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Novel approaches to densify powder metallurgical materials through hot isostatic pressing

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Materials Session 7 at 15:20

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Contents

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- Process Methods
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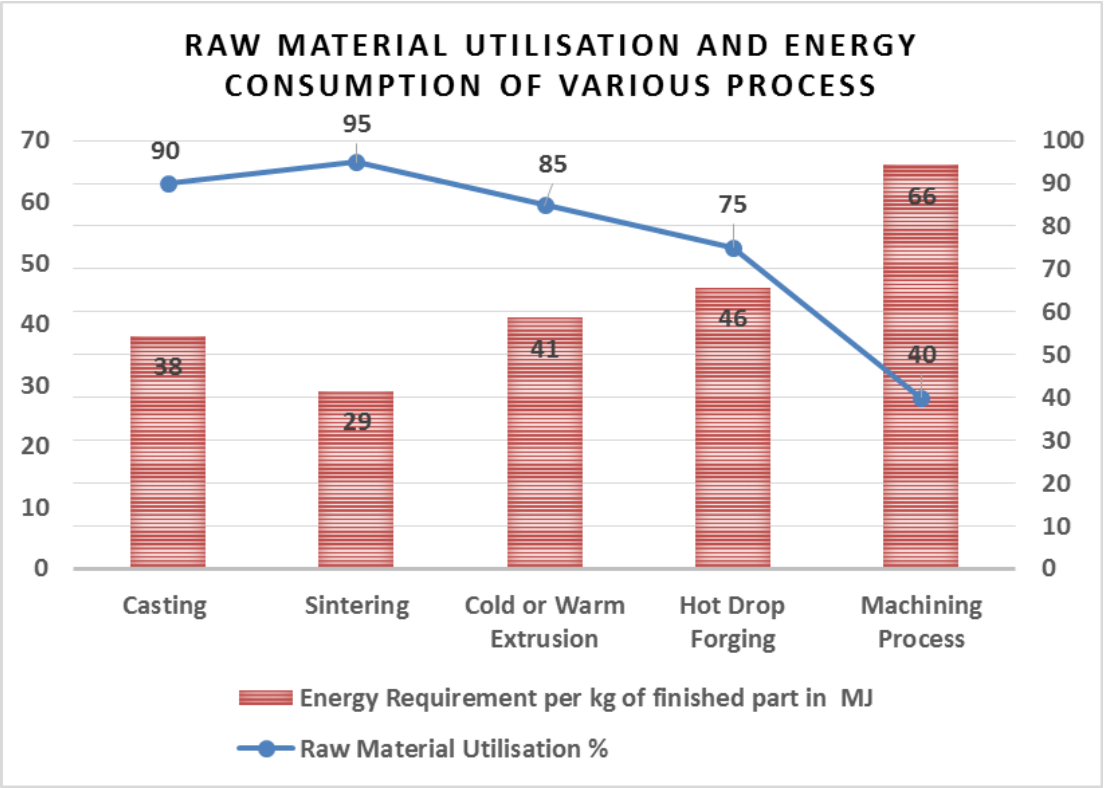
- Summary

Background: Why PM?

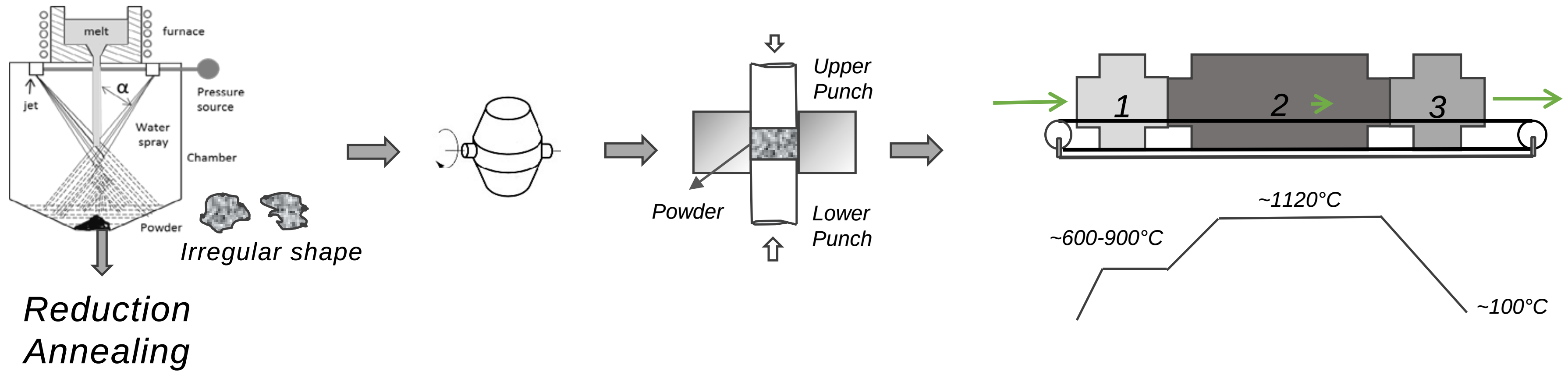
- Complex shapes
- High volume production
- Less energy consumption
- Less material waste
- Tailored properties
- Sustainable manufacturing process
- Need for high performance applications



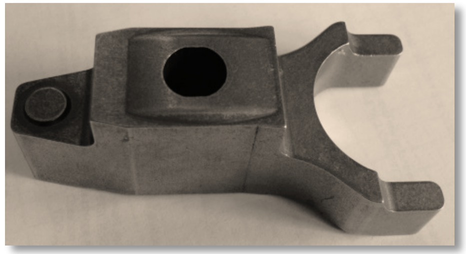
Source: European Powder Metallurgy Association

Powder Metallurgy Process

1. Powder Production 2.Admixing 3.Compaction 4.Delubrication and Sintering

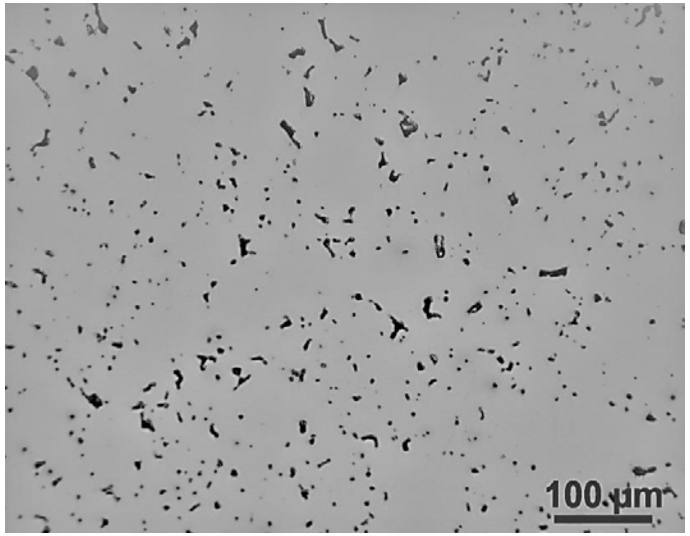


Sprocket



Injector Yoke

Challenge, Approaches and Benefits



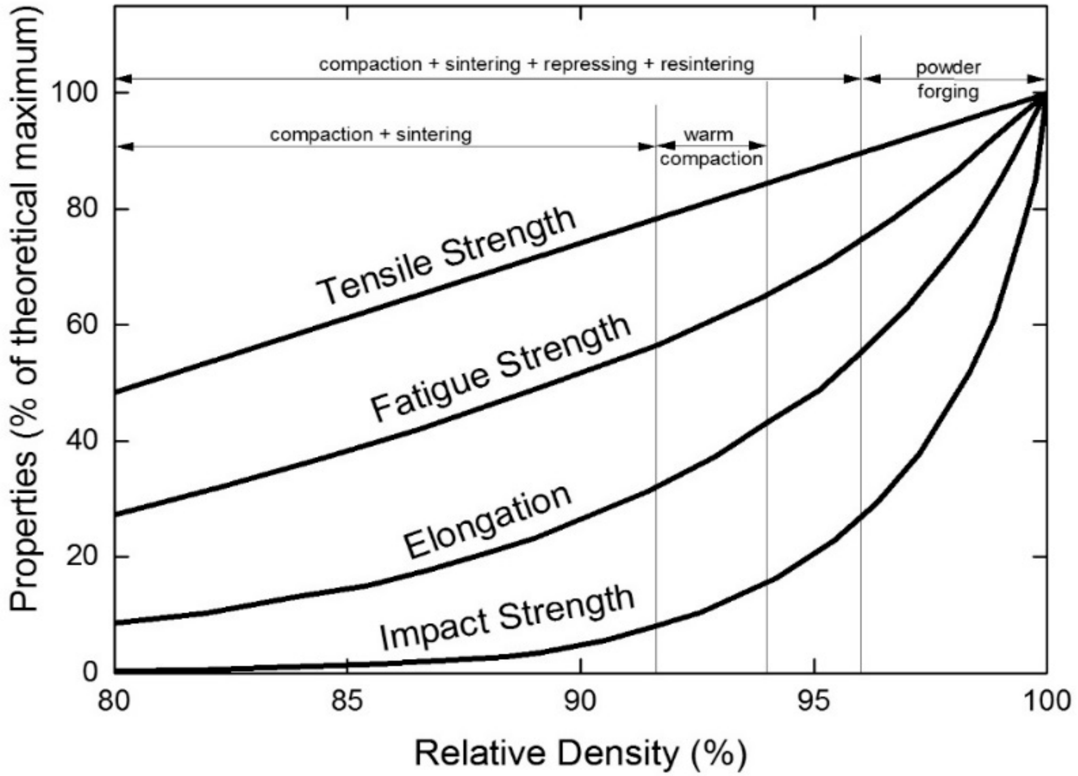
PM steel with density = 7.2 g/cm³

~ 90% theoretical density

"Porosity"

Limits the applications

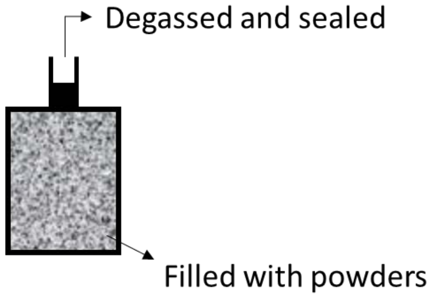
Alloying
+
Pressing
+
Sintering
+
Post Processing



Source: Höganäs Handbook for Sintered Components-3.Design and Mechanical Properties. Höganäs AB, 2004

Hot Isostatic Pressing

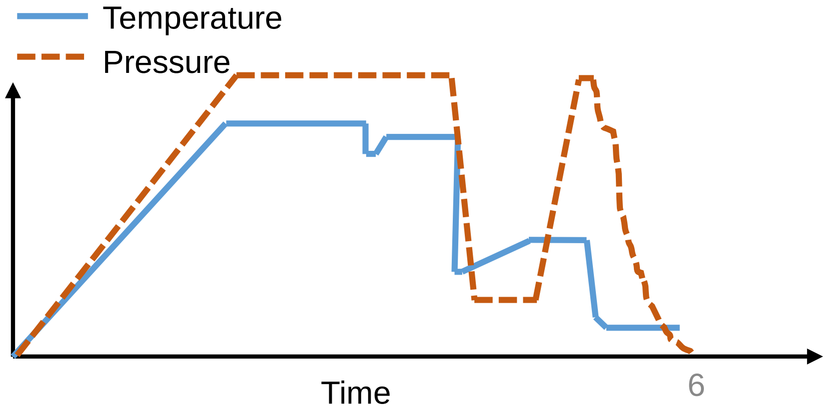
- ❖Post processing method
- ❖Simultaneous application of Pressure and Temperature (max 2000 bar and 2000 °C)
- ❖Capsule or a container is needed for powder consolidation
- ❖Cost competitive (< 5 SEK per Kg)
- ❖ Rapid cooling



HIP Capsule

QIH 15-L HIP press

Courtesy: Quintus Technologies AB



Objectives

- To achieve full density
- Surface pore closure for capsule free HIP
- Critical evaluation of the approaches

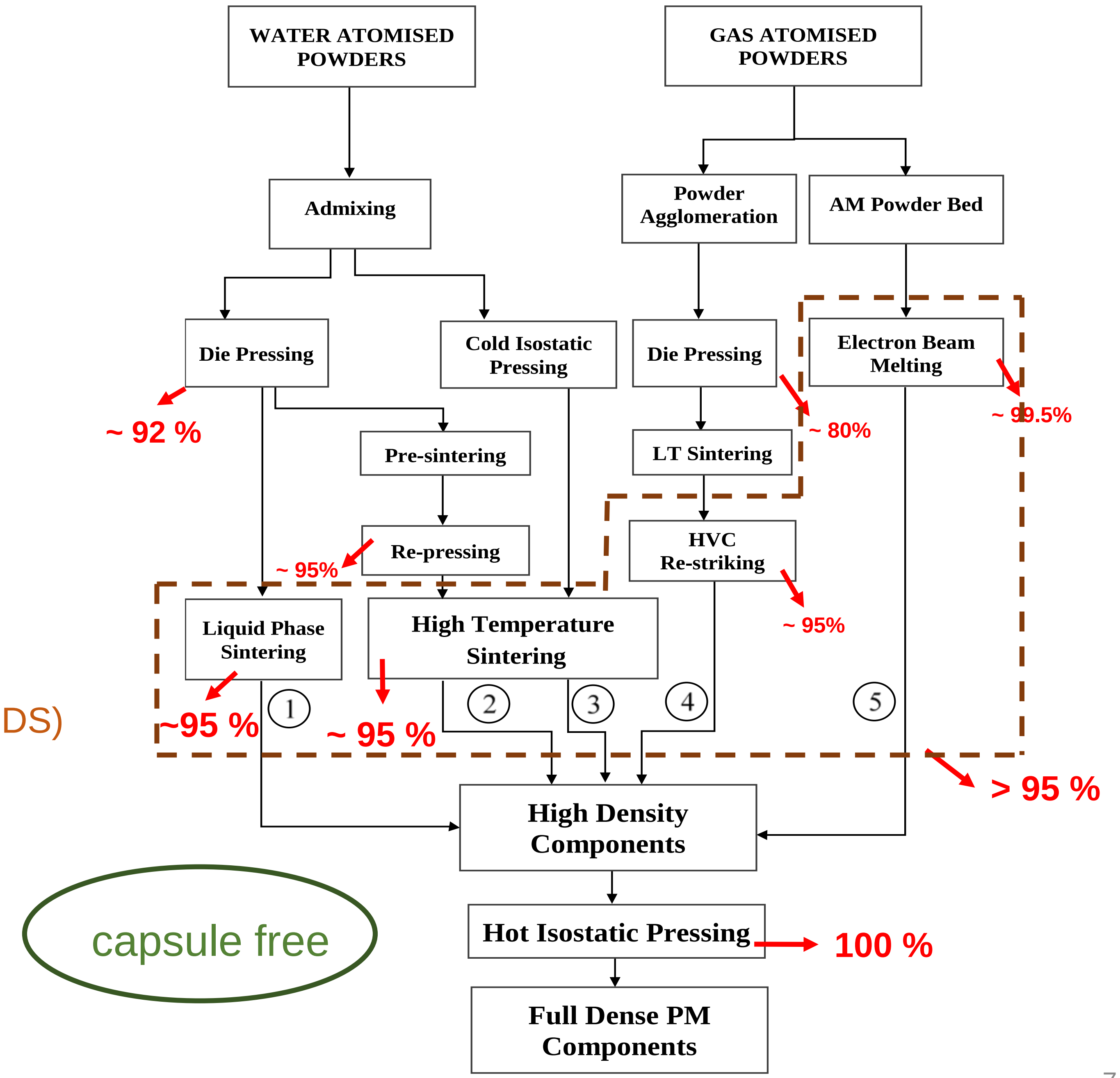
1 Liquid phase sintering (LPS)

2 Double pressing and Double sintering (DPDS)

3 Cold Isostatic Pressing (CIP)

4 High velocity compaction (HVC)

5 Additive manufacturing (AM)



Vattur Sundaram M. Licentiate Thesis 2017

High Density → Full density

1.LPS

2.DPDS

3.CIP

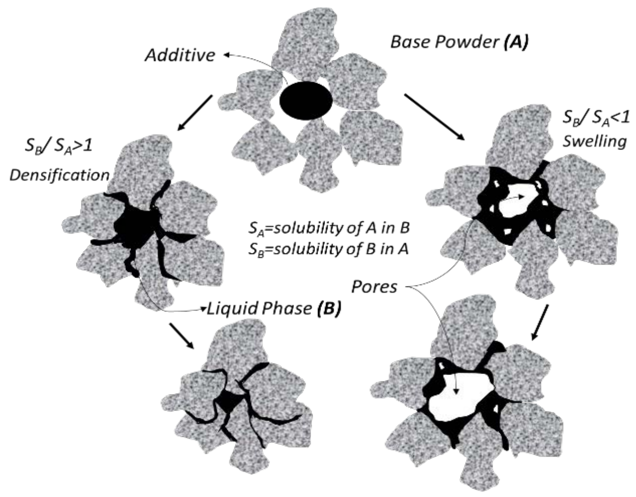
4.HVC

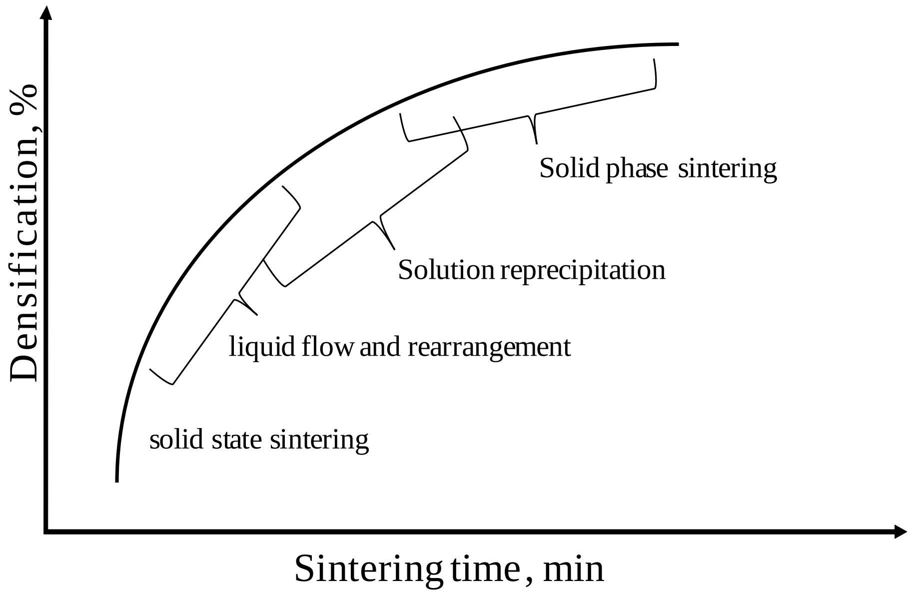
5.AM

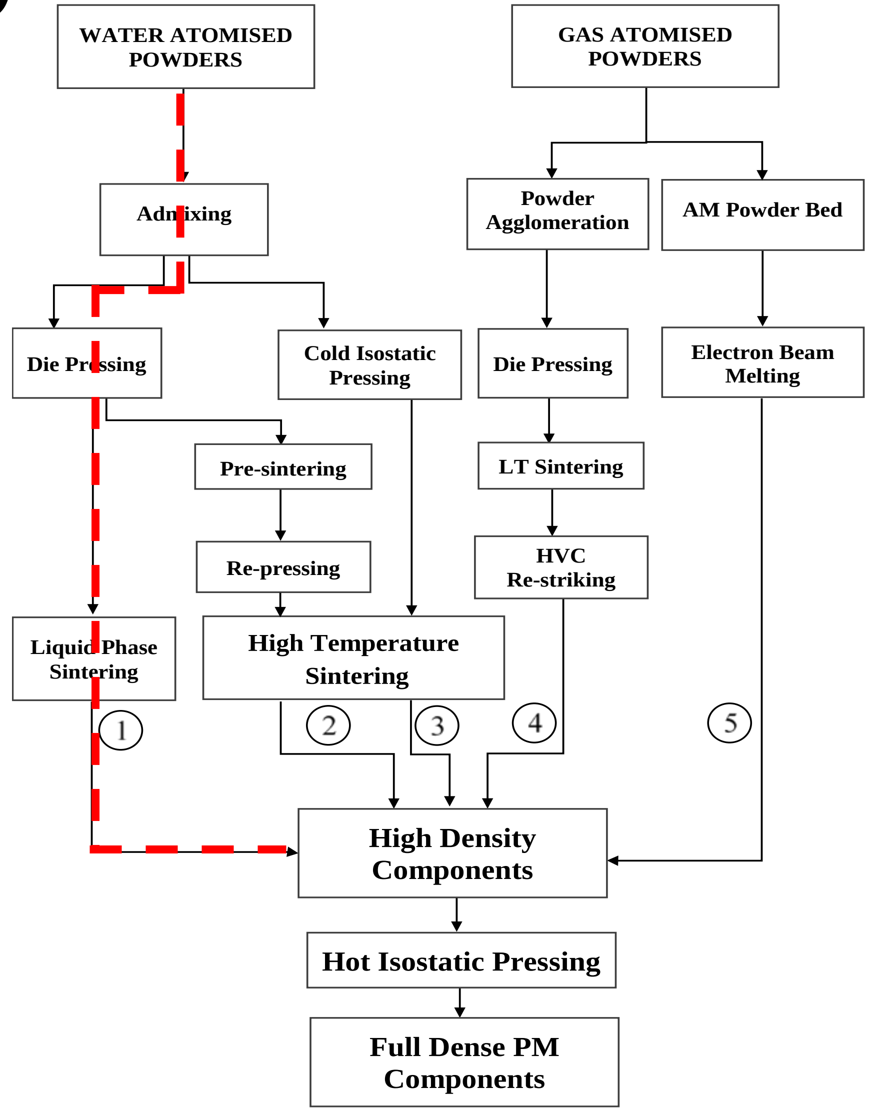
Capsule Free

HIP

1.Liquid Phase Sintering







Materials and Process

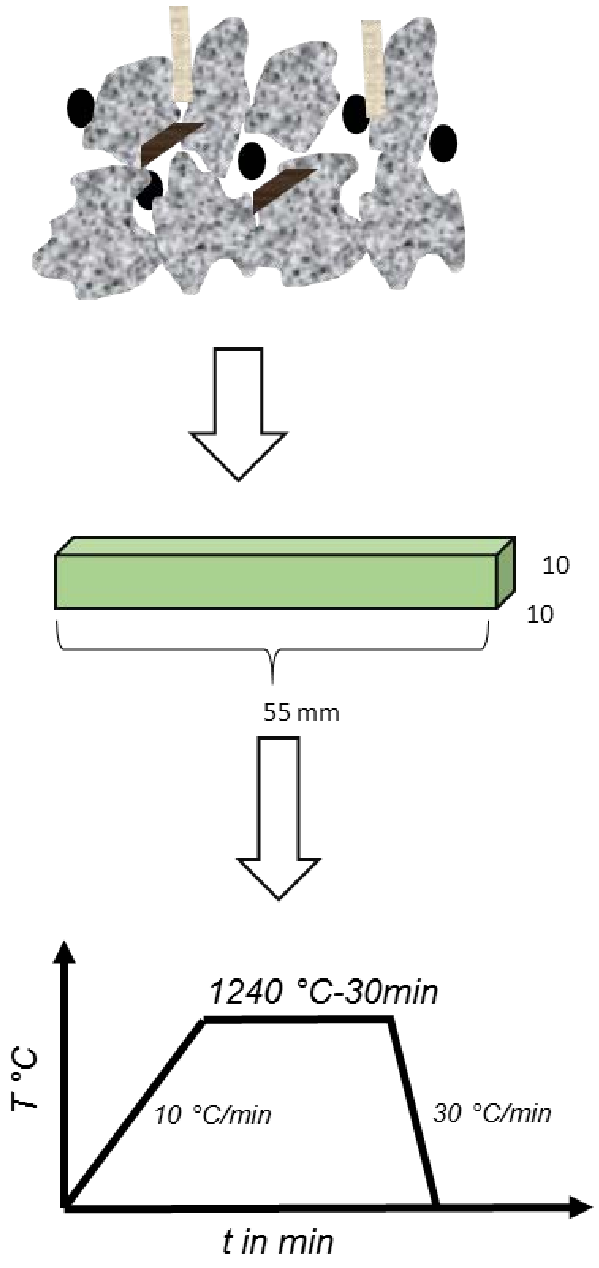
Ni-Mn-B *master alloy (MA)-CEITALLOY HD*

Elements	Ni	Mn	B	N	C	O	S
Wt. %	46.00	46.10	7.90	0.001	0.075	0.025	0.004

Fe and Mo prealloyed powders from Höganäs AB

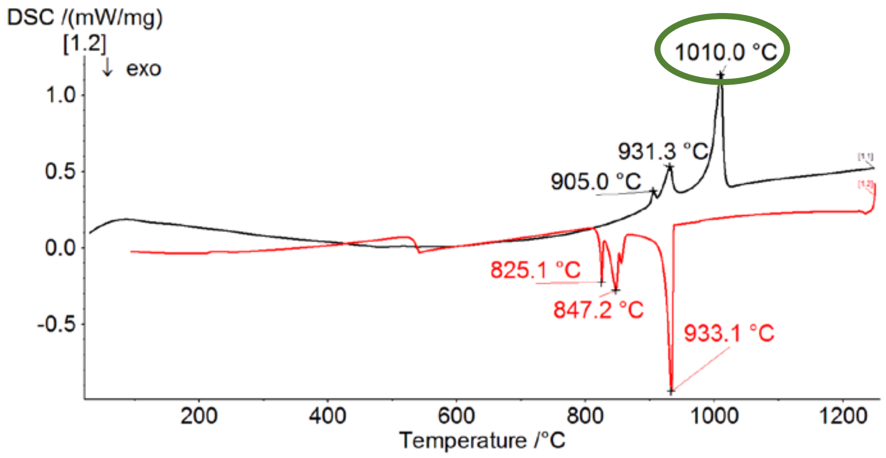
Material	**Elements in Wt.%**				**Designation**
	Mo	**C**	**MA**	**Fe**	
Fe (ASC100.29)	-	-	2.5	Bal	Fe+2.5MA
	-	0.3	2.5	Bal	Fe+2.5MA+C
Fe-0.45Mo (X-Astaloy 0.45Mo)	0.45	-	2.5	Bal	Fe-Mo+2.5MA
	0.45	0.3	2.5	Bal	Fe-Mo+2.5MA+C
	0.45	-	1.5	Bal	Fe-Mo+1.5MA
	0.45	0.3	1.5	Bal	Fe-Mo+1.5MA+C

- ***Effect of carbon***
- ***Effect of molybdenum***
- ***Effect of master alloy amount***



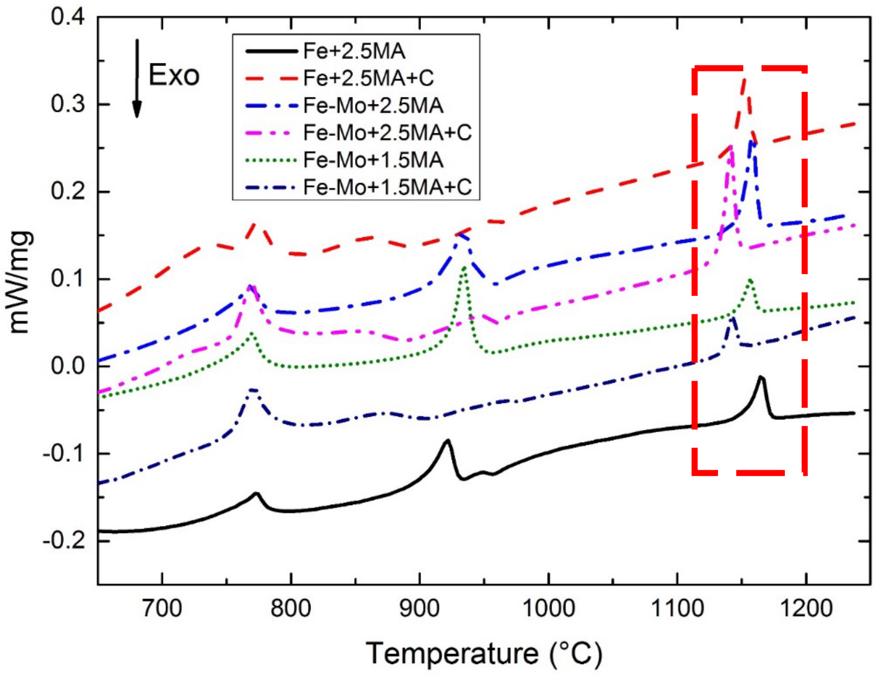
Results: DSC

Ni-Mn-B (< 45 µm) Master alloy



LP1-Master alloy melting

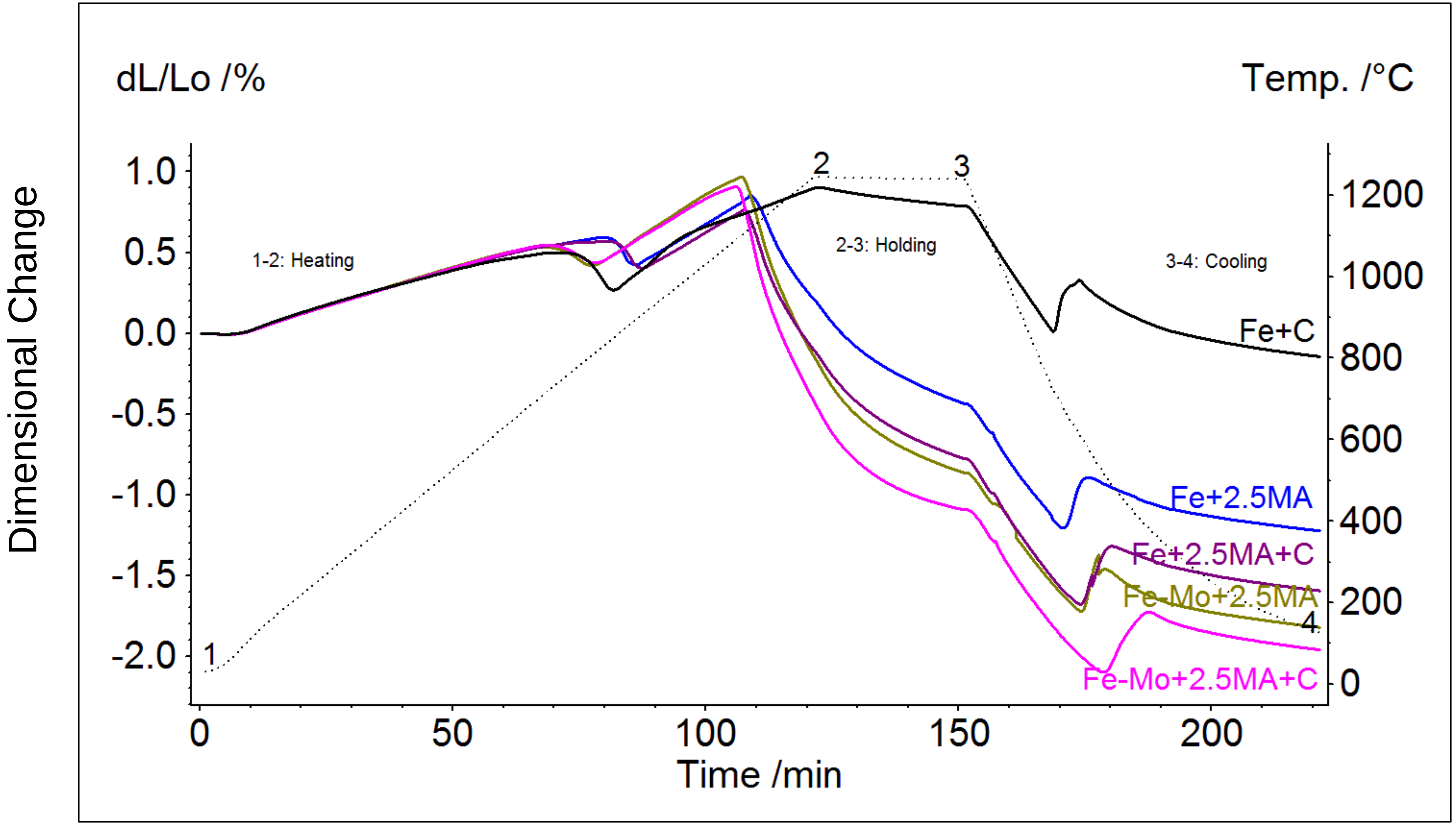
Fe+MA



LP2-Eutectic formation
1134 °C to 1154 °C

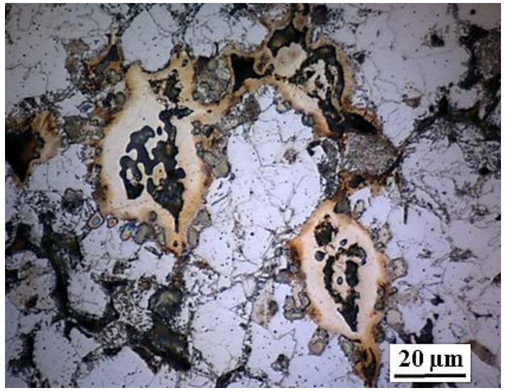
Vattur Sundaram M et al. Metall Mater Trans A 2017.

Dilatometry

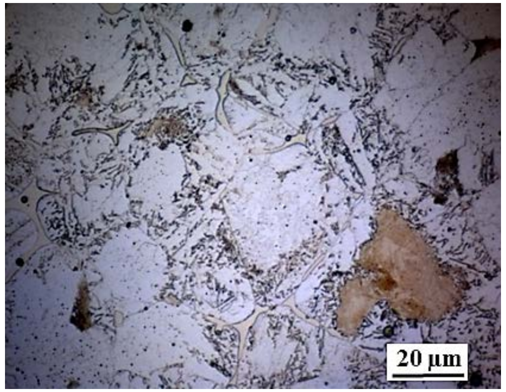


Microstructure

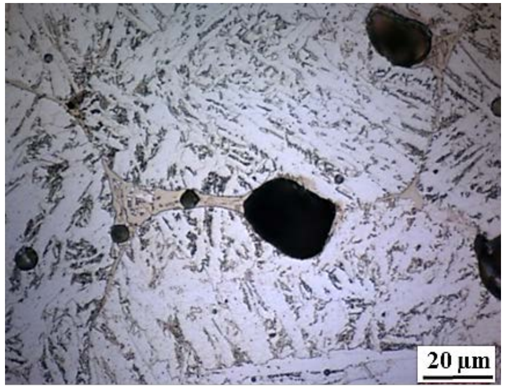
1000 °C-1 min



1100 °C-1 min



1240 °C-30 min

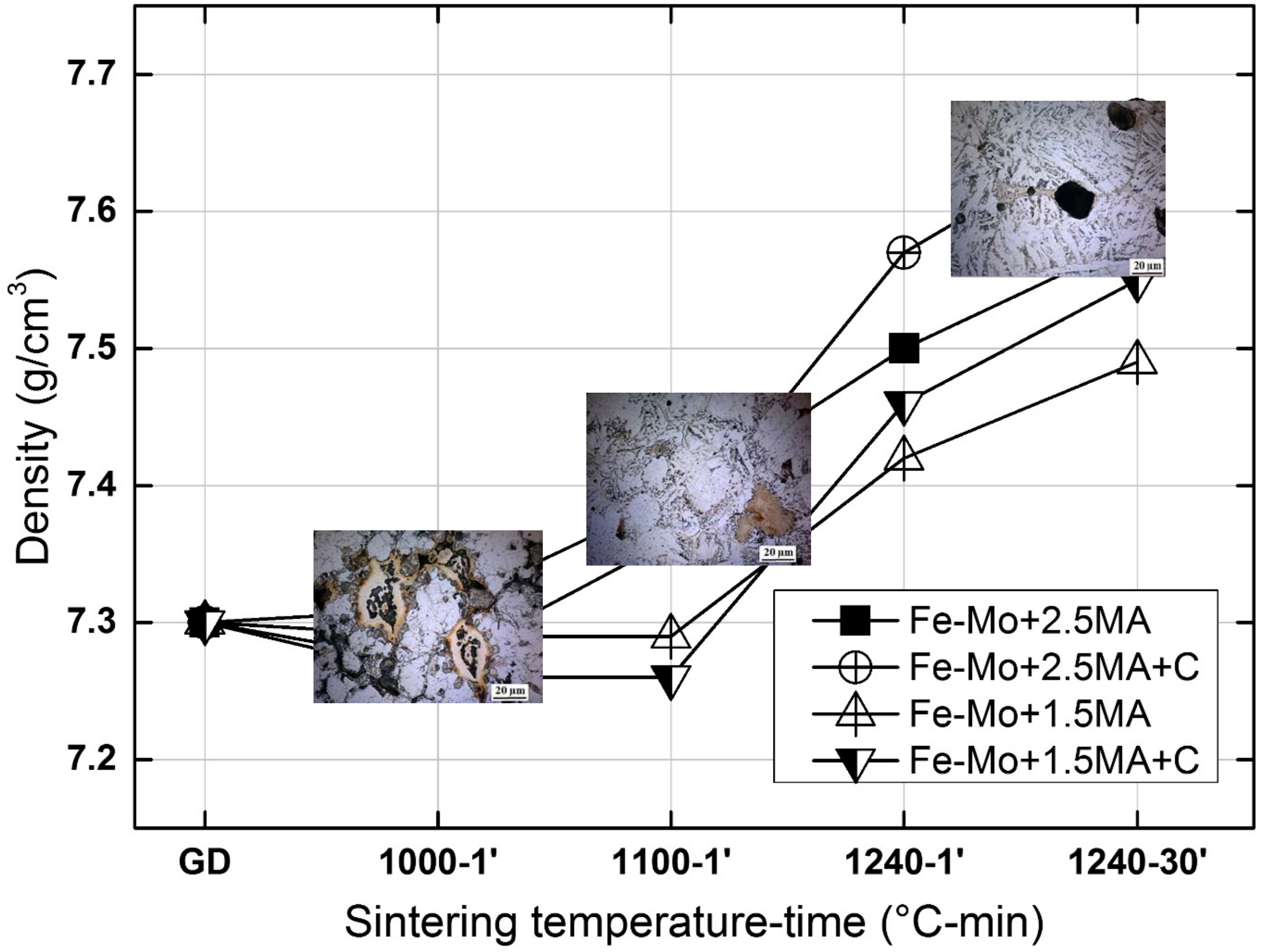


Two stages of Liquid formation occurs during Heating stage

LP-1 → Master alloy melting
LP-2 → Eutectic formation

Vattur Sundaram M et al. Metall Mater Trans A 2017.

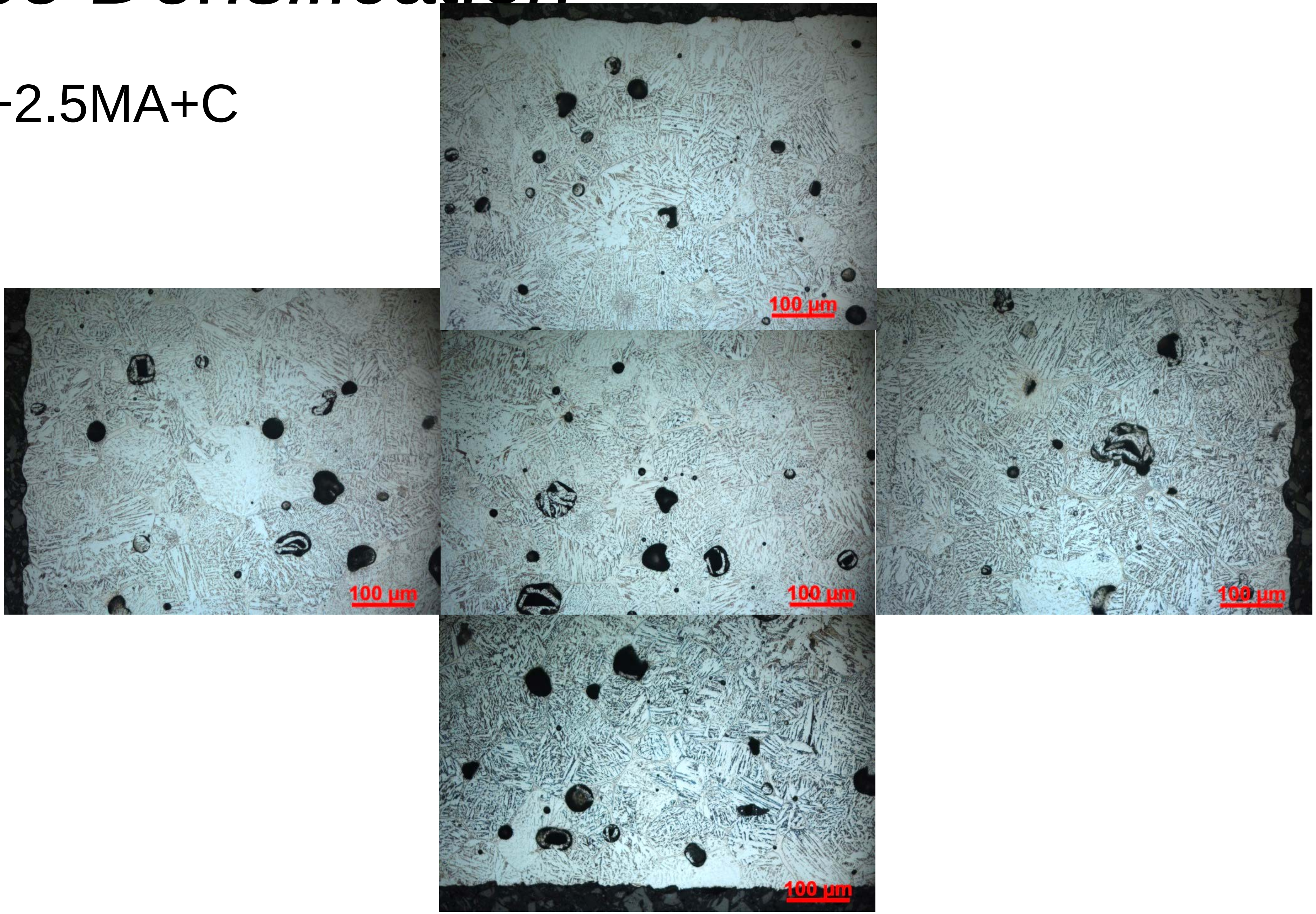
Density



Density (g/cm³)
7.7
7.6
7.5
7.4
7.3
7.2
GD
1000-1'
1100-1'
1240-1'
1240-30'
Sintering temperature-time (°C-min)
Fe-Mo+2.5MA
Fe-Mo+2.5MA+C
Fe-Mo+1.5MA
Fe-Mo+1.5MA+C
20 μm
20 μm
20 μm

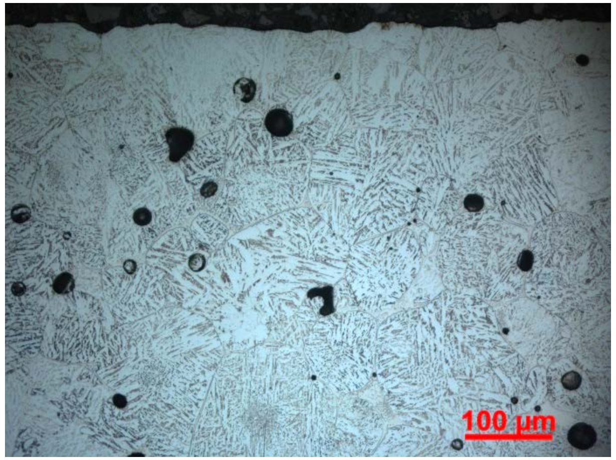
Surface Densification

Fe-Mo+2.5MA+C

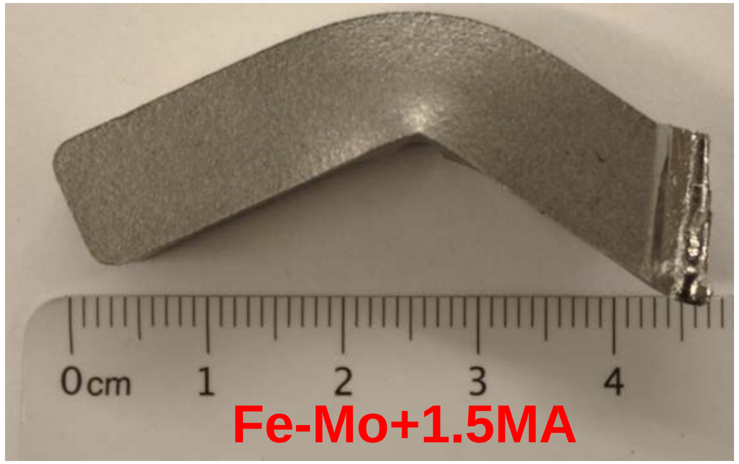


Densification after HIP

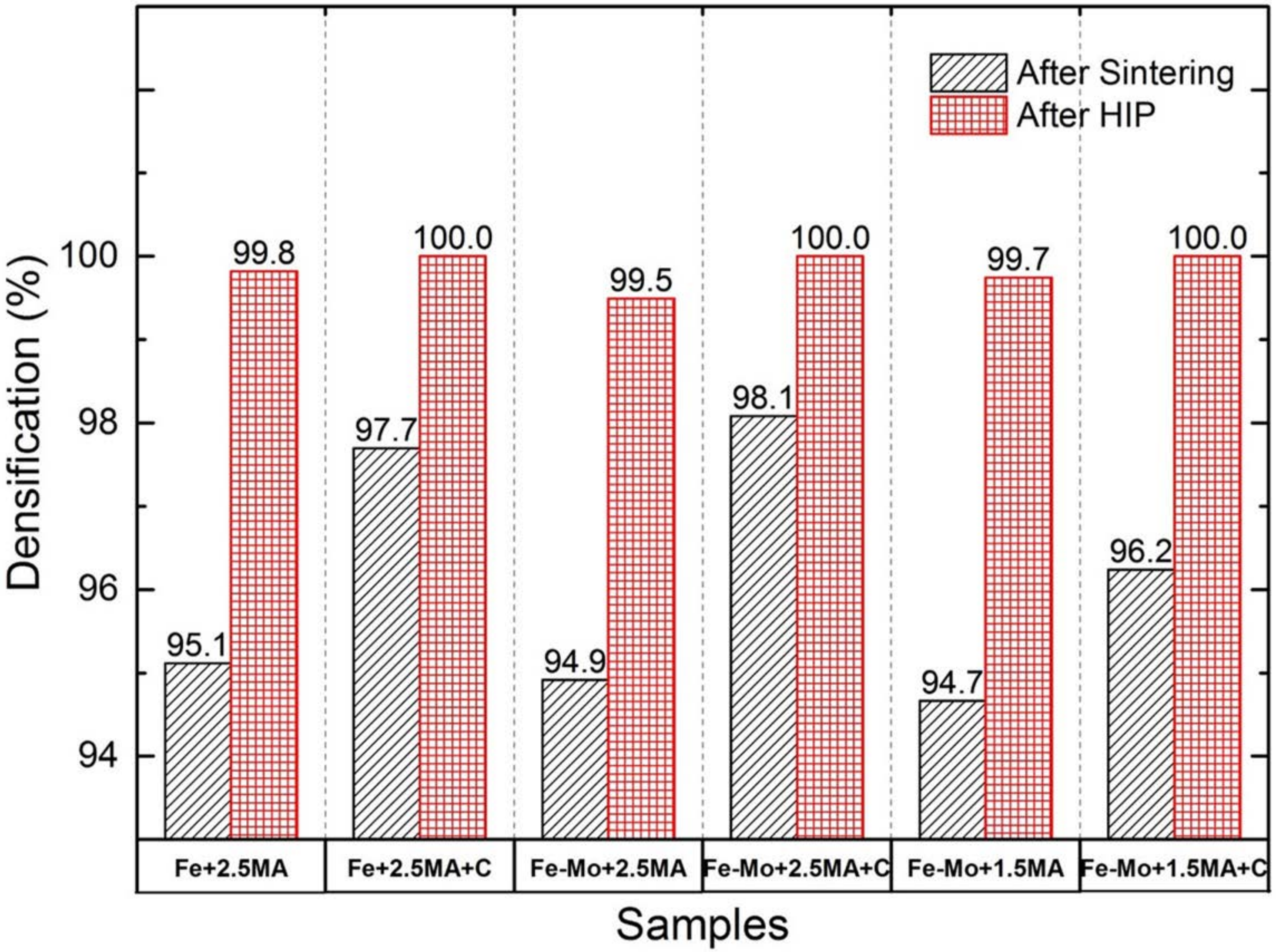
Surface Densification



After Impact testing



HIP at Quintus : 1000 bar, 1150 °C for 2 hours



Summary

- Liquid phase sintering of boron-containing master alloy powder enables the densification to > 95 % of the theoretical density and also surface pore closure.

- This enables full densification after HIP.

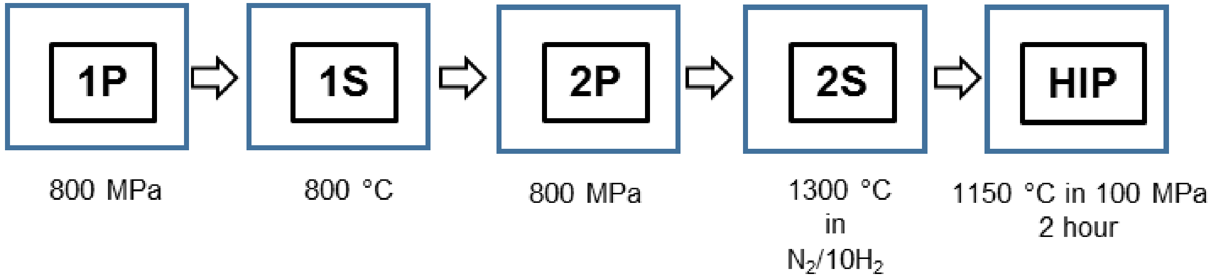
- Mo and C addition improves the densification and final properties.

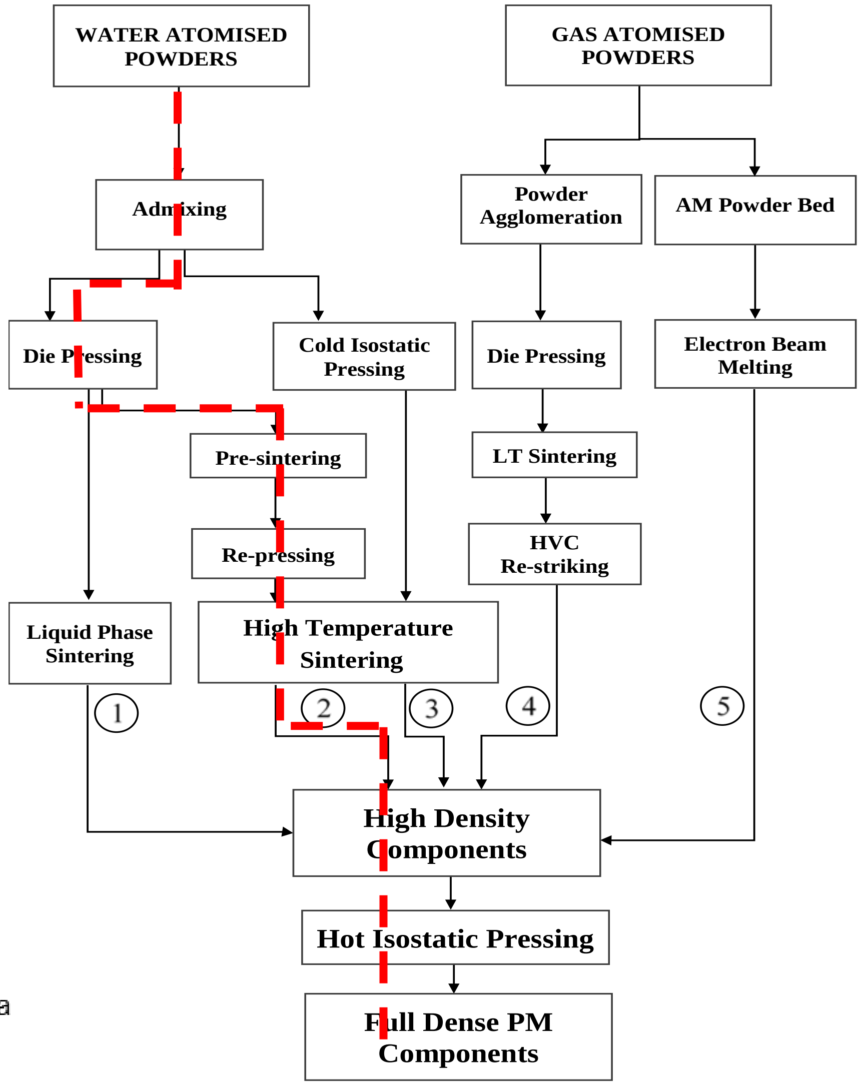
- Microstructural embrittlement occurs with 2.5 wt. % master alloy and it is more severe with carbon addition.

- For improved properties, lower boron content is needed, around 0.12 wt. % (1.5 wt. % master alloy addition).

2. Double pressing - Double sintering (DPDS)

- Pressure based densification
- Powder consolidation is from first pressing
- De-lubrication and stress relieving from pre-sintering
- Increase in density to enable pore closure after second sintering
- HIP for full densification



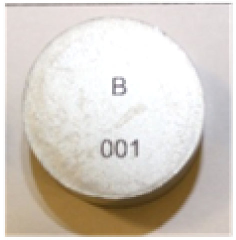
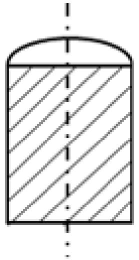
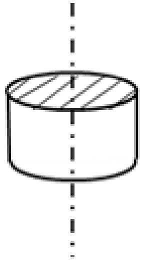
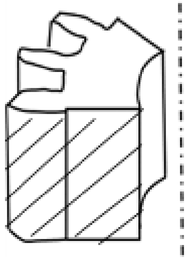
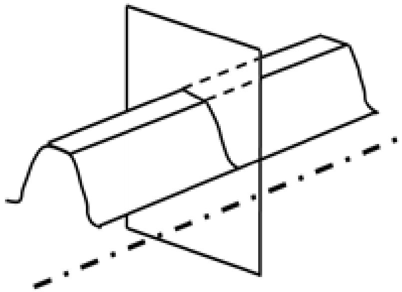


Materials and Process

Astaloy Mo (1.5 wt.% Mo Prealloyed+0.2 wt.% graphite) from Höganäs AB

Series	Powder	Lubricant	Sintering temperature and time
A	Standard	Densmix	1300°C, 1h
B	Standard	LubeE	1300°C, 1h
C	Fine (< 63µm)	LubeE	1300°C, 1h
D	Standard	Lube E	1250°C, 1h

1P	⇨	1S	⇨	2P	⇨	2S	⇨	HIP
800 MPa		800 °C		800 MPa		1300 °C in $N_2/10H_2$		1150 °C in 100 MPa 2 hour

	Samples	Parallel to axis	Perpendicular to axis
Cylinder			
Gear			

- Diameter→ 25 mm
- Height → 20 mm

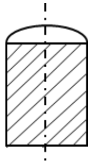
- Diameter→ 31.75 mm
- Axial width → 10 mm
- Module → 1.58 mm

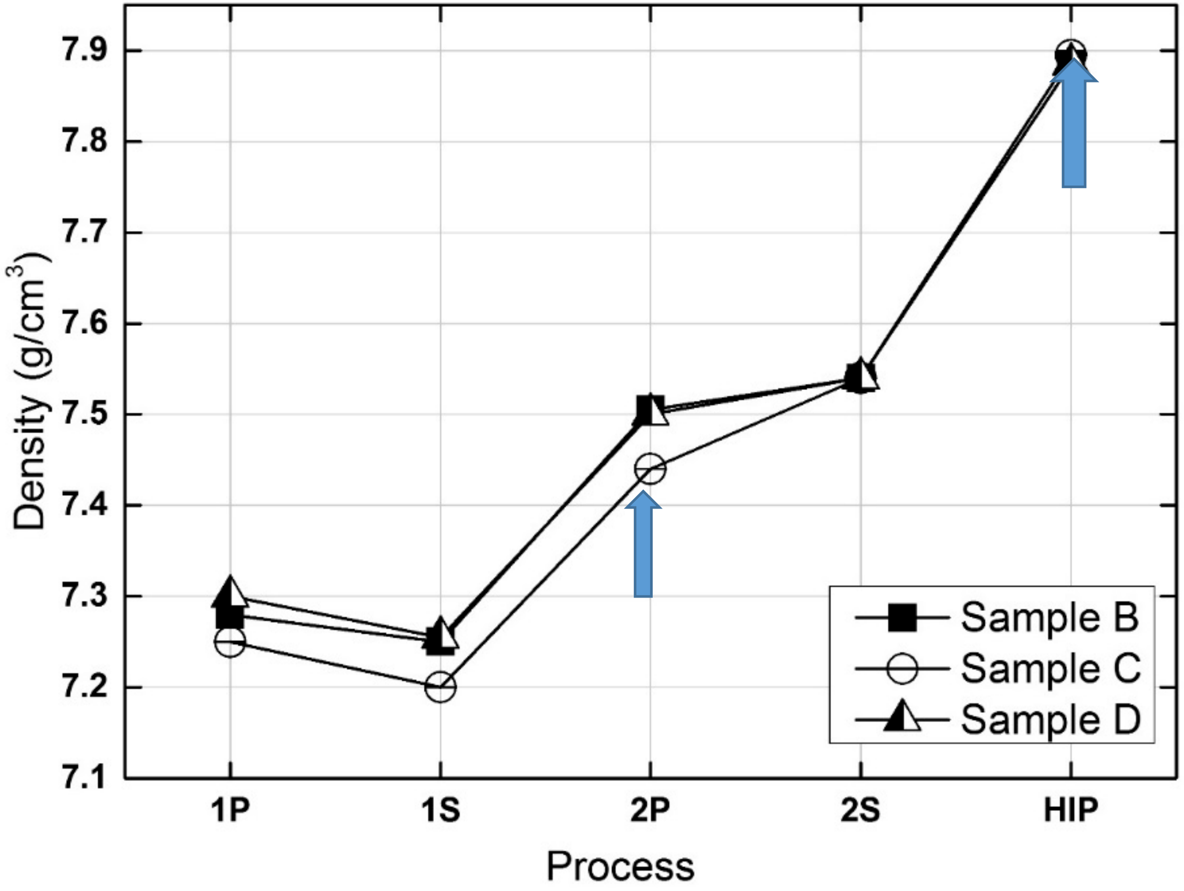
- Effect of lubricants
- Effect of powder size fraction
- Effect of sintering temperature

Density

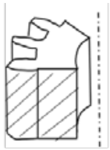
Sample	Powder	Sintering temperature and time
B	Standard	1300°C, 1h
C	Fine (< 63μm)	1300°C, 1h
D	Standard	1250°C, 1h

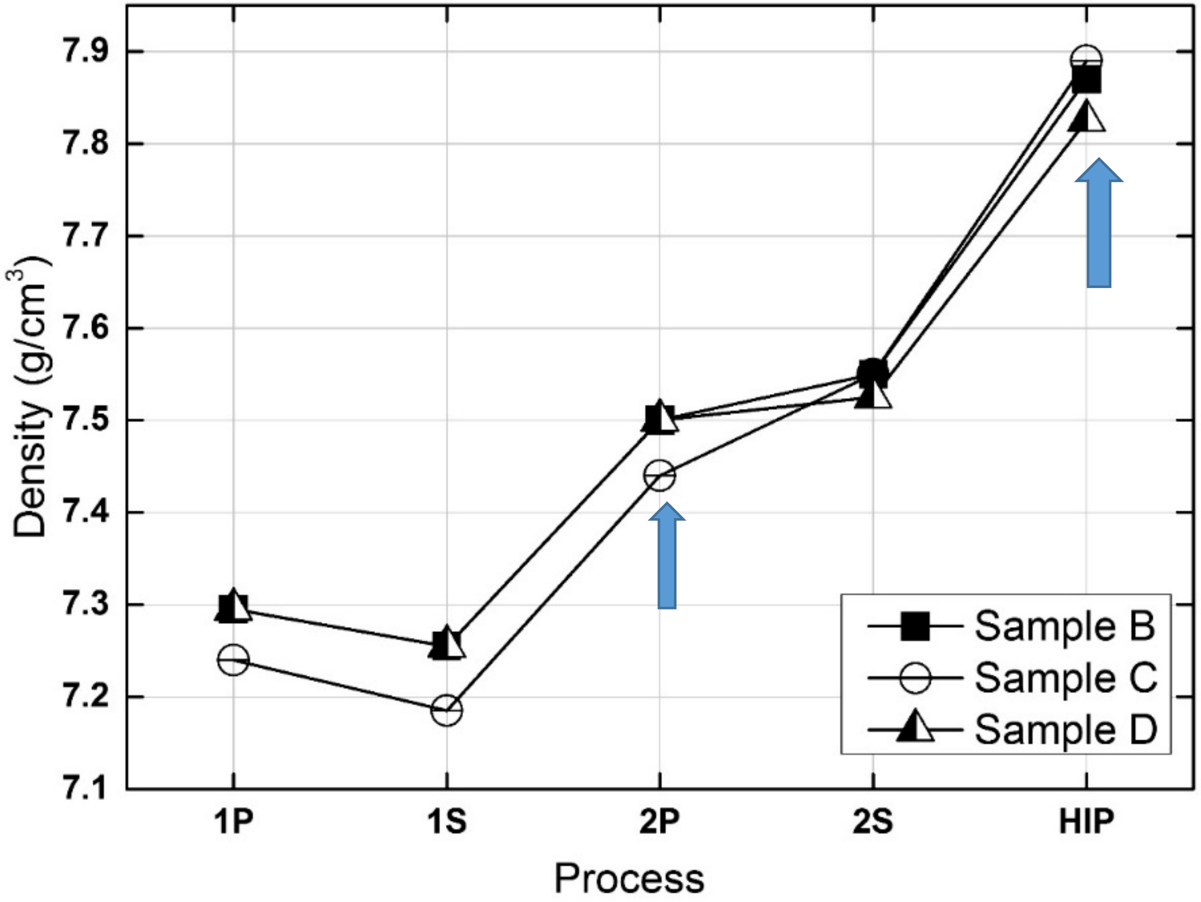
Cylindrical samples





Gear samples





Summary

- High density to close the surface pores after 1300 °C sintering and allows subsequent HIP without capsule.

- Near full density for both processed gears and cylinders.

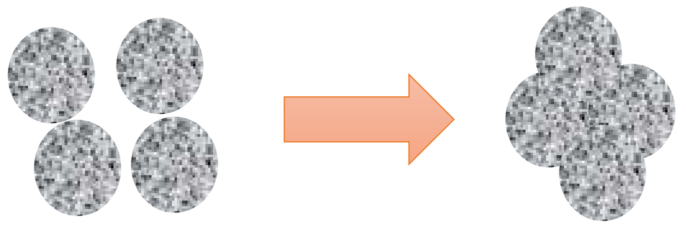
- Potential route for making fully dense PM parts.

- Neutral zone problems persists, that can be optimised through the geometrical optimisation.

4.High Velocity Compaction

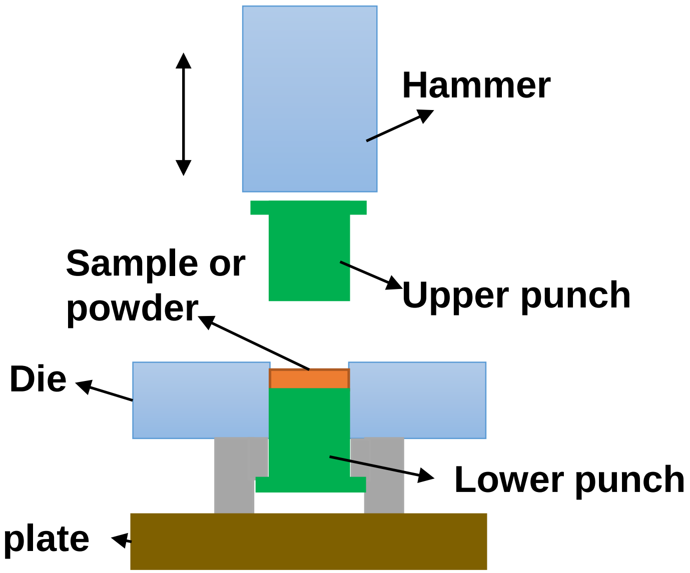
- Densification from intense shock waves
- Uniform density, lower spring back and ejection forces

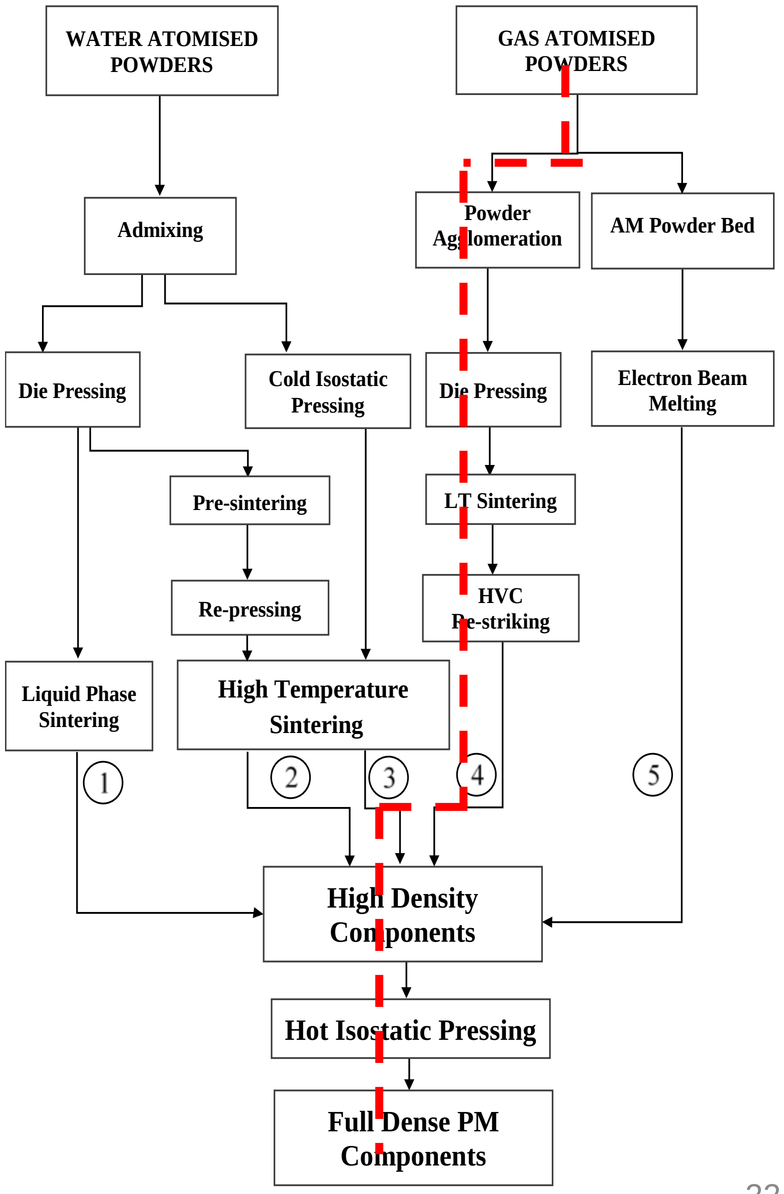
Scanpac Process



Gas atomised powders   Agglomerated using binders

HVC





Material and process

- 100Cr6 Gas atomised powder from Carpenter Powder Products AB
- Powder agglomeration through Scanpac process developed by Bofors Bruk AB

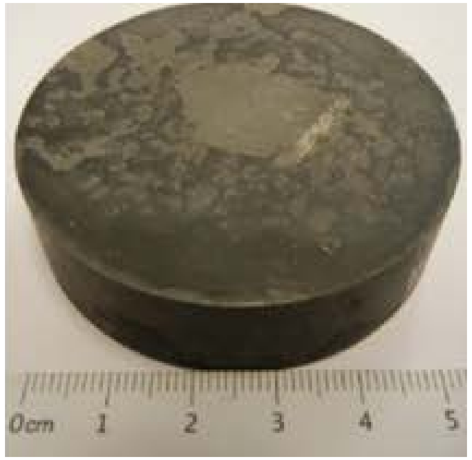
In wt.%	C	Si	Mn	Cr	Co	Fe
100Cr6	0.98	0.5	0.4	1.5	0.2	Bal

P ⇨ S ⇨ HVC ⇨ HIP

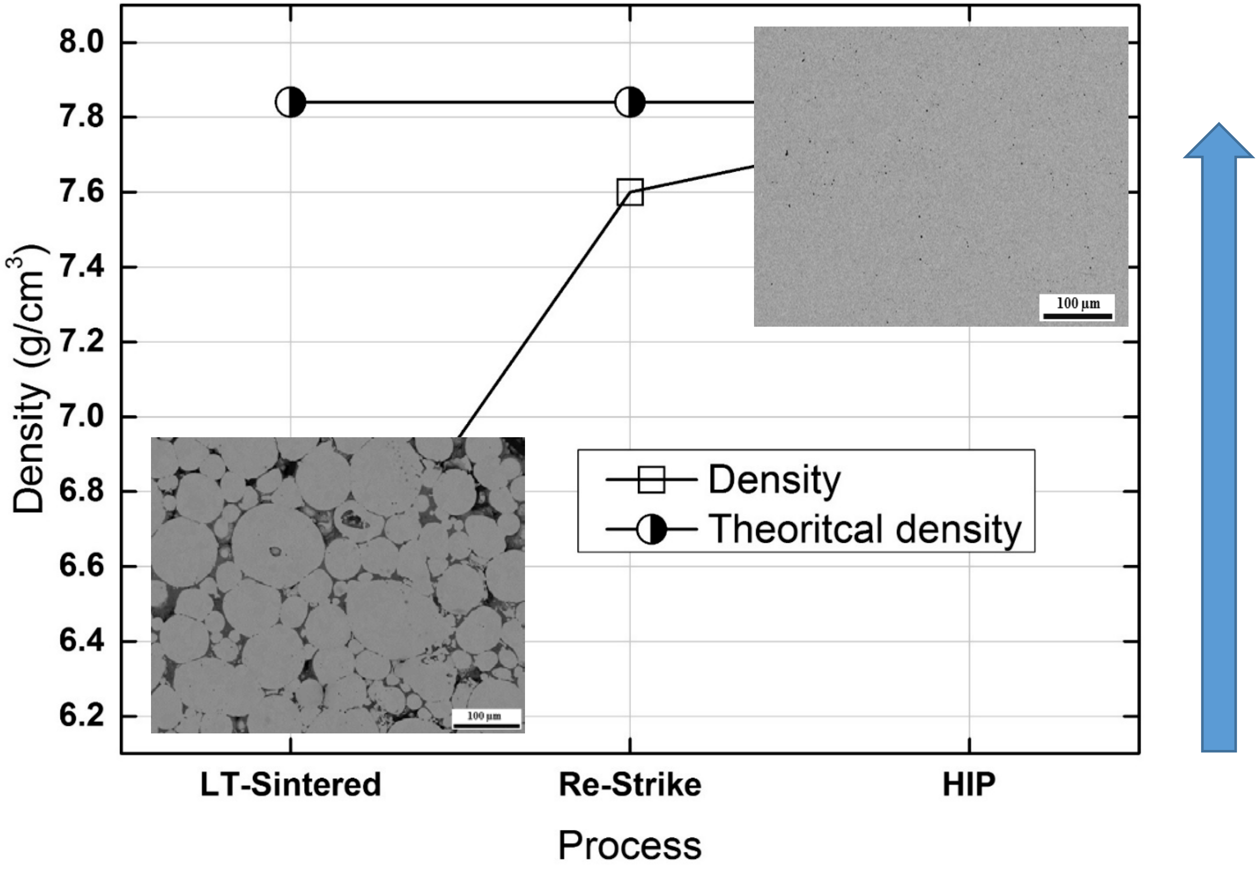
S: 1120 °C for 4h

N_2-H_2

HIP: 1000 bar,
1150 °C for 2 h



Density



Magnusson H, Frisk K, Vattur Sundaram M et al. World PM 2016.

Summary

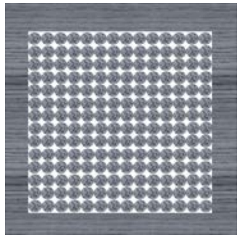
- HVC route using agglomerated powders up to density levels of ~97% of the theoretical density with the surface pore closure.

- Near full density ~99.4% is then reached after HIP without any capsule.

- Further control of sintering atmosphere is needed for efficient reduction of oxides.

5.Additive Manufacturing

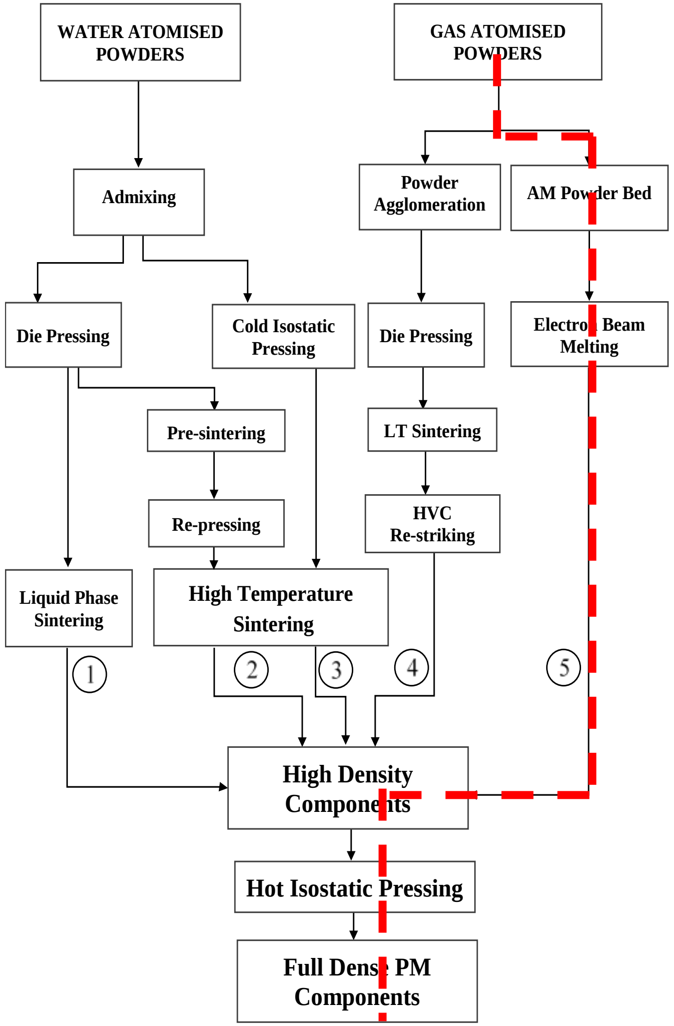
- Powder filled in shell structures for HIP
- Complex-shape full density components
- To improve productivity
- Ti-6Al-4V gas atomised powders (45-105 µm)



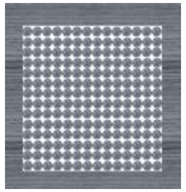
Diameter and height 24 mm

Shell sample filled with powders
With varying wall thickness

Arcam A2 EBM from Mid-Sweden University

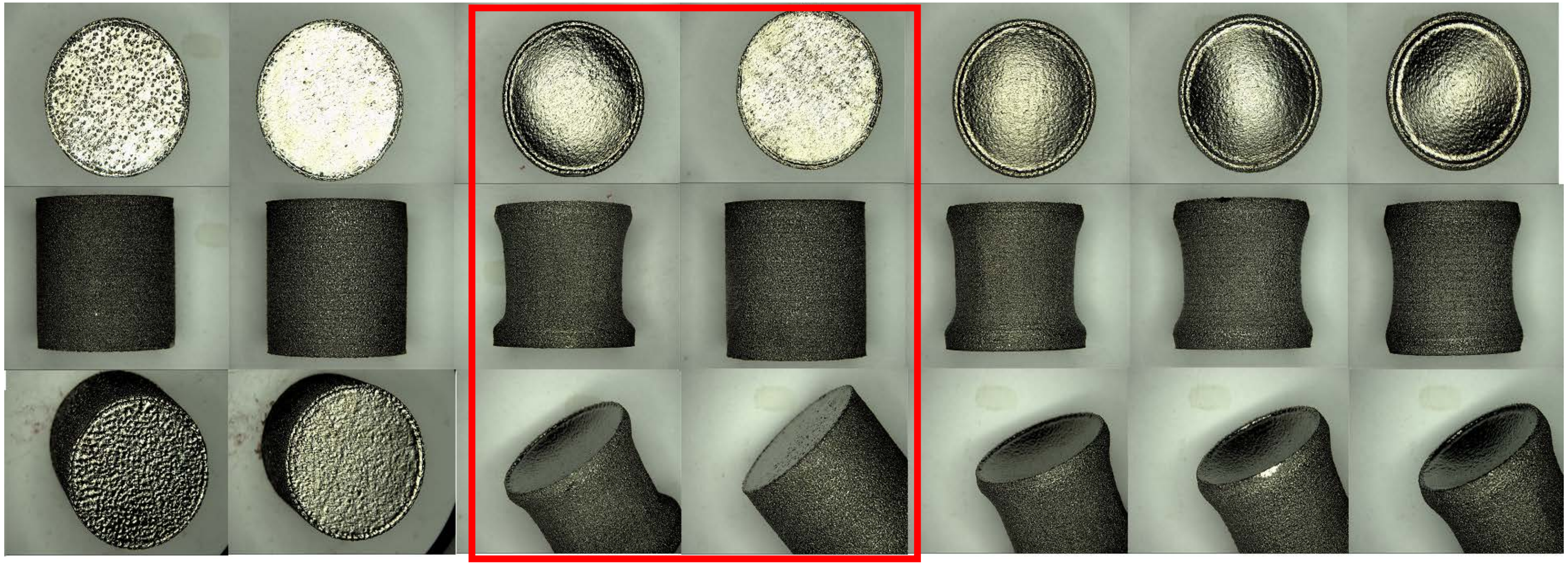


Shell structures after HIP



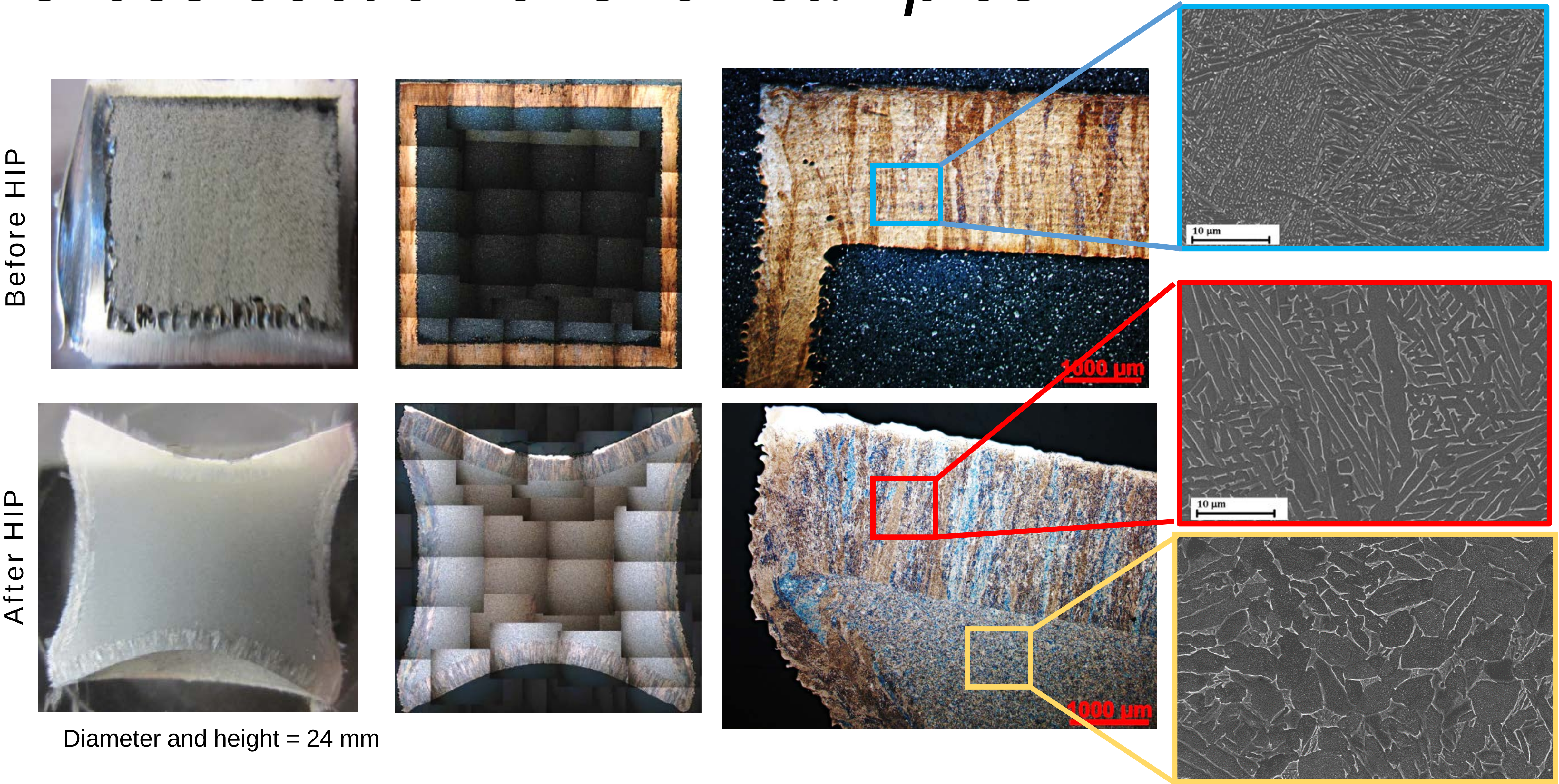
Diameter and height = 24 mm

0.5 mm ***0.7 mm*** ***0.9 mm*** ***0.9 mm*** ***1.1 mm*** ***1.3 mm*** ***1.5 mm***



Frisk K, et.al World PM 2016

Cross-section of shell samples



Diameter and height = 24 mm

Leicht A, Vattur Sundaram M et al World PM 2016.

Summary

- The concept works for HIP

- Critical wall thickness ~1 mm for pore free shell structure/gas tight

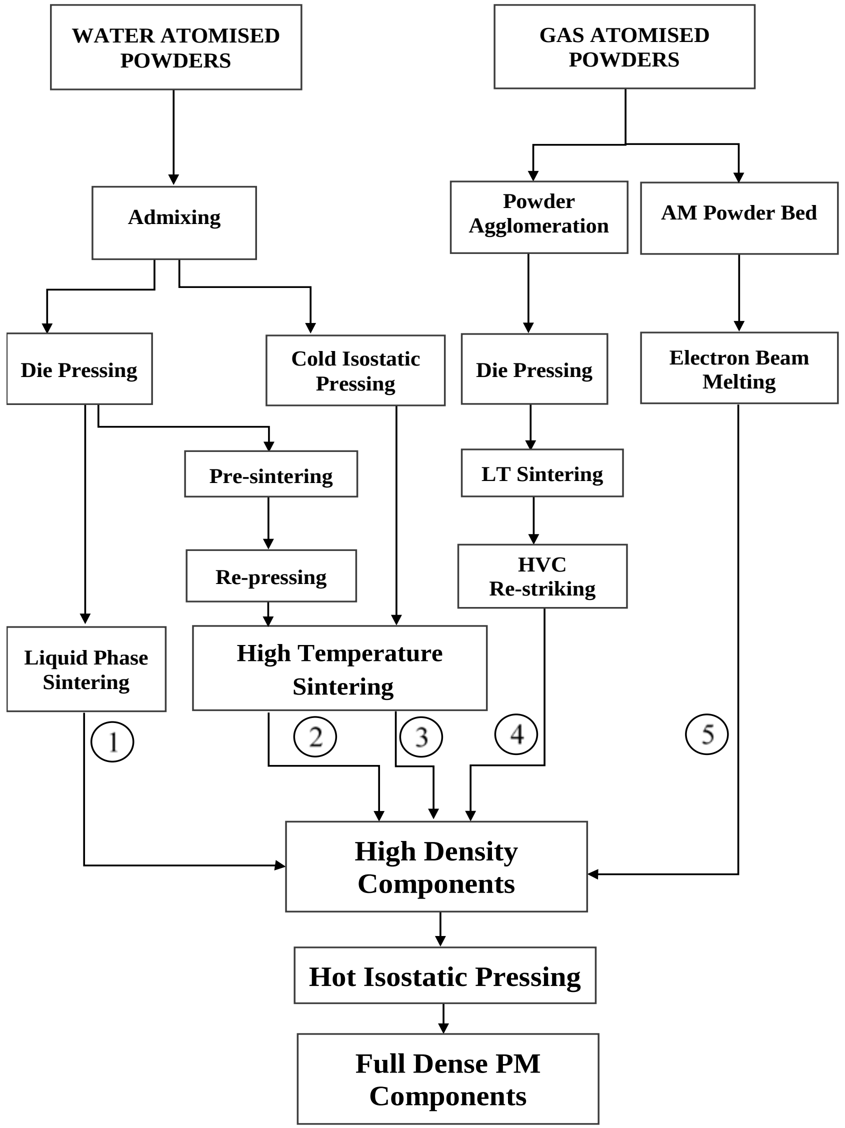
- HIP results in homogenous microstructure, eliminates defects and pores

- Defect free interface between the densified powders and printed shells

Overall Summary

- Full/High density is feasible through all the proposed processes routes.

- Large components, for High performance applications



Acknowledgements

Magnus Ahlfors, Quintus Technologies AB