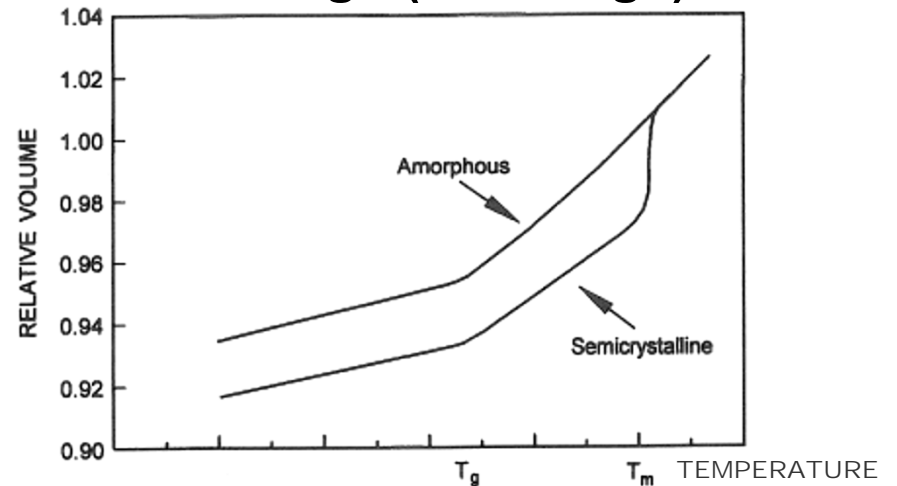
Mainly thermoplastics:

- (Semi-)crystalline
- Amorphous

Main SLS consolidation:

Partial or full melting

Volume change (shrinkage):



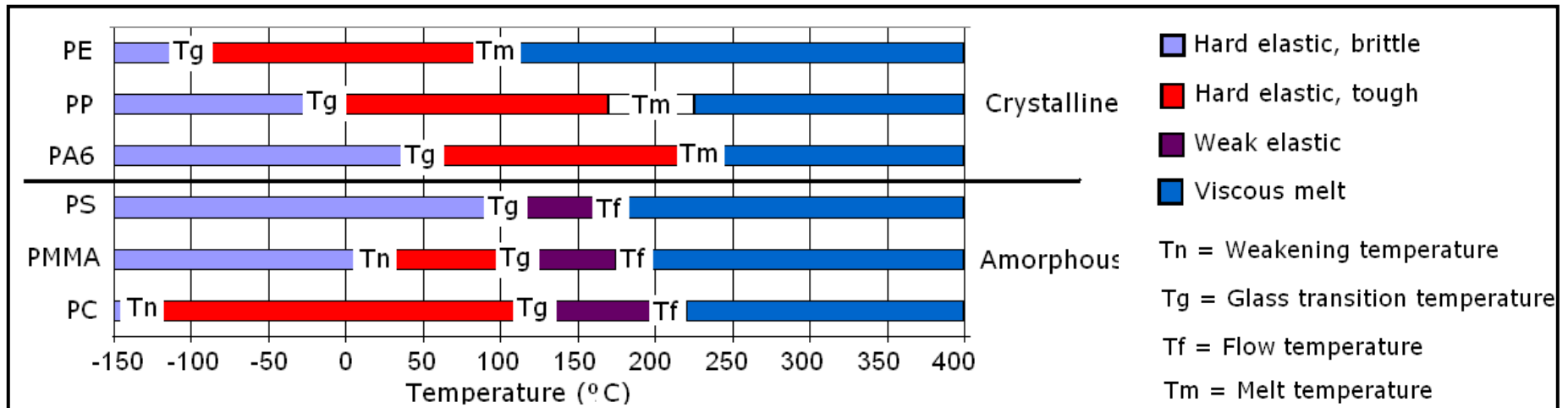
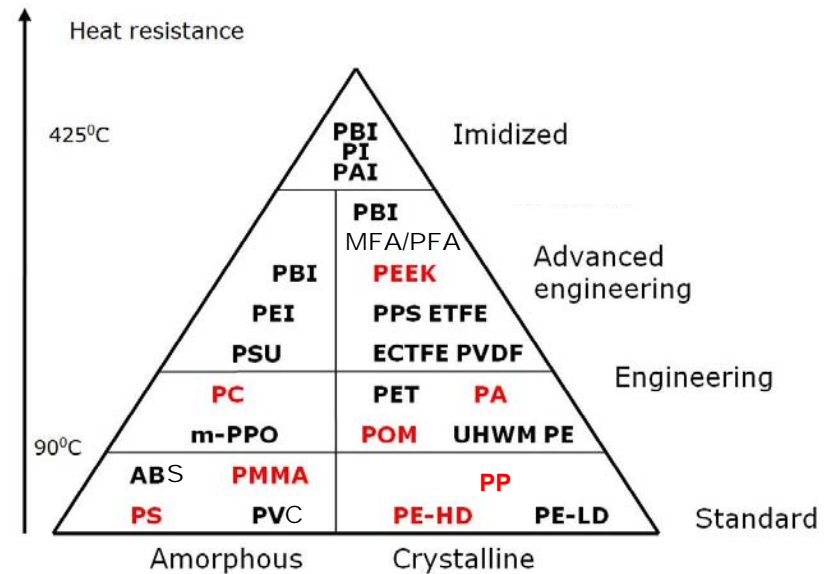
Polymers

Main consolidation:

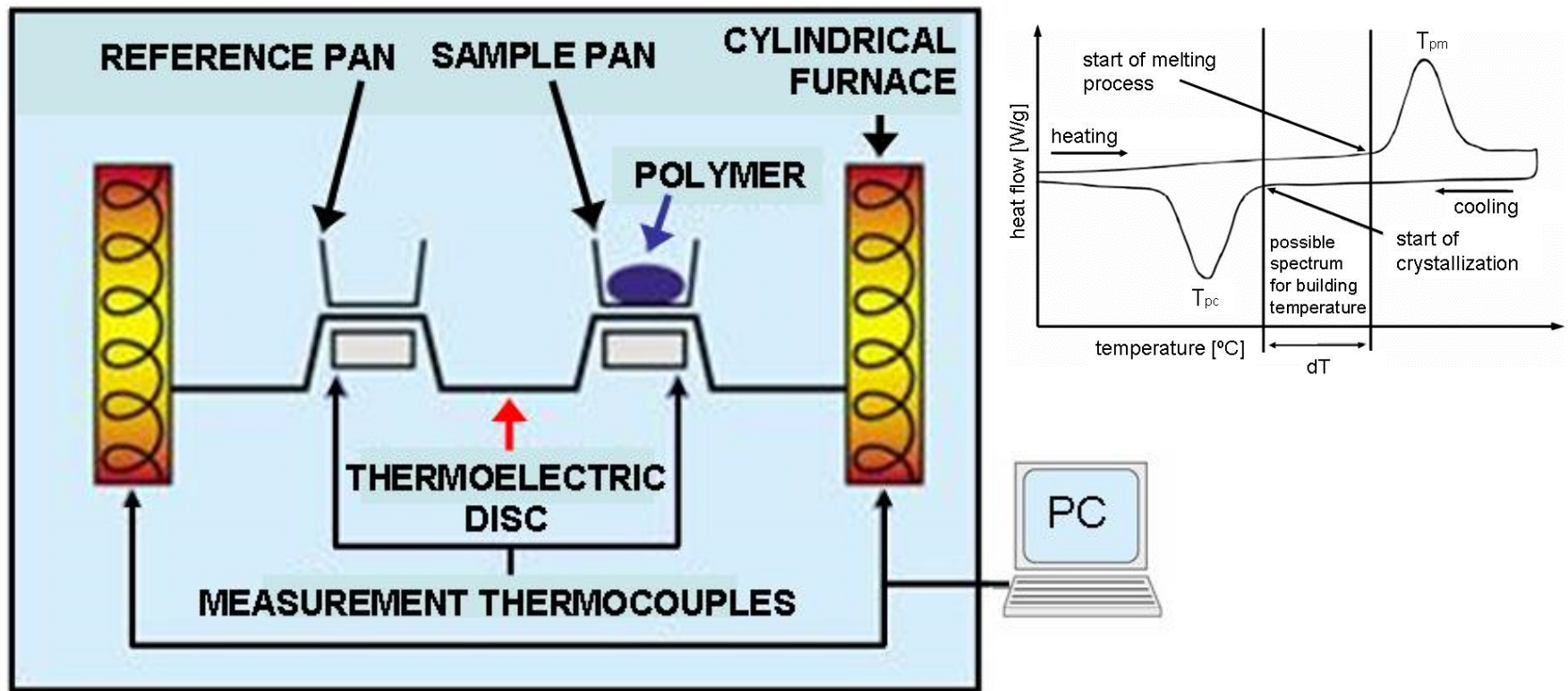
Partial or full melting

Major distinction:

- (Semi-)crystalline
- Amorphous

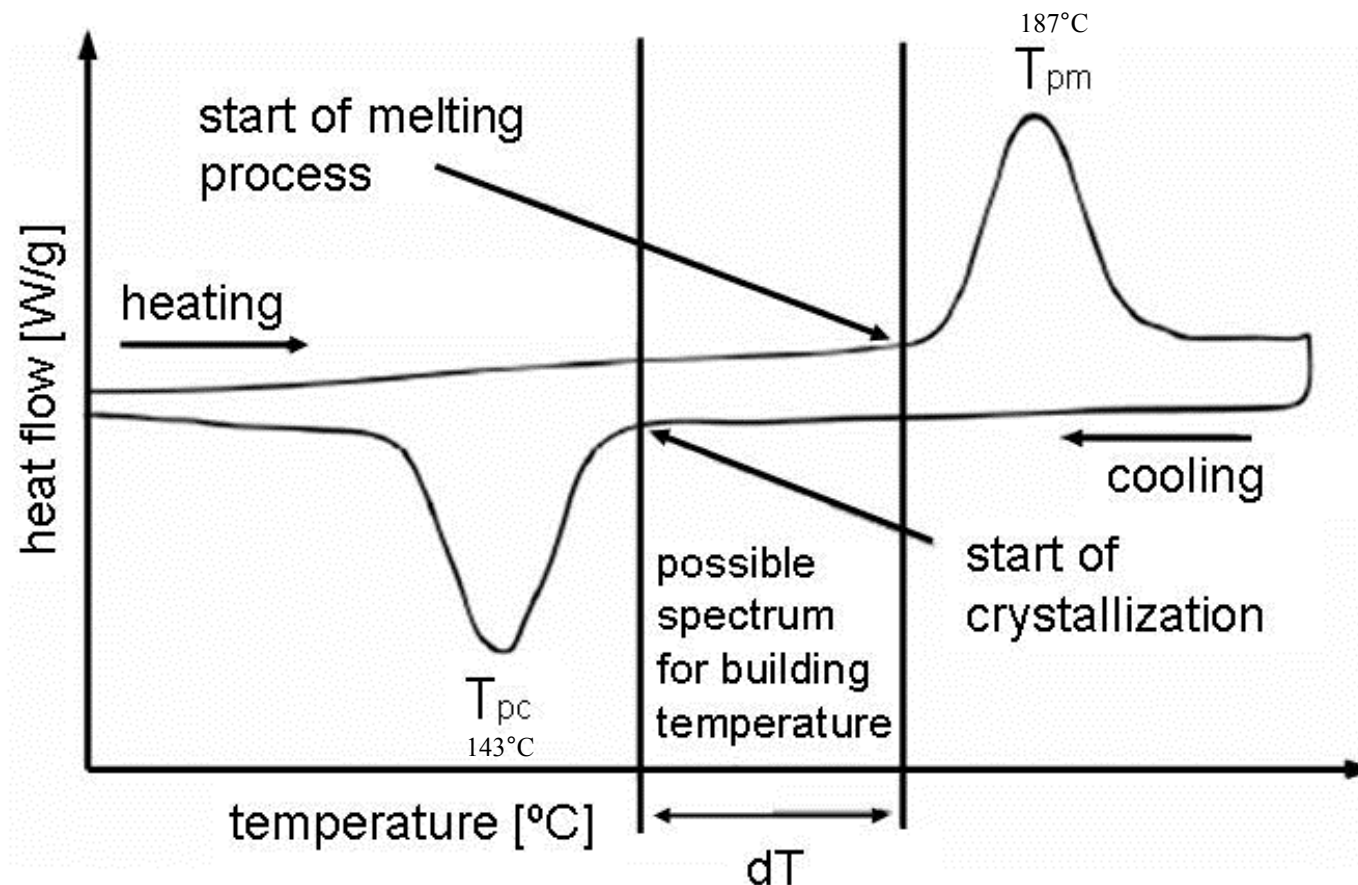


Differential Scanning Calorimetry (DSC)



DSC curve: melting & recrystallisation peaks

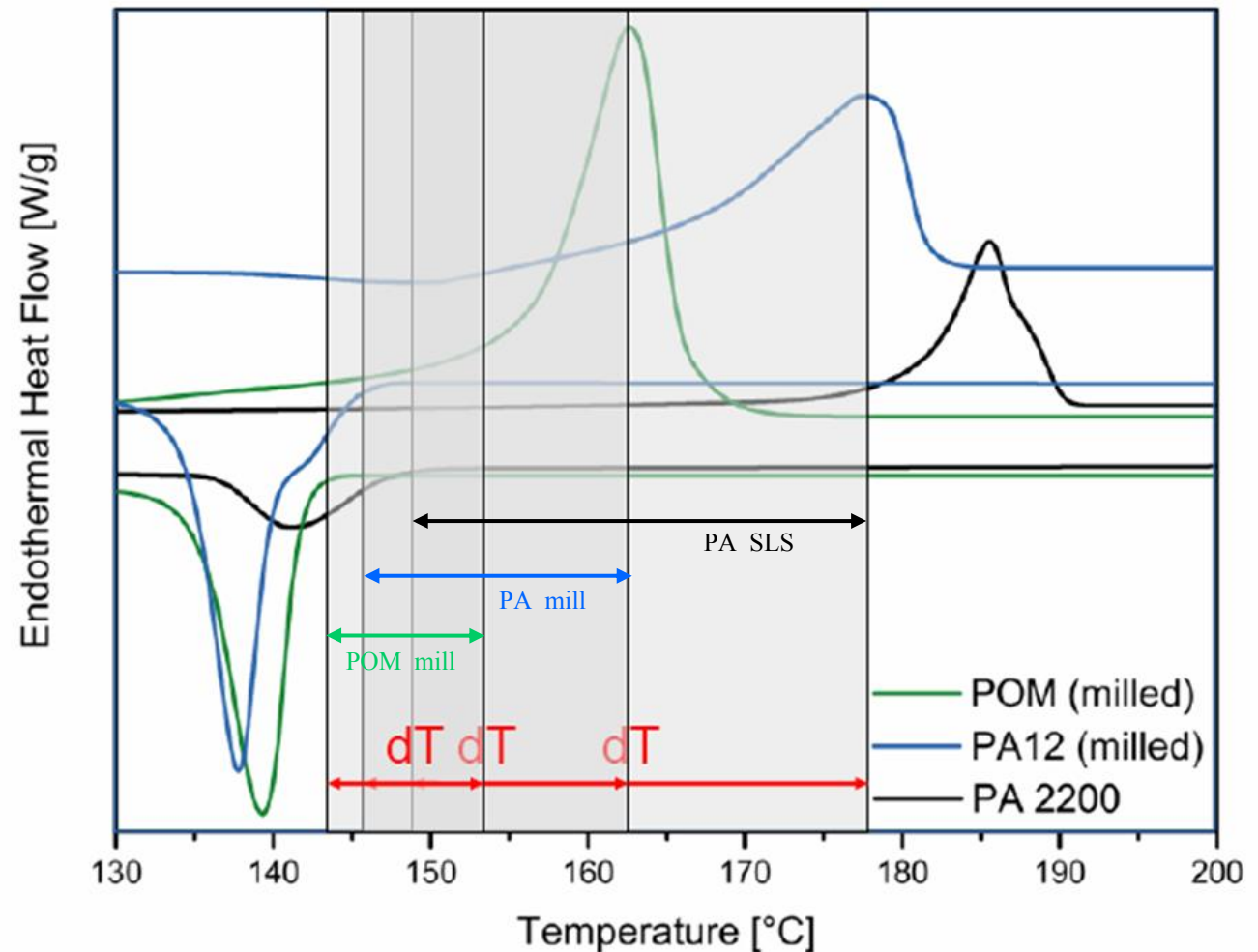
- DSC curve for PA12 (Differential Scanning Calorimetry)



Semi-crystalline polymers – DSC curves

Comparison of
DSC curves:

- PA12 for SLS
(PA 2200)
- PA12 milled
- POM milled

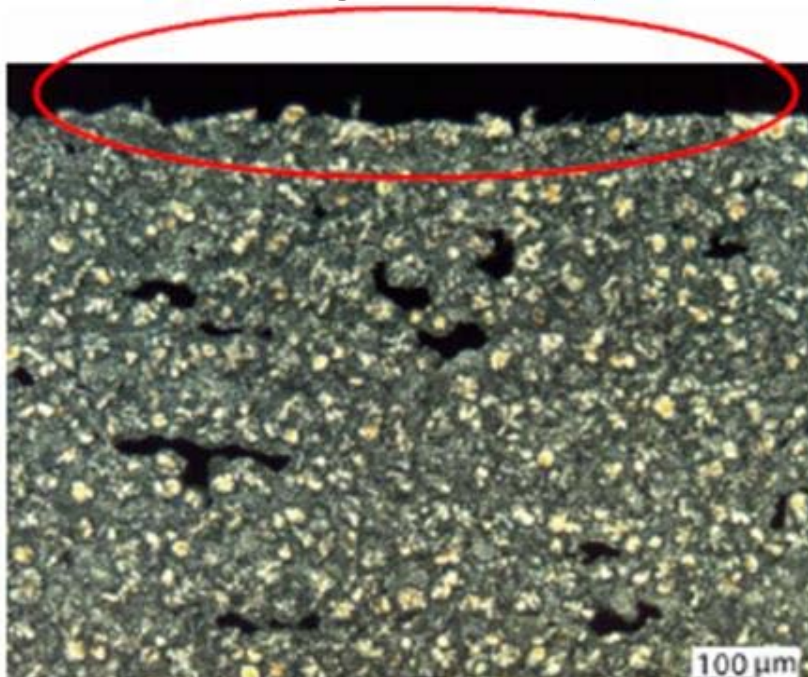


Source: University Erlangen

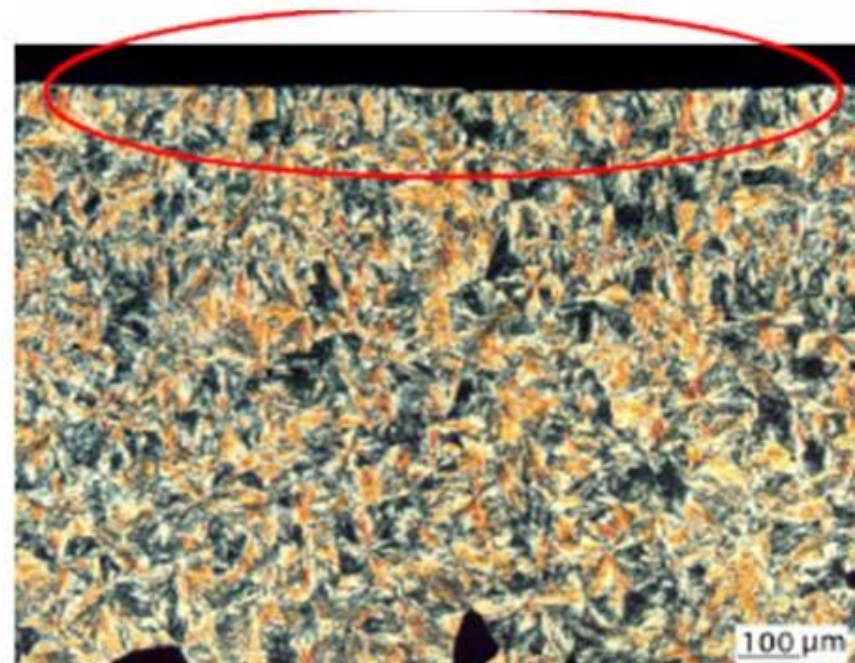
SLS of Semi-crystalline plastics (e.g. POM)

Transmission light microscopy images of microtome sections

PA (rough surface)



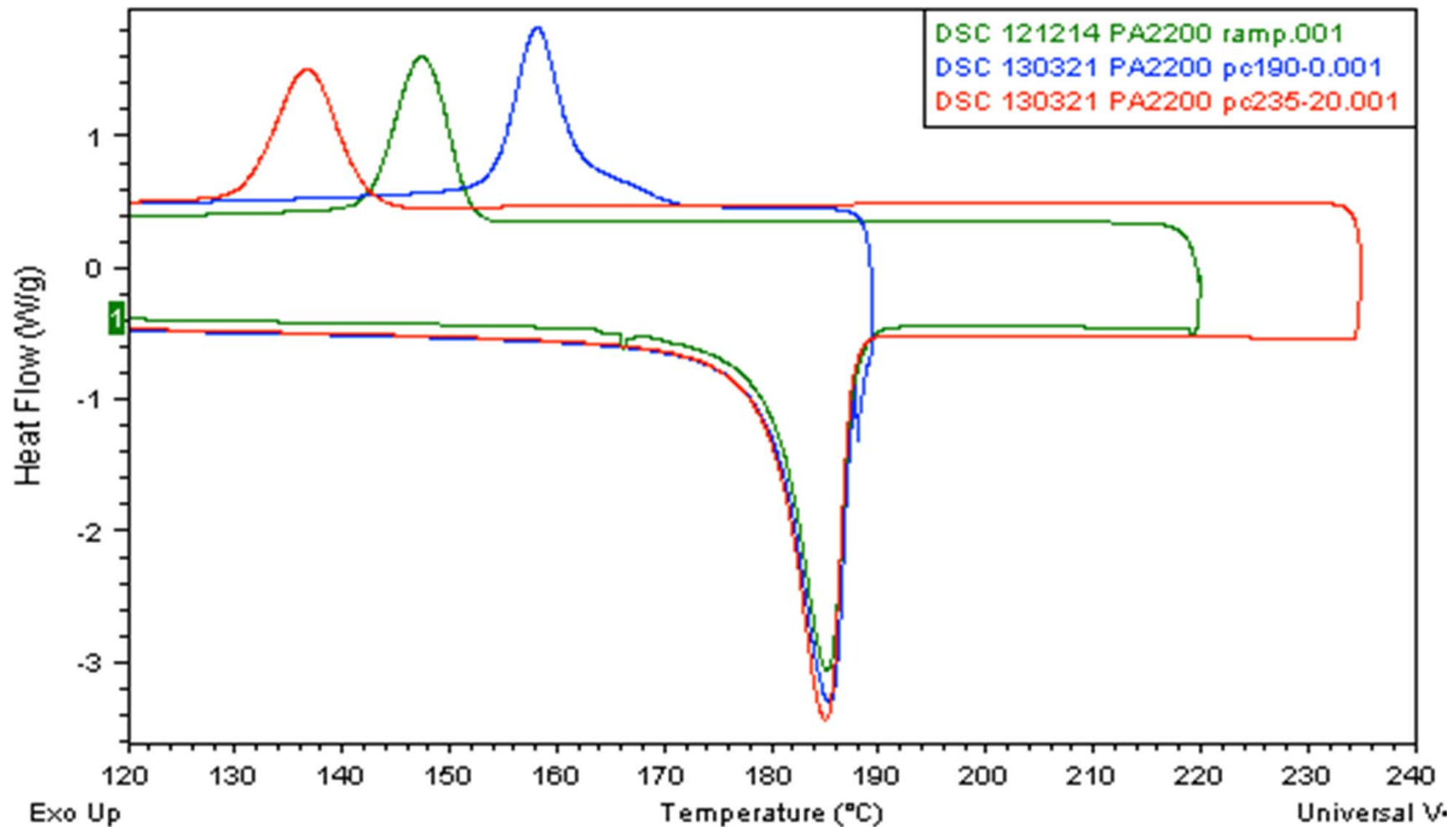
POM (smooth surface)








Source: University Erlangen

DSC curve: melting & recrystallisation peaks

- DSC curve for PA12 (Differential Scanning Calorimetry)

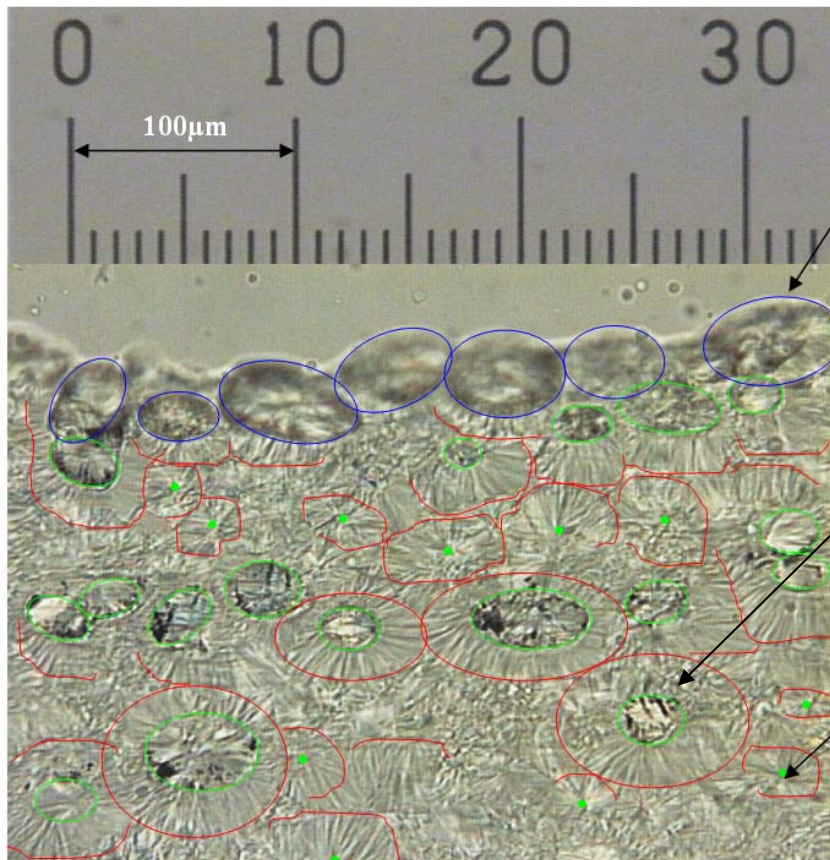


Polymers: types and applications

| Polymer powder material | Application field | Example | Main properties |
|---|--|---|---|
| Semi Crystalline Polymers e.g. PA-12 | (Semi-)Rigid polymer parts |  | Long term useable |
| Amorphous Polymer e.g. PS | Investment Casting Lost patterns |  | Accurate Partially porous |
| Sacrificial Polymers used as binder e.g. PMMA | Metal or Ceramic Parts |  | Thermally degradable amorphous polymers |
| Filled Semi Crystalline Polymers e.g. PA-GF, PA-Al, PA-Cu | Parts with special properties |  | Long term useable Can withstand high loads |
| Elastomeric Polymers e.g. Polyester | Elastic parts |  | Long term useable |
| Polymer-Polymer Blends | Emerging Extreme Applications | | |
| Thermo-setting Polymers e.g. epoxy resin | | | |

Polymers 1: Semi-crystalline (e.g. PA12)

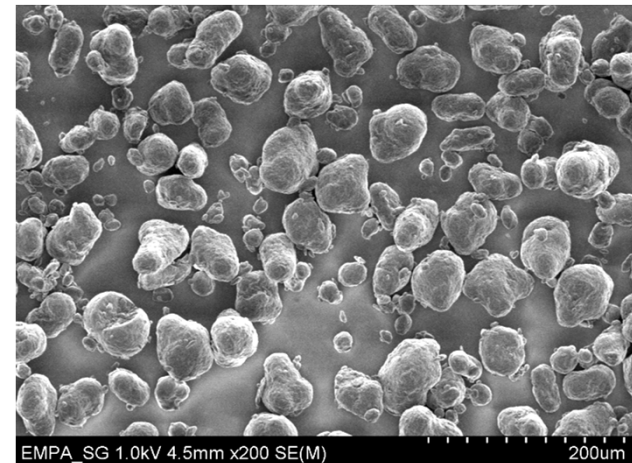
Partial or full melting



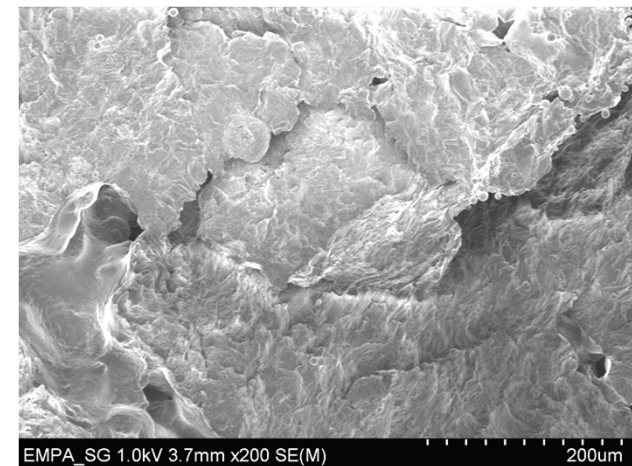
Un-molten
complete
particle
stuck to
edge

Un-molten
particle
core

Fully molten
particle (no
core)



Loose un-sintered PA-12 powder



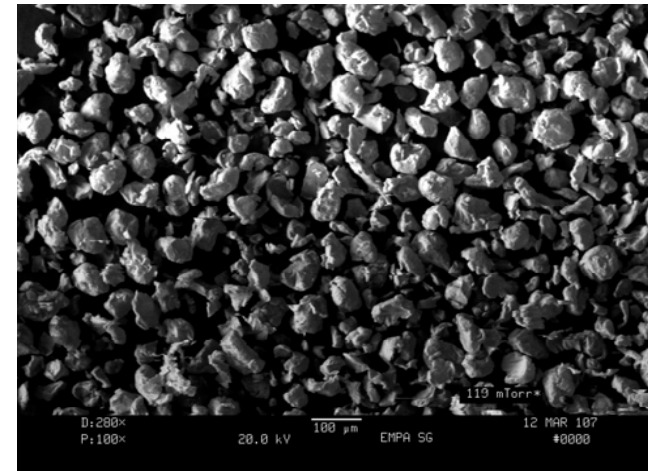
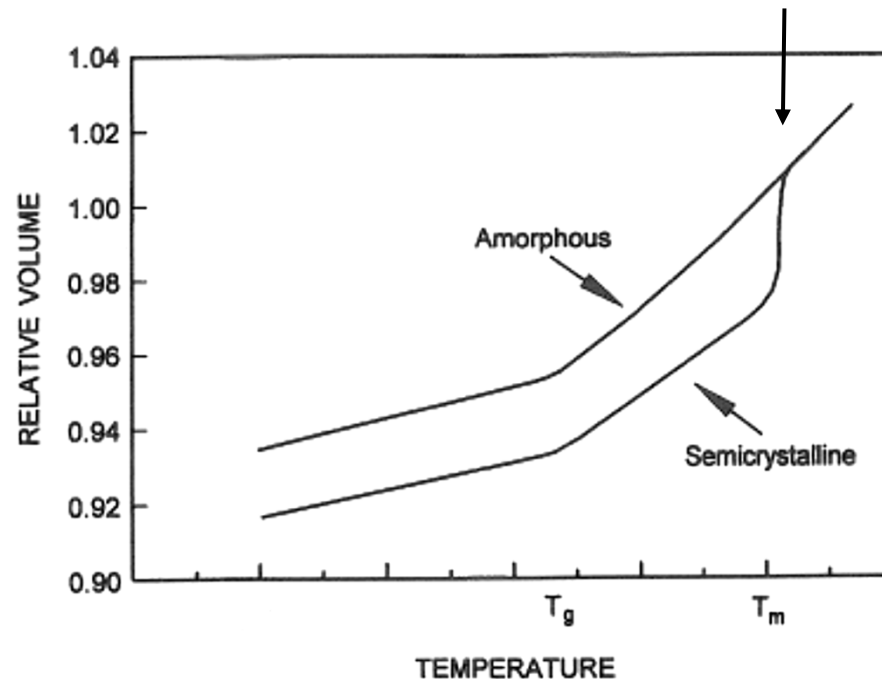
Tensile break surface showing some air voids

Polymers 2: Amorphous (e.g. PS)

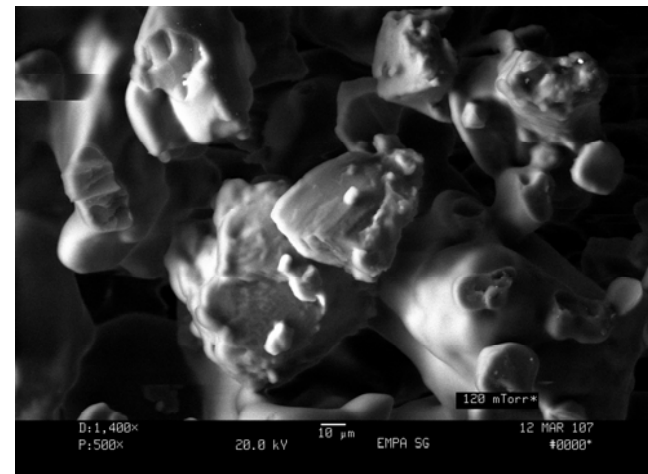
Partial melting

Low strength: only partial consolidation

Better accuracy: no sudden shrink (jump) when solidifying (crystalline shrink at T_m)



Loose un-sintered PS powder



Tensile break surface showing some air voids

Polymers 3: Debindable polymers (e.g. amorph. PMMA)

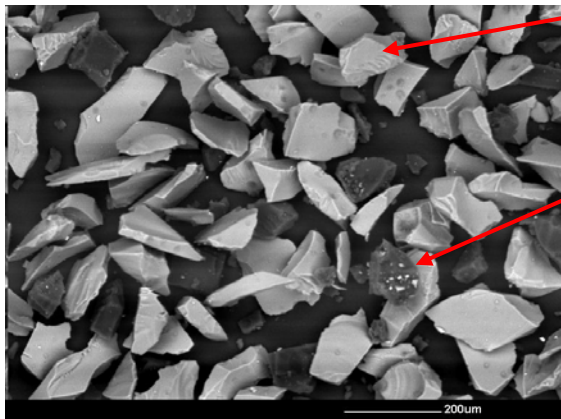
Partial melting / LPS

- Thermal debinding (depolymerisation) should occur in furnace at 350°-450°C, while not occurring during SLS
- Suited polymers: PMMA or MMA-BMA co-polymers, PA, PP
- May involve some cross-linkers (thermosetters)

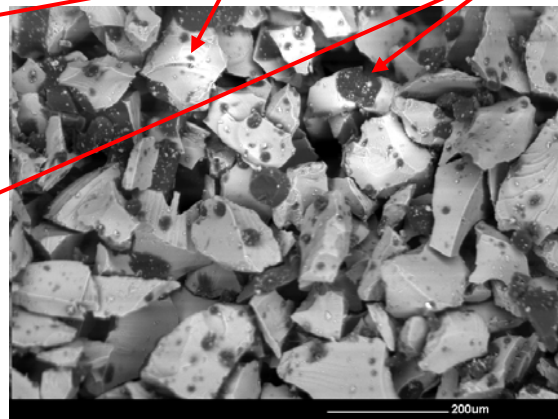
Examples:

- Steel (RapidSteel, Laserform)
- AW glass ceramics (Dalgarno)

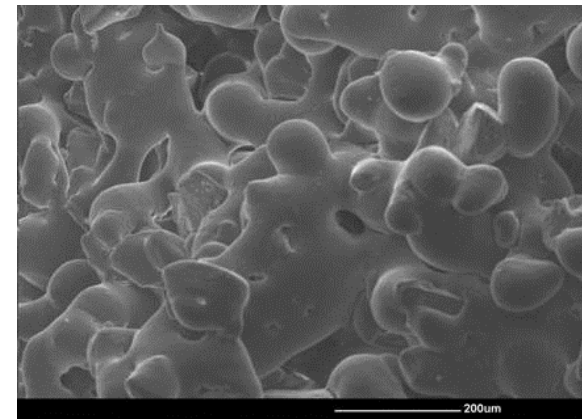
AW glass ceramic + MMA-BMA



Powder mixture



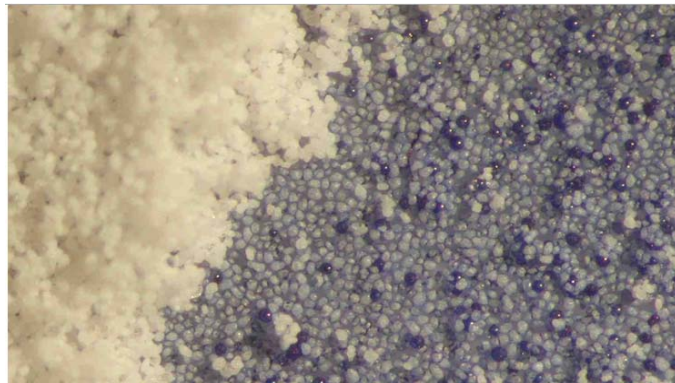
Green part (i.e. after SLS)



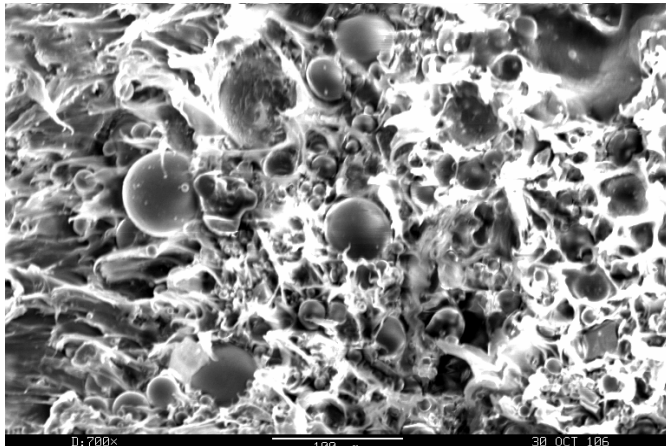
Brown part (i.e. after debinding & firing)

Polymers 4: Reinforced polymers (e.g. PA-GF)

Partial melting / LPS

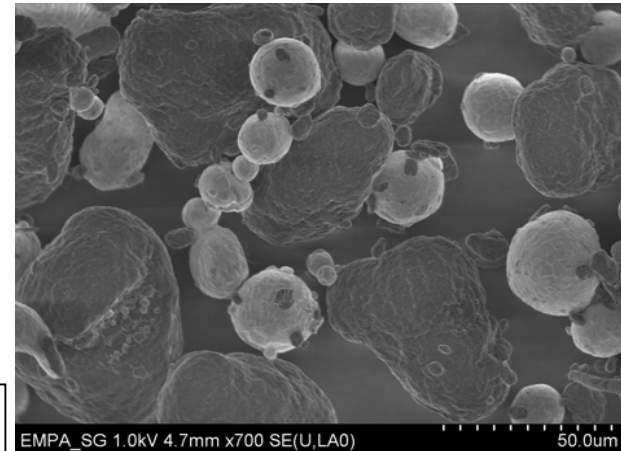


Loose un-sintered **PA-Glass** powder

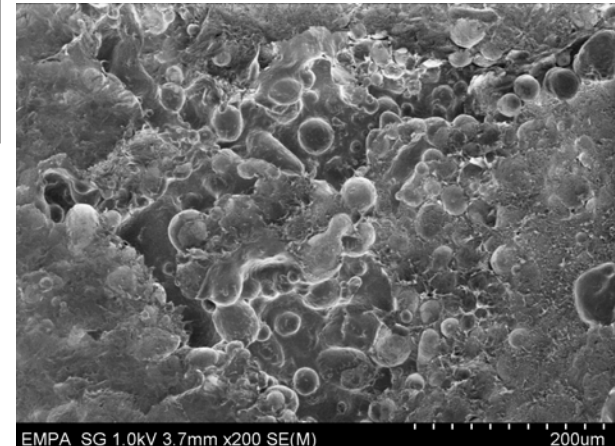


Tensile break surface showing some air voids

Polya
mide
+
Glass
beads



Loose un-sintered **PA-Al** powder (30% Al)



Tensile break surface showing some air voids

Polya
mide
+ Alu
beads

Polymers 4: Reinforced polymers (e.g. PA-Cu)

Partial melting / LPS

Injection mold made from
Cu-filled Polyamide and
Polypropylene molded parts
(injected at 2.76 MPa and 230°C)

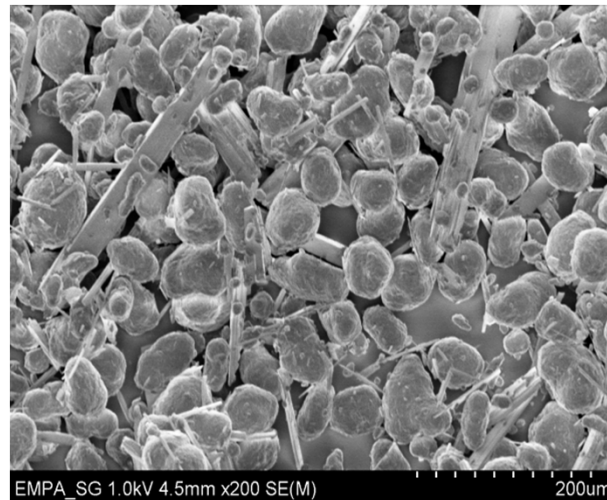


Polymers 4: Reinforced polymers (long fillers)

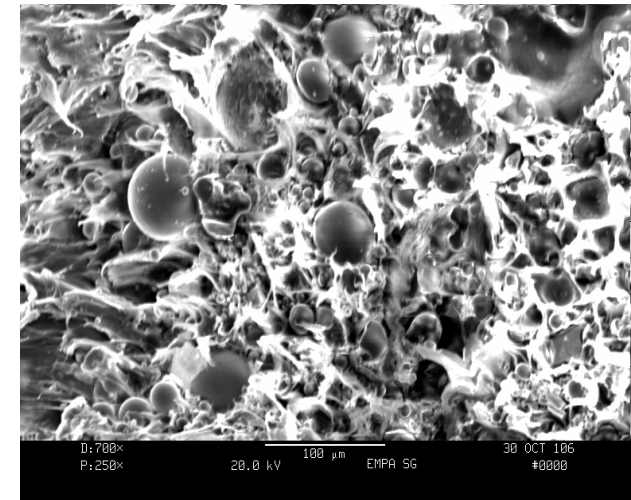
Polyamide (nylon) with elongated filler

Source: FHSG - Valspar

Elongated fibers (new)



Spherical glass particles (old)

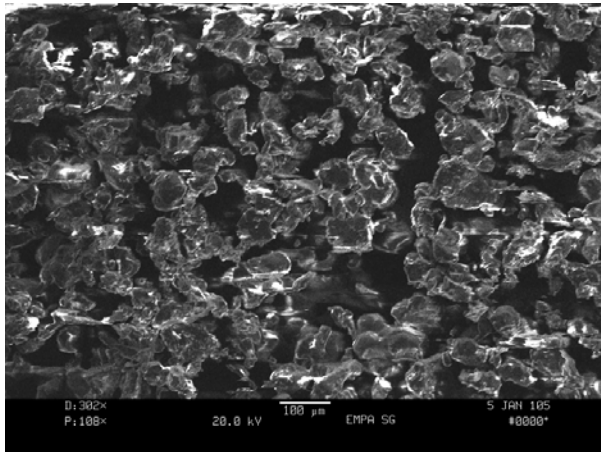


| Property | DF-M* | 3D PA | 3D GF | 3D AF |
|------------------------|-------|--------|-------|-------|
| Tensile strength (MPa) | 49.00 | 43.00 | 27.00 | 35.00 |
| Tensile elongation % | 5.00% | 14.00% | 1.50% | 1.50% |
| Tensile Modulus (MPa) | 5376 | 1586 | 4068 | 3960 |
| HDT [1.82 Mpa] | 165 | 95 | 134 | 137 |

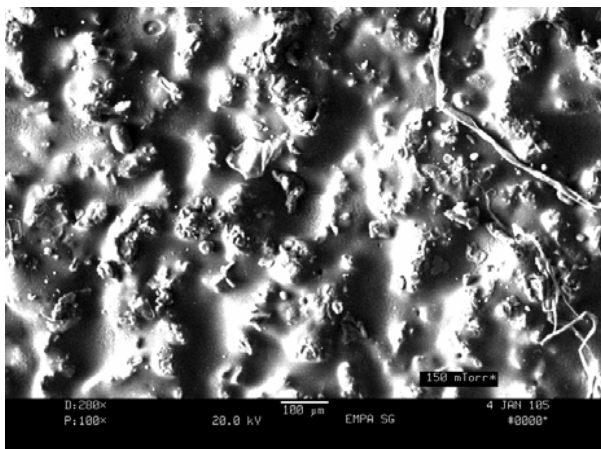
Polymers 5: Elastomeric polymer (e.g. polyester)

Partial melting

Polyester-based elastomer



Green part
(i.e. after SLS and
without infiltration)



Part after
infiltration with
polyurethane



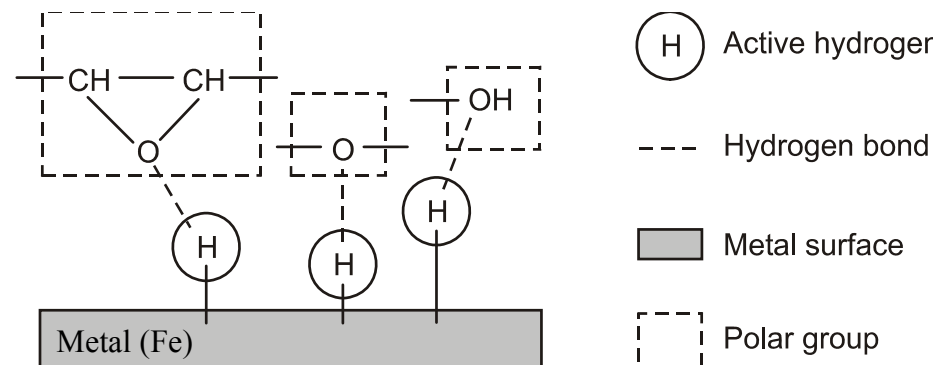
Polymers 6: Others (polymer blends, thermosets)

- **Polymeric blends: Partial melting**

- Multiphase materials → tuned microstructure!
- Example 1: mixed PA – HDPE (80/20, 50/50, 20/80 wt%)
- Example 2: polymer 1 coated with low melting (thermoplastic) polymer 2 ($T_m < 70^\circ\text{C}$, e.g. polyvinyl acetal, heptadecanoic acid,...)

- **Thermosetting materials: Chemical binding**

- E.g. mixture epoxy-iron
- Hydrogen bonds between polar O^- from resin and H^+ on iron surface



Polymers: Conclusion

- **Different classes of polymers covered**

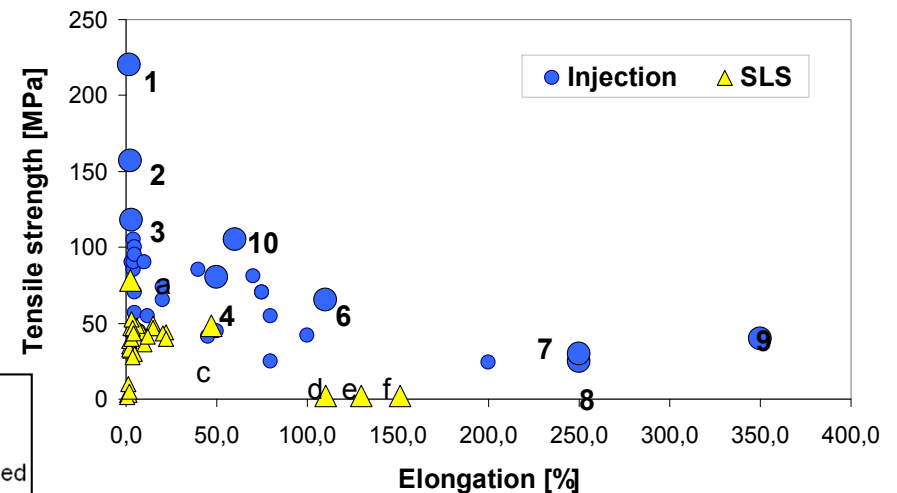
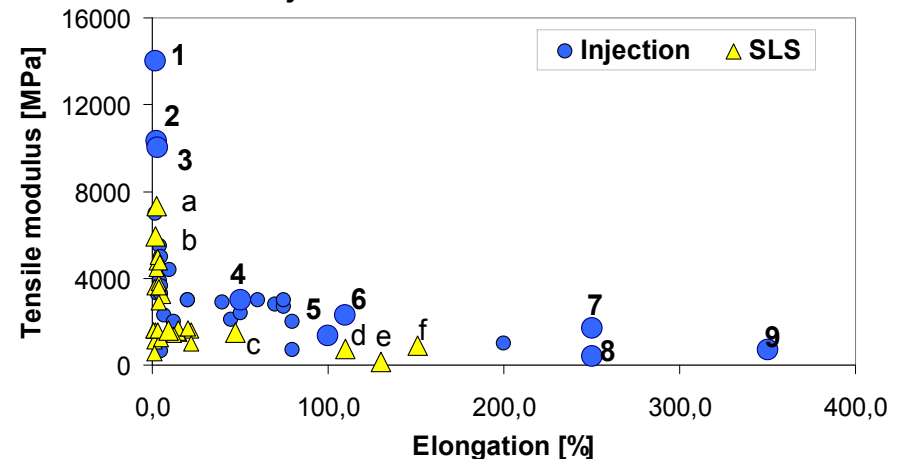
- Semi-crystalline
- Amorphous
- Debindable
- Filled polymers
- Elastomeric
- Polymer-polymer blends
- Thermosetting

- **Scope of applicable polymers still limited**

- Still mainly PA (plain or filled)

- **Good, but no extreme properties**

Injection vs. SLS Materials

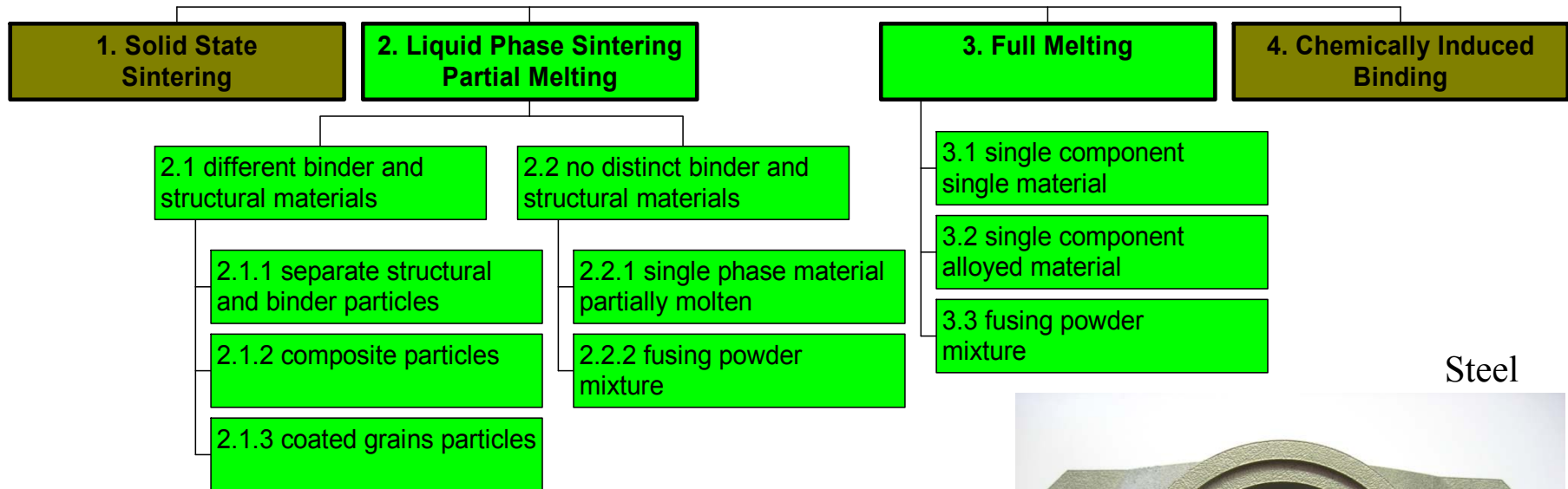


| INJECTION | | SLS | |
|-----------|---------------|-----|---------------------------|
| 1 | Grivory HTV | 6 | PC |
| 2 | Peek c | 7 | ECTFE a |
| 3 | Peek b | 8 | PTFE |
| 4 | PA 6 | 9 | PEUHMW |
| 5 | PP a | 10 | PEI |
| a | Windform PROB | d | DuraForm Flex |
| b | DuraForm GF | e | SOMOS 201 |
| c | DuraForm Ex | f | DuraForm Flex infiltrated |

Metals

Metals

Binding mechanism classification



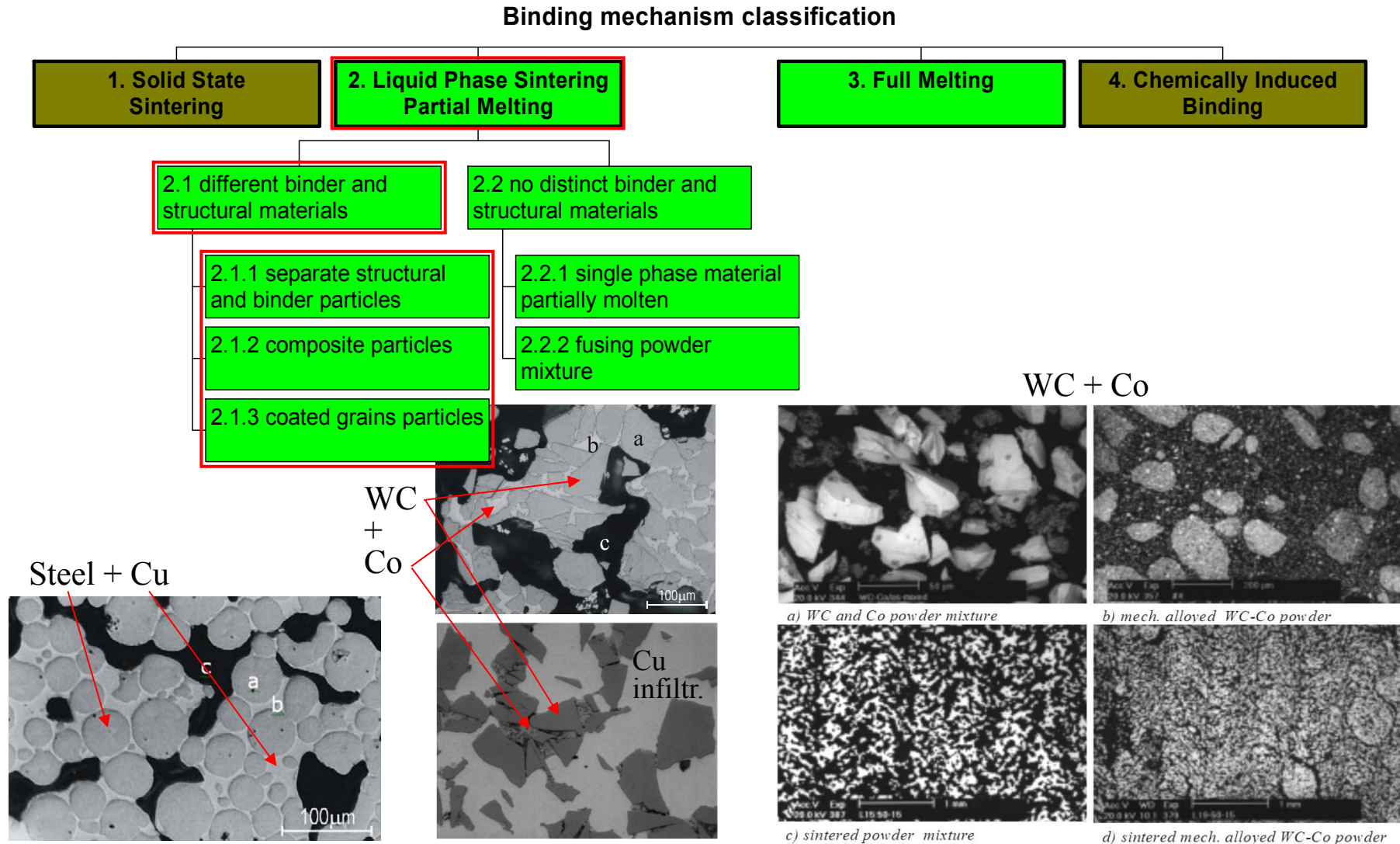
Titanium



Steel

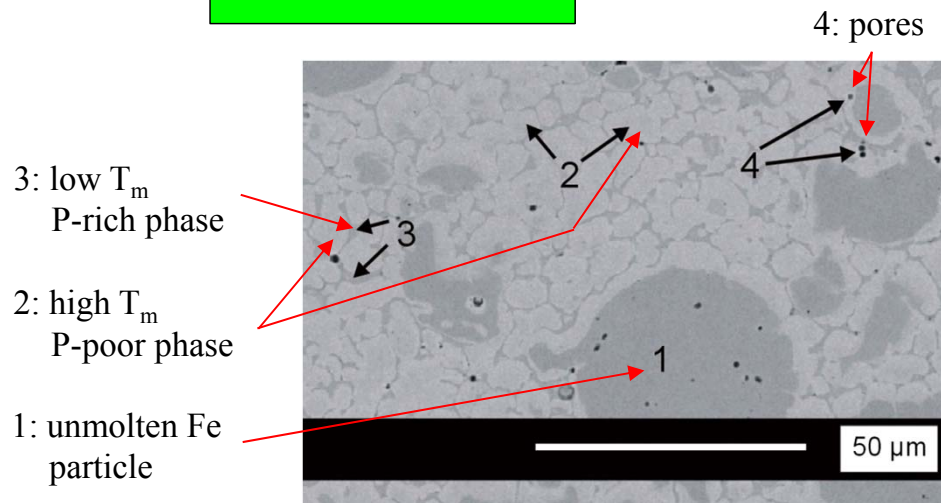
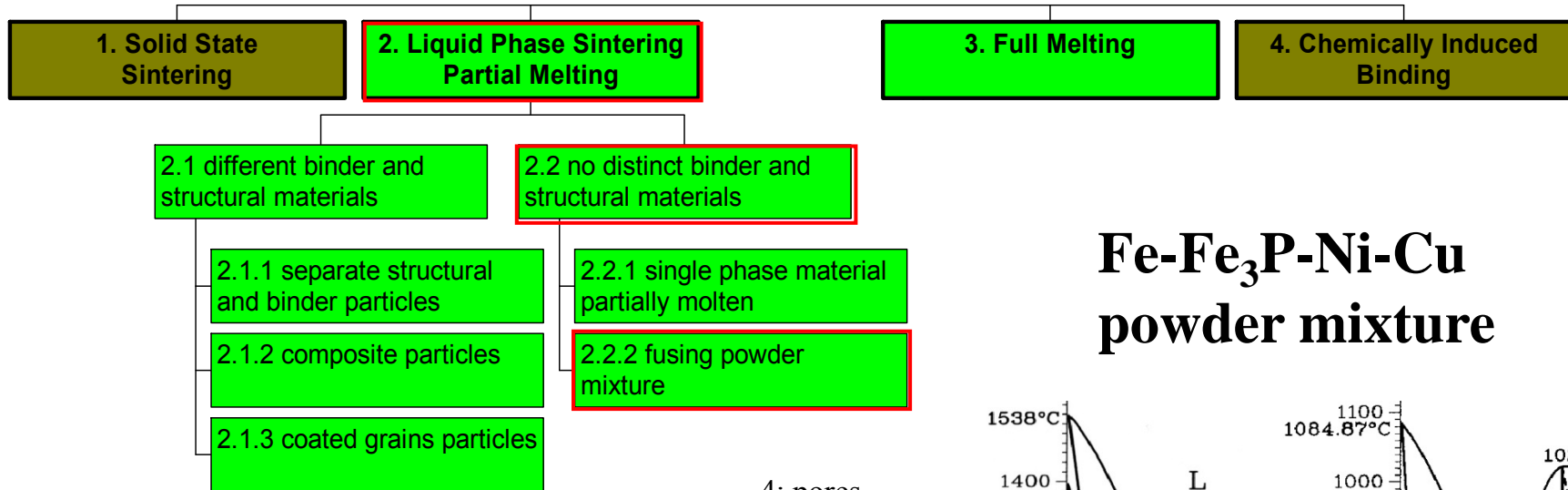


Metals 1: Liquid Phase Sintering (different materials)

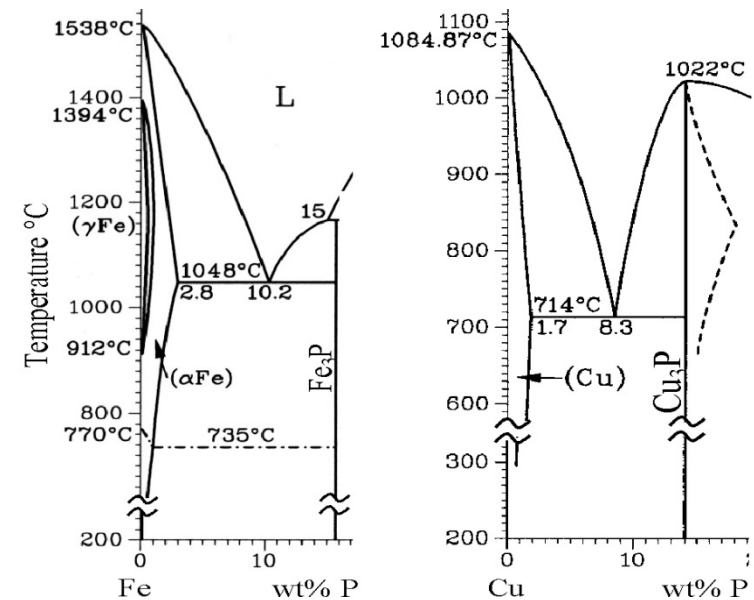


Metals 2: LPS / Partial melting (no distinct materials)

Binding mechanism classification

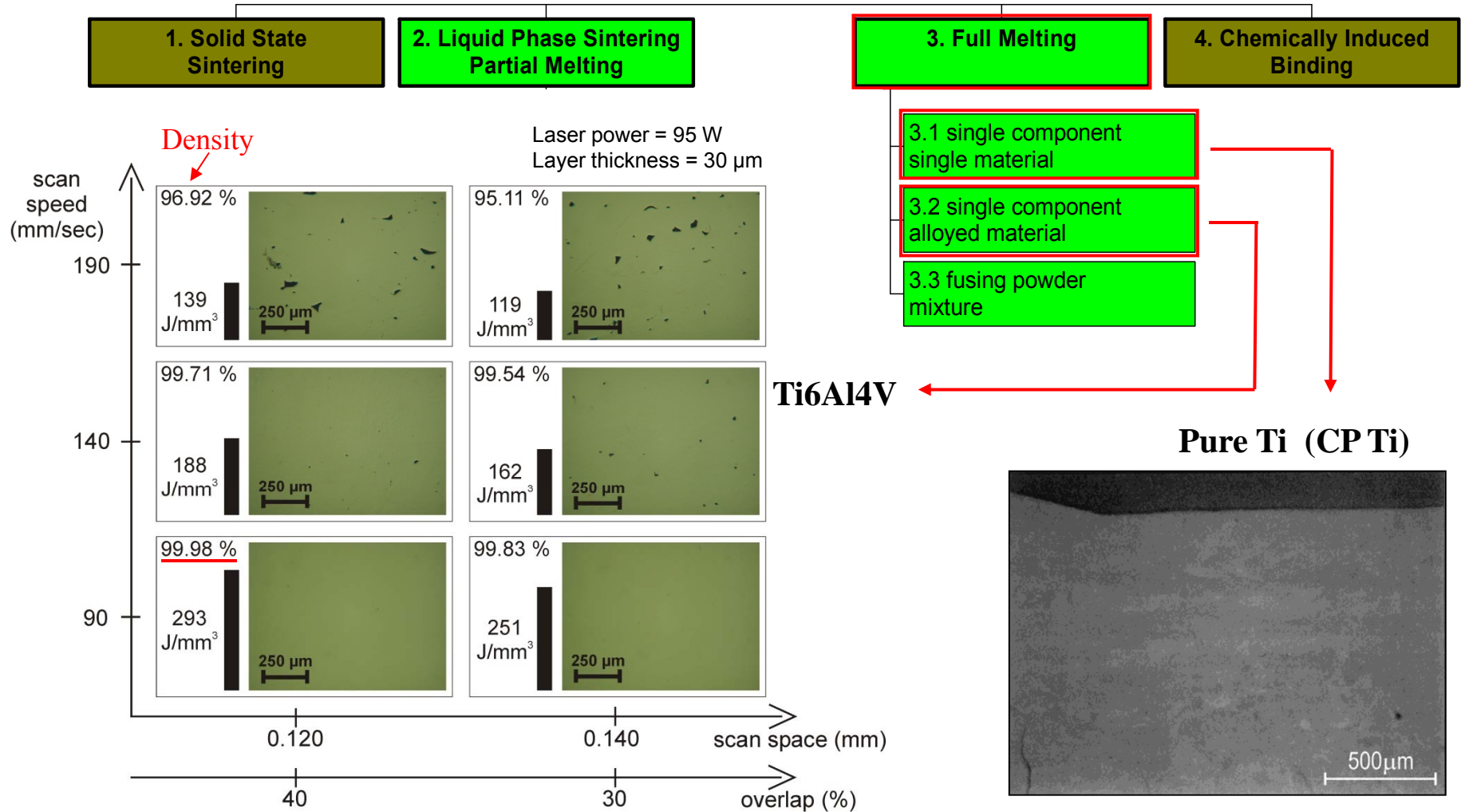


Fe-Fe₃P-Ni-Cu powder mixture



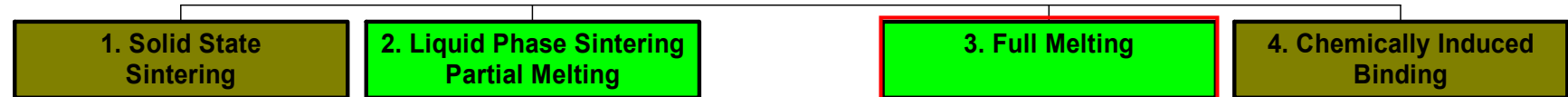
Metals 3: Full Melting (e.g. Titanium)

Binding mechanism classification



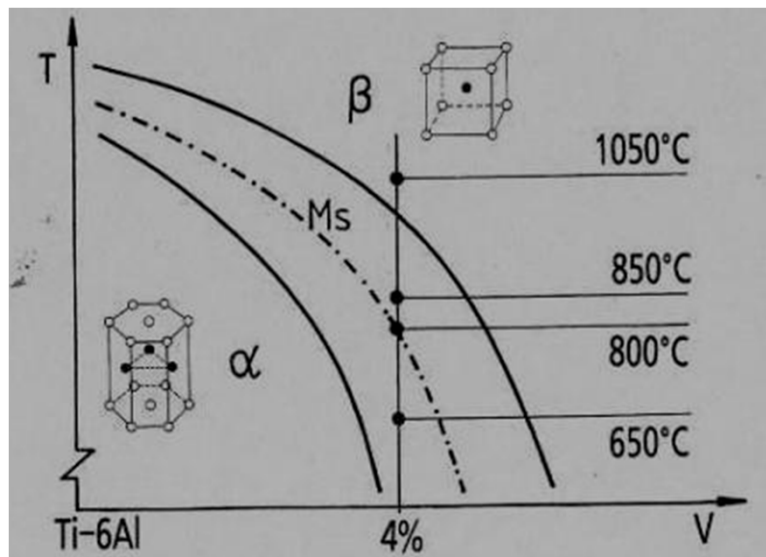
Metals 3: Full Melting (e.g. Titanium)

Binding mechanism classification



- 3.1 single component single material
- 3.2 single component alloyed material
- 3.3 fusing powder mixture

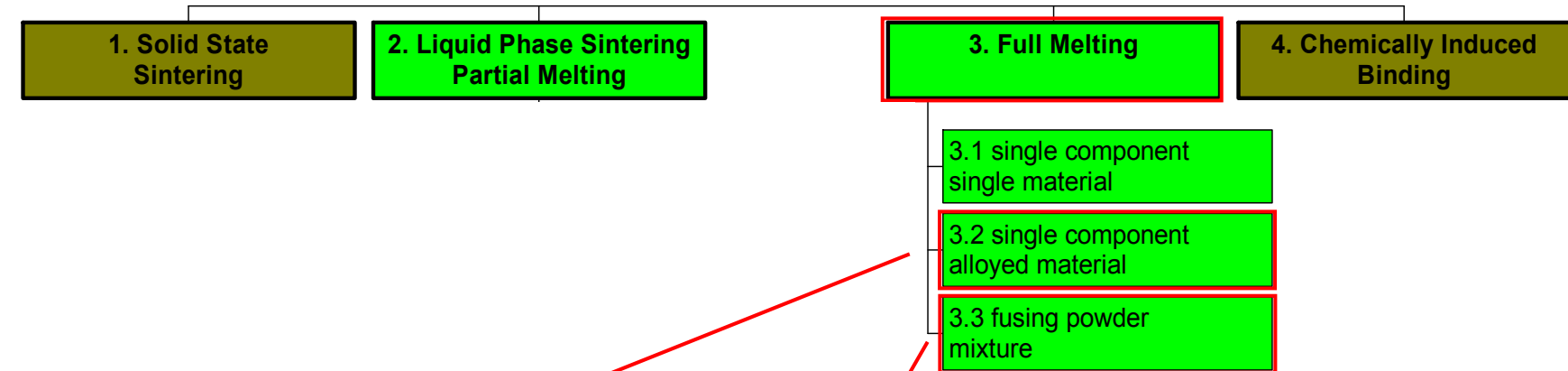
Ti6Al4V



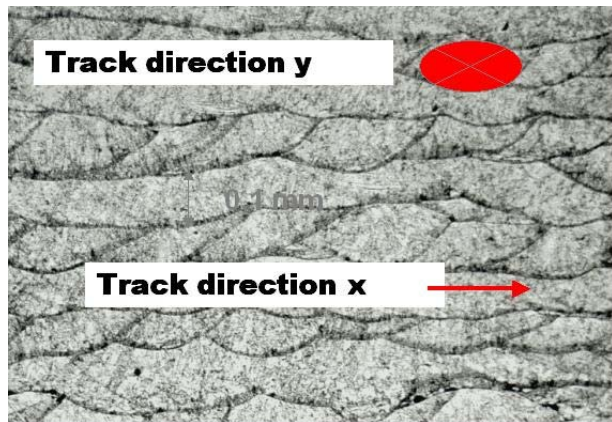
| Ti6AlV4 | SLM | | Bulk annealed |
|------------------------------|------|---|---------------|
| Density [kg/m ³] | 4415 | ≈ | 4430 |
| Hardness [Vickers] | 405 | > | 350 |
| Yields strength [MPa] | 1125 | > | 1035 |
| UTS [MPa] | 1250 | > | 1035 |
| Elongation [%] | 6 | < | 11 |
| E modulus [GPa] | 94 | < | 114 |

Metals 3: Full Melting (e.g. Fe alloys)

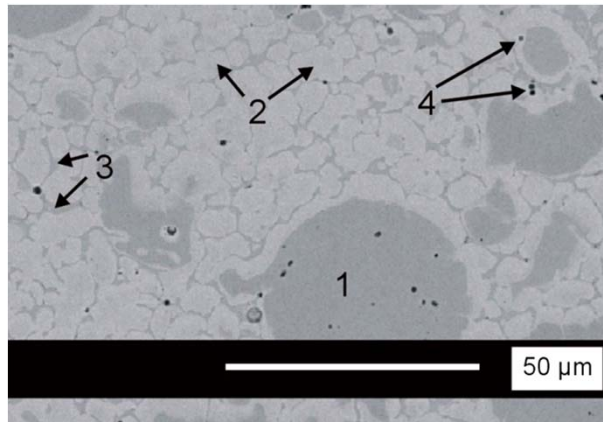
Binding mechanism classification



Stainless steel 316



Fe-Fe₃P-Ni-Cu powder mixture

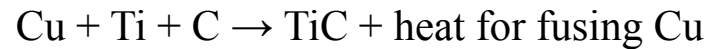


Metals 4: Chemical binding (e.g. Al, reinforced Cu)

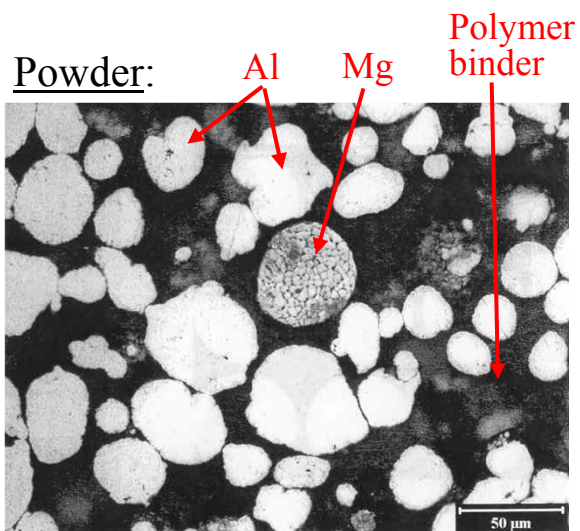
Binding mechanism classification



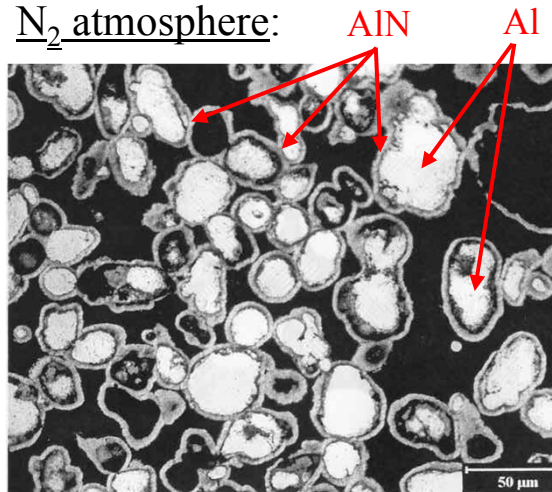
Cu-based composite:



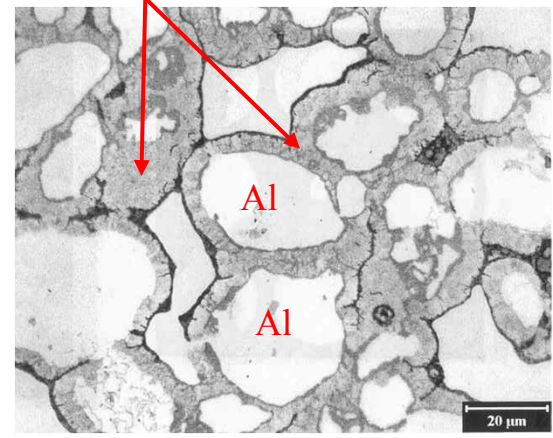
SLS of Aluminium:



Chemically bounded skeleton in N₂ atmosphere:



After infiltration with eutectic Al-13.8Si-4.7Mg infiltrant:



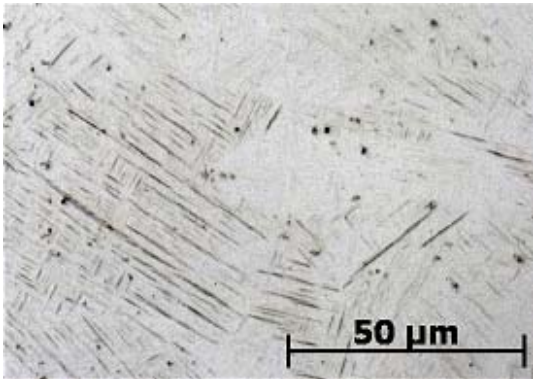
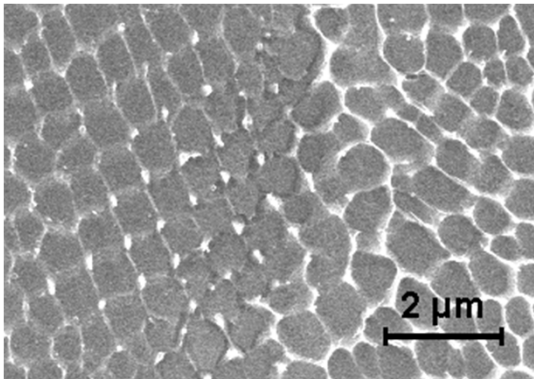
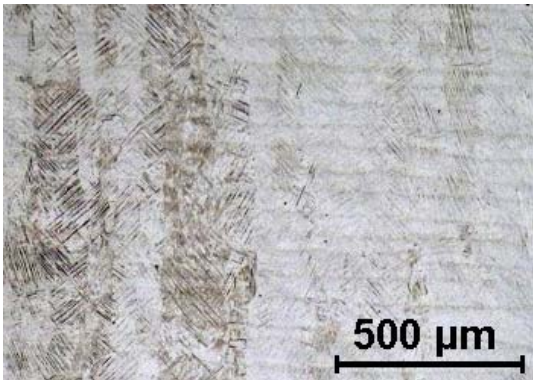
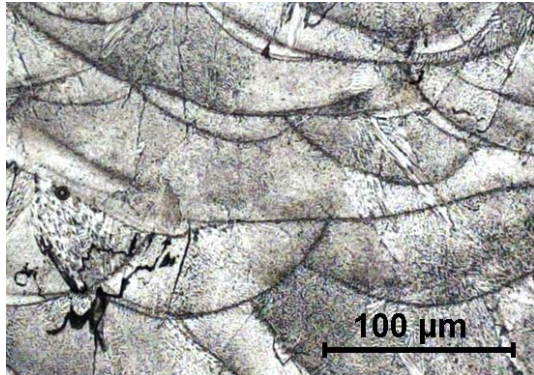
Mechanical properties of metals

(^{*}) Conventional material
(not heat treated)

| Alloy | Hardness | Charpy Impact | E-modulus | Tensile Strength | Elongation |
|--|-------------------------------|----------------------------------|---------------------------------|-----------------------------------|------------------------------|
| Titanium alloy Ti6Al4V | 410HV (396HV) [*] | 11,5±0,5J (21J) [*] | 96GPa (114GPa) [*] | 1250MPa (1170MPa) [*] | 6% (14%) |
| Stainless Steel 316L | | 59,2±3,9J (160J) [*] | | 719MPa (515MPa) [*] | 51% (60%) |
| Maraging Steel 18Ni300 | 390HV (324HV) [*] | 10,1±1,4J (18J) [*] | 163GPa (180GPa) [*] | 1290MPa (1000MPa) [*] | 1,6% (12%) [*] |
| Aluminium alloy AlSi10Mg | 127HV (86HV) [*] | | 56GPa (71GPa) [*] | 396MPa (317MPa) [*] | 2,75% (3,5%) [*] |
| Tool steel M2 | 760HV (250HV) [*] | | 110GPa (150GPa) [*] | 300MPa (750MPa) [*] | 0,35% (15%) [*] |
| Tantalum (Cold Worked) [*] | 207HV (200HV) [*] | | 168GPa (186GPa) [*] | 513MPa (900MPa) [*] | 29% |
| Cobalt Chrom | 392HV (477HV) [*] | | 169GPa (207GPa) [*] | 963MPa (925MPa) [*] | 20% (5%) [*] |

Other materials: Ni alloys (Inconel, Hastelloy), Pure CP-Ti, β -Ti, Nitinol, W, ...

Mechanical properties: microstructure

| | Ti6Al4V | CoCrMo |
|-------------------|--|---|
| Micro | <ul style="list-style-type: none">Fine, needle-like martensitic α'HCP  | <ul style="list-style-type: none">Fine, cellular α-CoFCC  |
| Macro (side view) | <ul style="list-style-type: none">Elongated prior β grains in the build direction  | <ul style="list-style-type: none">Melt tracks clearly visible in both side and top view  |

Mechanical properties – Heat treatments

- Heat treatments after SLM of Ti6Al4V

| | T [°C] | t [h] | Cooling Rate | E [GPa] | σ_y [MPa] | UTS [MPa] | $\epsilon_{\text{failure}}$ [%] |
|---|-------------|-------|--------------|--------------|------------------|-----------|---------------------------------|
| 1 | 540 | 5 | WQ | 112.6 ± 30.2 | 1118 ± 39 | 1223 ± 52 | 5.36 ± 2.02 |
| 2 | 850 | 2 | FC | 114.7 ± 3.6 | 955 ± 6 | 1004 ± 6 | 12.84 ± 1.36 |
| 3 | 850 | 5 | FC | 112.0 ± 3.4 | 909 ± 24 | 965 ± 20 | - (premature failure) |
| 4 | 1015 | 0.5 | AC | 114.9 ± 1.5 | 801 ± 20 | 874 ± 23 | 13.45 ± 1.18 |
| | followed by | | | | | | |
| | 843 | 2 | FC | | | | |
| 5 | 1020 | 2 | FC | 114.7 ± 0.9 | 760 ± 19 | 840 ± 27 | 14.06 ± 2.53 |
| 6 | 705 | 3 | AC | 114.6 ± 2.2 | 1026 ± 35 | 1082 ± 34 | 9.04 ± 2.03 |
| 7 | 940 | 1 | AC | 115.5 ± 2.4 | 899 ± 27 | 948 ± 27 | 13.59 ± 0.32 |
| | followed by | | | | | | |
| | 650 | 2 | AC | | | | |
| 8 | 1015 | 0.5 | AC | 112.8 ± 2.9 | 822 ± 25 | 902 ± 19 | 12.74 ± 0.56 |
| | followed by | | | | | | |
| | 730 | 2 | AC | | | | |

WQ = water quenching. AC = air cooling. FC = furnace cooling.

Treatment 6 to 8 are well known Ti6Al4V heat treatments [26].

Samples for treatment 3 were built in a different batch: building errors led to premature failure of components.

Traditional Ti-6-4 treatments

Mechanical properties – Toughness and fatigue

Ti6Al4V: ductility, toughness & fatigue (without thermal treatment)

- Charpy V-notch: the roughness of SLM parts does NOT act as a stress concentrator.

| | SLM | Investment cast | Wrought |
|--------------------|------------|-----------------|---------|
| Charpy V-notch [J] | 11,5 ± 0,5 | 15-19 | 15-20 |

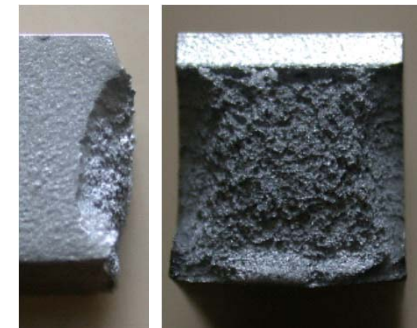
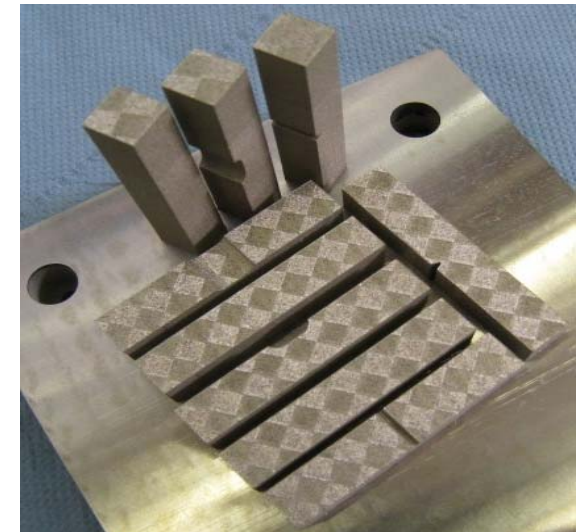
- Fracture toughness: (ongoing research)

| | SLM | Cast | Wrought |
|--------------------|-----|--------|---------|
| K_{Ic} [MPa√(m)] | 52 | 70-100 | 65-70 |

- Fatigue: (ongoing research)

| (Unnotched, R=0 or 0,1) | SLM | Cast | Wrought |
|-------------------------|------|------|---------|
| HCF limit [MPa] | >250 | >200 | >400 |

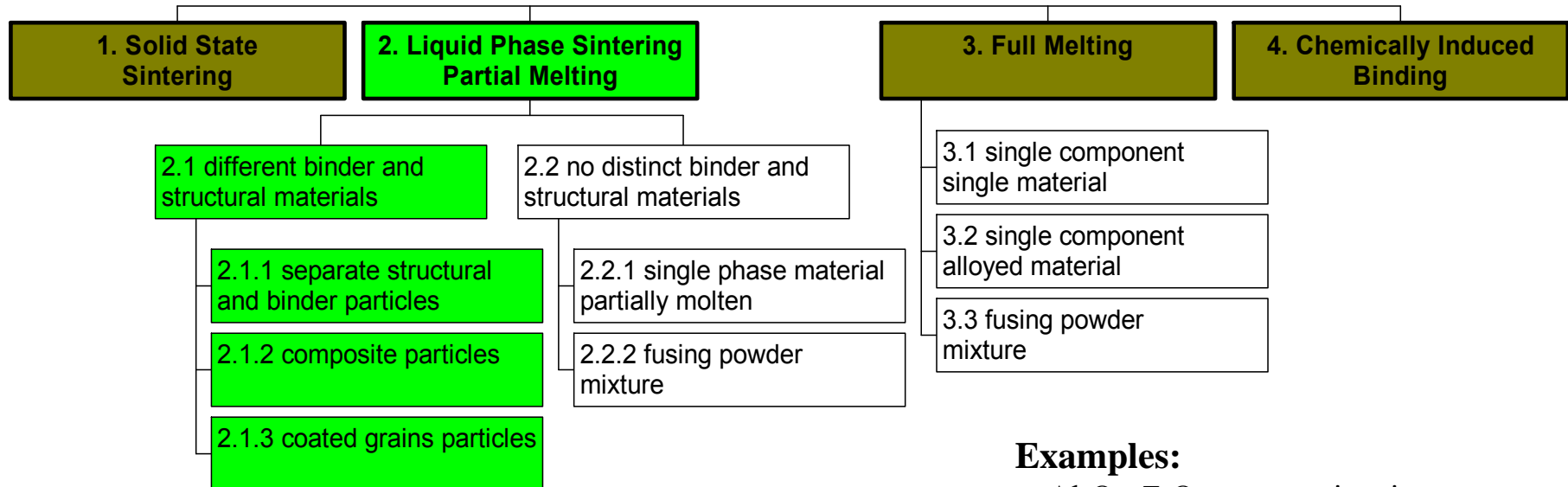
- Crack growth rate



Ceramics

Ceramics

Binding mechanism classification



Alumina part



Examples:

- Al_2O_3 , ZrO_2 , or eutectic mixture
- Micro SLS of SiC
- Transparent Ta_2O_5 dielectric ceramic
- SiO_2 investment casting shells
- HA biocompatible medical implants
- TCP/Glass biocompatible implants
- Bismuth-titanate ($\text{Bi}_4\text{Ti}_3\text{O}_{12}$) radiation detectors
- Bismuth-germanate ($\text{Bi}_4\text{Ge}_3\text{O}_{12}$)

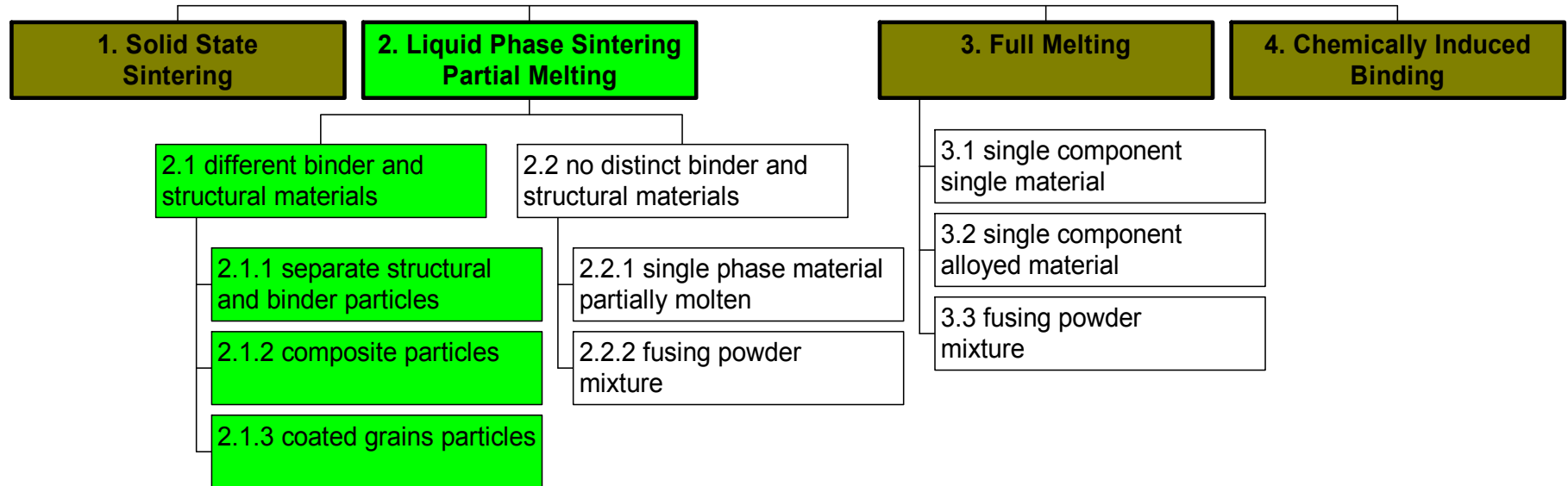
Ceramics : Classification

Major distinction:

| Ceramic type | Main Consolidation type |
|--|---|
| Silicate ceramics Multi-phase material made from clay, kaolin, silicate carriers (feldspar, soapstone) (+ Al_2O_3 , ZrSiO_4) | <ul style="list-style-type: none">• Liquid Phase Sintering• Partial Melting• Full melting |
| Oxide ceramics 90% single phase / single component metal oxides (Al-oxide, Mg-oxide, Zr-oxide, Al-titanate, Piezo-ceramic) | <ul style="list-style-type: none">• Solid State Sintering• Partial Melting• Full melting |
| Non-oxide ceramics Si and Al, with N or C <ul style="list-style-type: none">• Carbide ceramics• Nitride ceramics | <ul style="list-style-type: none">• Chemical Induced Binding• Partial Melting• Full melting |

Ceramics : Classification

Binding mechanism classification



Category 2
subdivided as

- | |
|--|
| 1. Solid State Sintering |
| 2a. SLS using sacrificial polymer binder |
| 2b. Liquid Phase Sintering / Partial melting |
| 2c. SLS of ceramic slurry |
| 3. Selective Laser Melting |
| 4. Chemical or self-induced binding |

Selective laser processing of ceramics (e.g. Al_2O_3)

- **Indirect SLS** of e.g. Al_2O_3 (partial melting of polym. binder)

- **Densification strategies**

- infiltration of green/brown/final parts with highly loaded Al_2O_3 suspensions
- isostatic pressing of green parts

- **Final Al_2O_3 densities after applying densification strategies**

- Carnauba wax: 75% => under investigation
- PS: 66% => 85%
- PA (ball milled): ... => 94%
(bad geometrical accuracy)
- PA : 48% => 71%
- PP: 48% => 82%
(bending strength: 96 MPa)

It's only the beginning.
Further improvements are expected!!

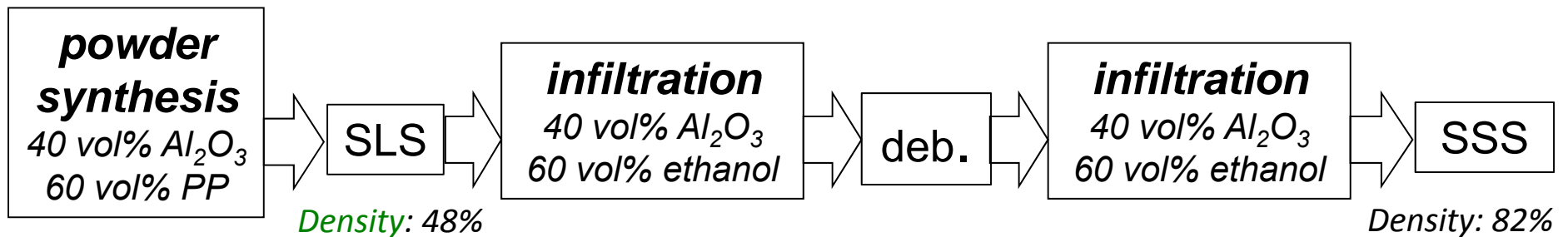
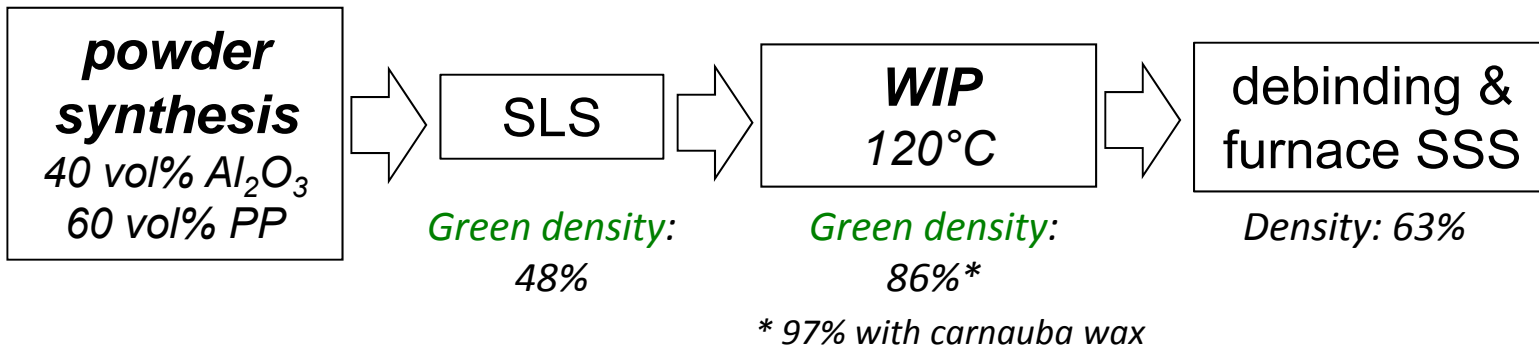
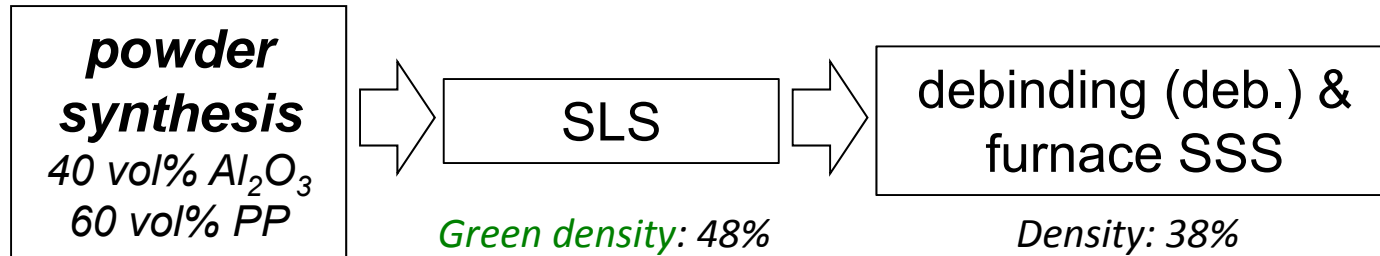


Emblem statue of Brussels city



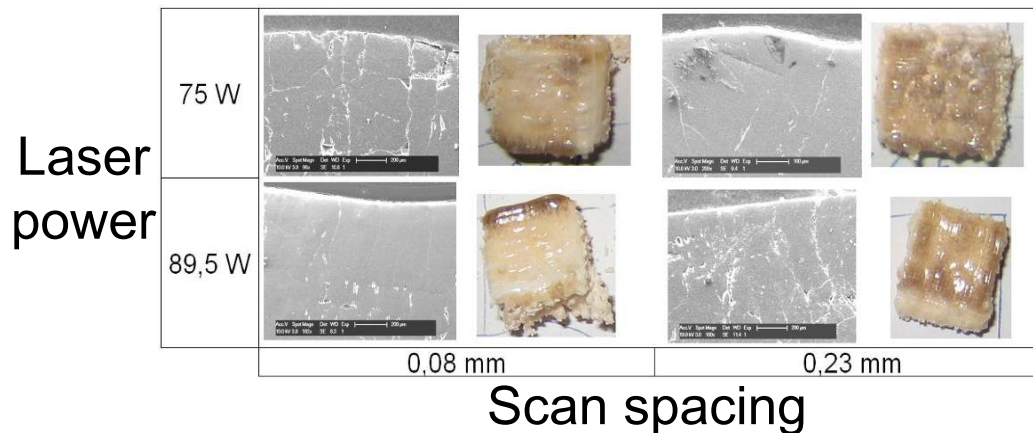
Selective laser processing of ceramics (e.g. Al_2O_3)

■ E.g.:



Selective laser processing of ceramics (e.g. Al_2O_3)

- Direct SLM of Al_2O_3 (full melting of ceramic itself, without polymer binder)



- DTM sinterstation 2000+
- $v = 25 \text{ mm/s}$

Requirements:

- high packing density of sub micrometer particles ($>50\% \text{ Al}_2\text{O}_3$)
- preheating $> 800^\circ\text{C}$
- no full melting

Experimental setup for
direct SLS of Al_2O_3 under
development...

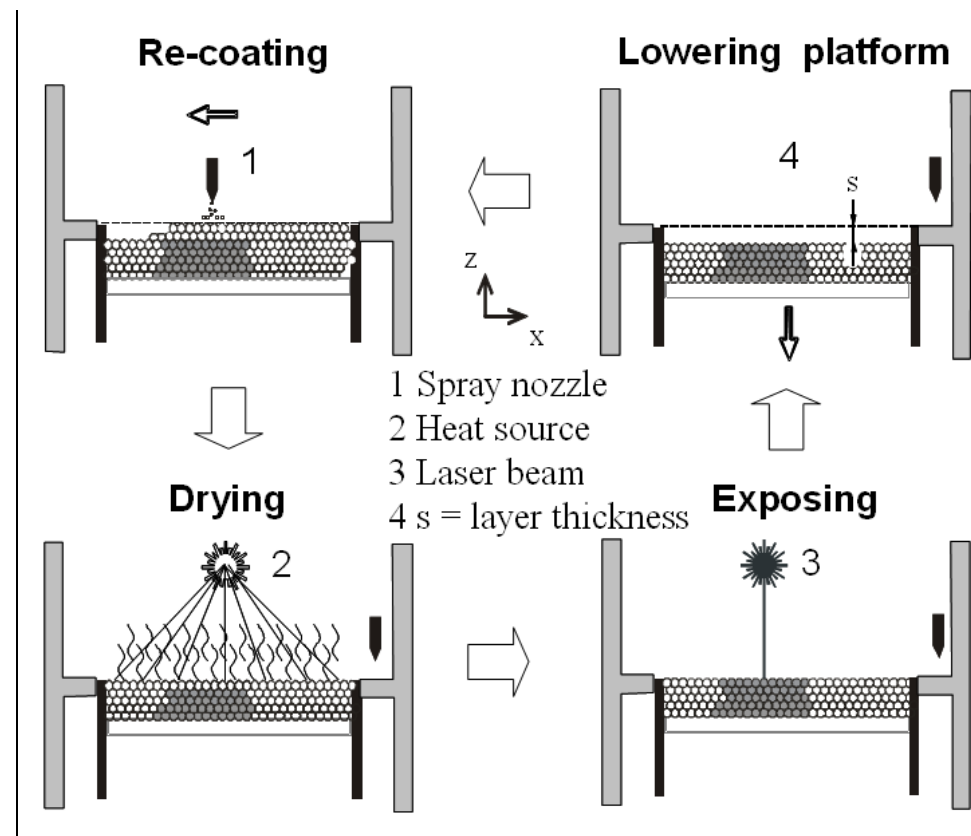
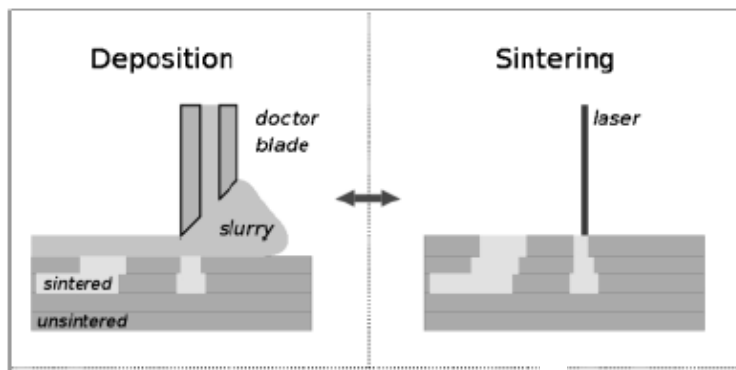
Ceramics: SLS of ceramic slurries

Principle:

- Replacing dry powder by slurry with smaller ceramic particles (high green density)
- Additional “drying” step, before “laser sintering”: capillary forces increase packing

Example (Univ. Clausthal):

- **Hydroxyapatite + H₂O**
(up to 66% solid loading)
- **Al₂O₃/SiO₂ + H₂O**
 - binder free, highly loaded slurry
 - + drying + SLS
 - low melting SiO₂ + reaction sintering with Al₂O₃



Ceramics: Chemically and Self-induced binding

Principle:

- Induce chemical reaction that binds powder particles

Examples:

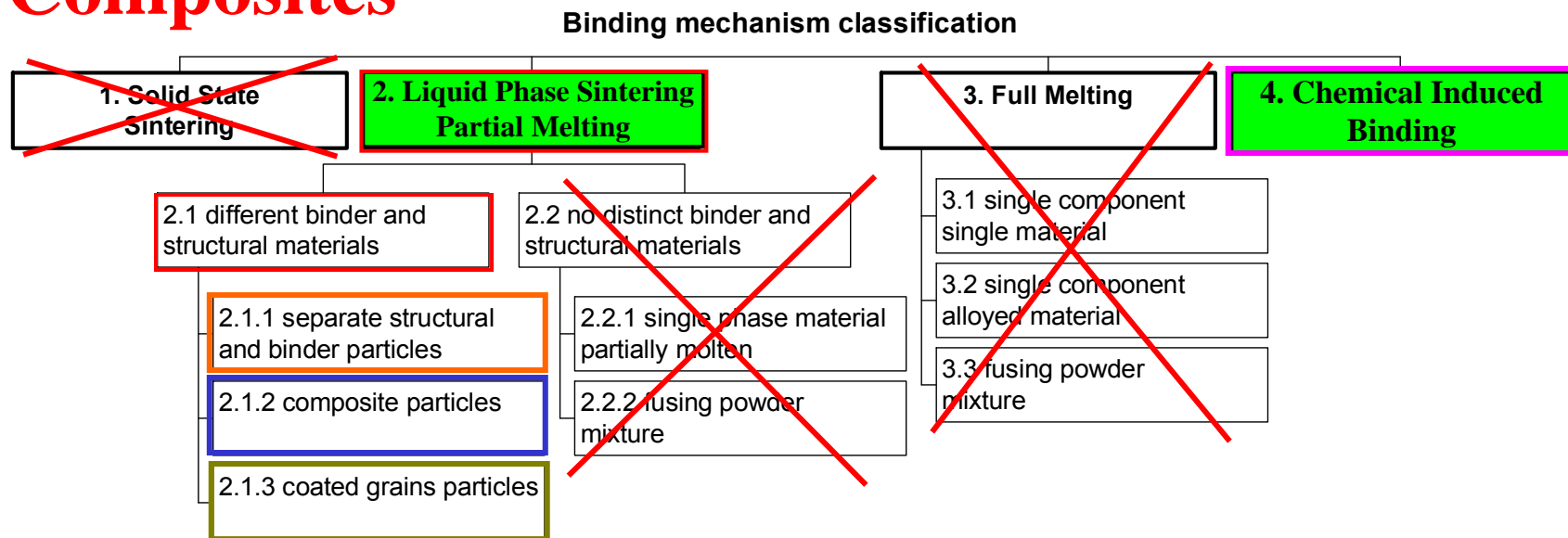
- $\text{SiC} \Rightarrow$
 - disintegration $\text{SiC} \rightarrow \text{Si} + \text{C}$
 - $\text{Si} + \text{O}_2 \rightarrow \text{SiO}_2$ binder for SiC
 - infiltration with Si + reaction bounded
- $\text{CuO} + \text{Al} \rightarrow \text{Al}_2\text{O}_3 + \text{Cu}$
 - Heat comes from laser + exothermal reaction
 - Self propagation controlled by addition of Cu
- $\text{Ti} + \text{Al} \rightarrow \text{TiAl}$
 - Heat comes from laser + exothermal reaction
 - Self propagation controlled by addition of TiAl
- Also tested: $\text{TiC-Al}_2\text{O}_3$ (mixture of TiO_2 , Al and C; self-propagating), ZrSiO_4 , MoSi_2



Investment casting shell and cast impeller

Composites

Composites



SLS/SLM well suited for all kind of composites:

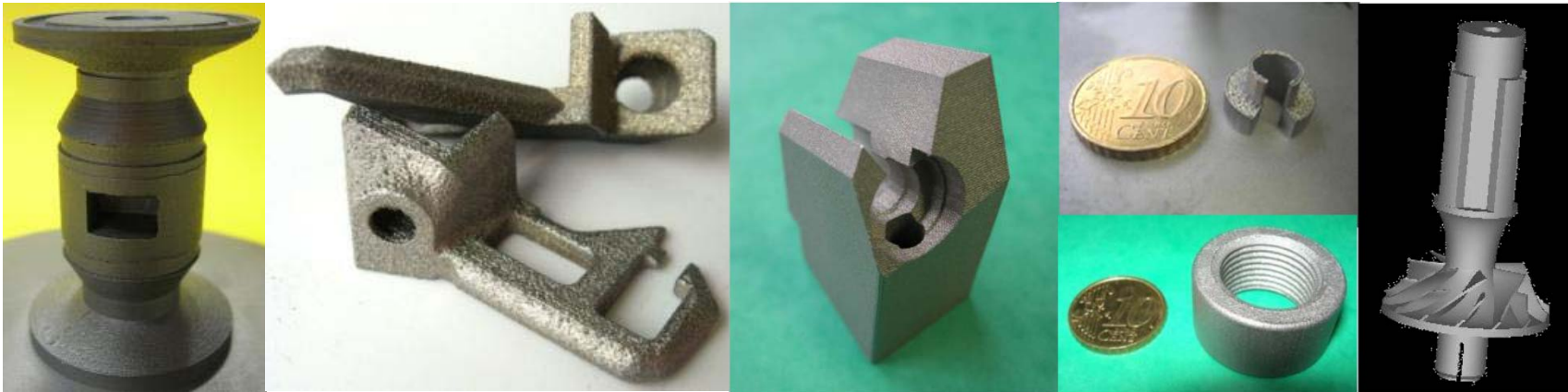
- polymer-metal (e.g. PA-Cu, PA-Al)
- polymer-ceramic (e.g. PE-HA, PCL-HA, PS-Al₂O₃)
- polymer-glass (e.g. PA-GF) → mixed → coated (no agglomeration; uniform distribution)
- metal-metal (e.g. Fe-Cu) → composite powder (uniform; no agglomeration; possible problems with fibers)
- metal-ceramic (e.g. WC-Co, Cu-TiC-TiB₂, Al₂O₃-Cu) → Chemical binding from mixture of CuO and Al
- ... → Liquid Phase Sintering → Chemical binding from mixture of Cu, Ti and B₄C

Conclusion

- **Laser powder-bed fusion is may be the most versatile AM technology**
- **It basically allows processing any material:**
 - **Polymers (semi-crystalline, amorphous, elastomeric, thermosetting)**
 - Although 20 years old, **still very limited pallet of polymers** (few semi-crystalline polymers)
 - Even with PA11-12, **density needs improvement** (still 5...8% porosity)
 - **Metal (ferro, non-ferro, reactive,...)**
 - **Pallet is increasing rapidly**
 - **Density mostly above 99 to 99.8%** (even up to 99.95% for several materials)
 - **Ceramics**
 - **Still under development**
 - **OK for porous parts** (filters and scaffolds), but **too low for structural parts** (94..97%)
 - **Composites** (polymer-metal, polymer-ceramic, metal-ceramics, metal-metal, reinforced)
 - **All kind of composites feasible** (see above)
 - **Technologically possible, but few industrial applications so far.**

Further developments may take decades, but this was also the case for subtractive and forming processes that have been developed for centuries.

Laser powder-bed fusion AM: SLS, SLM, ...



Thank you for your attention

