ANALYSIS OF SELECTIVE LASER MELTING OF RESORBABLE BIOCERAMICS

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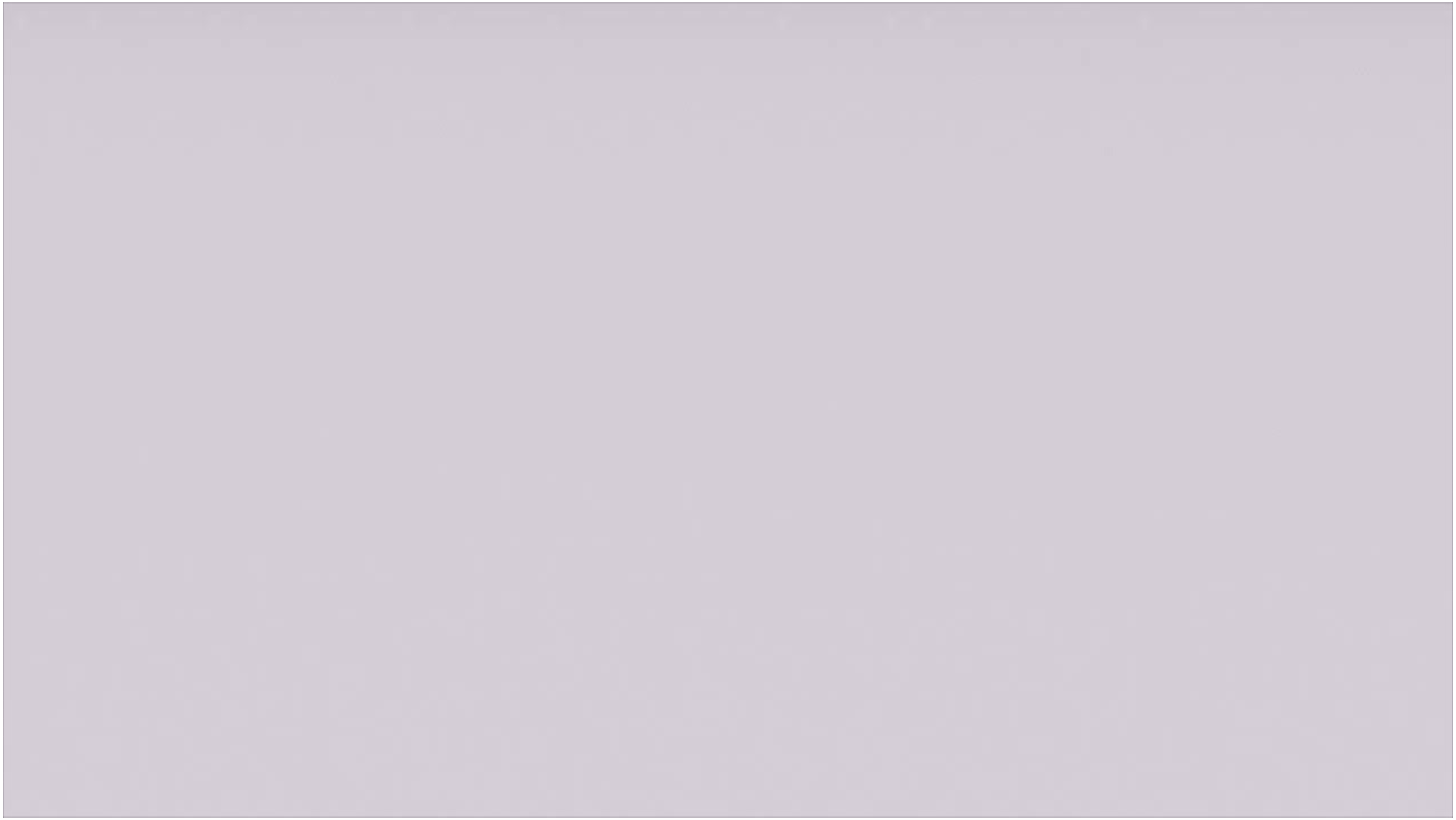
OBJECTIVES OF THE PROJECT

WHAT COULD BE POSSIBLE IN A NEAR FUTURE…



OBJECTIVES OF THE PROJECT

DESIGN/MANUFACTURING OF BIOCERAMIC IMPLANT

- **Better Osteointegration of the Implant (Compared to Metallic Implants)**
- **Implant as a Bone-repair-kit : Induction of Natural Bone Regeneratinon by the Implant**

OrthoFlase Project

Projet ANR-11-TECS-0005:OrthoFlase

SMEs

Project leader

Web-based software and data management

R&D partners

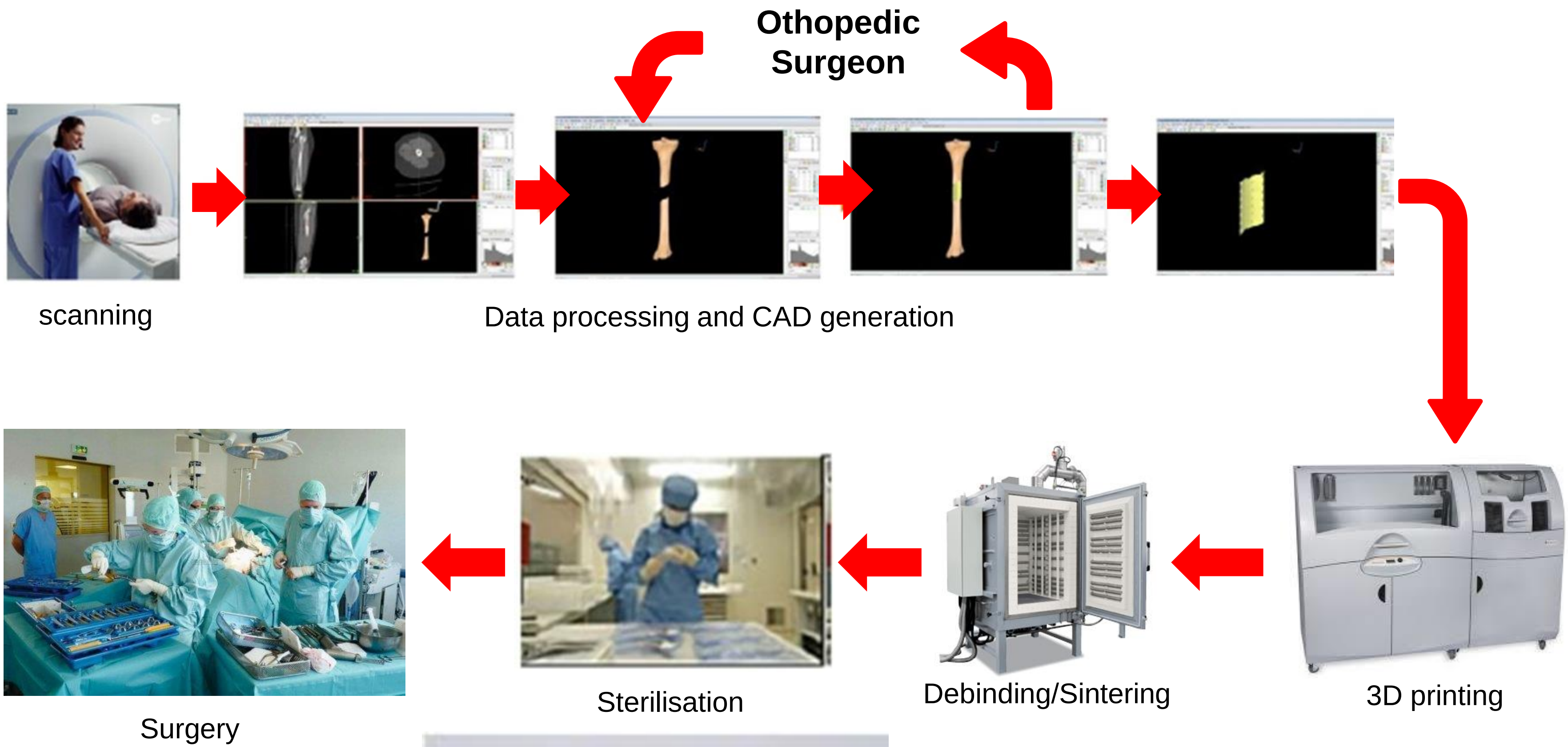
Powder

Biocompatibility

Process development

OBJECTIVES OF THE PROJECT

STATE-OF-THE-ART/EXISTING TECHNOLOGY



- **HAP (Hydroxyapatite)**
- **TCP Tricalcium Phosphate**

Miguel Castilho & al., Biofabrication 5 (2013) 035012 (12pp)

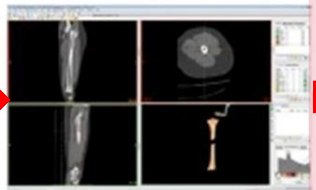
OBJECTIVES OF THE PROJECT

R&D AND INNOVATIONS PROPOSED

Web-based CAD/CAM and MMI

Othopedic Surgeon

scanning



Data processing and CAD generation

Direct Manufacturing

SLM of HAP or TCP

Sterilisation

Growing factors, bone cells

Bio-active implant

Surgery

+ Production of very pure TCP powder of SLM (very low content of metallic elements)

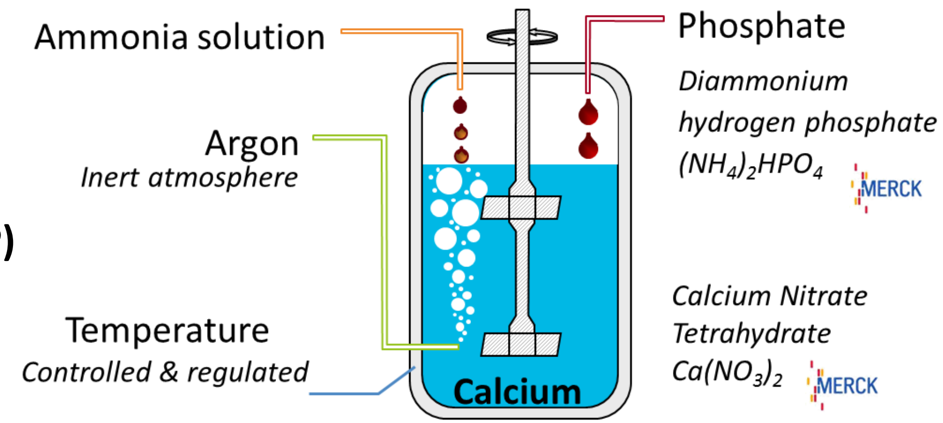
TCP POWDER MANUFACTURING

MANUFACTURING PROCESS OF TRICALCIUM PHOSPHATE Β (Β-TCP)

- *Chemical synthesis of Apatitic Tricalcium Phosphate*
- *Conversion into TCP*
- *Synthesis of macro-powder by spraying*

SYNTHESIS OF APATITIC TRICALCIUM PHOSPHATE (TCP AP)

Aqueous precipitation

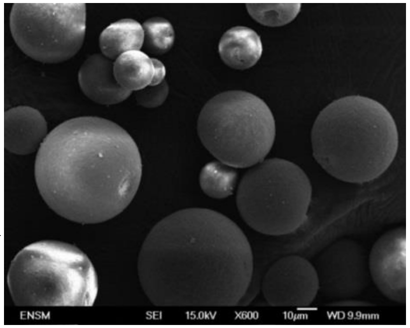


Powder synthesis → Filtration → 1st Heat -Treatment → Grinding → 2nd Heat -Treatment → Spray Drying

TCP ap

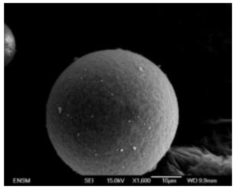
$6\,(NH_4)_2HPO_4 + 9\,Ca(NO_3)_2 + 6\,NH_4OH \rightarrow Ca_9\,(PO_4)_5\,(HPO_4)\,(OH) + 18\,NH_4NO_3 + 5\,H_2O$

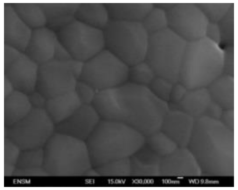
$Ca_9(HPO_4)(PO_4)_5OH,\ nH_2O \rightarrow 3\,Ca_3(PO_4)_2 + (n+1)H_2O$



β-TCP

- **Ultra pure composition (TCP >99.5% wt., Metals <50ppm)**
- **Dense and spherical agregates**
- **Size distribution: $1 \leq Ø \leq 50$ µm**

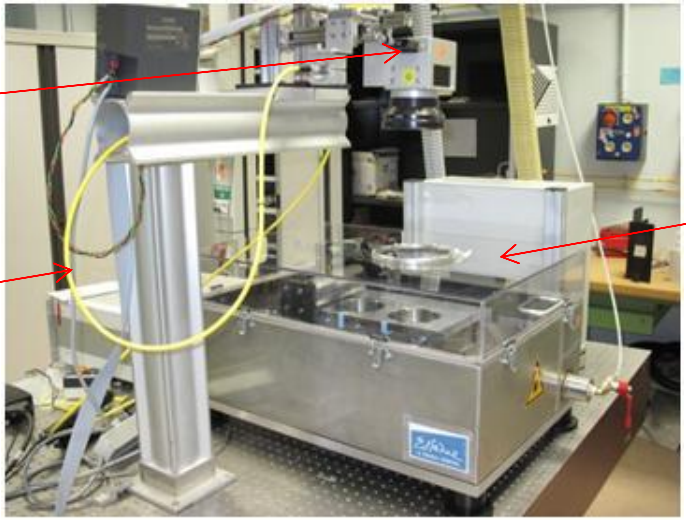


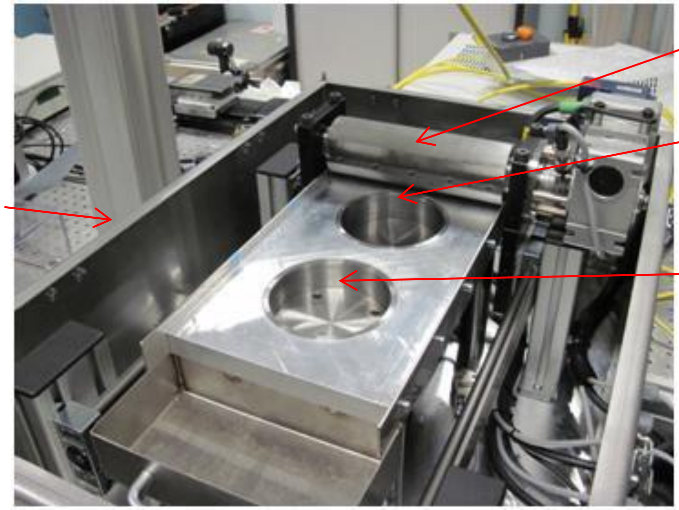


Demonstrator Setup

SLM demonstrator system

- Laser : CW (100W), CO_2 (50W) (nanoseconde fiber laser, CW Nd:YAG, NIR diode : available)
- Manufacturing Chamber : Ø100mm, stainless steel with HAP

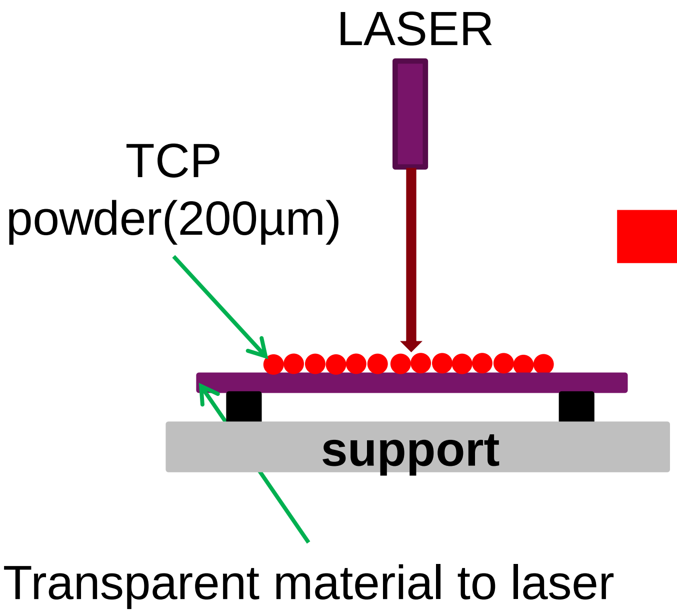




Process parameter search

- Instrumentation with high speed camera
- Fusion lines
- Main parameters : Power, Scanning speed, Layer thickness, Laser spot diameter,…

ABSORBTIVITY OF TCP POWDER :STATIC TEST



- **838nm (Thalès laser diode) (spot ~1mm)**
  - **No abosption at 50W**
- **1,064µm (laser Nd:YAG 3kW Lamp pomped) (spot diam ~1,5mm)**
  - **No absorption up to 750W**
- **10,6 µm (CO2 laser 50W max)**
  - **Fusion at few Watts**

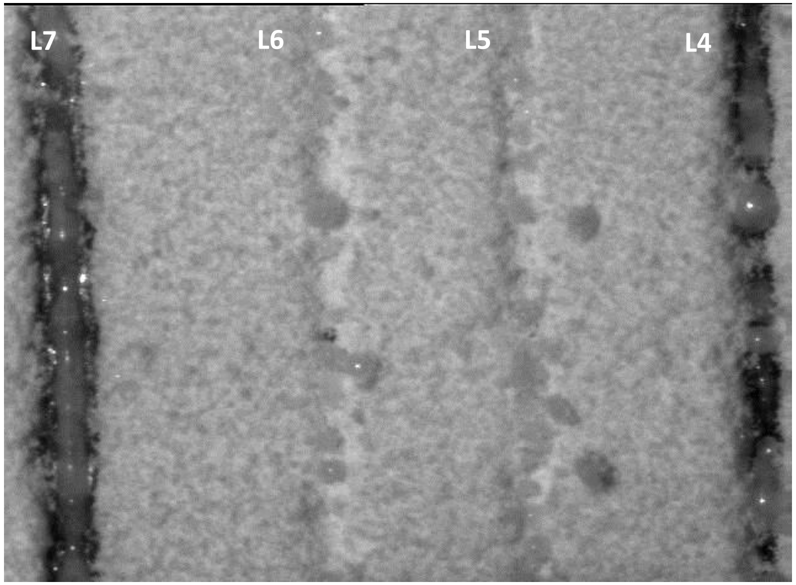
- ✓ **Study with CO2 laser possible**
- ✓ **For NIR lasers (1,064µm,1.07µm,838nm,…) : need to modifiy the powder…**

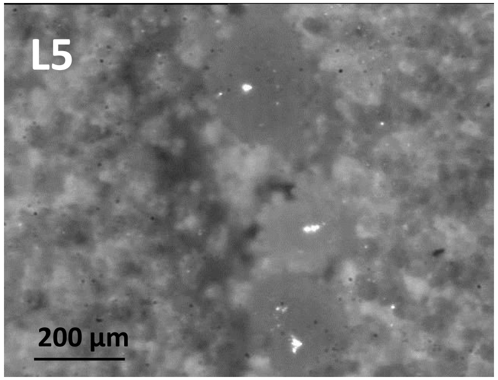
SLM PROCESS STUDY : CW CO_2 LASER

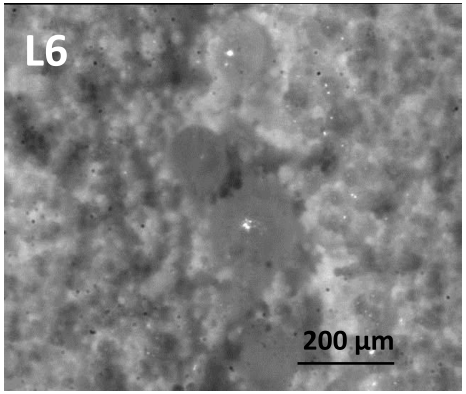
Initial trials

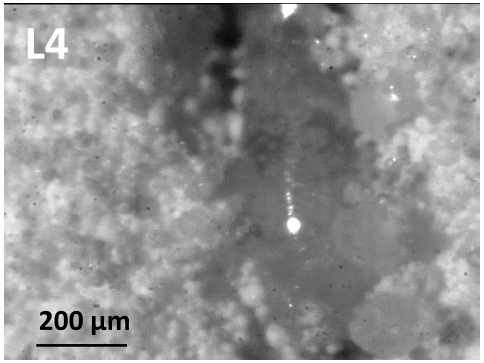
Powder thickness : 200µm
Support : HAP coating

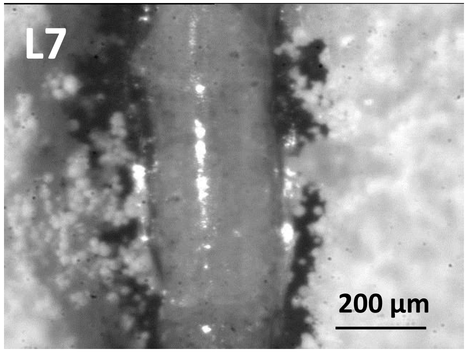
Trial	Power	Speed		Foacal spot diam.	T° heating
TCP_CO2_C1_L4	52 W	0,1 m/s	6 m/min	200 µm	25°
TCP_CO2_C1_L5	52 W	0,25 m/s	15 m/min	200 µm	25°
TCP_CO2_C1_L6	52 W	0,25 m/s	15 m/min	200 µm	25°
TCP_CO2_C1_L7	52 W	0,05 m/s	3 m/min	200 µm	25°











Results :

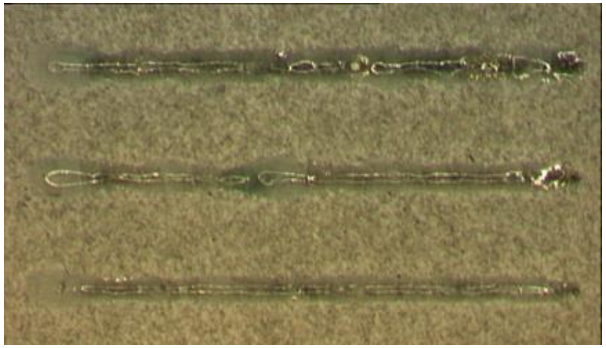
- Fusion in dropplets for highest speed
- No enough energy density to get stable fusionTCP
- Need to decrease speed

SLM PROCESS STUDY : CW CO_2 LASER

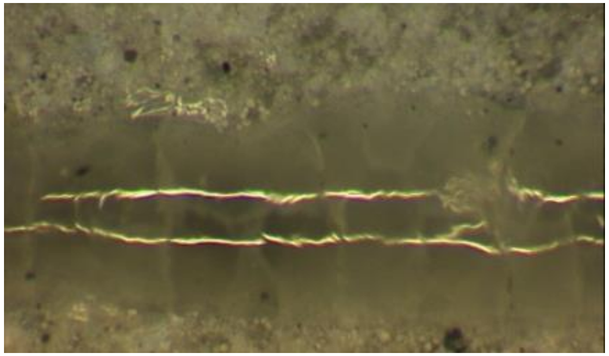
Reducing travel speed

Trial	Power	Speed	Focal spot diam.	Width
TCP_CO2_C1_L1	52 W	1 m/min	200 µm	~300µm
TCP_CO2_C1_L2	52 W	1.5 m/min	200 µm	~250µm
TCP_CO2_C1_L3	52 W	2 m/min	200 µm	~200µm

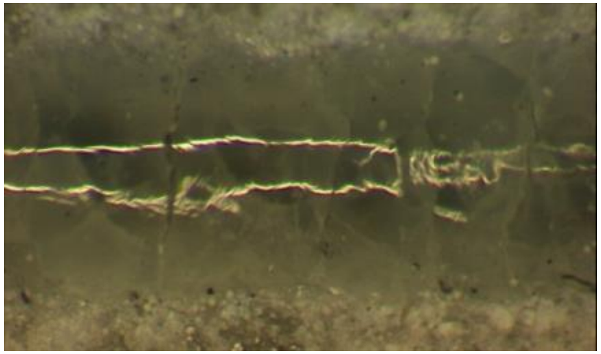
Powder thickness : 200µm
Support : HAP coating



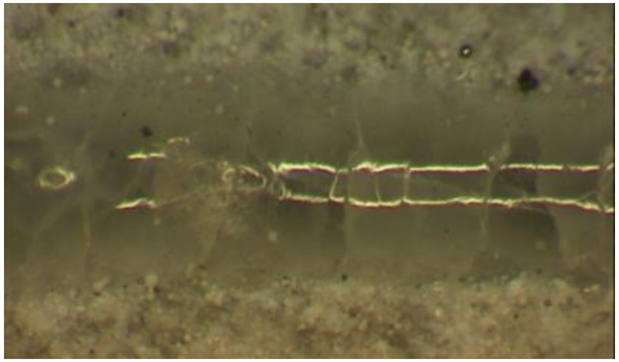
overview



L1 param



L2 param



L1 param

Results

- **Generation of continuous and stable fusion of TCP**
- **Numerous cracks** → **Heating of manufacturing chamber**
- **Further process parameter optimization** → **Need to decrease powder thickness, increase travel speed andlaser power…**

ADDITION OF PARTICLES TO TCP POWDER

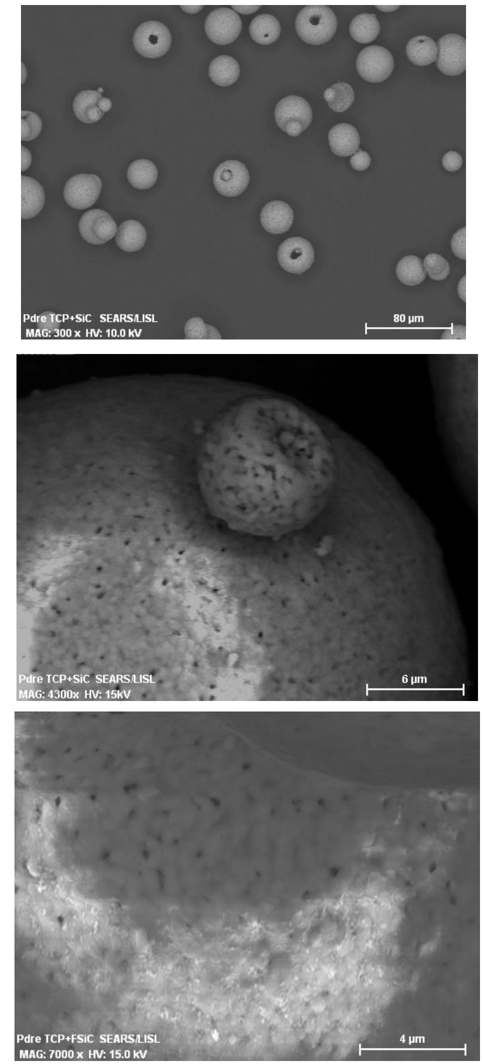
- **Biocompatibility**
- **Sub-micro,nano**

TiC,SiO_2, SiC ….

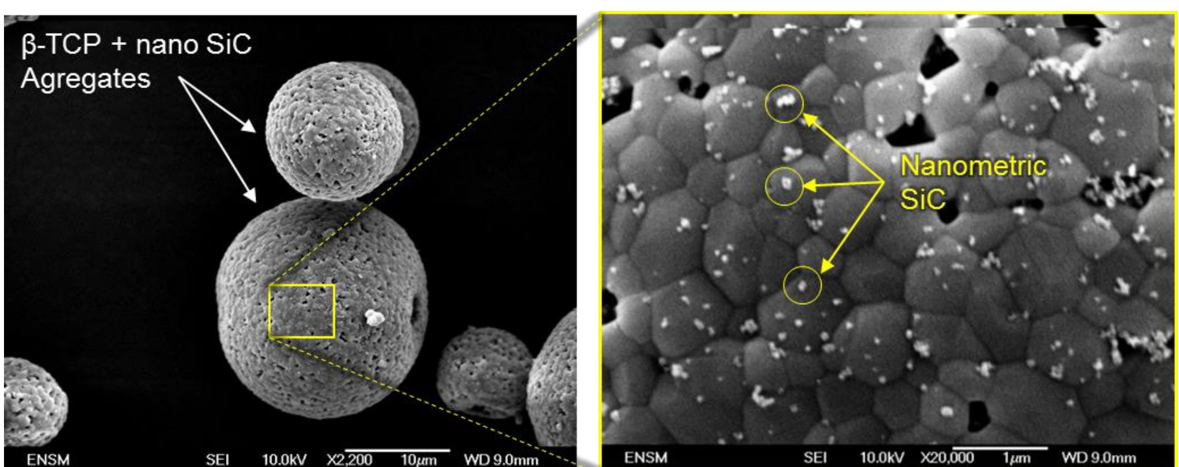
B-TCP+nano SiC

- First method
  - Use of β TCP powder
  - Mechanical mixing with nano-SiC powder
- Second method
  - Co-spray drying SiC - β TCP

- Elaboration of dense and spherical agregates
- Particle size distribution: **1 ≤ Ø ≤ 50 µm**



Mechanical mixing



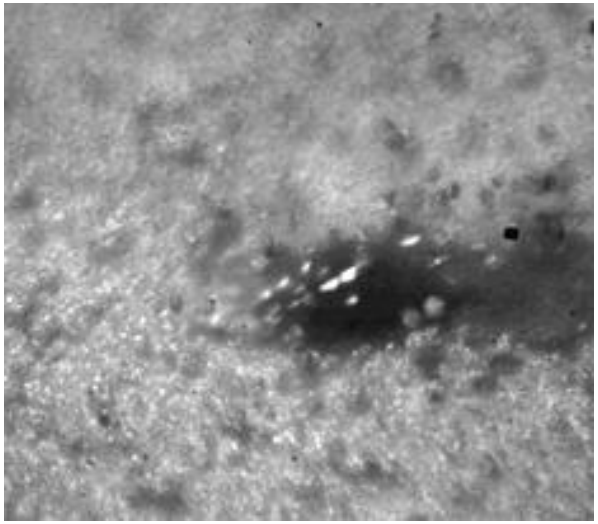
Co-spraying

SLM PROCESS STUDY : CW FIBER LASER

1. PARAMETER SEARCH FOR 2D LINE ON SUBSTRATE

- HAP coating stainless steel plates
- Power : 30-100W
- Focal spot diameter: ~100µm
- Scanning speed : 1-200mm/s

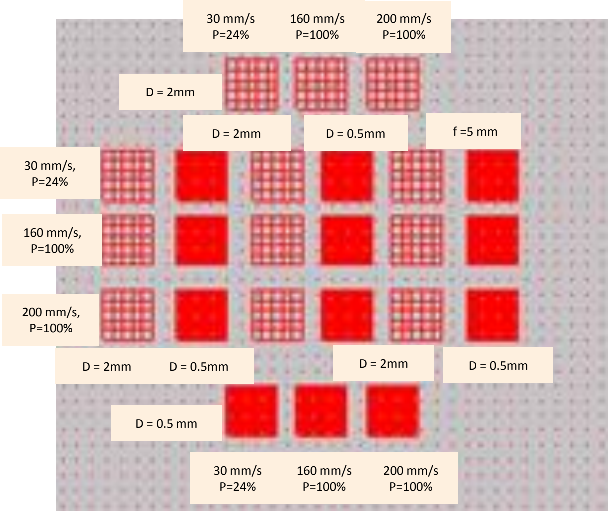
- Selection of best fusion profile (stability)



TCP+SiC (f=10kHZ, P=50W, V=100mm/s)

2. PARAMETER SEARCH FOR 2D SURFACE

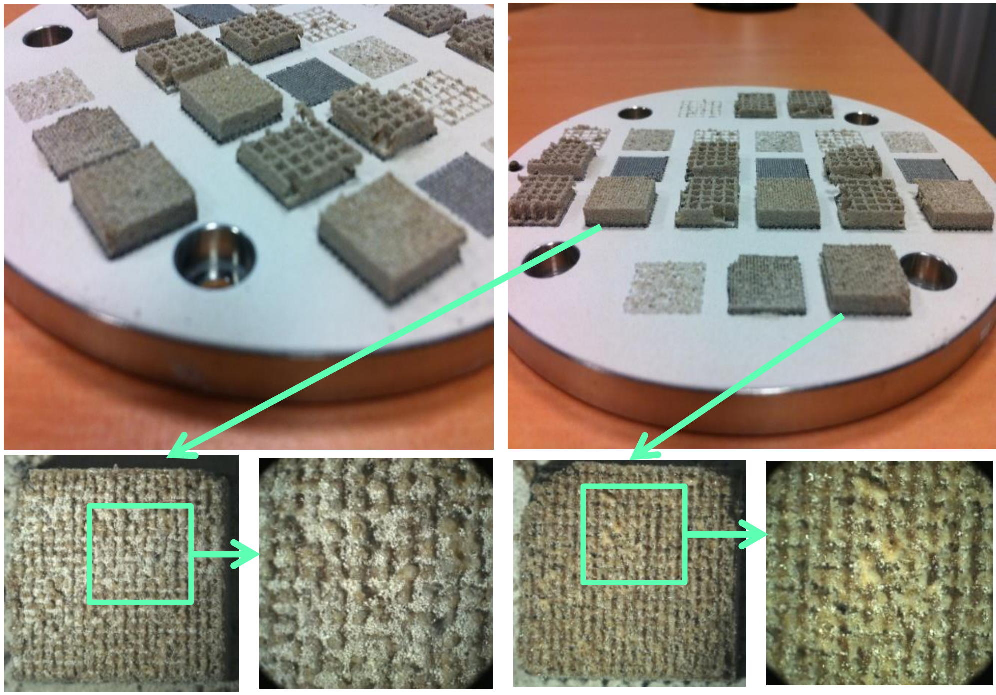
- Power : 30-100W
- Focal spot diameter: ~100µm
- Scanning speed : 100-200mm/s
- Distance between 2D line : 50%-100% of line width and grids

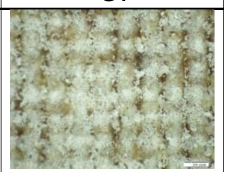
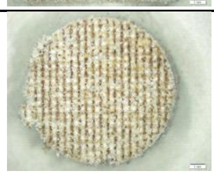
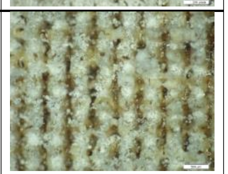
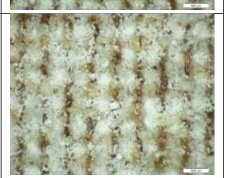


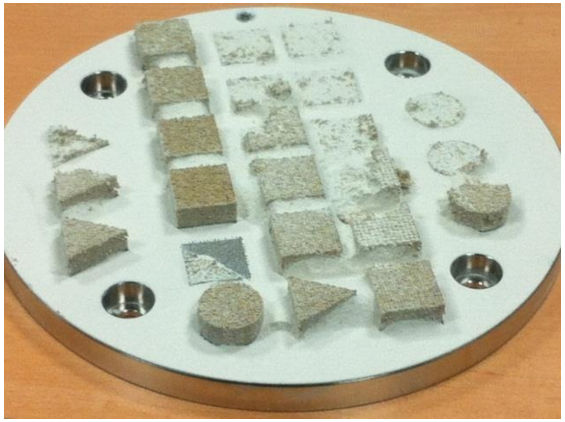
SLM PROCESS STUDY : CW FIBER LASER

3. BUILDING 3D SIMPLE GEOMETRIES

- Squares, cylinders,…
- Focal spot diameter: ~100µm
- Scanning speed : 1-200mm/s

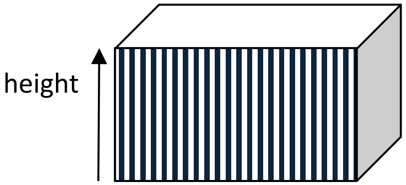


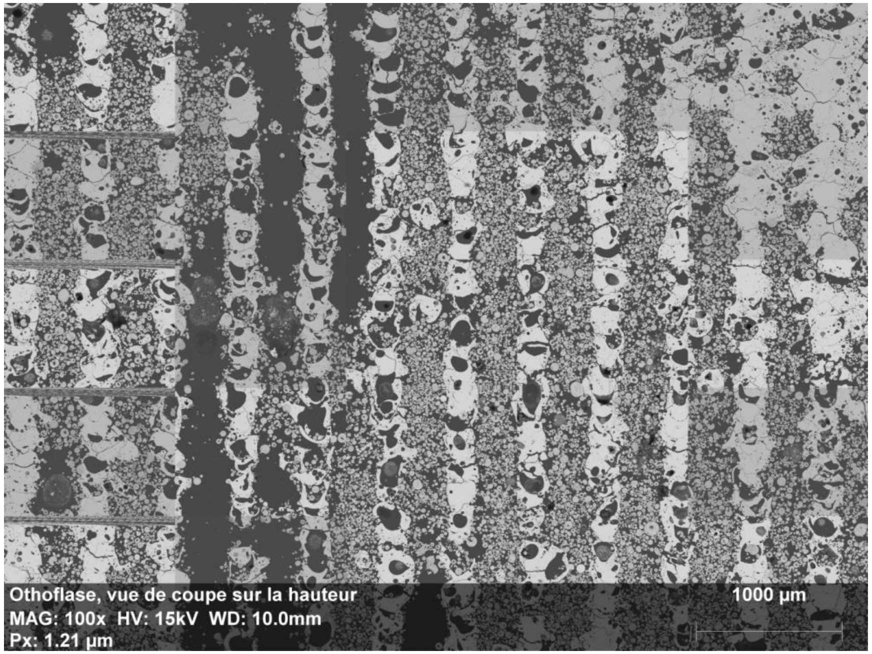
Sample	G1.2	G4
200mm/s – Ec=0.5mm - carré		
200mm/s – Ec=0.5mm - cylindre		
200mm/s – Ec=0.5mm - triangle		
220mm/s – Ec=0.5mm		

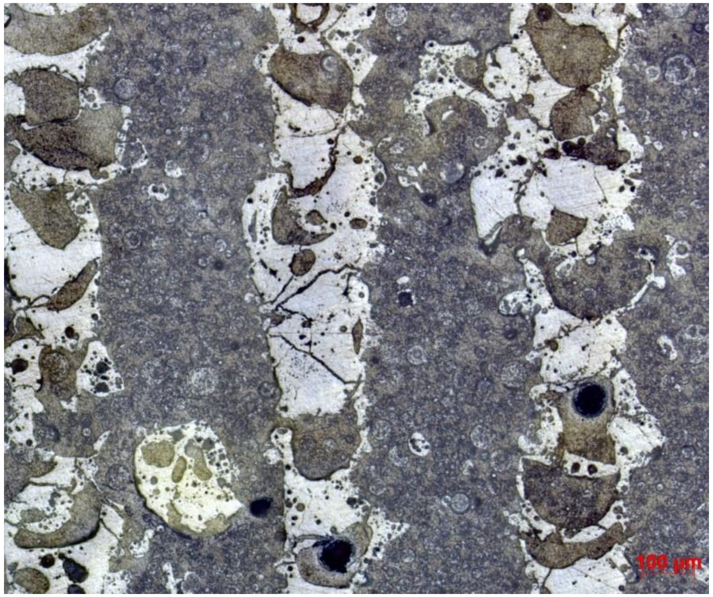


SLM PROCESS STUDY : CW FIBER LASER

MATERIAL ANALYSIS



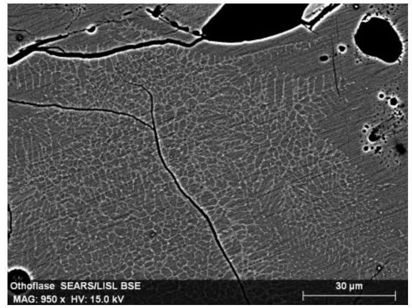


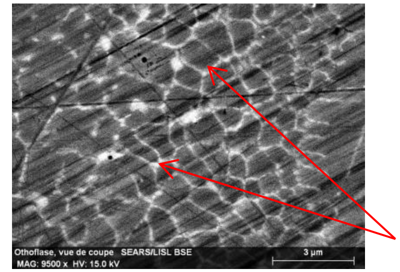


SLM PROCESS STUDY : CW FIBER LASER

MATERIAL ANALYSIS

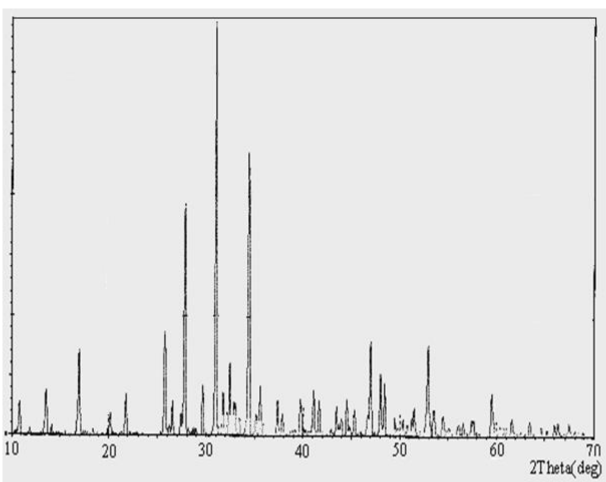
Detail of structure



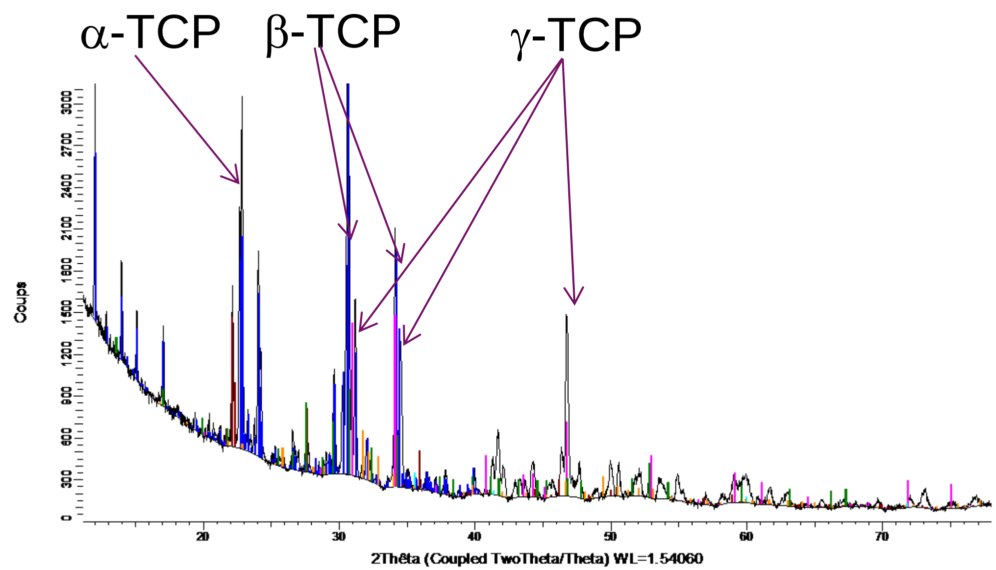


Detail of the microstructure

Rich Ca,O zone



Pure β-TCP XRD spectrum



XRD spectrum

CONCLUSION AND PERSPECTIVES

Process parameter search

1. CO2 laser : Continuation of process parameter search for 3D part manufacturing
   - Decrease powder thickness
   - Increase of travel speed
   - Decrease of cracks
   - Manufacturing of 3D parts
2. Fiber laser
   - New additional material
   - Optimization of mixture (TCP+add)
3. Material study
   - Composition
   - Fraction of B-TCP, HAP, TTCP,…
4. Manufacturing complex part

Biological tests

1. 3D samples
2. Study of porosity of the sample