# BIOLOGICAL PROPERTIES OF NANOSTRUCTURED TITANIUM MADE BY SPD

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## SUMMARY

MECHANICAL BACKGROUND:

This paper reports the results of mechanical and biological testing of nanostructured titanium (n-Ti), and demonstrates its applicability for medical application and dental implants.

Nanostructuring involved SPD processing by equal-channel angular pressing followed by thermo-mechanical treatment (TMT) resulted in a major reduction in grain size. The initial 25 micrometer equal-axed grain structure of the titanium rods was reduced to 150 nm after SPD. Nanostructuring of CP titanium by SPD processing is improving the mechanical properties mainly sufficient mechanical strength.

The values of mechanical properties parameters of nanostructured CP Grade 4 titanium (UTS, YS, Elongation) have increased more than 2 times, the fatigue strength is almost 80 pct. higher than conventionally processed CP titanium and even higher than that of Ti-6Al-4V alloy.

## Biological properties:

OBJECTIVES:

The biological testing of nanostructured titanium (n-Ti) was performed on 2 levels:
Cytocompatibility Tests and animal study on pigs to prove the applicability for medical application and dental implants.

METHODS:

1. material samples of comercialy pure Titanium (cpTi) and Nanostructured Titanium (n-Ti) were exposed to mice fibroblast cell cultivation, the surface cell occupation was mesured and quantified.
2. Sets of different implants - warious surface treatmend ( includind simple machinated, etched, laser pitched..) were implanted into Pigs mandibles and tibias.
After sacrificing , the Bone / Implant Contact (BIC) parameter was calculated.

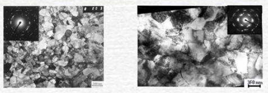
RESULTS:

The preliminary results are demonstrating excelent properties of Nanostructured Titanium surface in both tests.
Cytocompatibility tests utilizing fibroblast mice cells L929 were undertaken to verify the biological properties of nanostructured CP titanium compared to conventionally processed grained CP Ti. The cell investigation shows that fibroblast colonization of the CP Grade 4 titanium surface dramatically increases after nanostructuring and suggest that a high osteointegration rate should be expected with nanostructured CP Grade 4 titanium.

CONCLUSION:

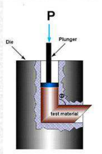
Nanostructured commercial purity titanium produced by severe plastic deformation (SPD) is a material considered to be mechanically and biocompatibility suitable for medical applications.

## Equal Channel Angular Pressing ( ECAP) method



a) b)
Typical nanostructures of CP Ti (a) and Ti –64 (b).

| Material | State | Grain size, nm | Strength,MPa | El., % |
|---|---|---|---|---|
| CP Ti | CG | 1500 | 450 | 24 |
| | ECAP+CR | 150 | 1100 | 8 |
| Ti-64 | CG | 1000 | 900 | 18 |
| | ECAP+ upsetting | 400 | 1400 | 8 |




$$\varepsilon = \frac{N \cdot 2}{\sqrt{3} \cdot ctg\,\phi}$$

Fig 1. Principle of ECAP method for Nanostructure Titanium production

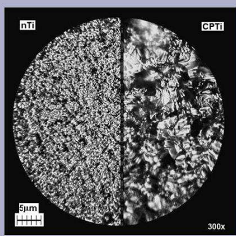



Fig. 2. Surface relief after hydrofluoric acid treatment of nanostructured (left) and CP Grade 4 titanium (right) surfaces.

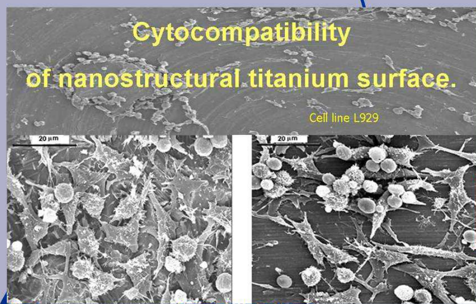



Fig. 3. Occupation of the mice fibroblast cells L929 after 24 hours; Nanostructured (left) and conventional (right) CP Grade 4 titanium.

What is a plane occupied by cells on the different surface?

After 72 hours

| MATERIAL | surface | finishing | % of occupied surface |
|---|---|---|---|
| ASTM F67-00 | A1 | turning | 49,07 |
| ASTM F67-00 | A2 | turning&plasma | 53,11 |
| ASTM F67-00 | A3 | turning&HF-etching | 53,01 |
| ASTM F67-00 | A4 | turning&HF&plasma | 54,16 |
| NANOMATERIAL | N1 | turning | 68,76 |
| NANOMATERIAL | N2 | turning&plasma | 65,41 |
| NANOMATERIAL | N3 | turning&HF-etching | 87,22 |
| NANOMATERIAL | N4 | turning&HF&plasma | 86,96 |

- Cell adhesion is affected by a character of surface
- Nanomaterial has a significance to quickly cell adherence

## Preclinical tests for Bone Implant Contact Calculations

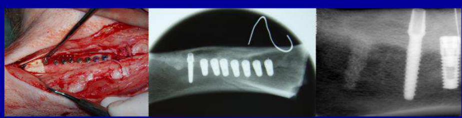


Fig 4. Preclinical tests for Bone Implant Contact Calculations
Implant samples inserted in pig tibia; X-Ray after sacrifice; X-ray detail of nanoimplant and implant F.

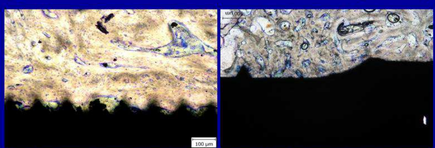



Fig 5. Implant A, left mandible, pig 3 ( longitudinal section)

Fig 6. Nanoimplant, left tibia, pig 4 ( longitudinal section)

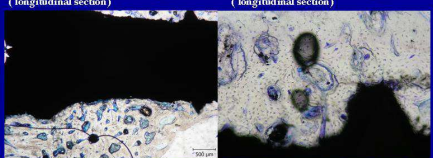



Fig 7. Nanoimplant, left tibia, pig 4 ( longitudinal section)

Fig 8. Same image as in figure 7 but with a higher magnification

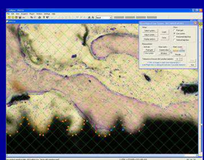


**Fig 9. BIC measurement**
**In order to obtain results for the evaluation of osteointegration**
The BIC was evaluated by the use of the programme Elipse in a rectangular network with each retangle being 75 x 75 px to 150 x 150 px. Stereologically there are 200 evaluation points for the 1 histological preparation. We have evaluated the meeting of network-margin of implant by the following formula:
**Contact of network-Bone implant contact / network-margin of implant x 100**
**Or alternatively Bone/Implant x 100**

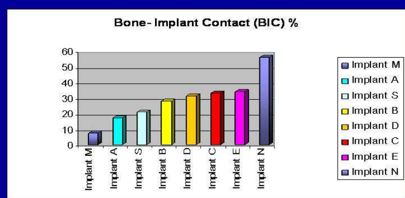



**Fig 10. BIC results**
**A** = laser 5 µm pores, pitch 15 µm; **B** = laser 10 µm pores, pitch 20 µm; **C** = laser 20 µm pores, pitch 30 µm; **D** = laser 5 µm pores, pitch 5 µm; **E** = laser 10 µm pores, pitch 10 µm; **F** = laser 20 µm pores, pitch 20 µm; **S** = sandblasted; **M** = machined ; **N** = machined etched NANO

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