Doctoral Program in Mechanical Engineering
30th August 2021

XIX Brazil MRS Meeting
August 30th to September 03rd
IUMRS ICEM 2021
VIRTUAL

Optimization of zirconia surface textured designs using Nd:Yag laser for biomedical applications

Ana Cristina Marques, Ângela Cunha, Bruno Henriques, Sara Madeira, Diana Faria, Narayan Sahoo, Alexandrina Barbosa, Paulo Pinto, João Pinto, Nathalia Hammes, Óscar Carvalho, Filipe Samuel Silva

Universidade do Minho

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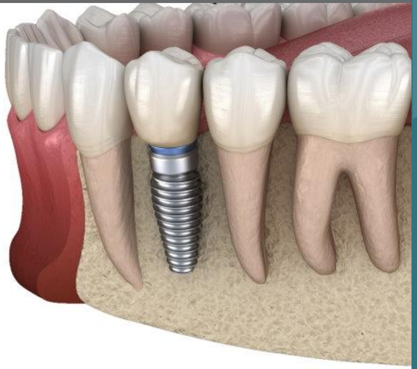
CENTER FOR MICROELECTROMECHANICAL SYSTEMS

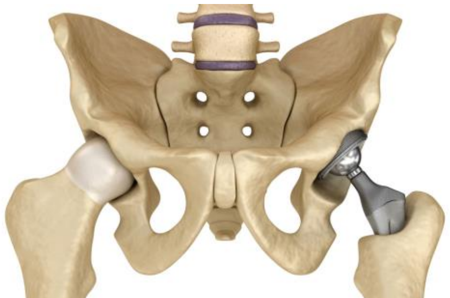
Main Goal



Developing of surface textured designs

Primary stability of surgically anchored implants

Mechanical interlocking between bone and implant body



Summary

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Guideline

Introduction

- Yttria-stabilized Zirconia (Y-PSZ)
- Laser potentialities
- Biomedical applications

Materials and Methods

- Production of Zirconia samples
- Laser texturing
- SEM analysis

Summary

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Guideline

Preliminary evaluation of laser processing conditions

Power, scanning speed and distance between lines

Textures

Laser Blasting

Thin Patterns

Patterns

Grooves

Summary

Guideline

Cellular structures

Laser Machining parameters

SEM analysis

Summary

Guideline

Conclusions

Acknowledgments

Summary

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Introduction

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Yttria-stabilized Zirconia (Y-PSZ)

Bioceramic with …

Ceramic properties

- Translucent
- Biocompatible

Metallic properties

- High hardness
- High wear resistance
- High fracture and flexural strength (900-1200 MPa) (Cionca et al., 2000)
- High coefficient of thermal expansion

Promising alternative to titanium due to …

- High chemical stability
- Aesthetics properties
- Bionertness

Summary

Yttria-stabilized Zirconia (Y-PSZ)

Osseointegration

Biological fixation through continuous bone formation and remodeling toward the implant

The surface properties of the biomaterials are essential for the osseointegration process

Surface modification

- Sandblasting
- Chemical etching
- Laser treatment

(Moon et al., 2016), (Pereira et al., 2020) (Depprich et al., 2008b), Meshramkar et al., 2019; Parithimarkalaignan and Padmanabhan, 2013).

Summary

Laser Potentialities

Laser Surface Texturing

- Normal geometric shapes such as pits, grooves, pillars, cavities, ridges, rips and others
- Operate at high speed values with elevated precision and promote local heat treatment
- Textured surfaces contribute to improve the surface tribological (friction and wear) behavior. Moreovoer, the wettability, osseointegration and bacterial adhesion are enhanced by surface modification.

Summary

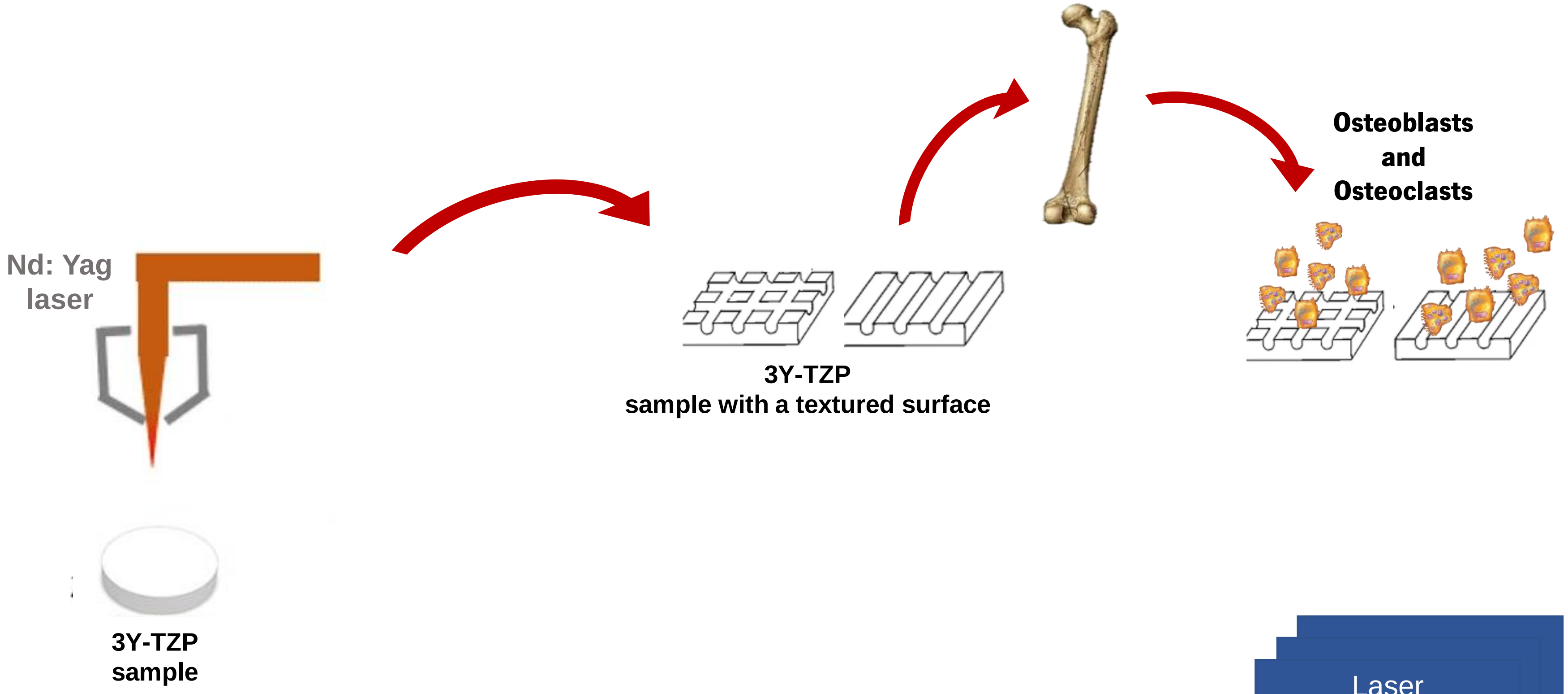
Laser Potentialities

Laser Surface Texturing - Nd: Yag Laser

- ❑ Nd: Yag laser is one of the most used in ceramic surface texturing

- ❑ Pulsed Nd: Yag laser has the capacity to control processing parameters when compared with continuous mode and improve the machining quality

So, the aim of this study is …



Laser Potentialities of Zirconia surface texturing

Summary

Materials and Methods

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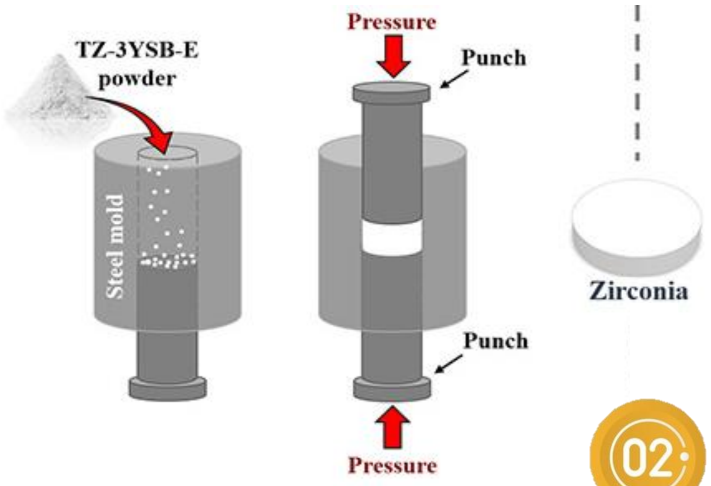
Materials and Methods

01 Production of **green zirconia compacts**



02 Laser beam was focused on the surface of green zirconia compacts

Nd:YAG laser

Output power: 6W
Spot size: 3 μm
Pulse: 35 ns.

Nominal focal length: 160 mm **Wavelength:** 1.064 μm **Pulse energy:** 0.3 mJ/pulse

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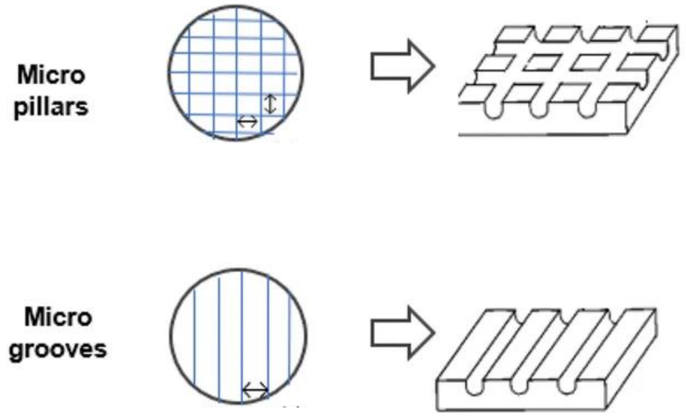
Materials and Methods

04 Design of **crosslines** was defined in a **computer-aided design system**

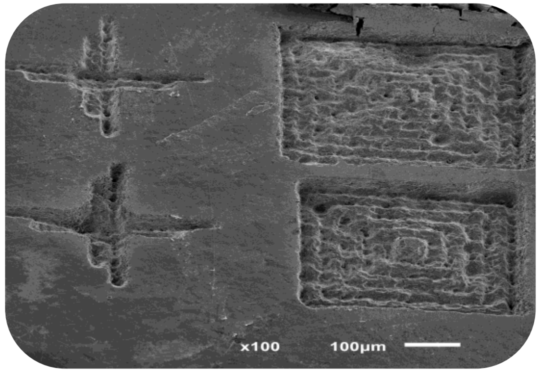


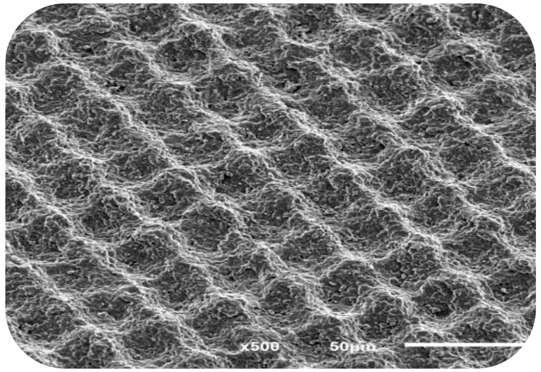
05 **Define several strategies** and different laser parameters (laser power, scan speed, distance between lines and laser passages)

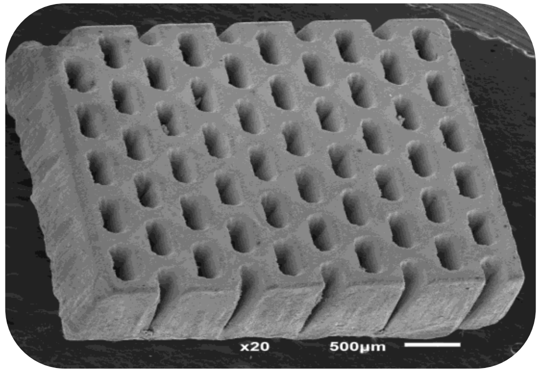
06 **SEM** analysis

Summary

Results







Preliminary evaluation of laser processing conditions

Textures

- Laser Blasting
- Thin Textures
- Textures
- Grooves

Cellular Structures

- Laser Machining

Summary

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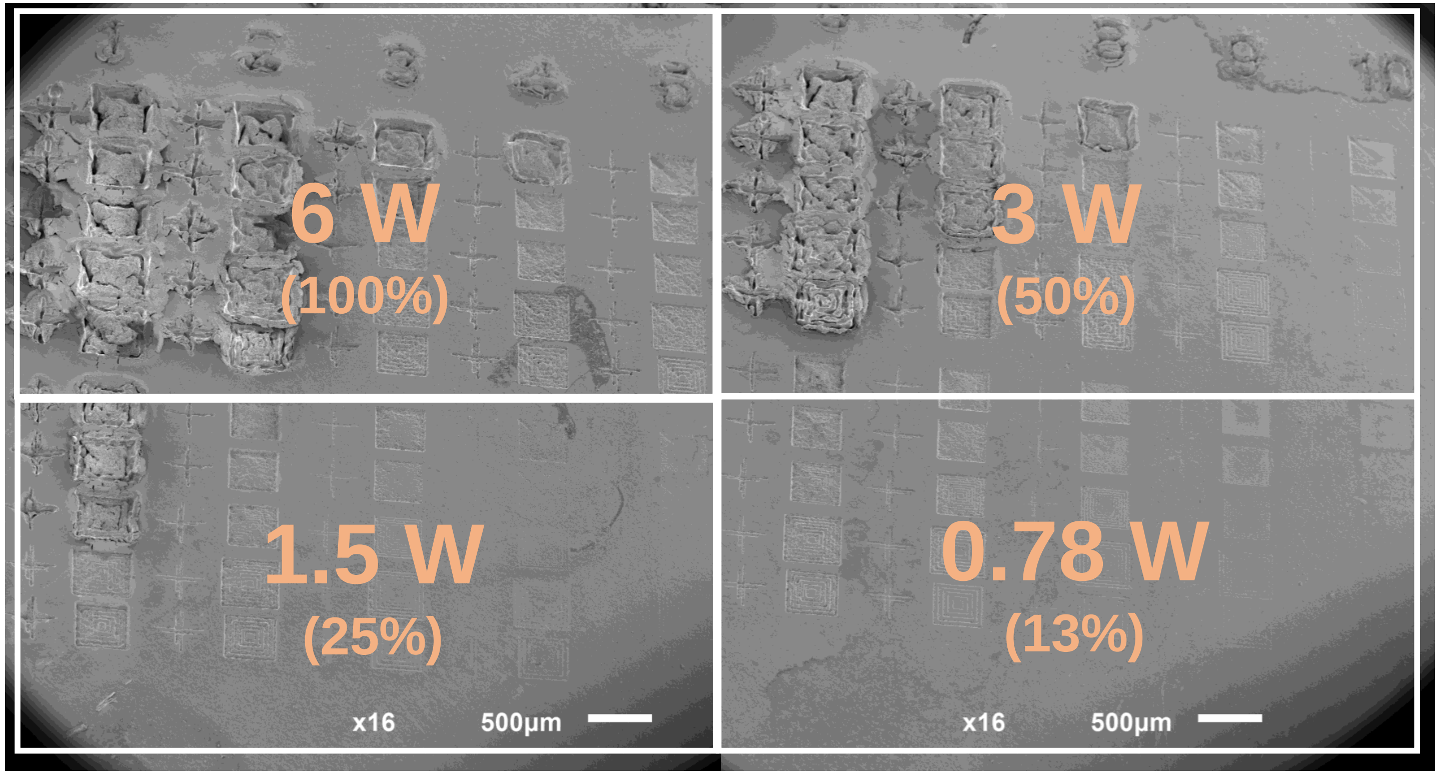
Preliminary evaluation of laser processing conditions

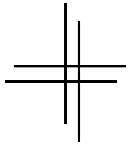
Preliminary evaluation of laser processing conditions

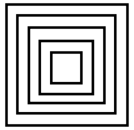
Scanning speed (mm/s): 32, 64, 128, 256, 400, 32, 64, 128, 256, 400

Distance betwen lines (µm): 10, 20, 30, 40, 50, 10, 20, 30, 40, 50



Each condition evaluate …





Summary

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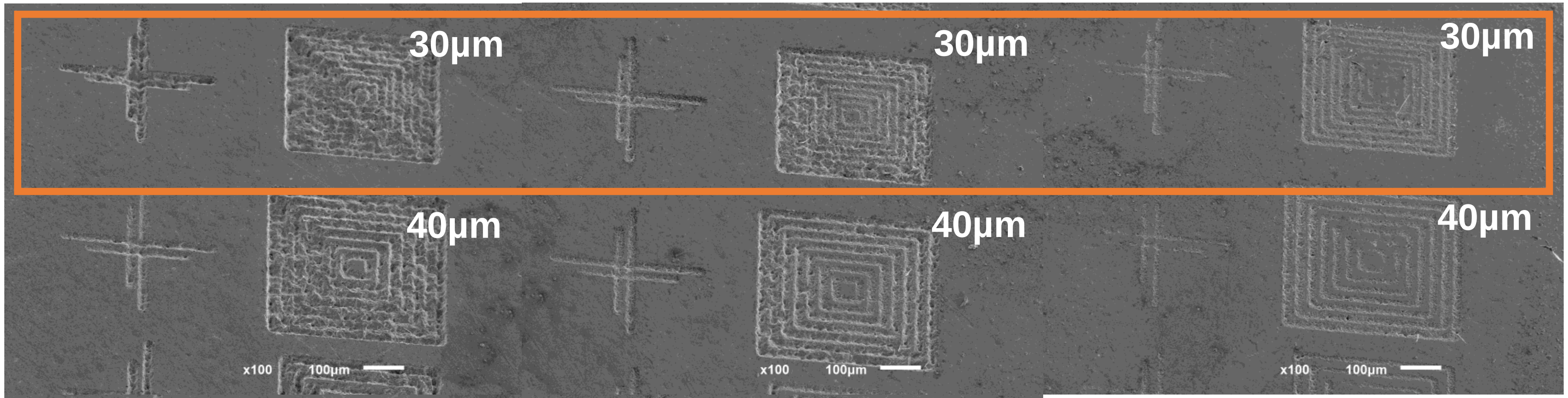
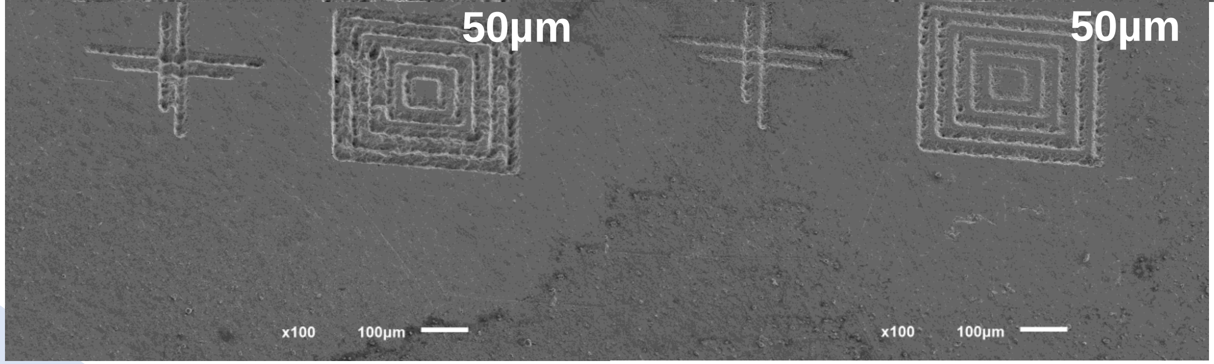
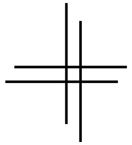
Preliminary evaluation of laser processing conditions

Influence of scanning speed – 0.78 W

Column 6 – 32 mm/s **Column 7 – 64 mm/s** **Column 8 – 128 mm/s**

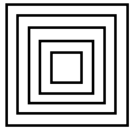



A line spacing inferior to 40 µm (S40) the groove walls overlap and cease to exist.



Lines

A line spacing superior to 30 µm (S30) do not produce holes



Holes

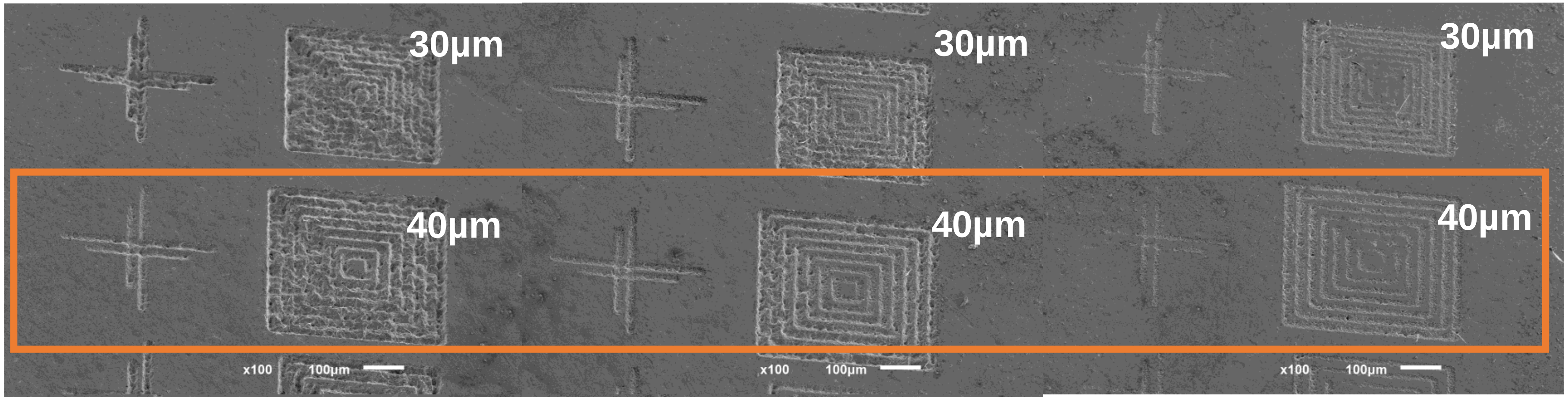
Scan speed

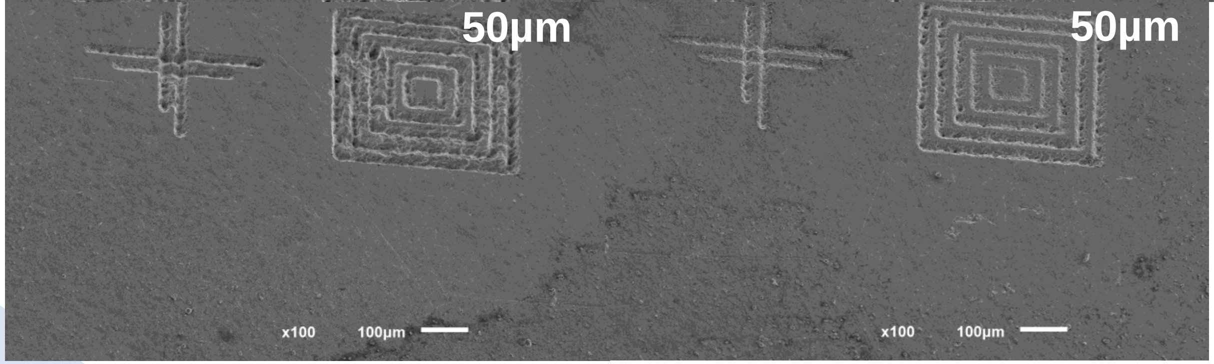
Summary

Preliminary evaluation of laser processing conditions

Influence of scanning speed – 0.78 W

Column 6 – 32 mm/s **Column 7 – 64 mm/s** **Column 8 – 128 mm/s**





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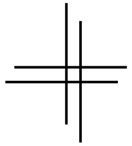

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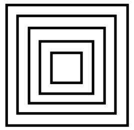

Holes

Scan speed

Summary

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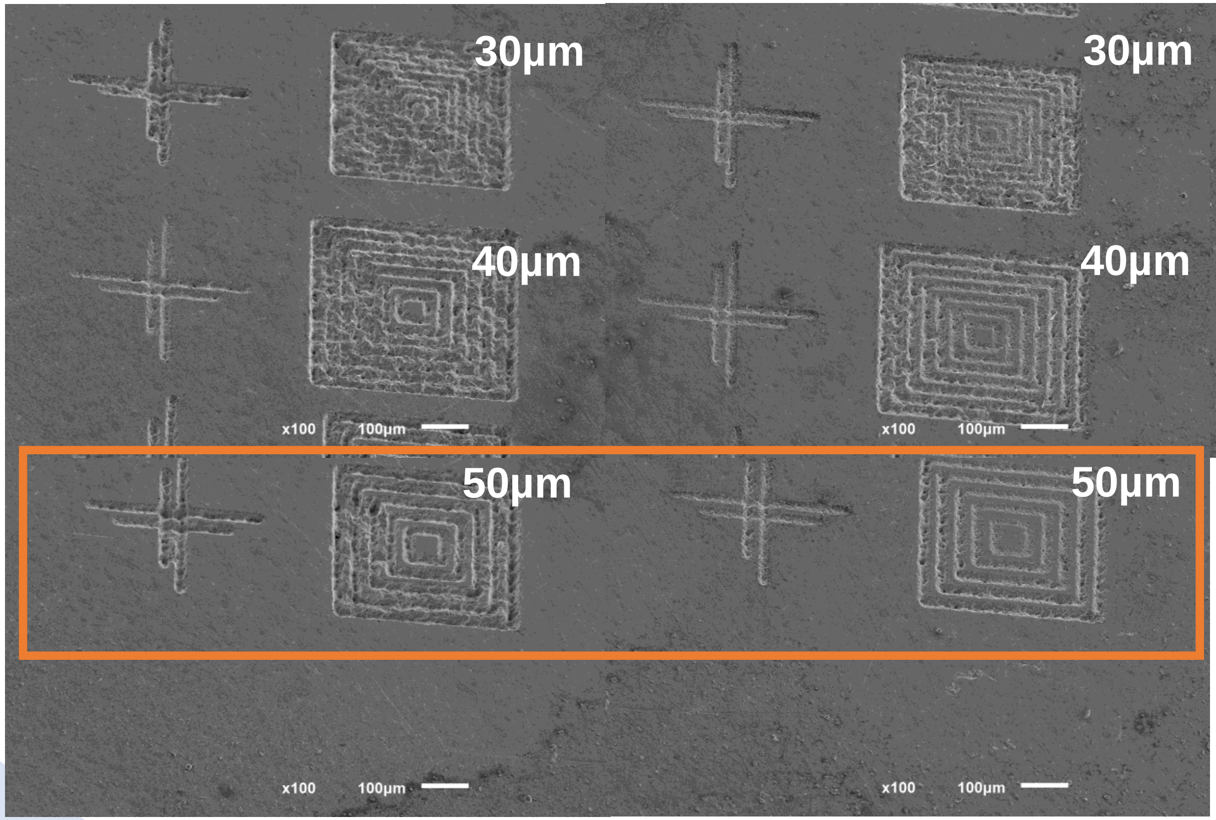
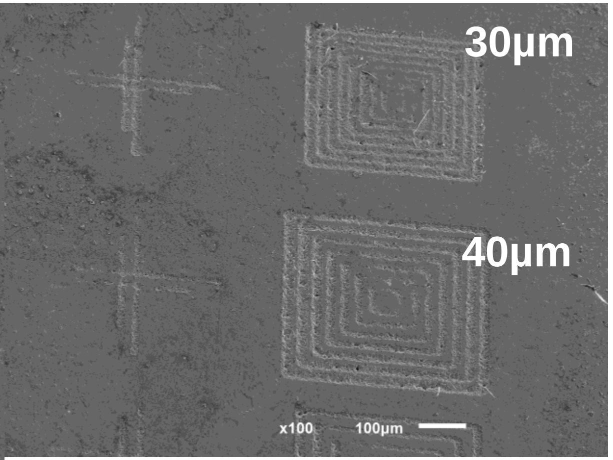
Preliminary evaluation of laser processing conditions

Influence of scanning speed – 0.78 W

Column 6 – 32 mm/s | **Column 7 – 64 mm/s** | **Column 8 – 128 mm/s**





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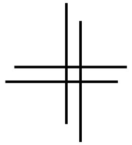

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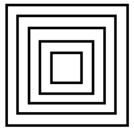


Holes

Scan speed

Summary

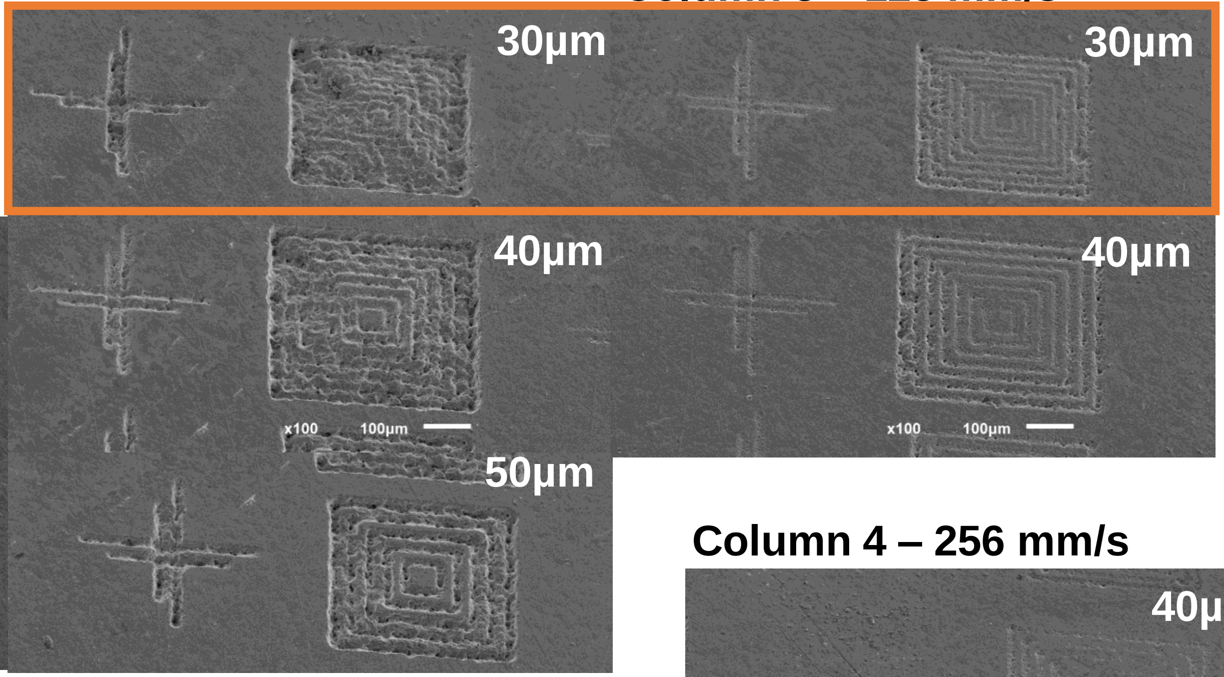
UMINHO CMEMS CENTER FOR MICROELECTROMECHANICAL SYSTEMS

Preliminary evaluation of laser processing cond

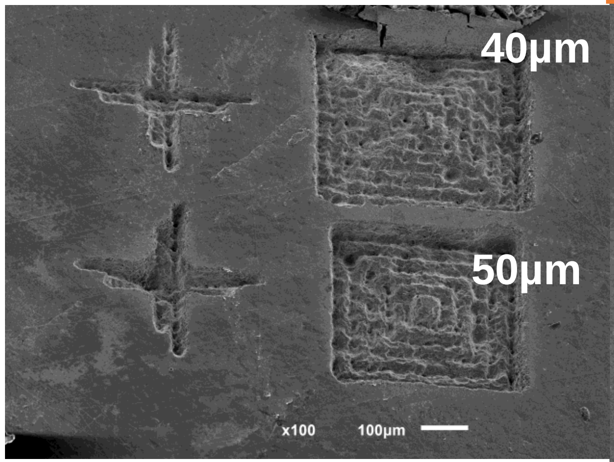
Influence of scanning speed – 1.5 W

Column 2 – 64 mm/s

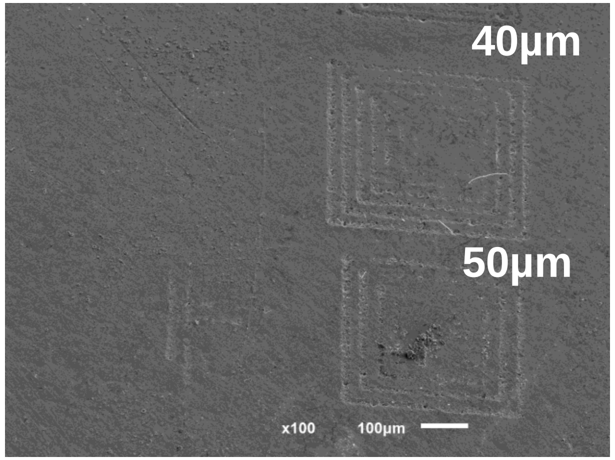
Column 3 – 128 mm/s



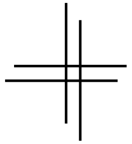
Column 1 - 32 mm/s



Column 4 – 256 mm/s



A line spacing inferior to 40 µm (S40) the groove walls overlap and cease to exist.



Lines

Scan speed

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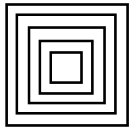

Holes

Summary

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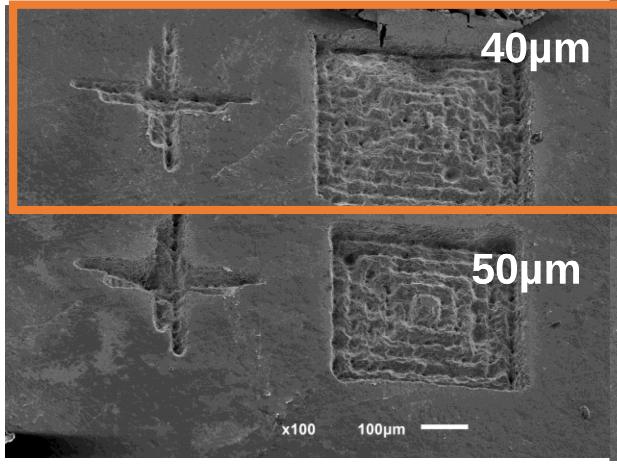
Preliminary evaluation of laser processing conditions

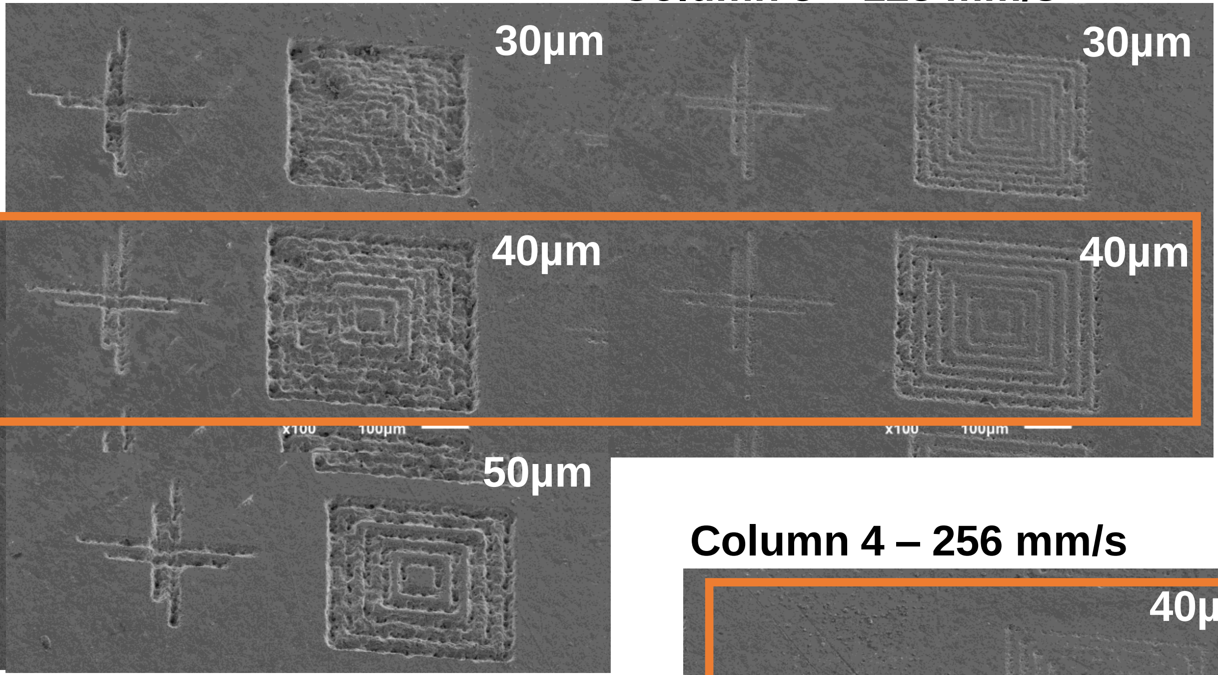
Influence of scanning speed – 1.5 W

Column 1 - 32 mm/s

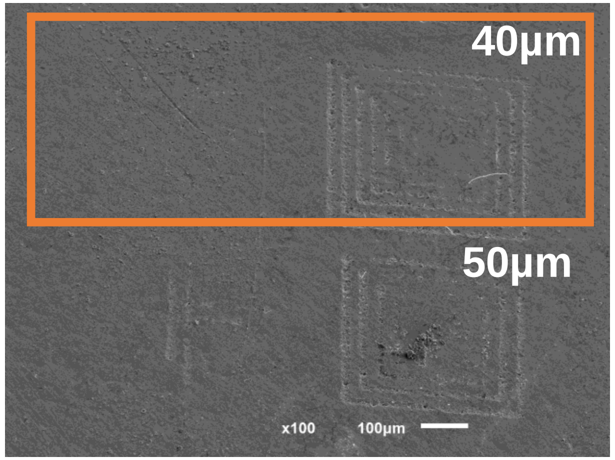


Column 2 – 64 mm/s

Column 3 – 128 mm/s



Column 4 – 256 mm/s



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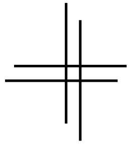

Lines

Scan speed

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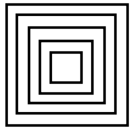

Holes

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Preliminary evaluation of laser processing cond

Influence of scanning speed – 1.5 W

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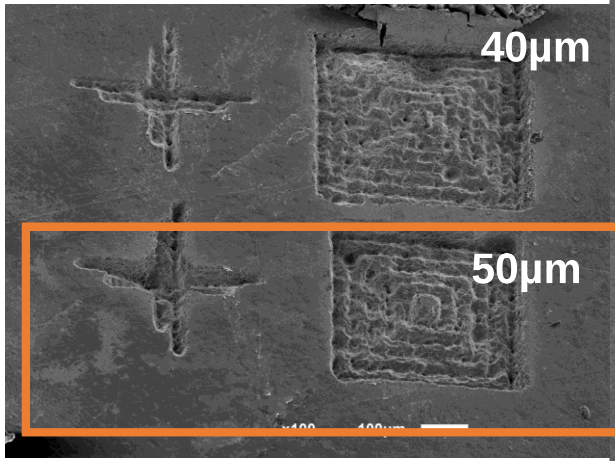
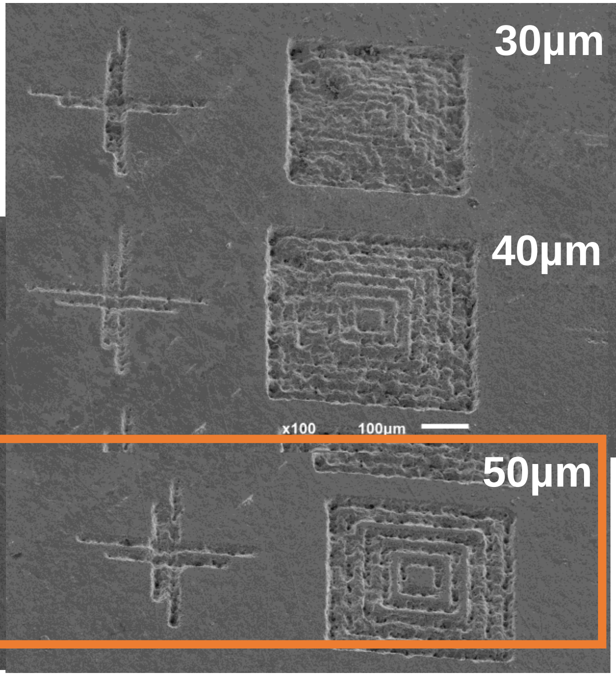
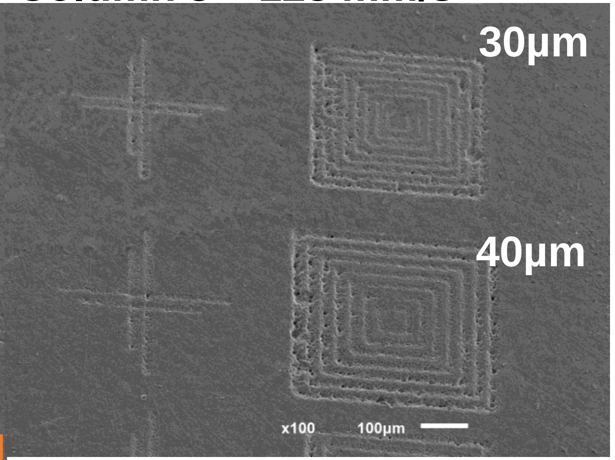


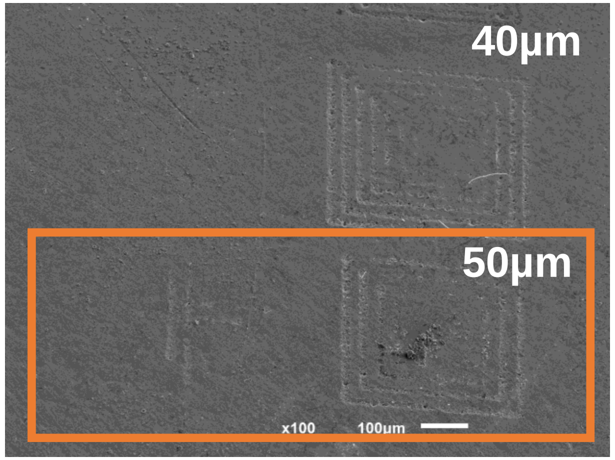
Column 2 – 64 mm/s



Column 3 – 128 mm/s



Column 4 – 256 mm/s



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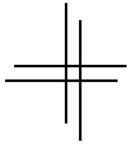

Lines

Scan speed

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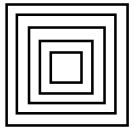

Holes

Summary

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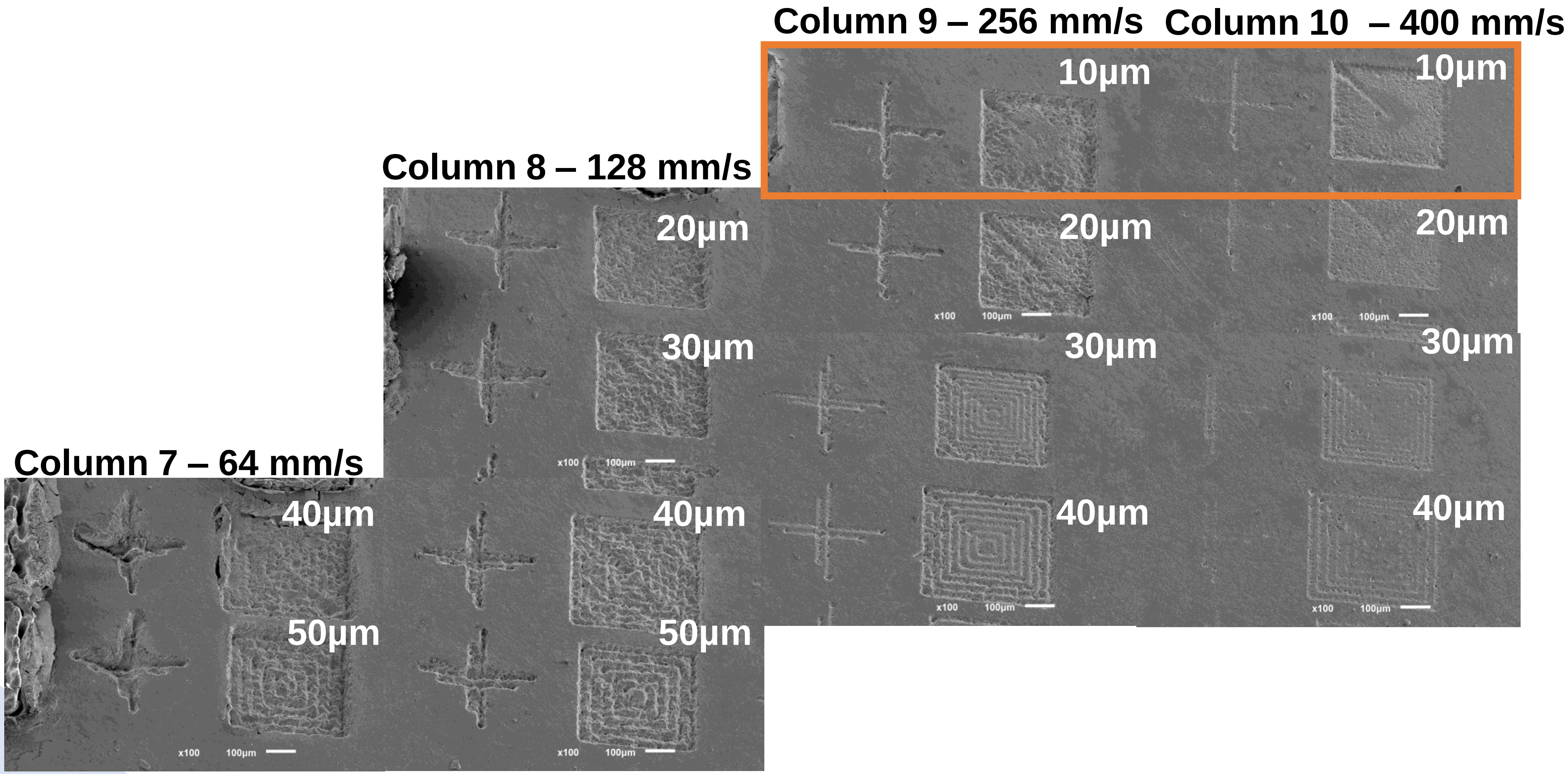
Preliminary evaluation of laser processing conditions

Influence of scanning speed – 3 W



Summary

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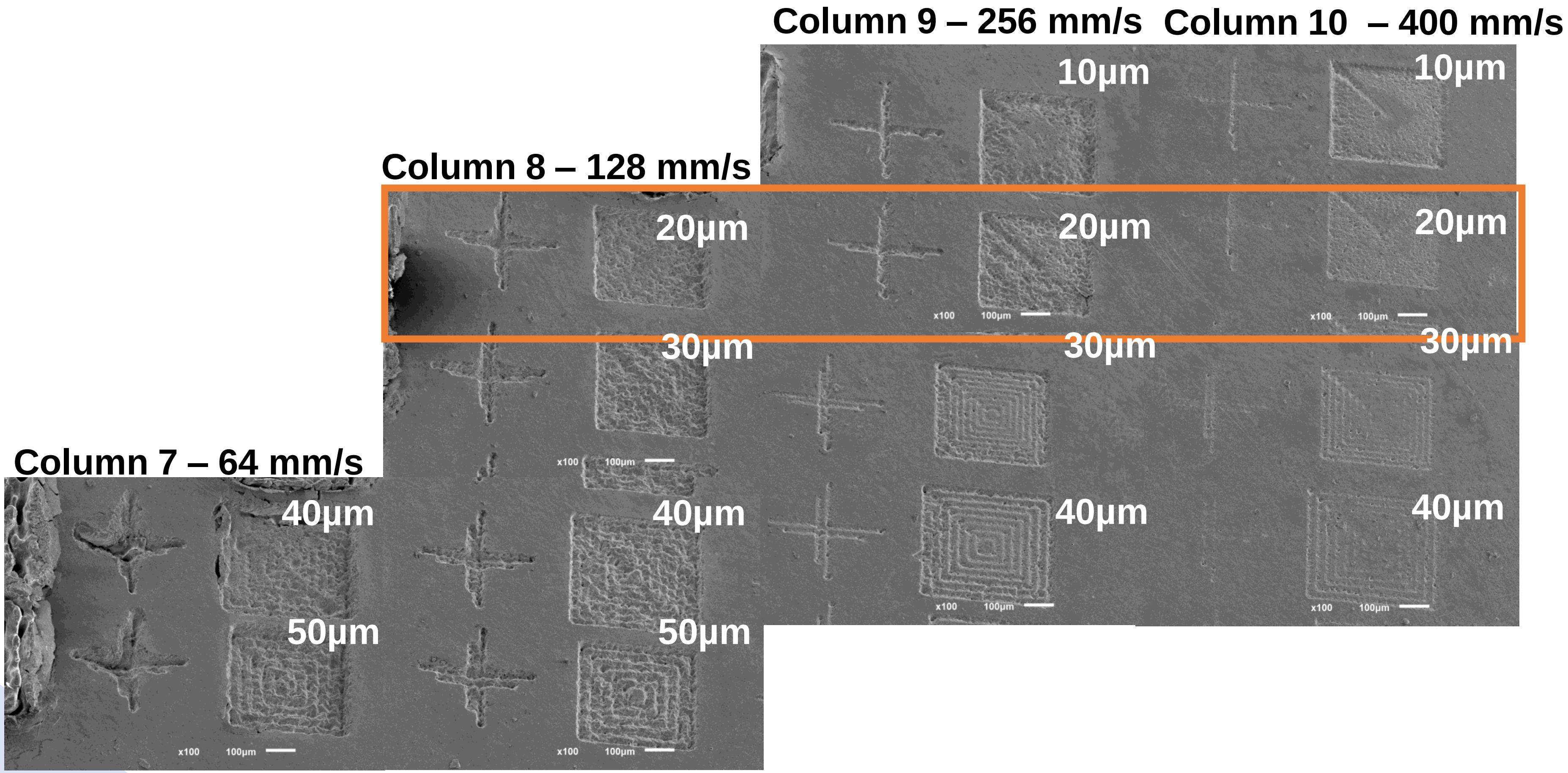
Preliminary evaluation of laser processing conditions

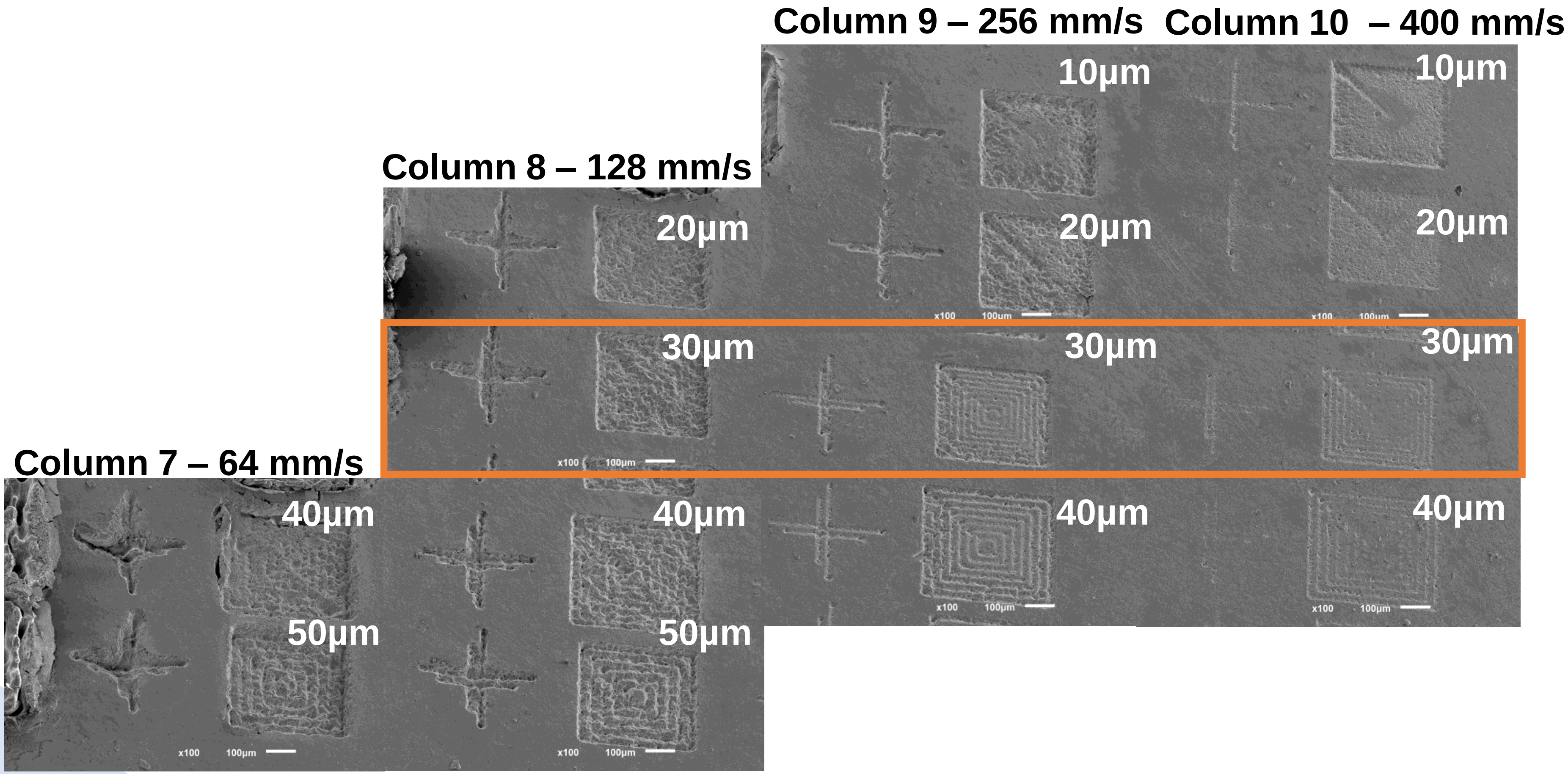
Influence of scanning speed – 3 W



Summary

Preliminary evaluation of laser processing conditions

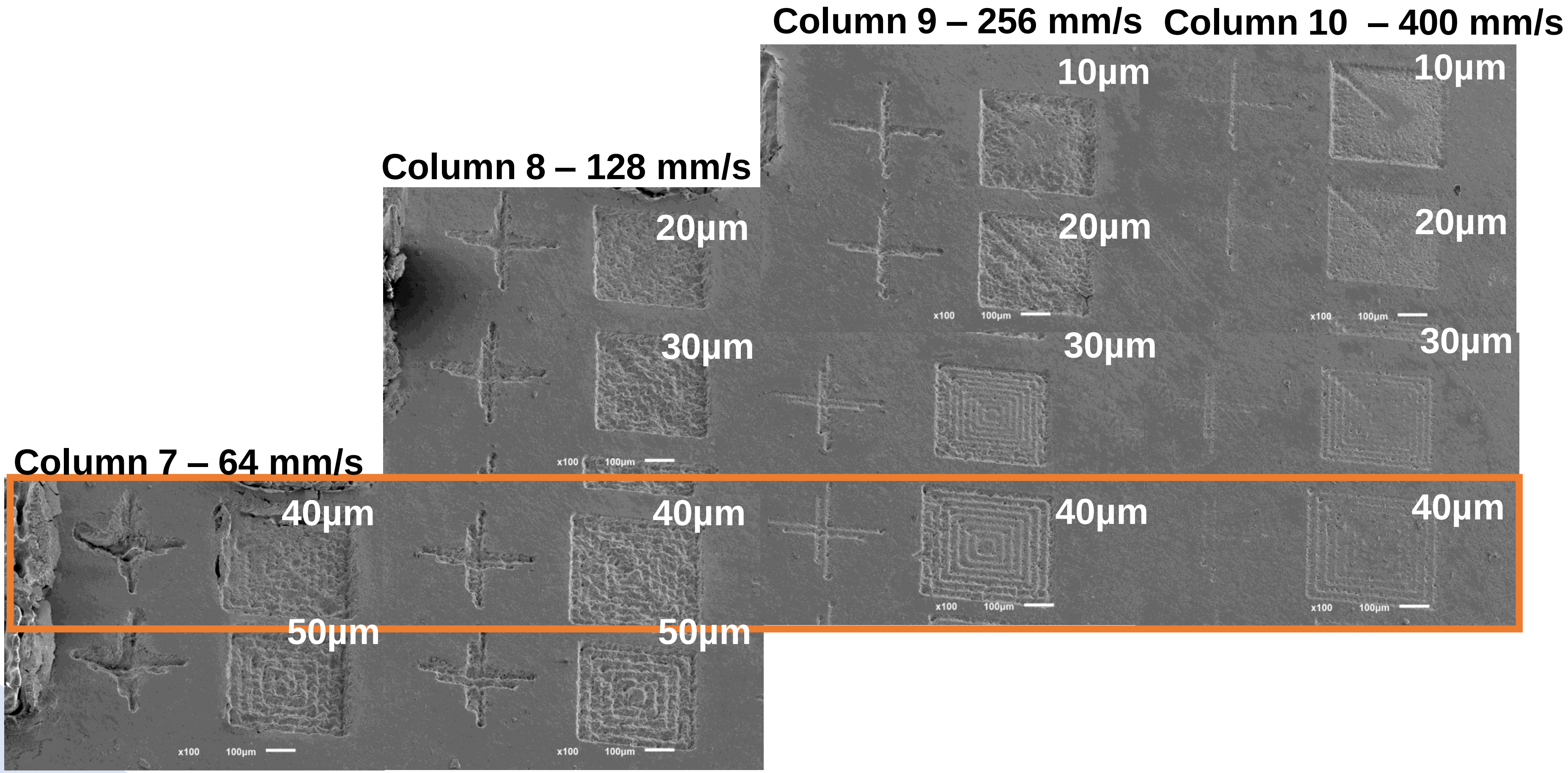
Influence of scanning speed – 3 W



Summary

Preliminary evaluation of laser processing conditions

Influence of scanning speed – 3 W



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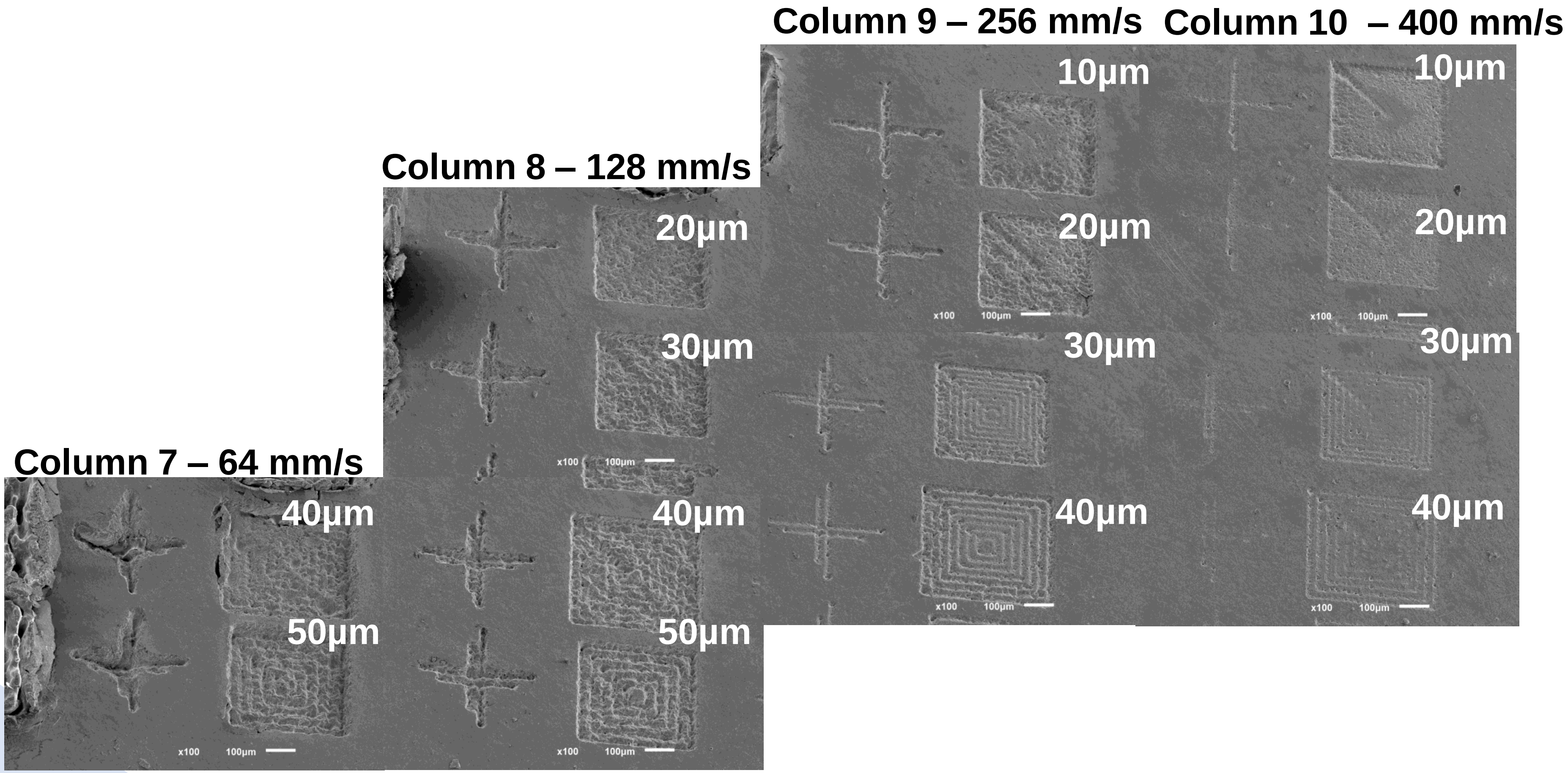
Preliminary evaluation of laser processing conditions

Influence of scanning speed – 3 W



Summary

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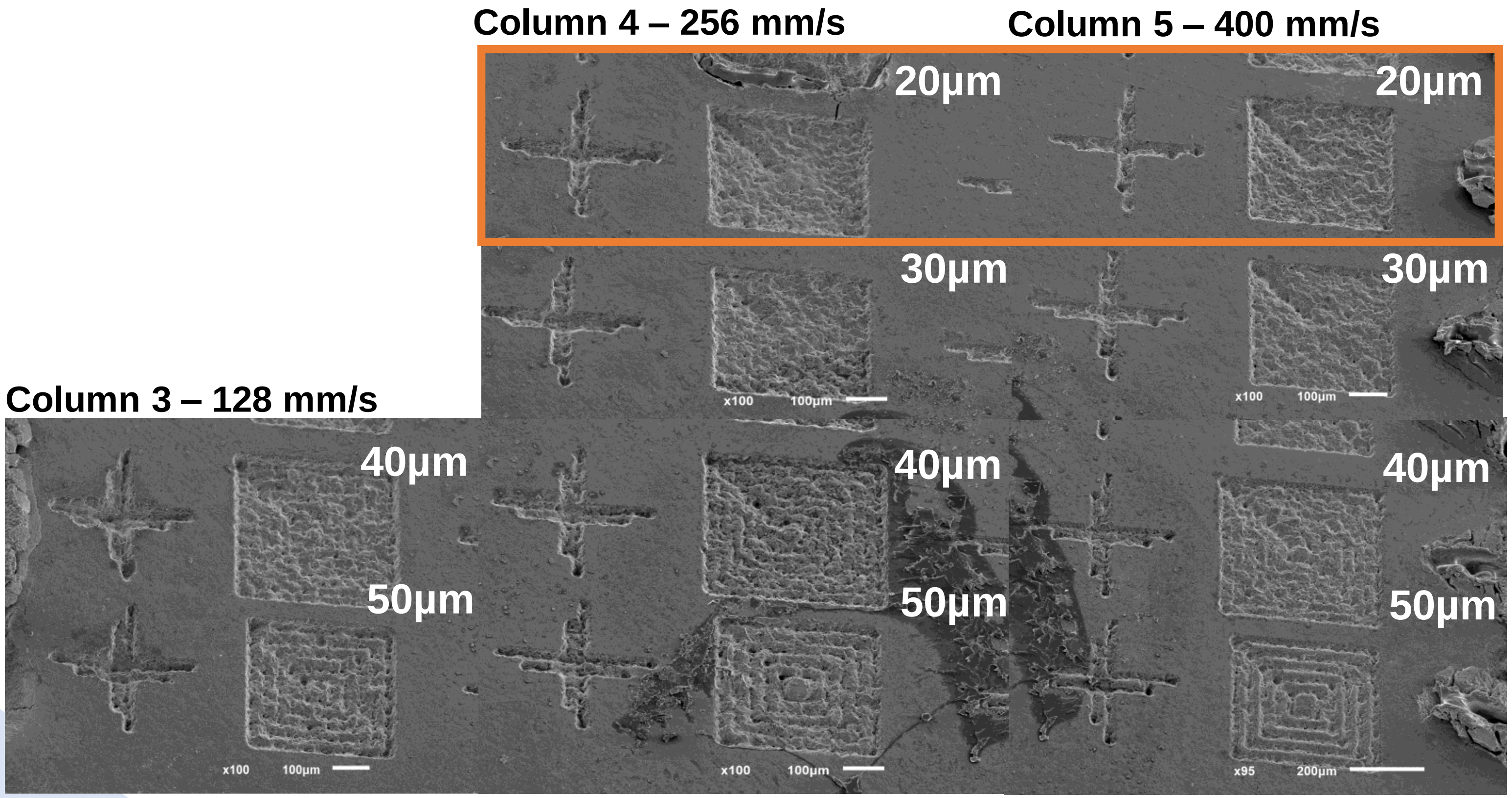
Preliminary evaluation of laser processing conditions

Influence of scanning speed – 6 W



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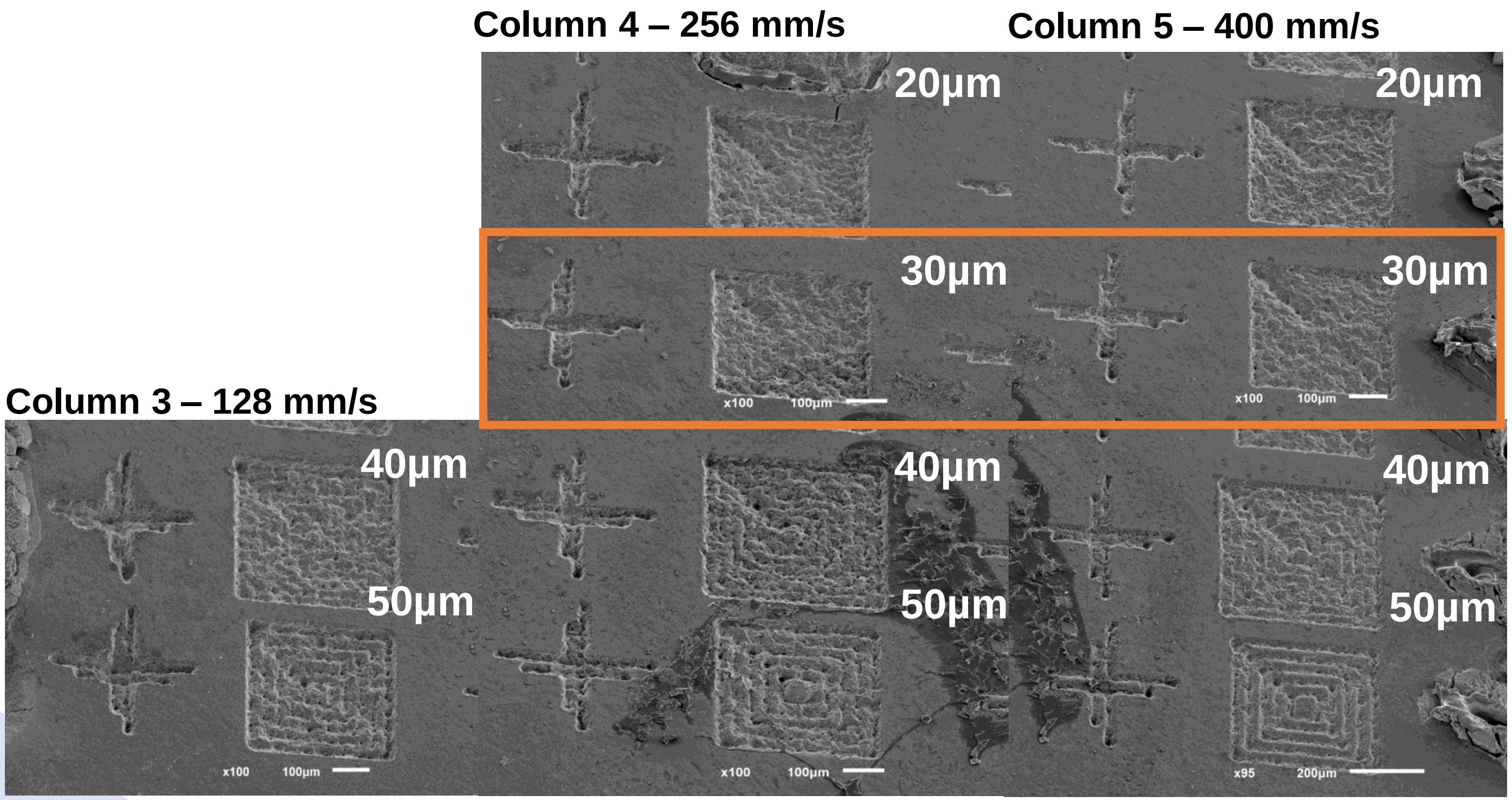
Preliminary evaluation of laser processing conditions

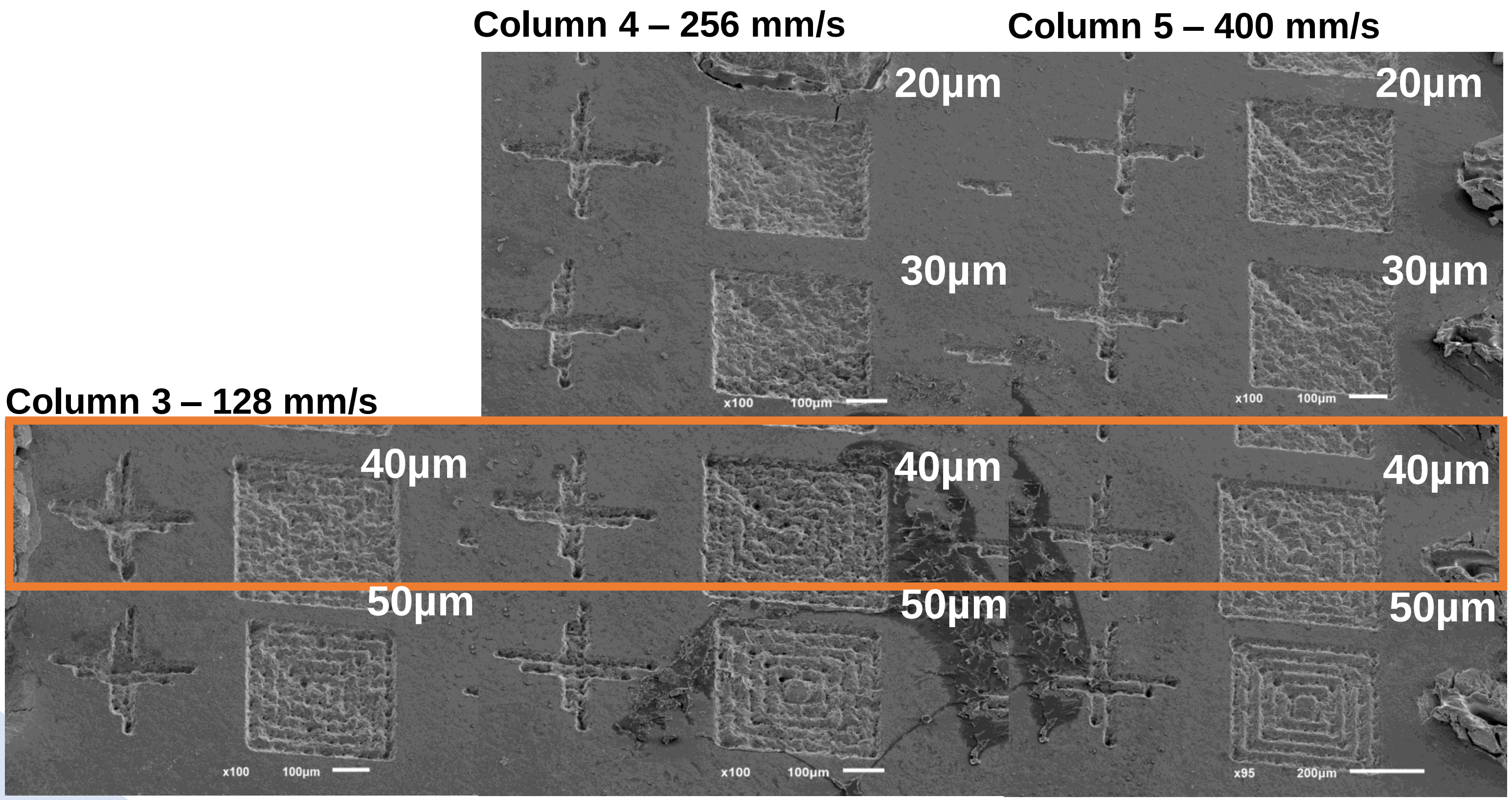
Influence of scanning speed – 6 W



Summary

Preliminary evaluation of laser processing conditions

Influence of scanning speed – 6 W

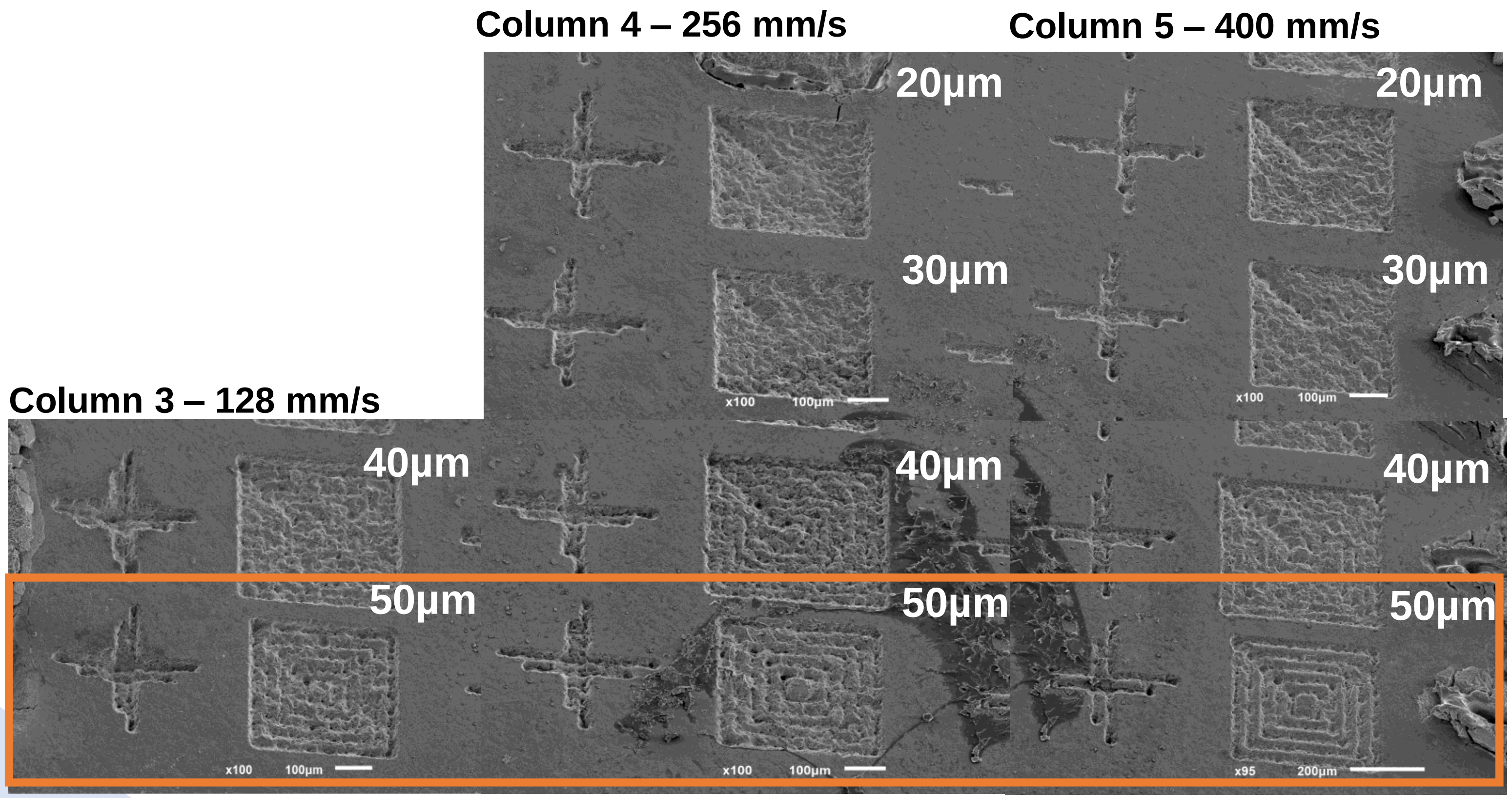


Summary

UMINHO CMEMS CENTER FOR MICROELECTROMECHANICAL SYSTEMS

Preliminary evaluation of laser processing conditions

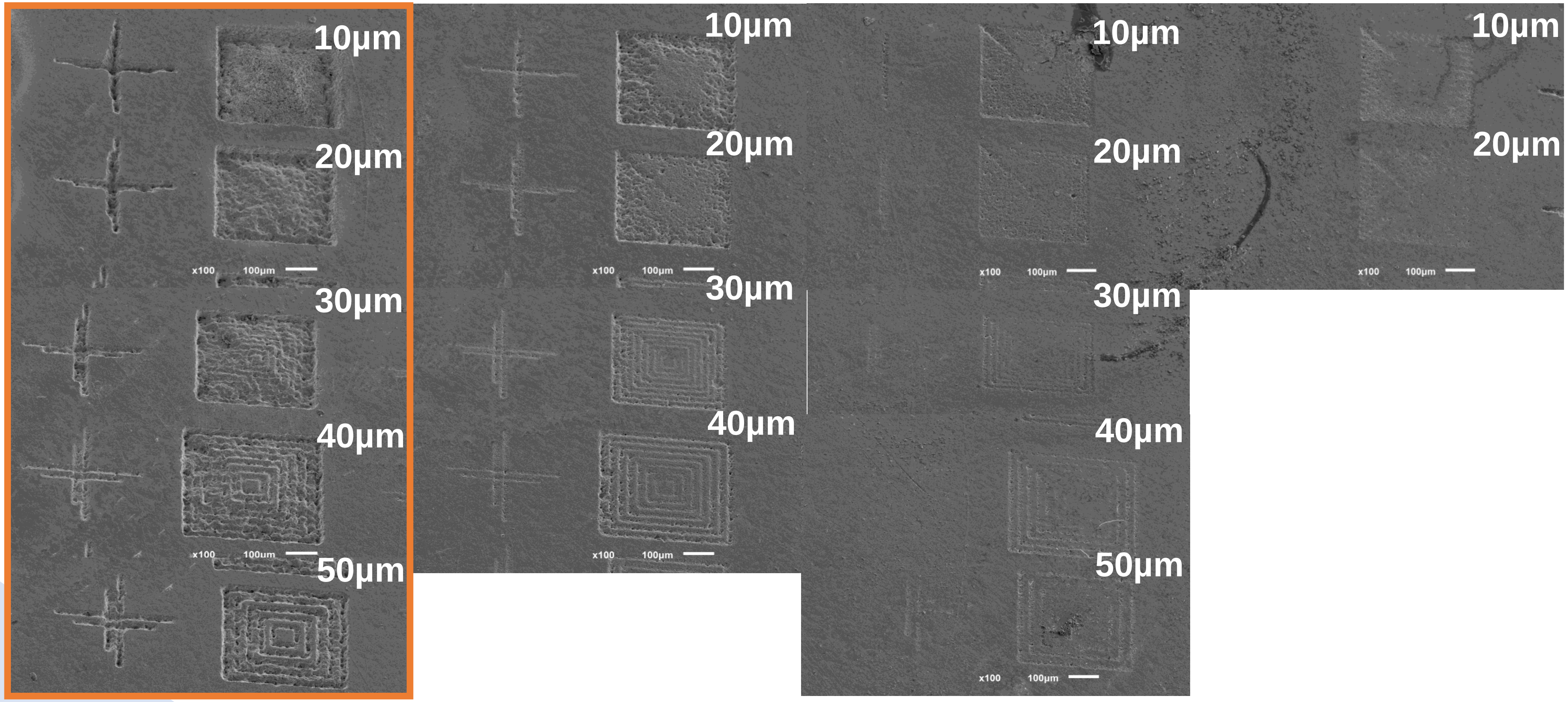
Influence of scanning speed – 6 W



Preliminary evaluation of laser processing conditions

Influence of distance between lines – 1.5 W

Column 2 - 64 mm/s | **Column 3 - 128 mm/s** | **Column 4 - 256 mm/s** | **Column 5 - 400 mm/s**

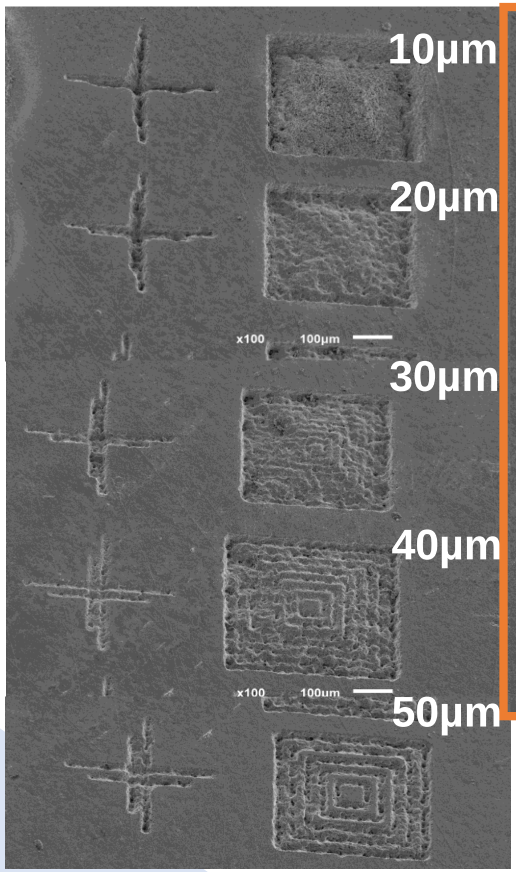


Summary

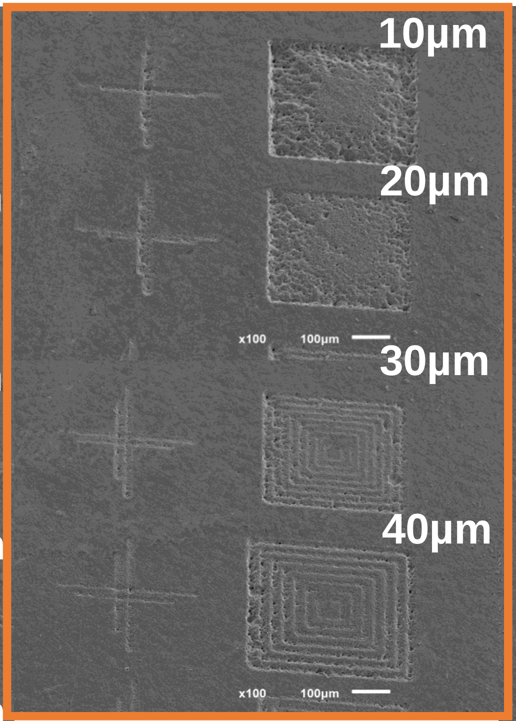
Preliminary evaluation of laser processing conditions

Influence of distance between lines - 1.5 W

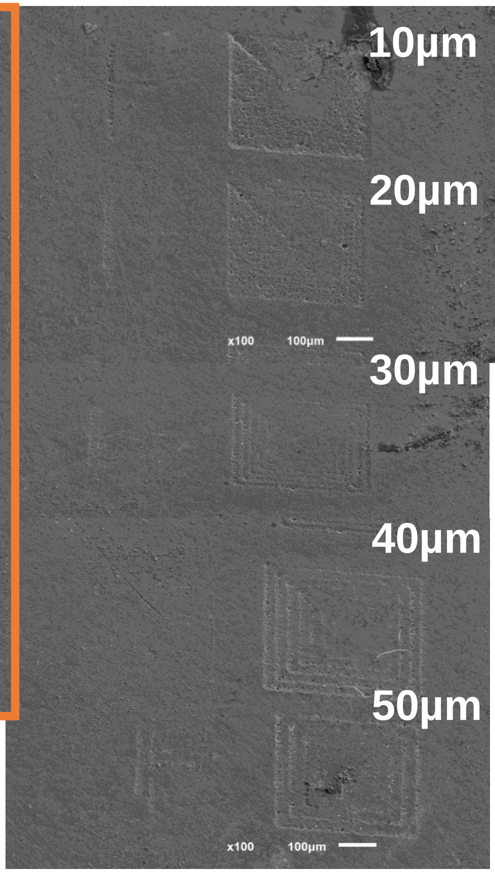
Column 2 - 64 mm/s



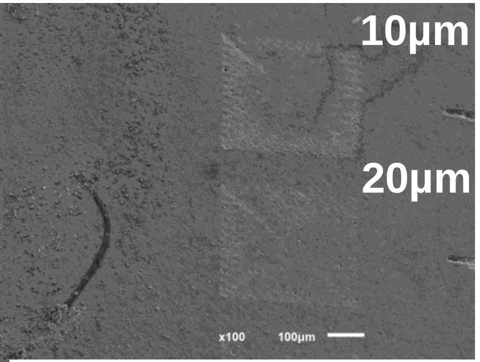
Column 3 - 128 mm/s



Column 4 - 256 mm/s



Column 5 - 400 mm/s



Summary

Preliminary evaluation of laser processing conditions

Influence of distance between lines - 1.5 W

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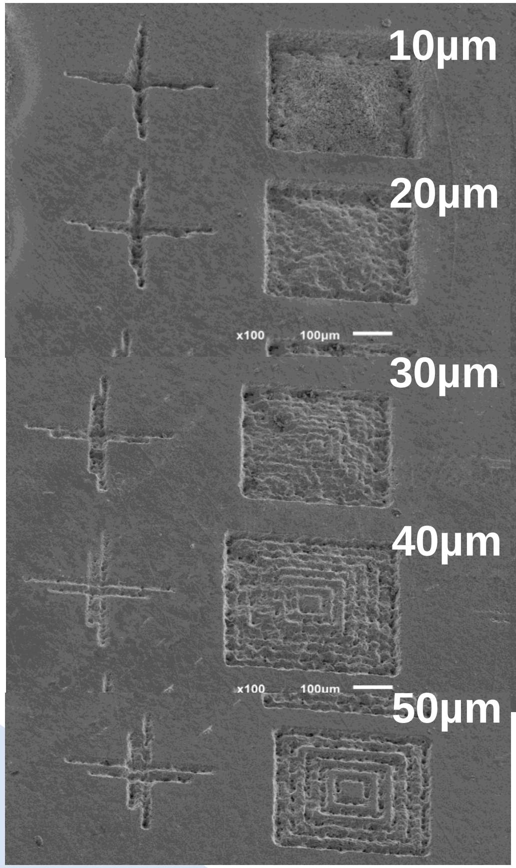
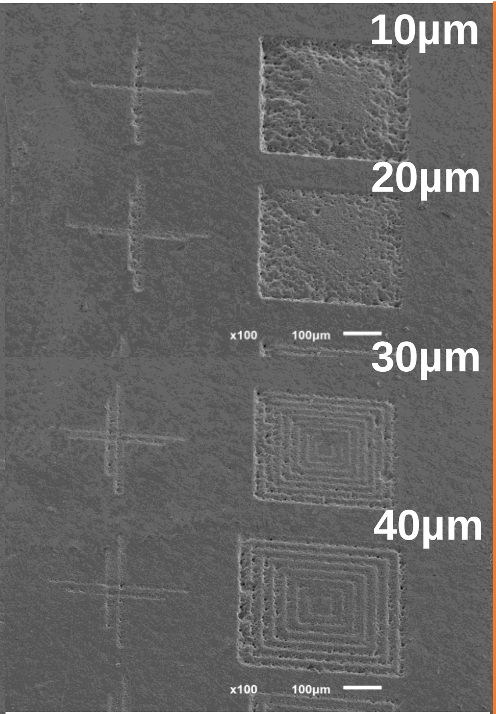
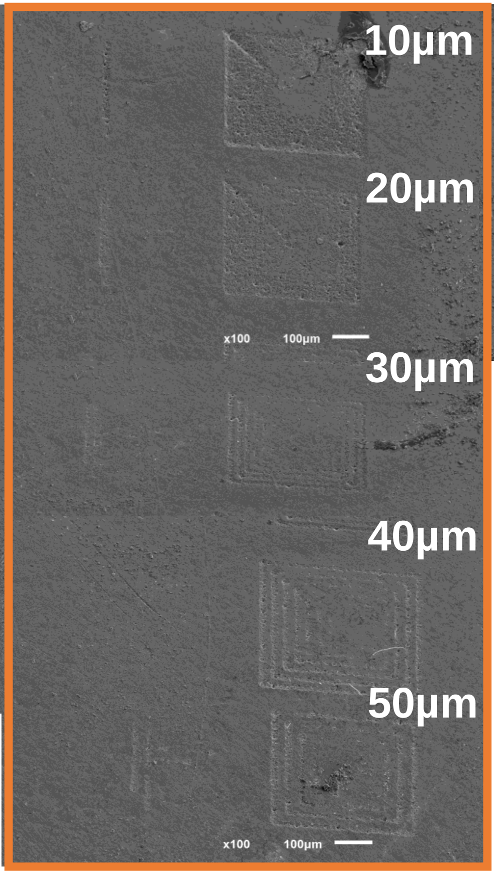


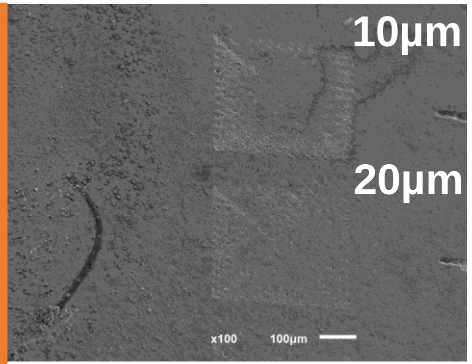
Column 3 - 128 mm/s



Column 4 - 256 mm/s



Column 5 - 400 mm/s

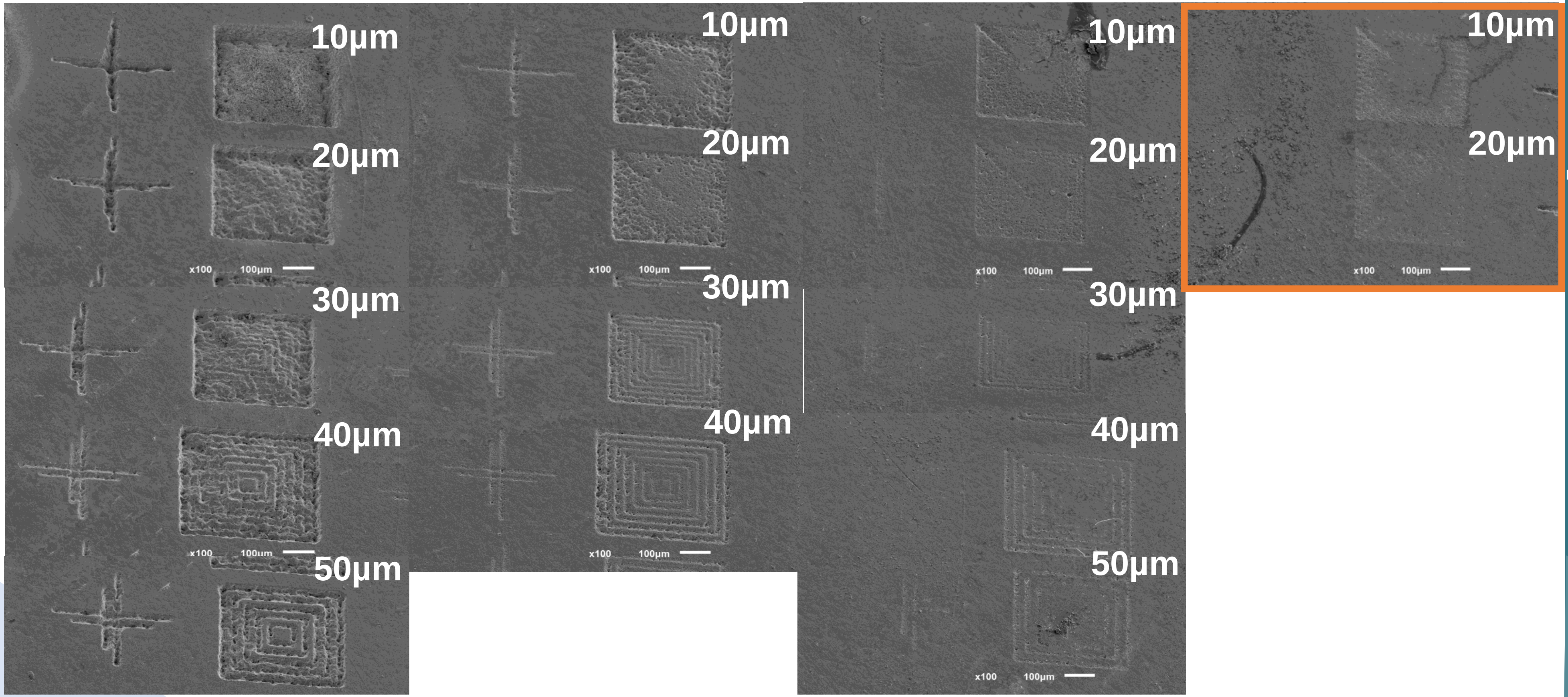


Summary

Preliminary evaluation of laser processing conditions

Influence of distance between lines - 1.5 W

Column 2 - 64 mm/s | Column 3 - 128 mm/s | Column 4 - 256 mm/s | Column 5 - 400 mm/s



Summary

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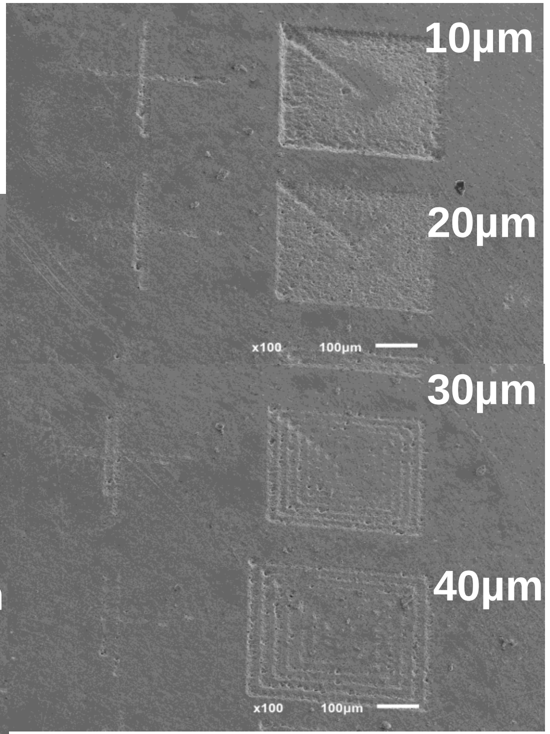
CENTER FOR MICROELECTROMECHANICAL SYSTEMS

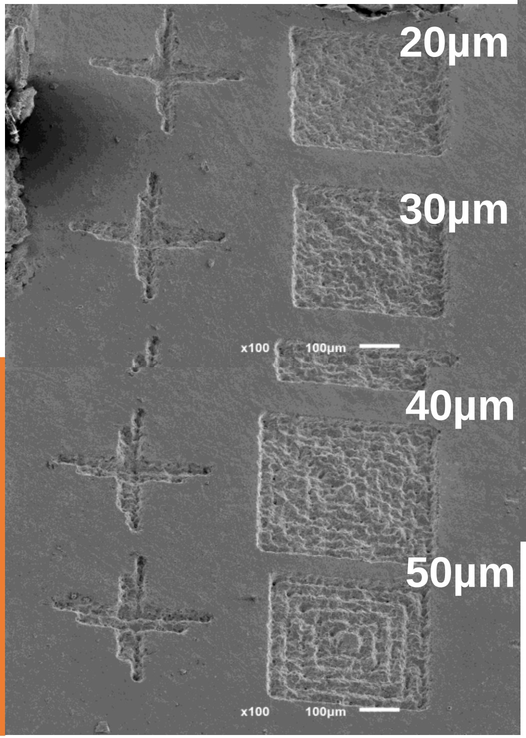
Preliminary evaluation of laser processing conditions

Influence of distance between lines – 3 W

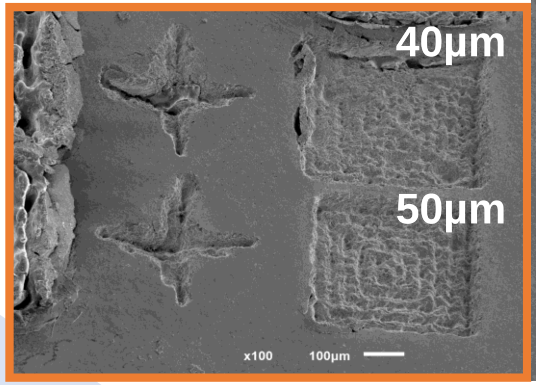
Column 10 - 400 mm/s



Column 8 - 128 mm/s



Column 7 - 64 mm/s

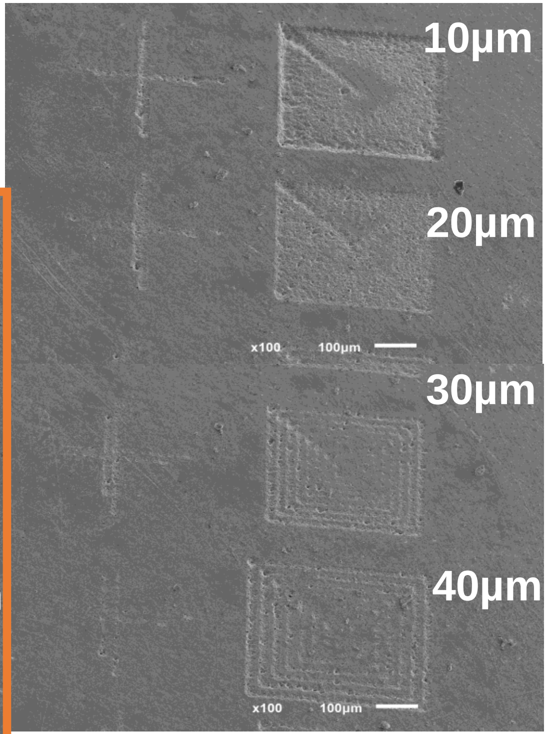


Summary

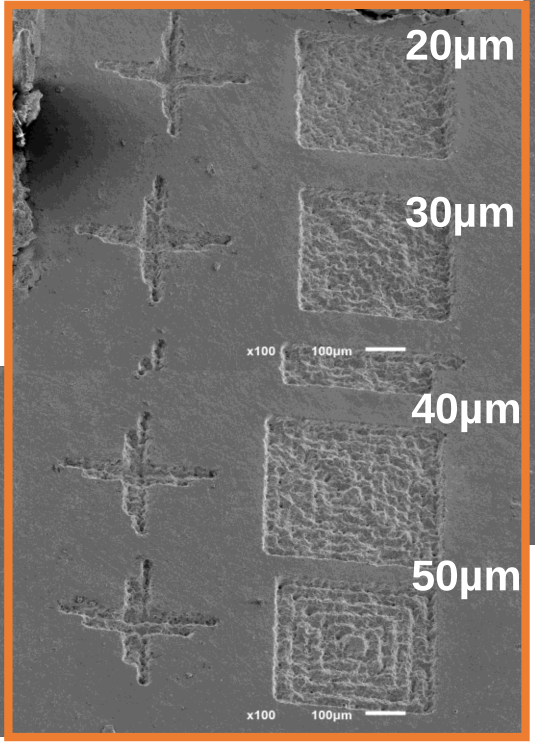
Preliminary evaluation of laser processing conditions

Influence of distance between lines - 3 W

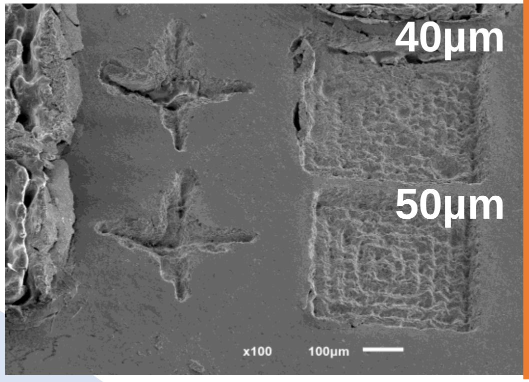
Column 10 - 400 mm/s



Column 8 - 128 mm/s



Column 7 - 64 mm/s



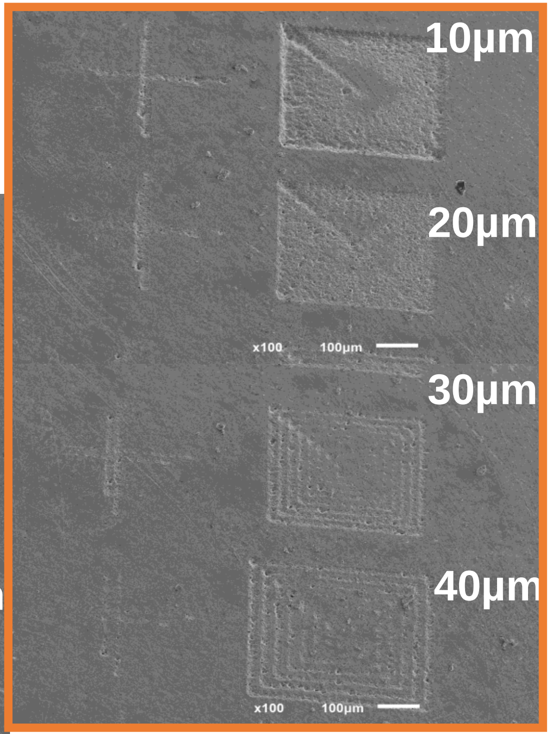
Summary

UMINHO CMEMS CENTER FOR MICROELECTROMECHANICAL SYSTEMS

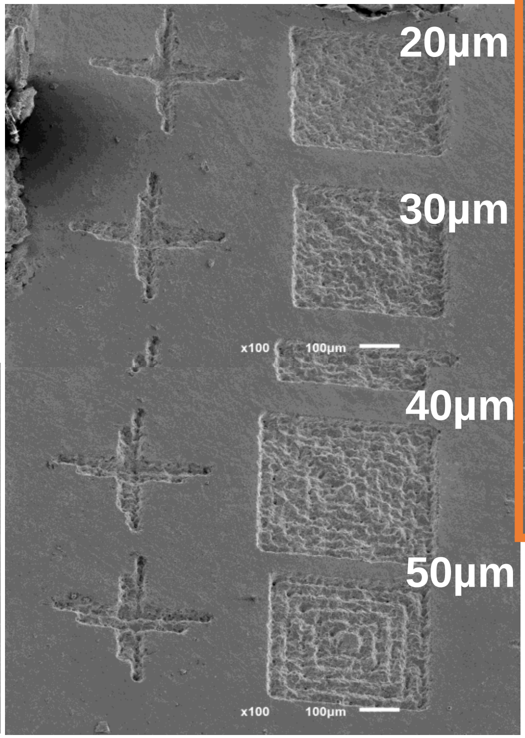
Preliminary evaluation of laser processing conditions

Influence of distance between lines - 3 W

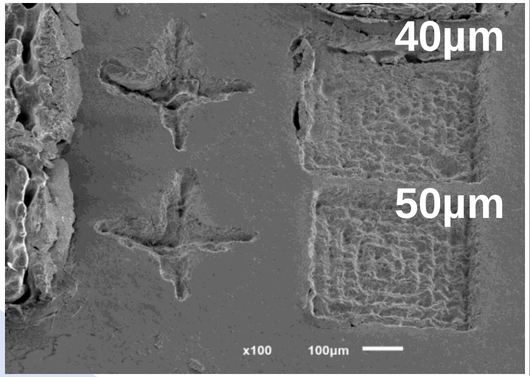
Column 10 - 400 mm/s



Column 8 - 128 mm/s



Column 7 - 64 mm/s



Summary

UMINHO CMEMS CENTER FOR MICROELECTROMECHANICAL SYSTEMS

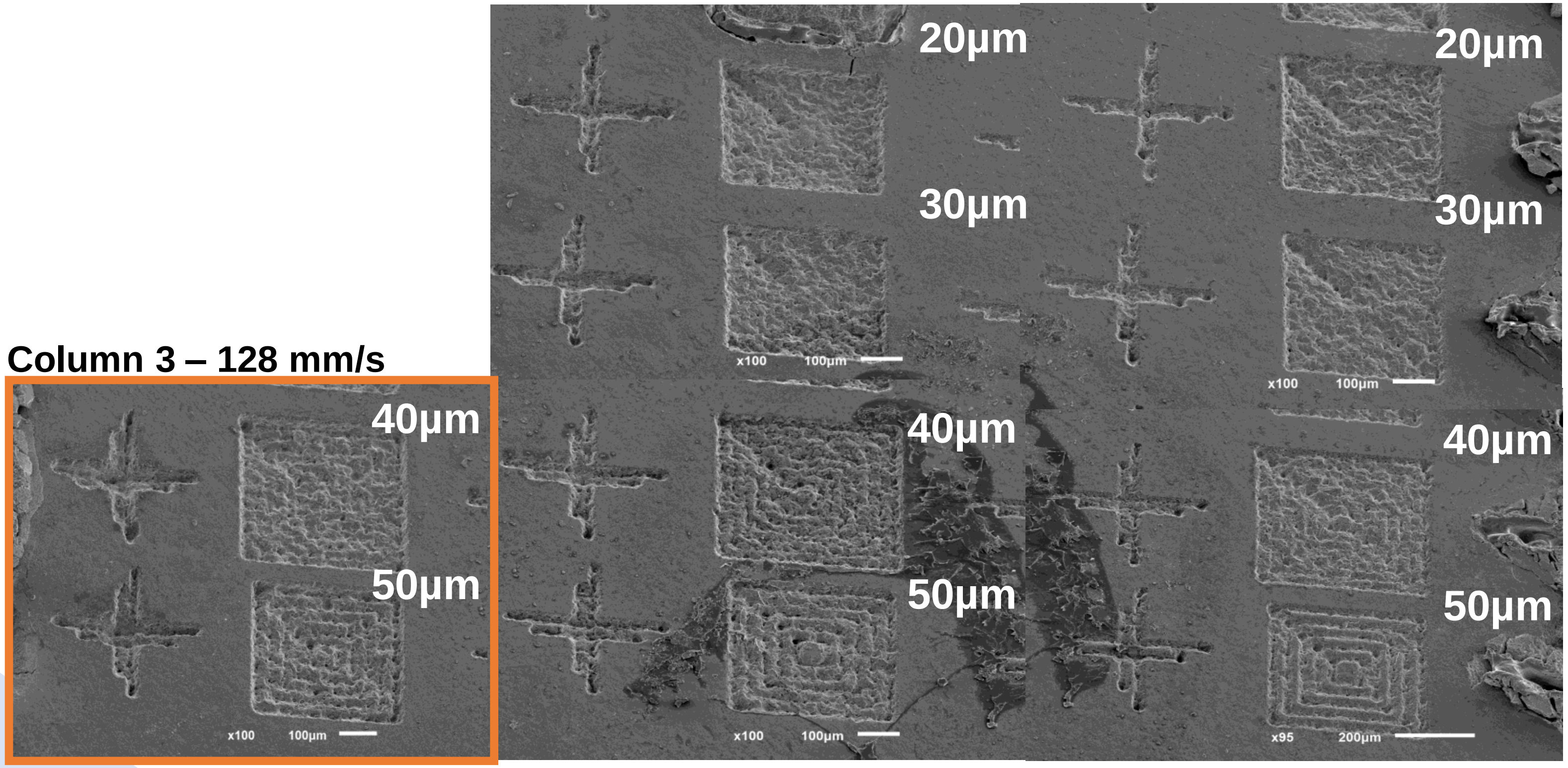
Preliminary evaluation of laser processing conditions

Influence of distance between lines – 6 W

Column 4 – 256 mm/s

Column 5 – 400 mm/s

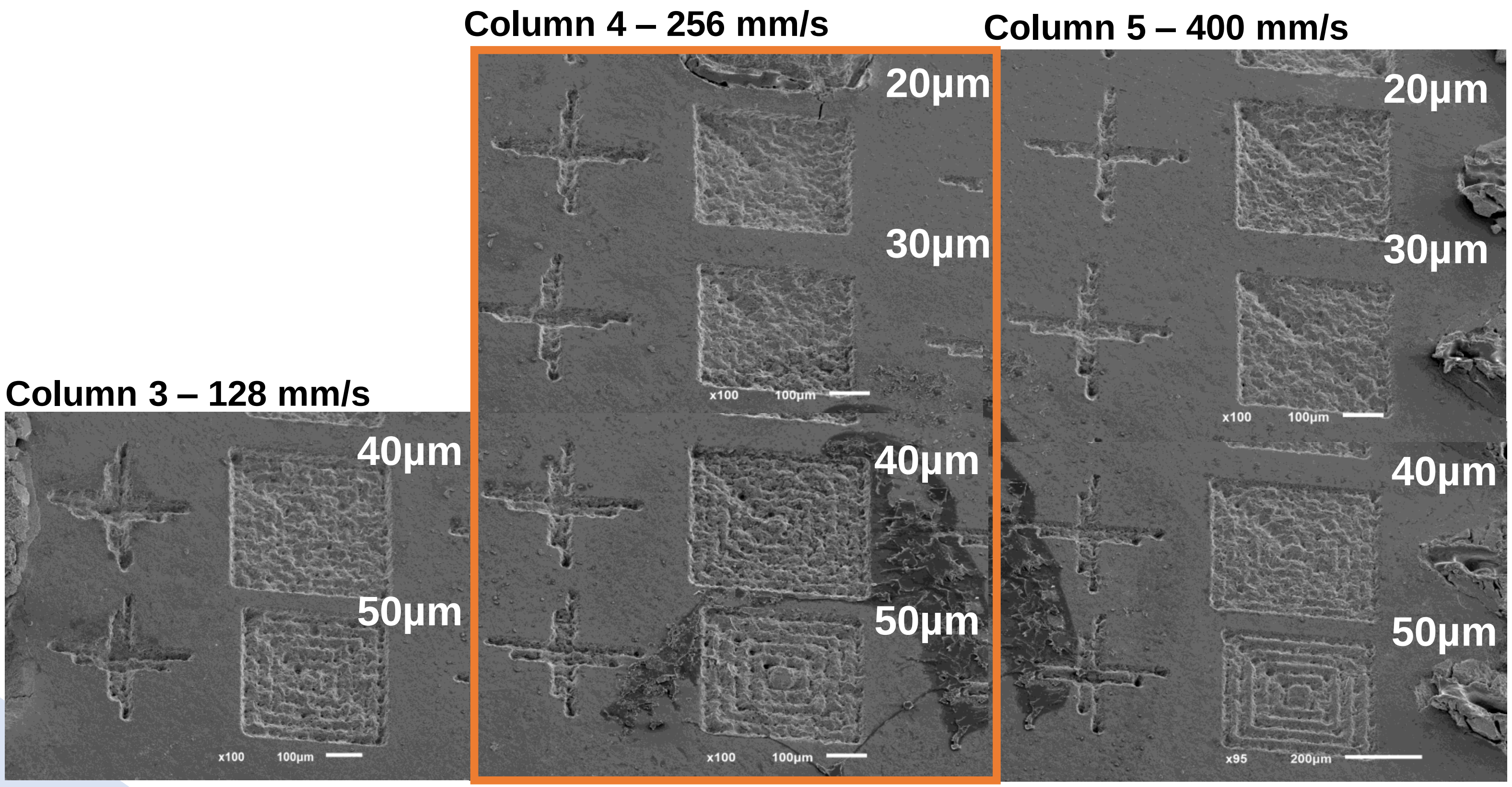
Column 3 – 128 mm/s



Summary

Preliminary evaluation of laser processing conditions

Influence of distance between lines – 6 W



Summary

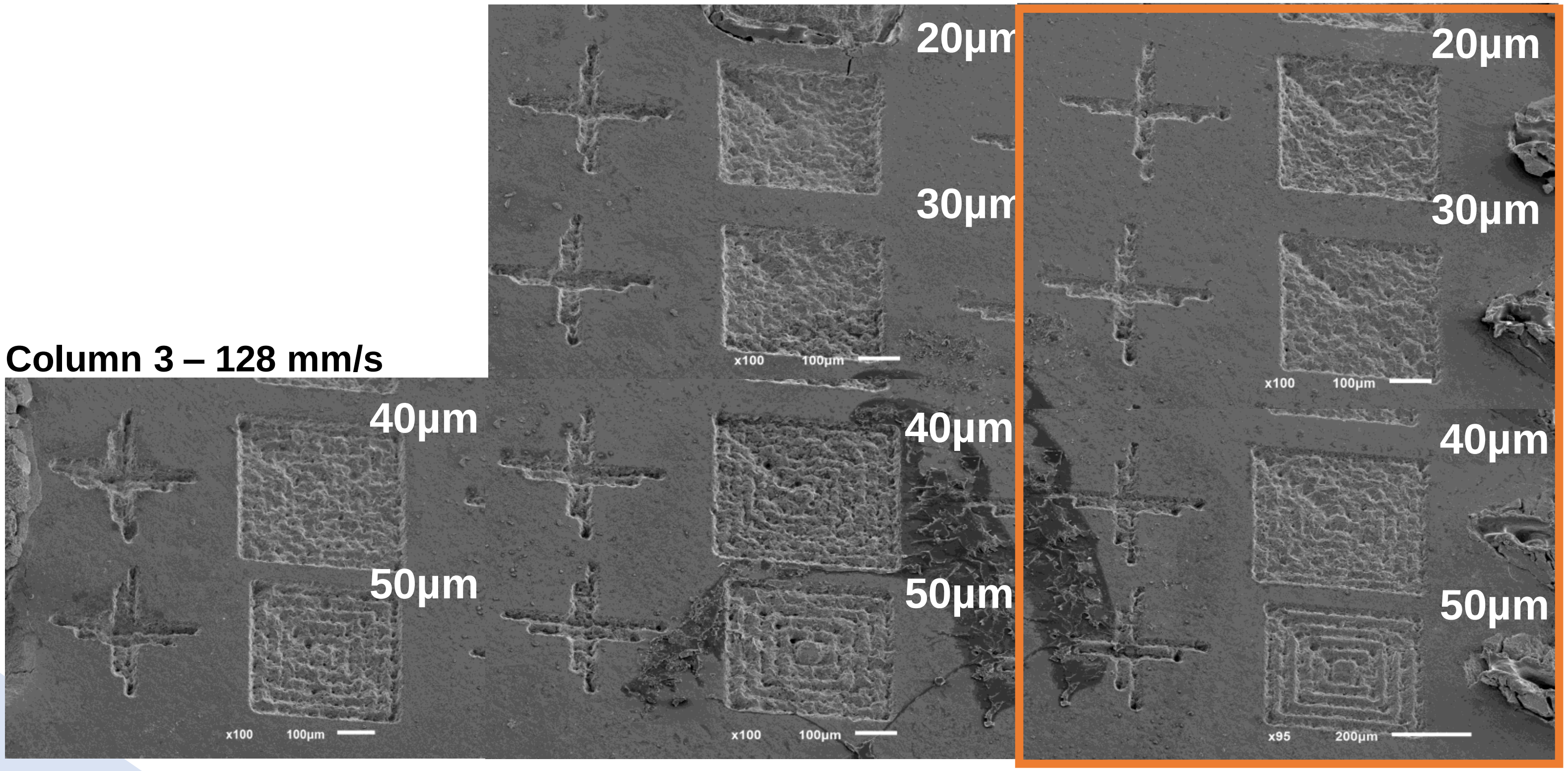
Preliminary evaluation of laser processing conditions

Influence of distance between lines – 6 W

Column 4 – 256 mm/s

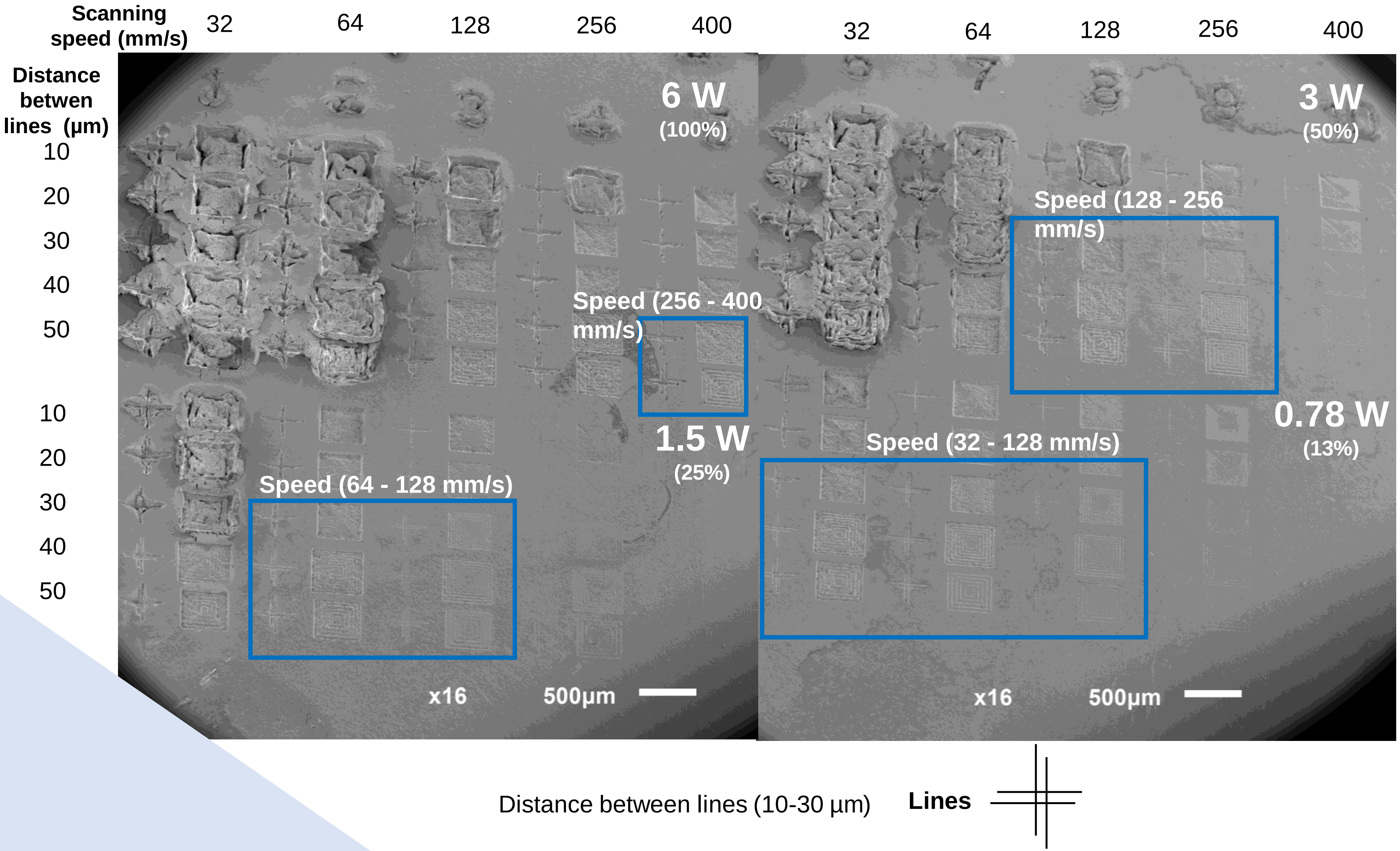
Column 5 – 400 mm/s

Column 3 – 128 mm/s



Summary

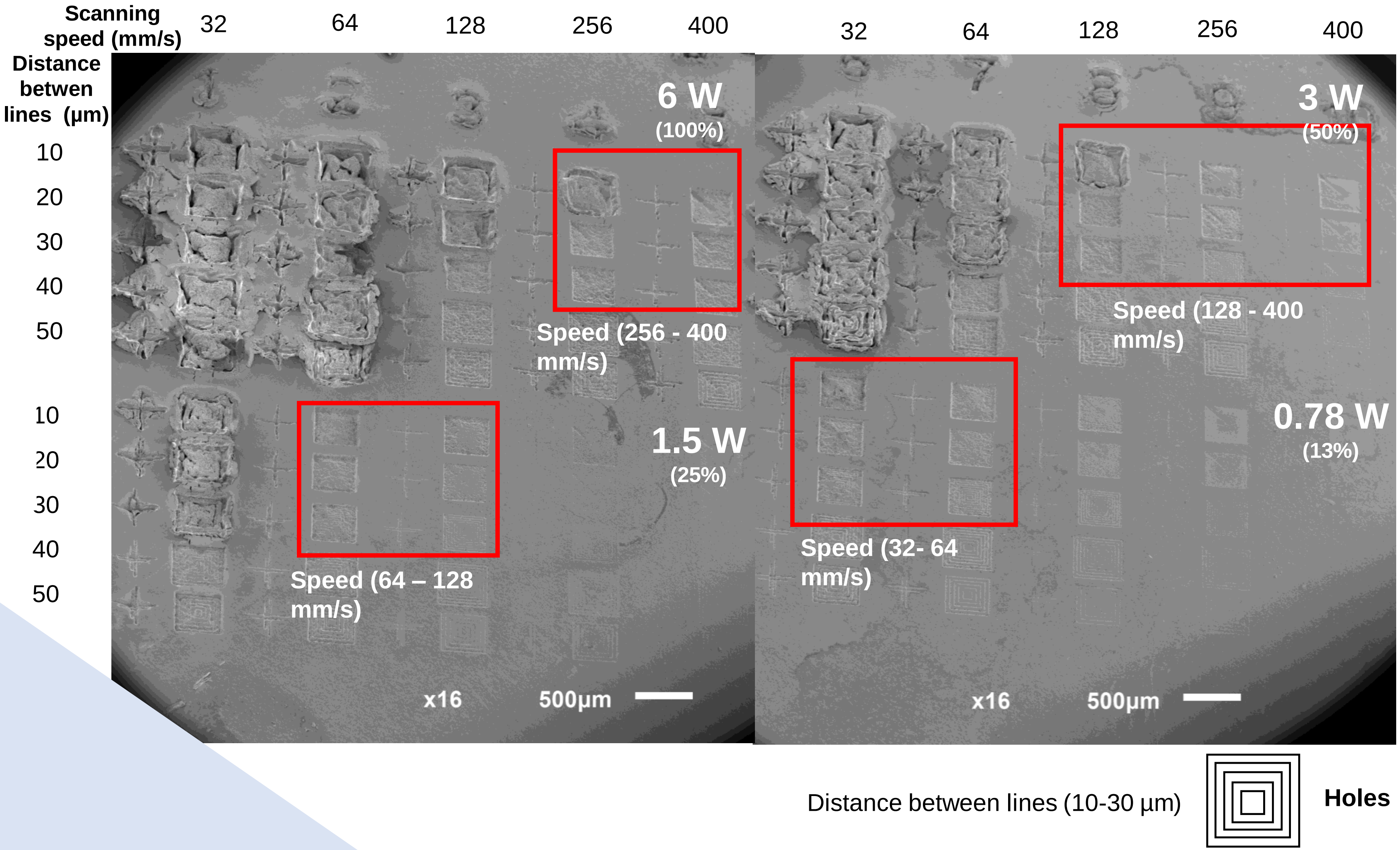
Preliminary evaluation of laser processing conditions



Summary

UMINHO CMEMS CENTER FOR MICROELECTROMECHANICAL SYSTEMS

Preliminary evaluation of laser processing conditions



Summary

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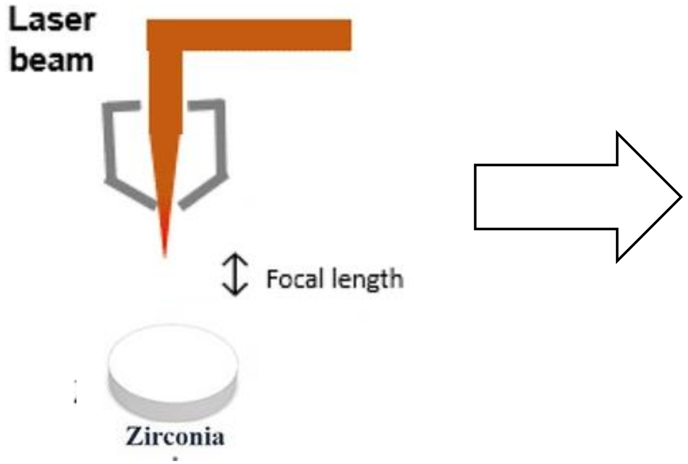
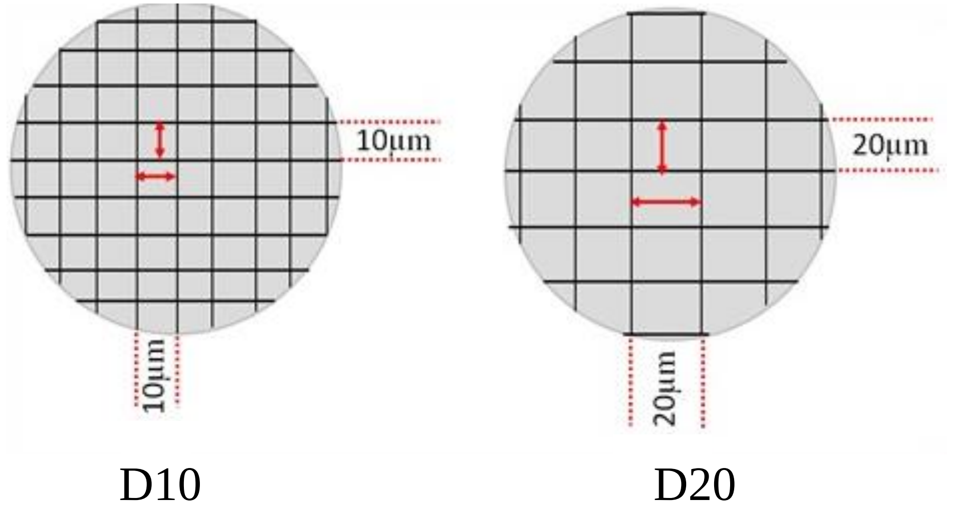
Textures

Textures



Design



Parameters

Distance between lines (µm): 10 (D10) and 20 (D20)

Number of passages: 1

Laser Power (5) : 1, 3, 10, 25

Scan speed (mm/s) : 32, 64, 128, 256

Textures

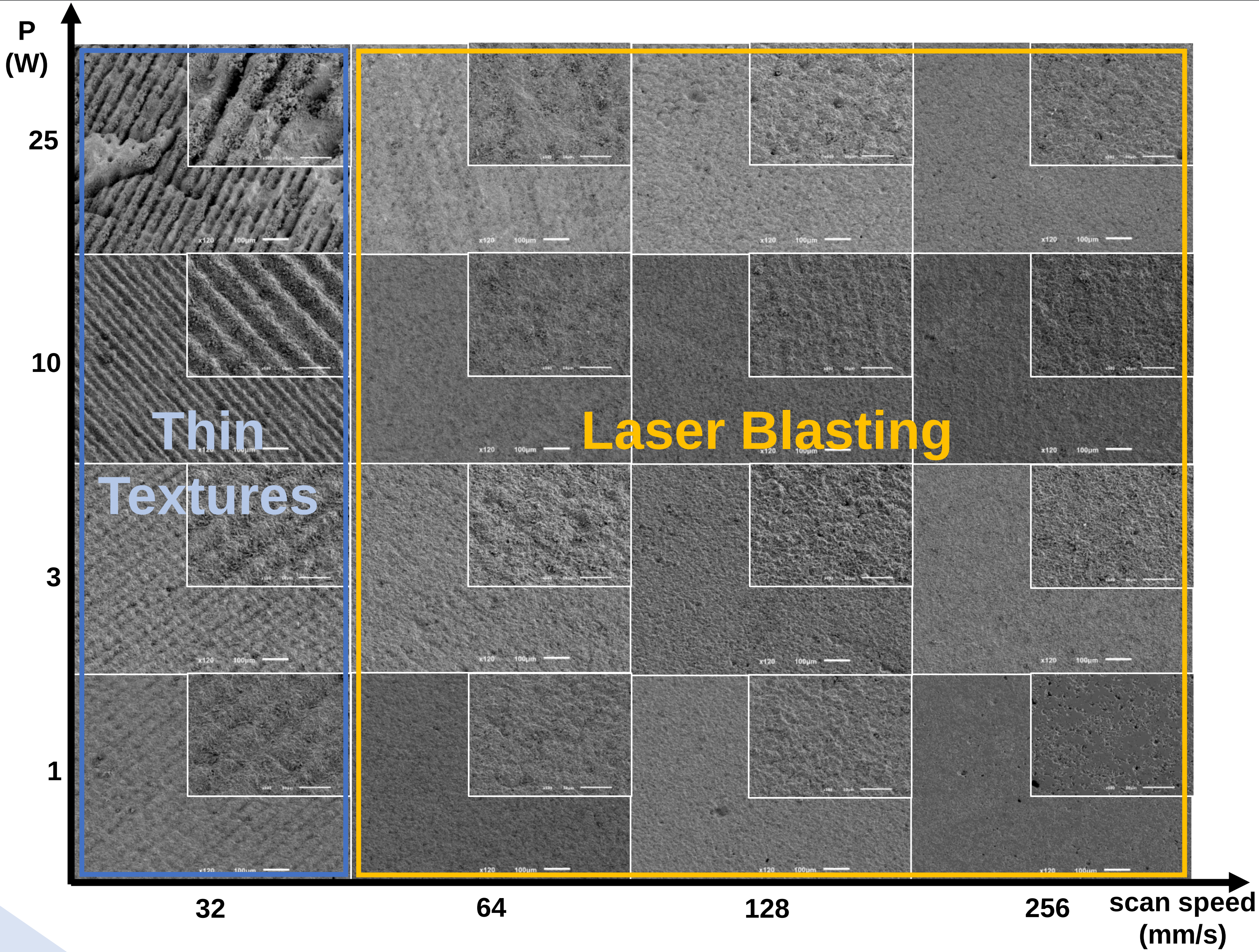
D10

Low Scan speed

Thin textures

High Scan speed

No laser effect or rough surface



UMINHO CMEMS CENTER FOR MICROELECTROMECHANICAL SYSTEMS

Textures

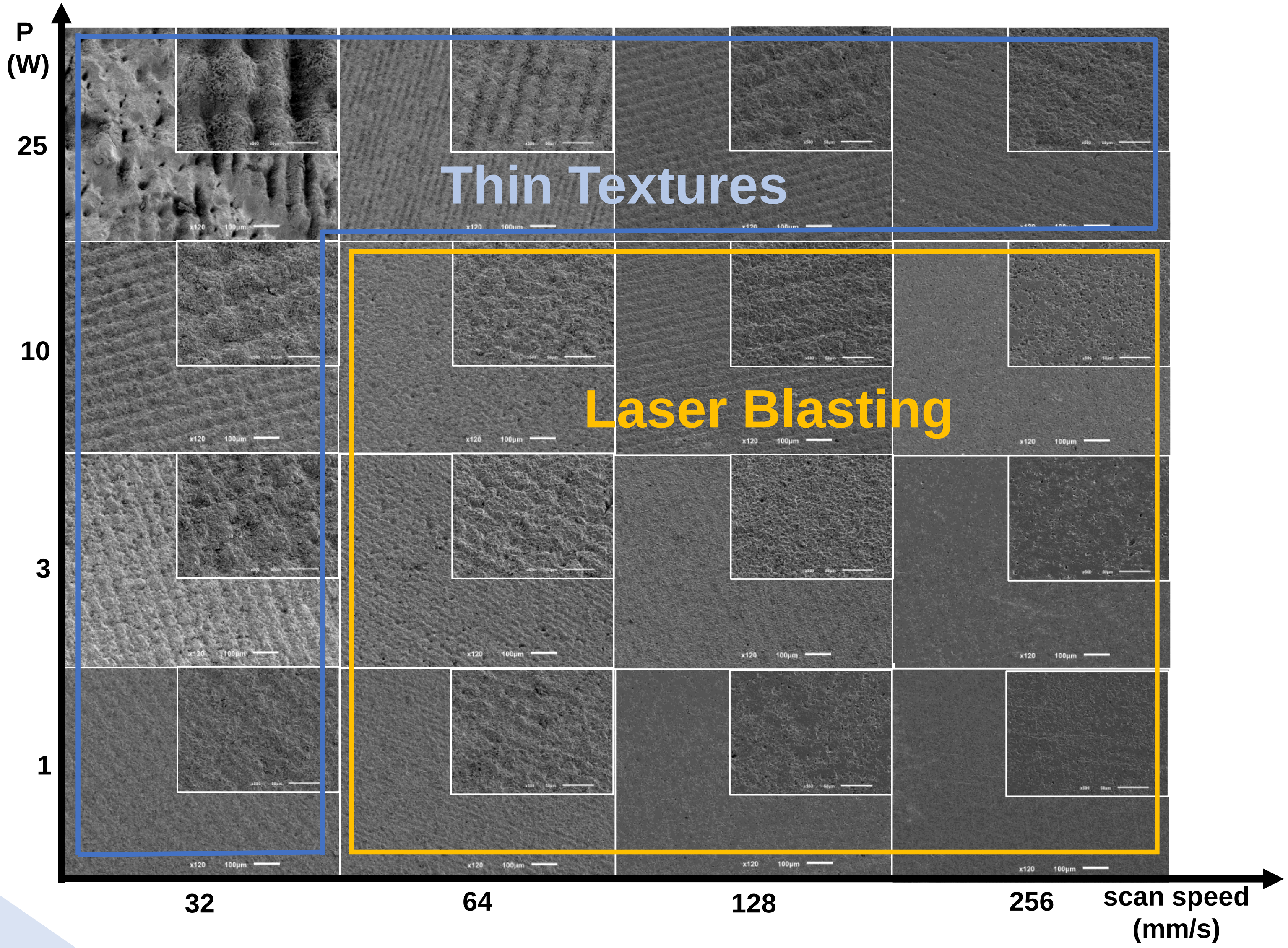
D20

Low Scan speed

Thin textures

High Scan speed

No laser effect or rough surface



Textures

Laser blasting advantages

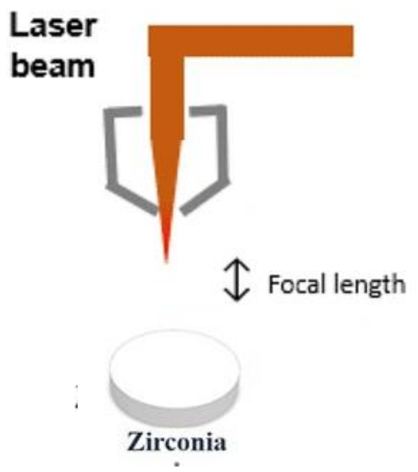
- ✓ It was verified that for both **D10** and **D20** distance between lines conditions are suitbale to obtain laser blasted surfaces;
- ✓ It is possible to make different structures with **less concave surfaces**
- ✓ Sandblasting induces stresses in the Zirconia surface which lowers the mechanical strength.
- ✓ Surface patterns produced by laser create more mechanical interlocking than sandblasting which promotes greater adhesion in facets or dental crowns
- ✓ Laser blasting technique allows to create distinct type of textures at the same part. This is harder to obtain by sandblasting

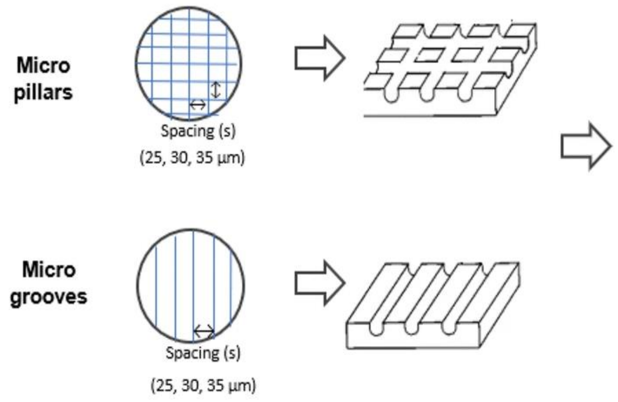
D10	D20
Scan speed (64-256 mm/s)	**Scan speed** (64-256 mm/s)
Power (1-25 W)	**Power** (1-20 W)

Textures

2.2 Thin textures

Design





CONVENTIONAL SINTERING

- Temperature:1500 °C,
- Heating and cooling rate: 8 °C/min
- Holding time: 2h.

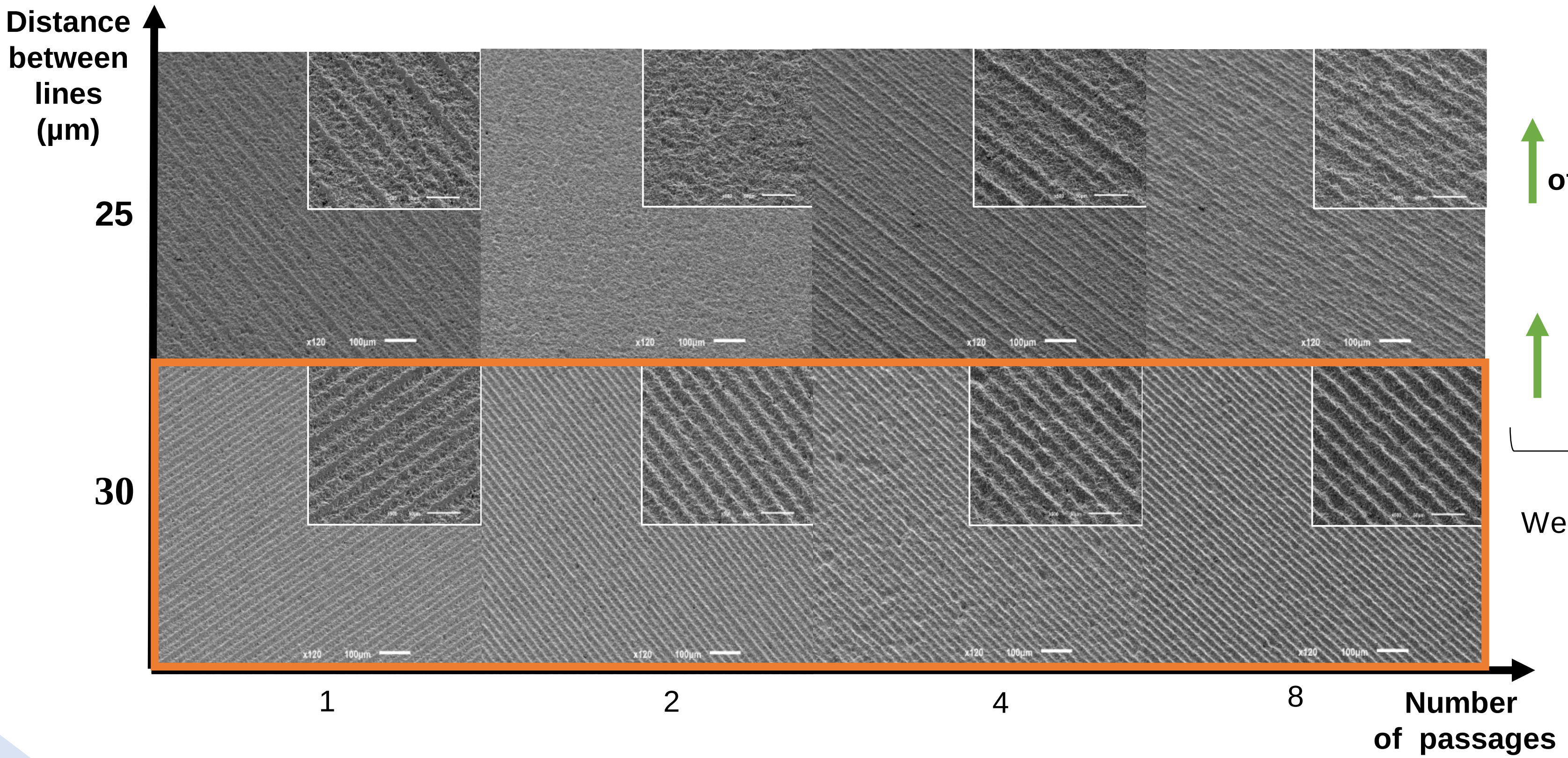
Parameters

Distance between lines (μm): 25 (D25) and 30 (D30)
Number of passages: 1
Laser Power (W): 1
Scan speed (mm/s): 128
Wobble frequency (Hz): 75

Summary

Textures

2.2 Thin textures



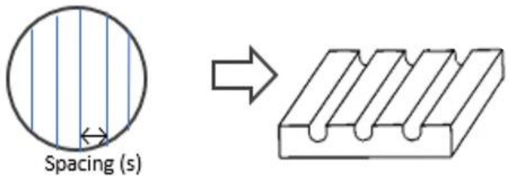
Number of passages

+

Distance between lines

Well defined thin textures

Micro grooves



25 e 30 µm

Summary

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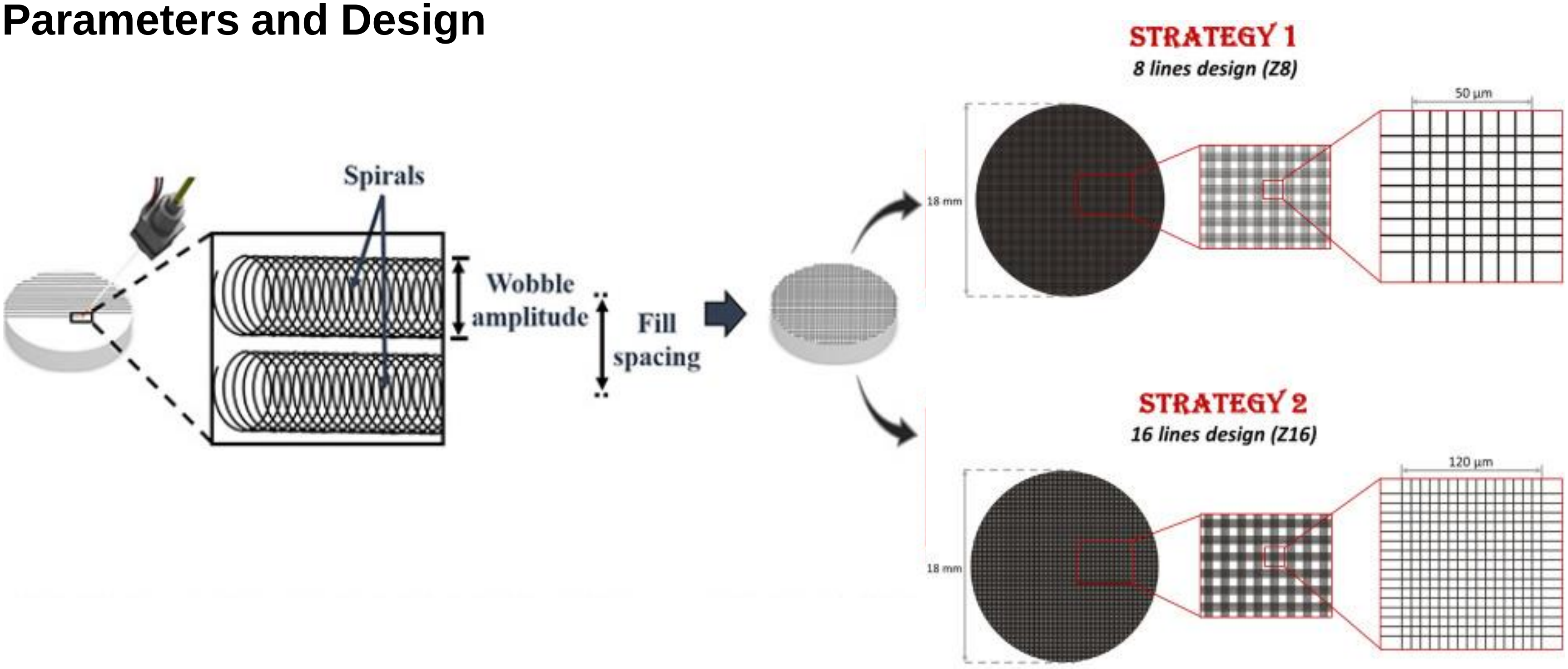
Textures

2.3 Patterns

Parameters and Design



Summary

Textures

2.3 Patterns

Parameters and Design

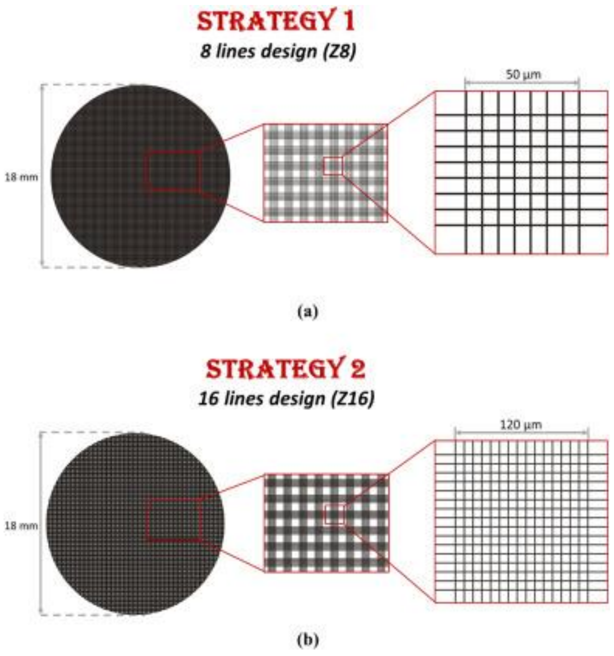


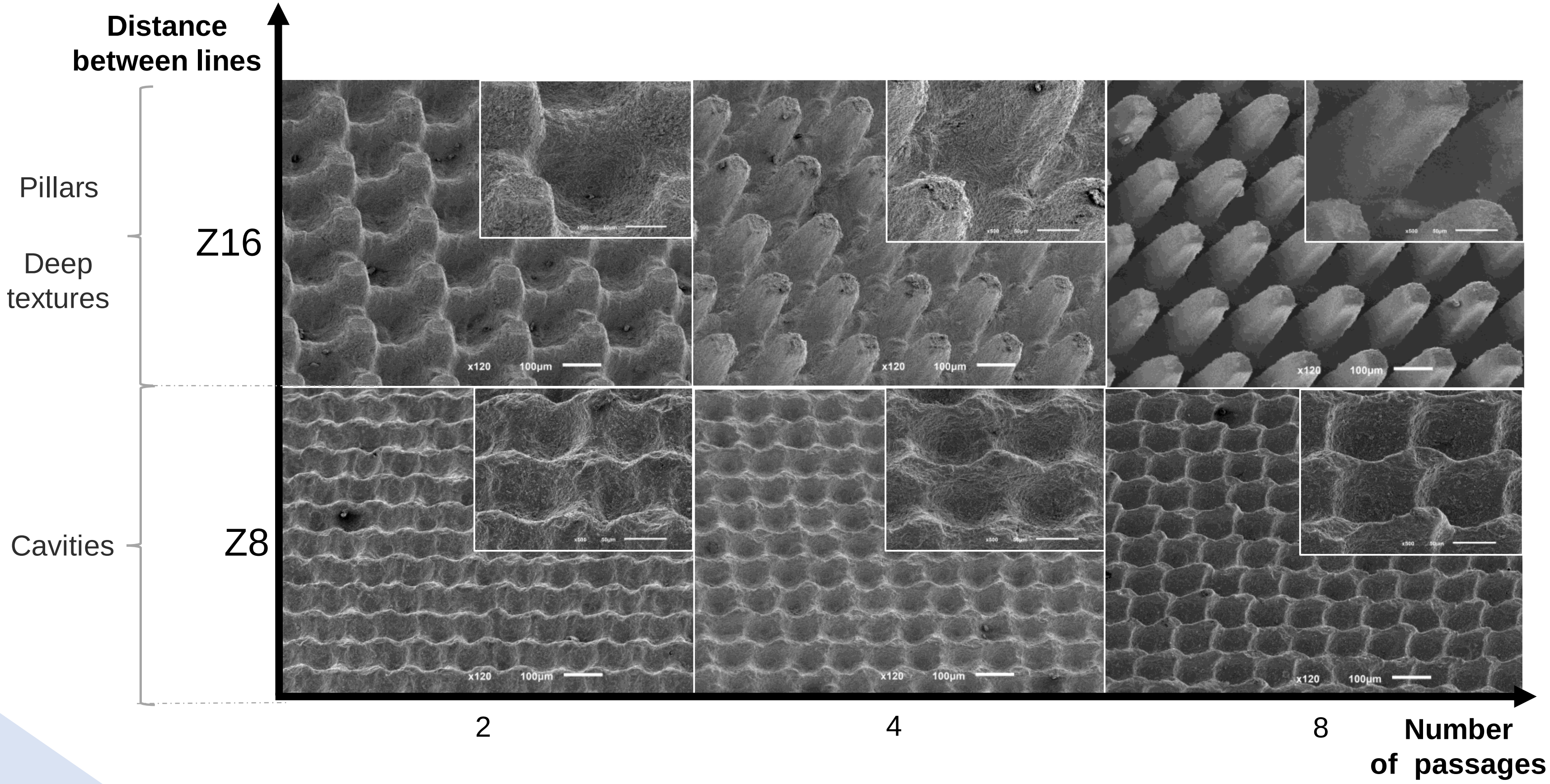
Table 1. Summary of the laser texturing parameters tested

Experiment	Strategy	Laser Power	Number of laser passages	Scan Speed (mm/s)	Fill spacing (mm)	Wobble frequency [Hz]
Z8P1.5L2	Z8 (8 lines design)	1.5	2	200	8	550
Z8P1.5L4			4			
Z8P1.5L8			8			
Z16P1.5L2	Z16 (16 lines design)		2			
Z16P1.5L4			4			
Z16P1.5L8			8			

Summary

Textures

2.3 Patterns



Summary

Textures

2.3 Patterns

Increasing the number of laser passages from **L1 to L8**

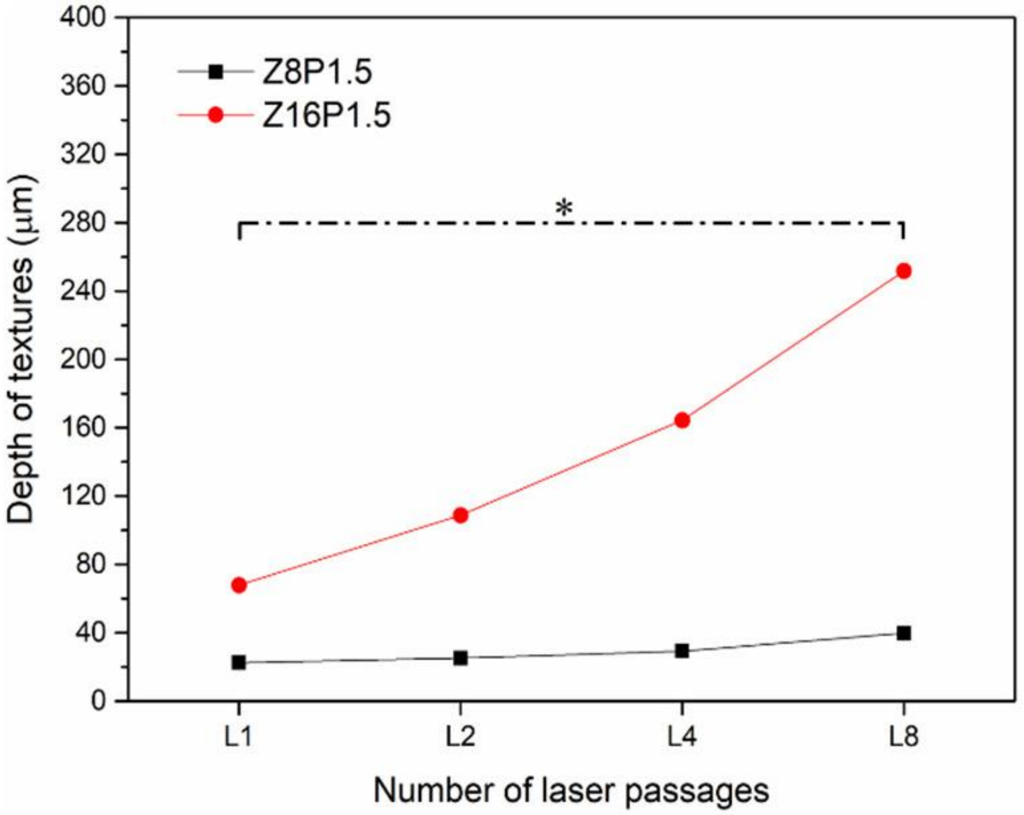
↓

High marking depths

↓

Higher irradiated energy on those areas

The increase of texture depth with number of laser passages is more significant for Z16



Graph 1. Evolution of depth of textures with the number of laser passages

Summary

Textures

Conclusions

- Increasing the number of laser passages from **L1 to L8**, while maintaining constant the laser power (P1.5) led to high marking depths, as a result of the higher irradiated energy on those areas;
- The opposite effect was achieved for a smaller number of **laser passages (L1)**
- The textures produced with **Z16** have much higher depth than the ones produced with **Z8**.
- It can be observed, in the case of Z8, an increase on **depth of cavities from L1 to L8** (from 22.4 to 39.8 μm) while in the case of Z16 there is a significant increase on depth of pillars from L1 to L8 (from 67.9 to 251.8 μm) ($p < 0.05$).

Summary

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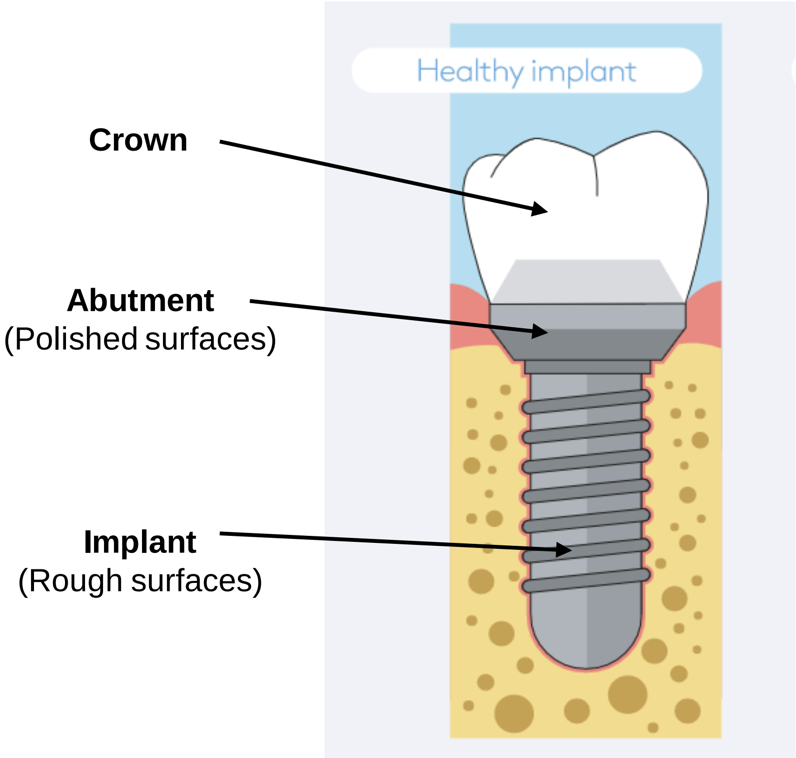
Textures

Conclusions

- Laser technology showed to be an **effective** and **versatile** method to produce different and complex textured zirconia surfaces under green state, without compromise zirconia mechanical performance
- The laser parameters and strategies showed to have a great influence on **geometrical definition** and **depth** of created textures
- Regarding the laser strategy, **cavities** were obtained from strategy 1 **(Z8),** while **pillars** were created from strategy 2 **(Z16).**

Textures

Biomedical application



Implant

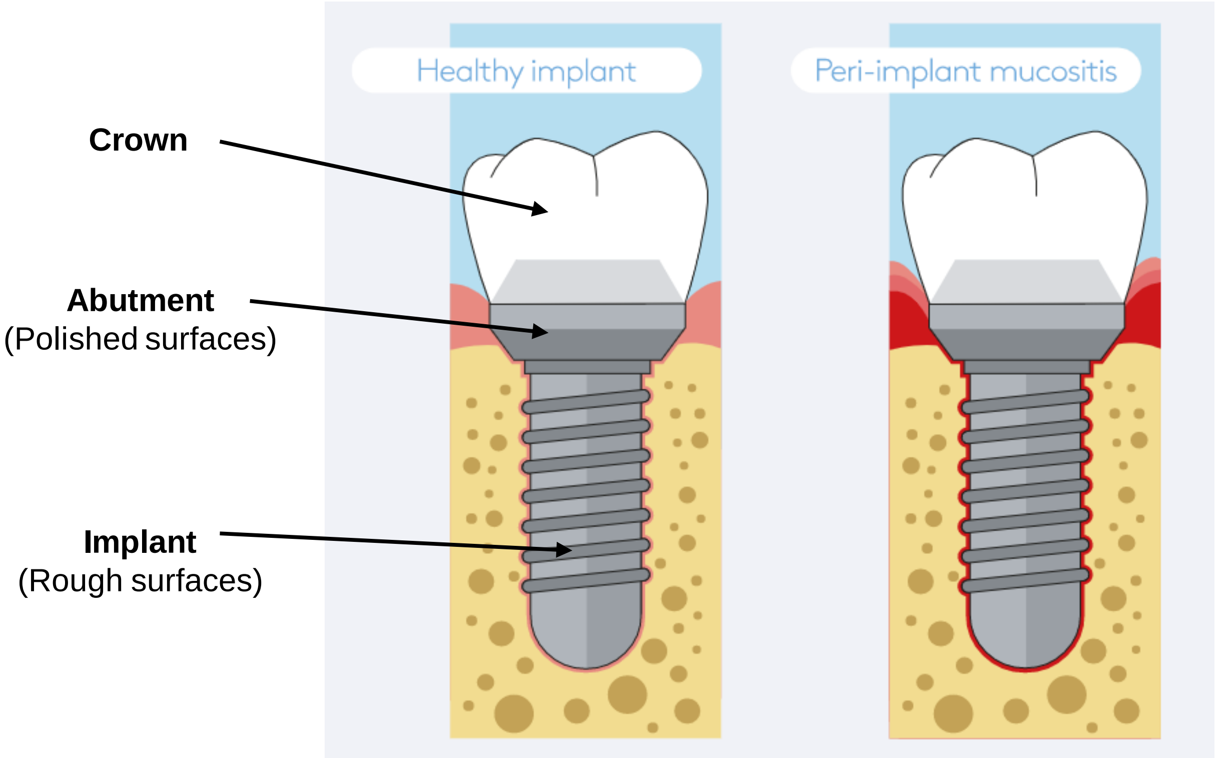
Rough surfaces are expected to help to enhance the primary and secondary stabilities.

Abutment

Commonly, bacterias tend to adhere to rough surfaces. In this sense, smooth surfaces are reported in literature as preferable solutions to avoid this bacterial adhesion.

Textures

Biomedical application



The bacteria adhere to the implant-bone interface and form a biofilm.

Summary

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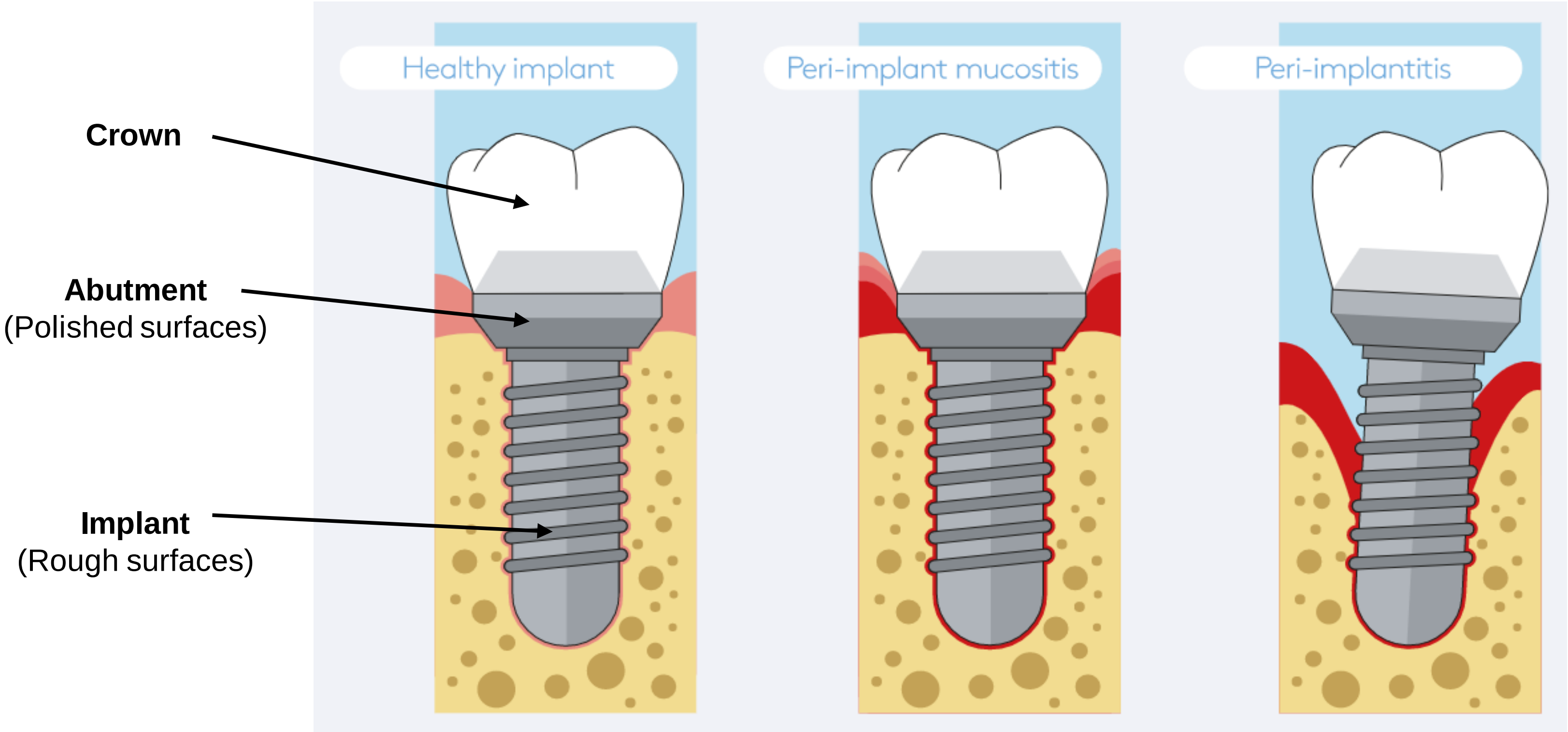
Textures

Biomedical application

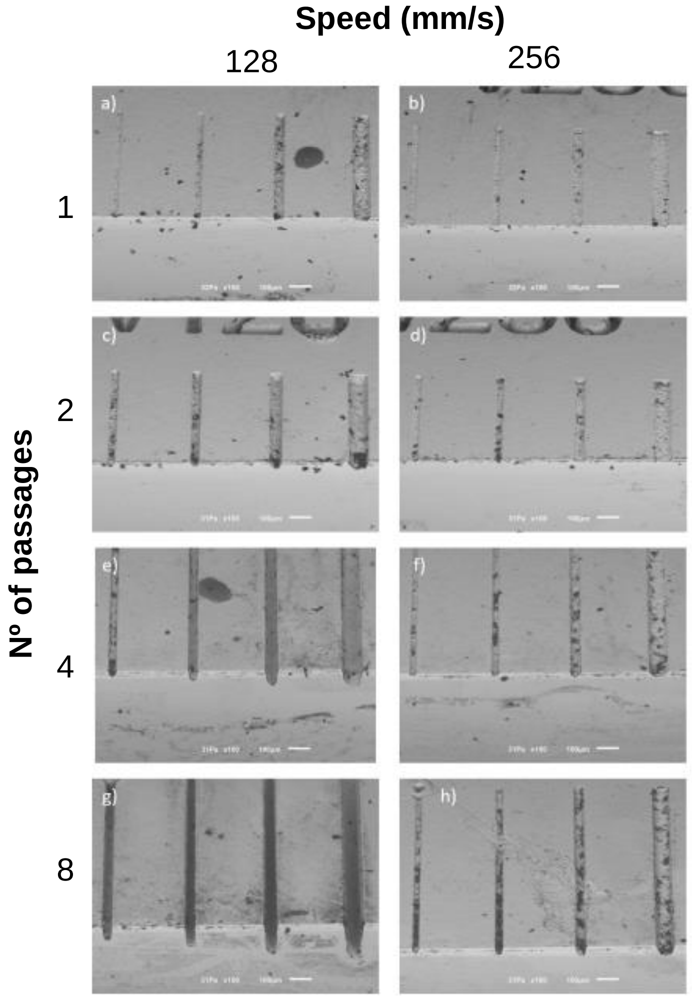


The bacteria adhere to implant-bone interface and form a biofilm. Consequently, gingival and bone recession occcur and the implant may lose its stability.

Summary

Textures

3.3 Groove



SEM images of the microgrooves produced via laser ablation: 128 and 256 mm/s; 1, 2, 4 and 8 passages; 1, 2, 4 and 8 lines

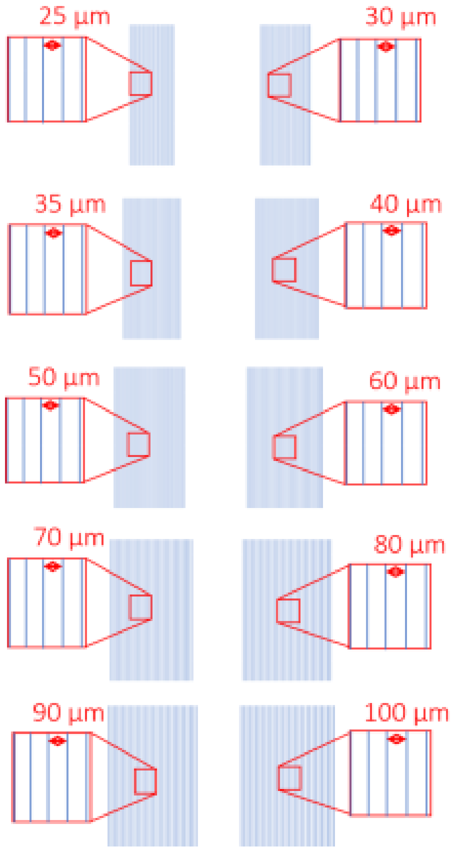
From all the obtained results, the most adequate profile dimensions considering the physical barrier correspond to condition

N1 S256 L1 (1 scan, 256 mm/s scan speed and 1 line).

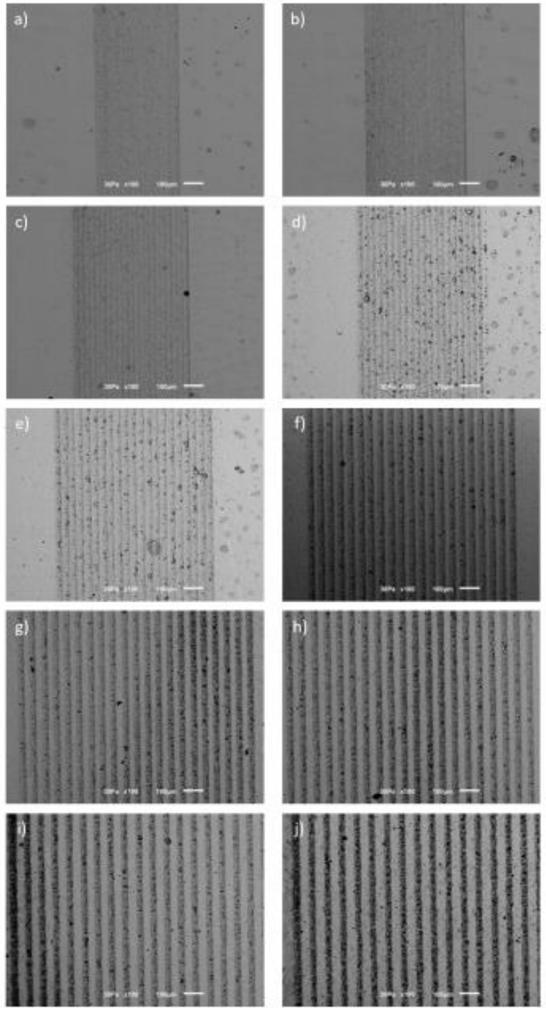
Summary

Textures

3.3 Groove



General representation of the drawing reproduced in software inskape to use in groove interspace study.

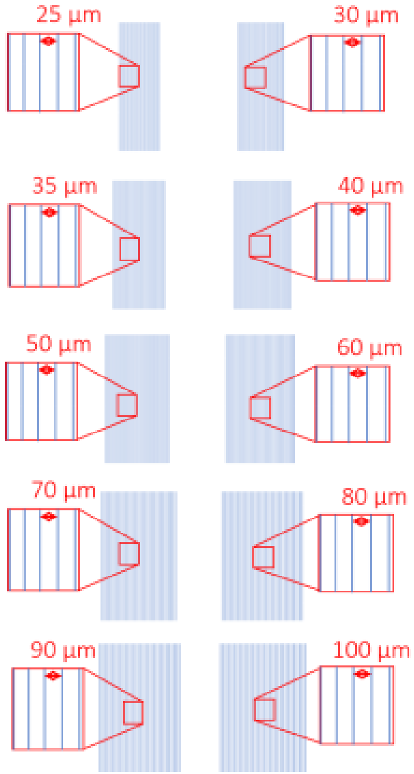


Top SEM images of Y-PSZ micromachined surface with 2D drawing line spacing parameter from sample: a) S25; b) S30; c) S35 d) S40 e) S50 f) S60 g) S70 h) S80 i) S90 j) S100.

Summary

Textures

3.3 Groove



General representation of the drawing reproduced in software inskape to use in groove interspace study.

Table with line spacing and ridge width average values (mean±SD) of micro machined Y-PSZ obtained by software Image J

2D drawing	Grooves interspacing ± SD (µm)	Ridge width ± SD (µm)
S25	-	
S30	-	
S35	-	
S40	28.8 ± 2.1	≈ 0
S50	38.3 ± 1.0	8.5 ± 0.2
S60	49.7 ± 1.7	21.4 ± 1.5
S70	57 ± 1.5	22.7 ± 1.1
S80	62.4 ± 1.0	28.1 ± 0.8
S90	72.7 ± 1.7	35.8 ± 2.4
S100	75 ± 1.5	40.7 ± 0.8

A line spacing inferior to 40 µm (S40) the groove walls overlap and cease to exist.

Therefore, successive grooves closer than approximately 28.8 µm are not attainable by this specific technology

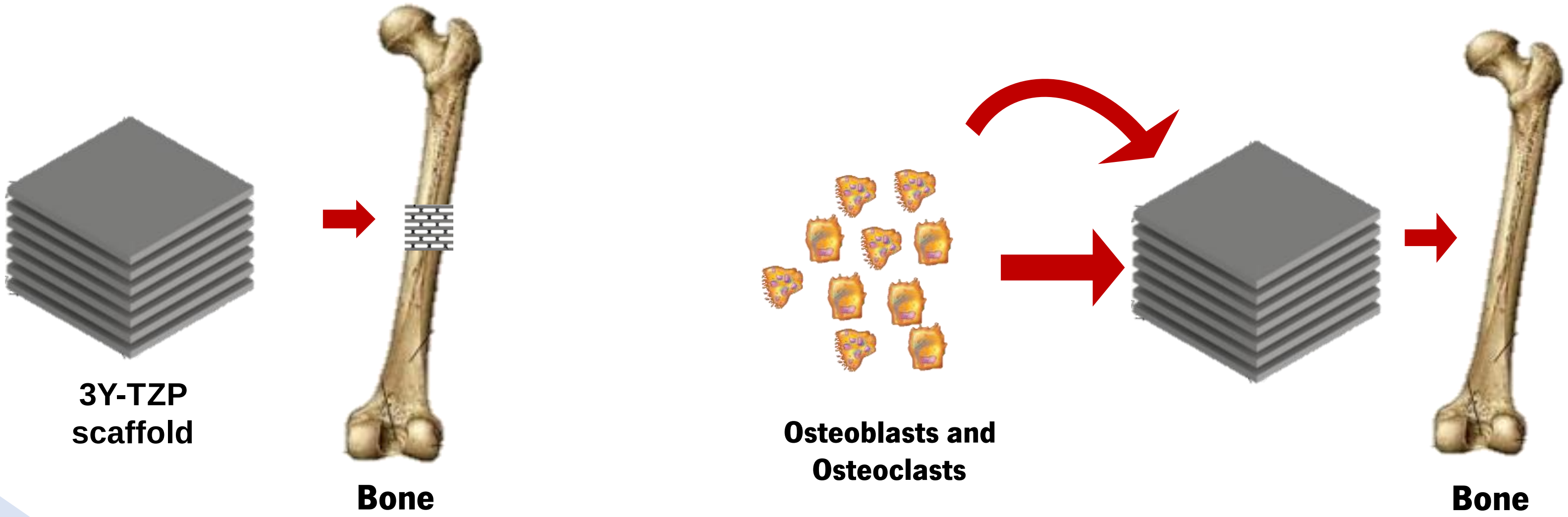
Summary

Cellular Structures

Cellular Structures

Scaffold features and properties

- Complex interconnected structure can allow **bone ingrowth**, **vascularization** and provide a **good initial stability**
- Novel thin-walled zirconia scaffold
- Zirconia scaffold lower than zirconia bulk material
- Less stiff geometry with small (walls and floors) dimensions.

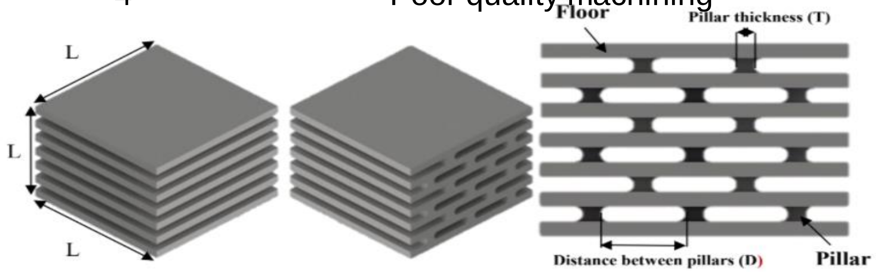


Summary

Cellular Structures

Laser Machining Parameters

Iterations	Power	Speed (mm/s)	Wobbel	Height	Observations
1	0.6 W (10%)	2-20	200-20	2	Optimum conditions
2	0.6 W (10%)	2-20	200-20	4	Desfocused
3	0.6 W (10%)	2-20	200-20	3	Desfocused
4	0.9 (15%)	3-30	300-30	3	Desfocused
5	0.9 (15%)	4-40	300-30	3	Desfocused
6	1.2 (20%)	10-100	300-30	4	Poor quality machining
7	1.2 (20%)	20-200	300-30	4	Poor quality machining
8	1.2 (20%)	4-400	300-30	4	Poor quality machining



Summary

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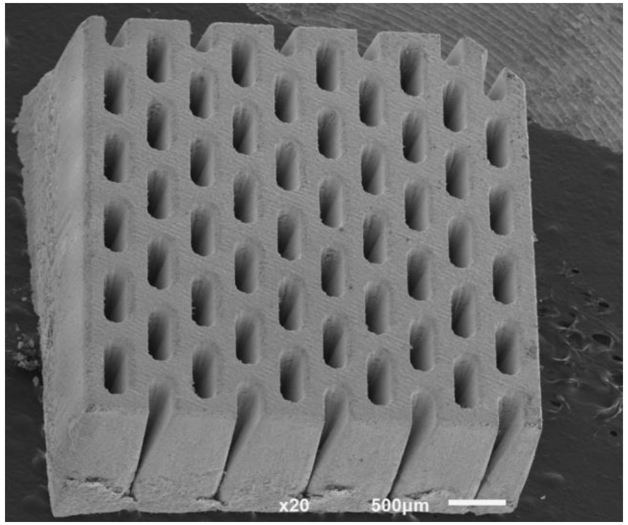
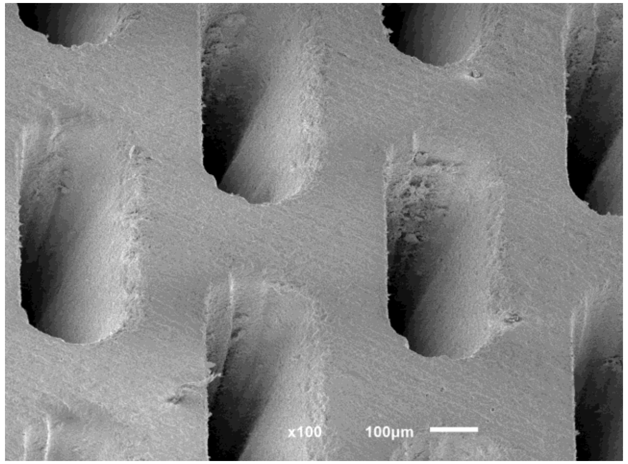
Cellular Structures

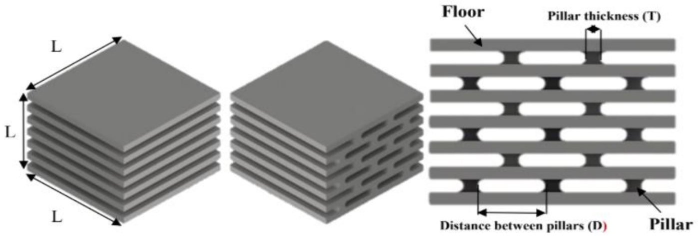
Results

Laser machining allows to obtain holes with smaller dimensions when compared with CNC machining

Difficulty to machine parts with high height







Summary

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Conclusions

- ✓ **Regular** geometries such as **cavities** or **pillars** were observed
- ✓ **High quality** machined structures (laser does not machine cavities but crosses the entire bulk).
- ✓ Some texturing strategies are suitable to obtain high quality surface textured patterns

Summary

Conclusions

Cell adhesion and proliferation on biomedical surfaces

↓

well-defined geometric features like pits , grooves pillars , cavities and ridges

↓

controlled response in the bone-implant contact region ✓

Summary

Conclusions

Cell adhesion and proliferation on biomedical surfaces

↓

well-defined geometric features like pits , grooves pillars , cavities and ridges

↓

controlled response in the bone-implant contact region ✓

Sandblasting and chemical etching

↓

Scar tissue formation ✗

Production of regular and defined features by laser

↓

Reproducible surfaces

↓

Precise control over the human body response ✓

Summary

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Acknowledgments

This work is supported by FCT (Fundação para a Ciência e a Tecnologia) through the grant SFRH/BD/148031/2019, the project UIDB/00285/2020, UIDB/04436/2020 and, also by project UIDP/04436/2020.

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NORTE 2020 PROGRAMA OPERACIONAL REGIONAL DO NORTE

Summary

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September 2020

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UMINHO CMEMS CENTER FOR MICROELECTROMECHANICAL SYSTEMS