Mechanical characterization of nanostructured composite materials for potential applications as a bone implants

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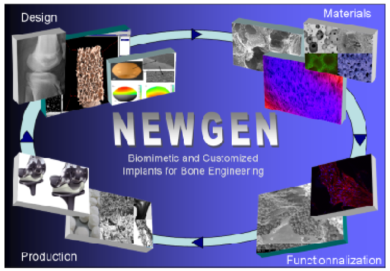

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OUTLINE

- Aim/Motivation
- Introduction
- Synthesis
- Characterization
- Results
- Conclusion

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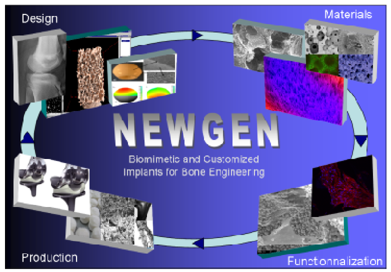

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AiM/Motivation

- **AIM** of this research is to obtain nanostructured mesoporous CaP coated zirconia or titania scaffolds which have all the properties of the hydroxyapatite with enhanced mechanical properties.

- Currently, there is no single material which can meet all the required properties for good scaffold :

  1.bioactivity

  2. biocompatibility

  3. mechanical properties

for applications in bone tissue engineering.

cost
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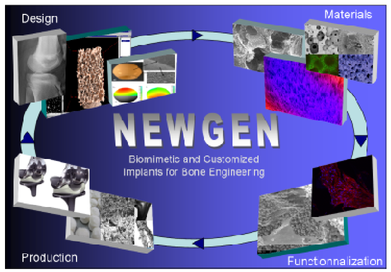

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Introduction - Zirconia

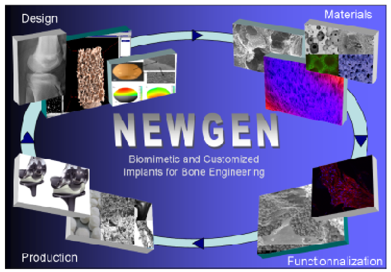
- ZrO_2 is bioinert and due to its low reactivity, together with its good mechanical and optical properties, is widely applied for orthopedic and dental restorative
- Bioactivation with CaP (HAP)

- Stabilizing agents such as magnesia, ceria, yttria and calcium to retain the tetragonal phase in a metastable condition at room temperature

- Calcium phosphate can be prepared and applied to the surface of the material by various methods whereby the last decade emphasizes biomimetic process

- Advantages of porous Zirconia: less specific weight, and increased surface roughness, which makes it suitable for different surface modifications

- Problem with stabilized ZrO_2 ceramics is changes in the structure of the surface that may occur after connecting with the tissue, decrease mechanical properties.



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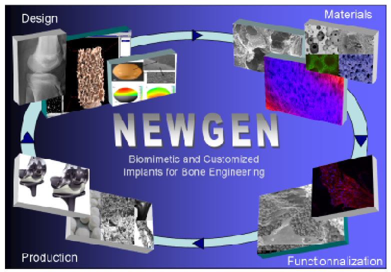
Introduction - Titania

- Titanium and titanium alloys are frequently used as orthopedic and dental implants
- Favorable properties: good ductility, tensile and fatigue strength, modulus of elasticity matching that of bones, low weight, and good biocompatibility.
- Nanotubular surface enhances adhesion, growth and differentiation of the cells
- Increase the roughness of titanium implants on the nanoscale, providing the surface similar to that of a human bone
- Nanotubular layers provide a high surface-to-volume ratio with controllable dimensions
- Further enhancement bioactivity of titanium implant with nanotubular surface by hydroxyapatite deposition into the titania nanotubes which further promotes bone ingrowth.



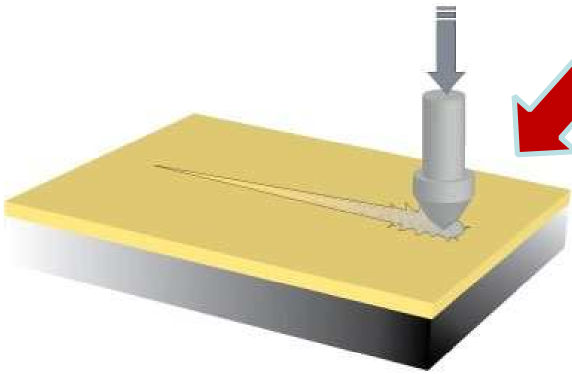
Characterization

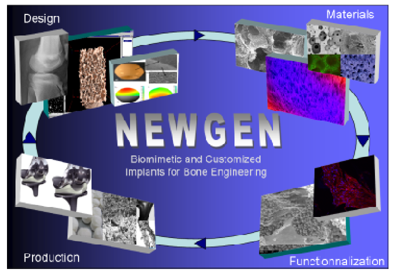
Raman spectrometer Horiba Jobin Yvon T64000

SEM TM-Hitachi

CSM Revetest scratchtester

Monitoring
F_z (applied load)
F_x (lateral force)
Z -penetration depth
AE-acoustic emission





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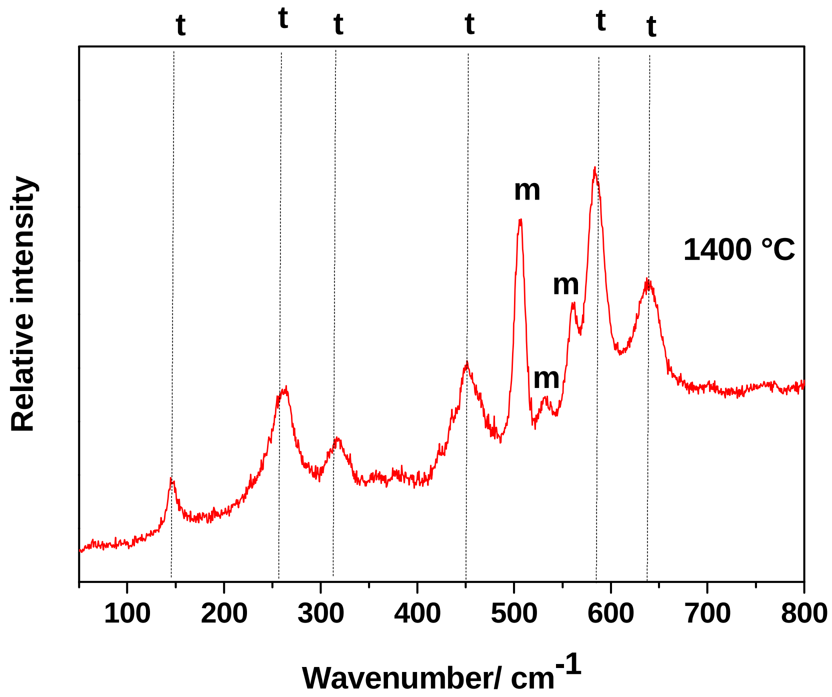
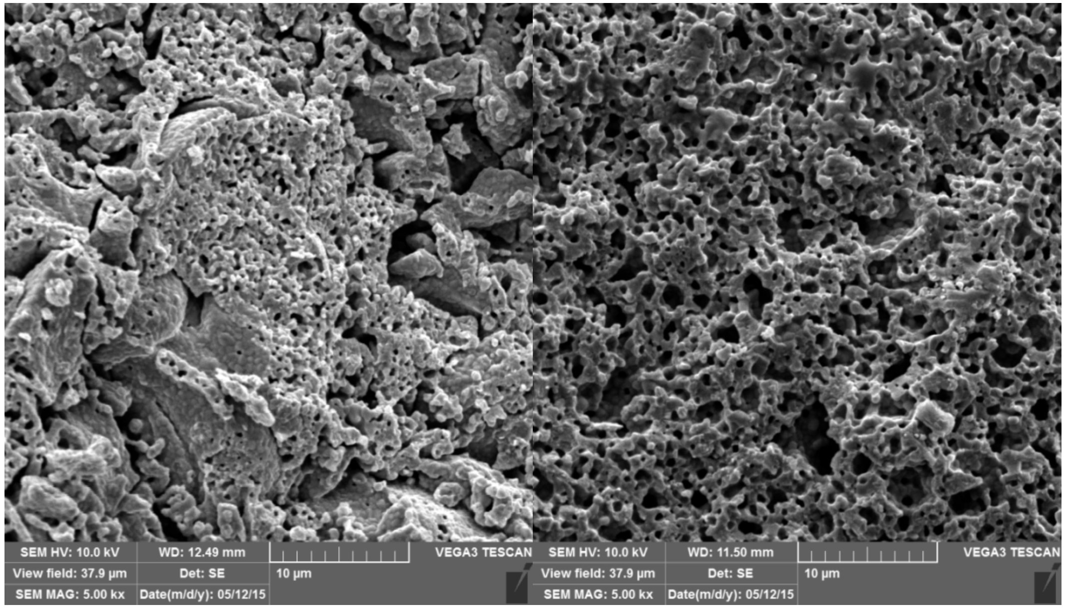
Synthesis

➢ Sol-gel porous ZrO_2+10%Y_2O_3+annealing at 1400°C





Porosity 15% - Arhimed method

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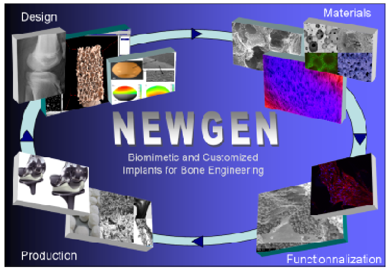

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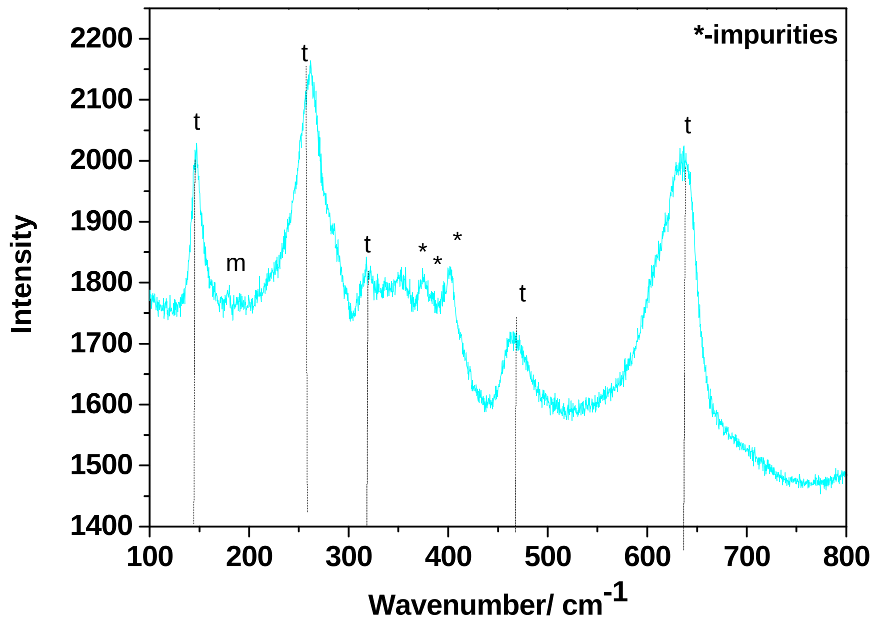
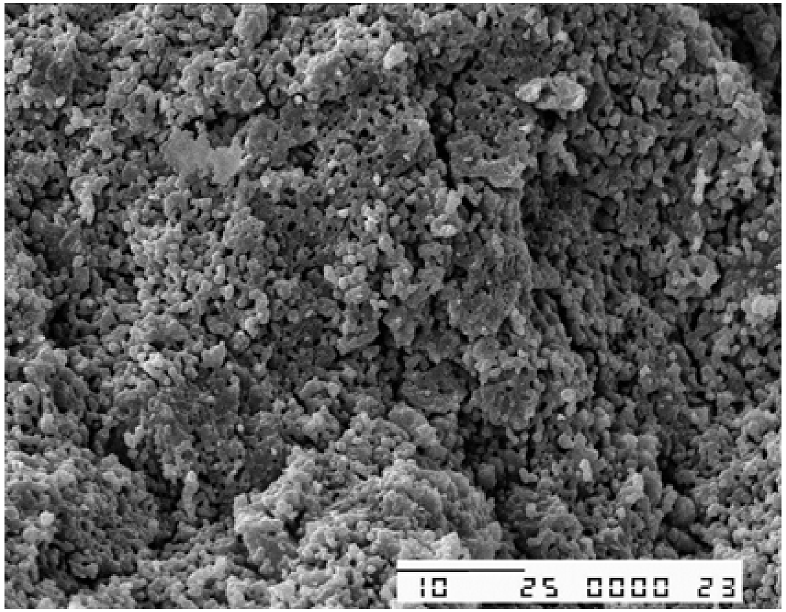
Synthesis

➤ Mechanochemical synthesis of porous ZrO_2+10%Y_2O_3+30%Al_2O_3 +annealing at 1400°C





Porosity 11% - Arhimed method

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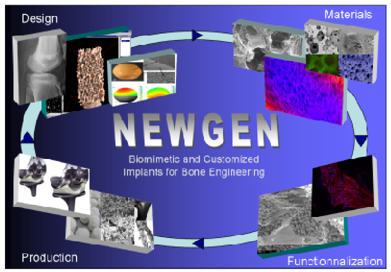

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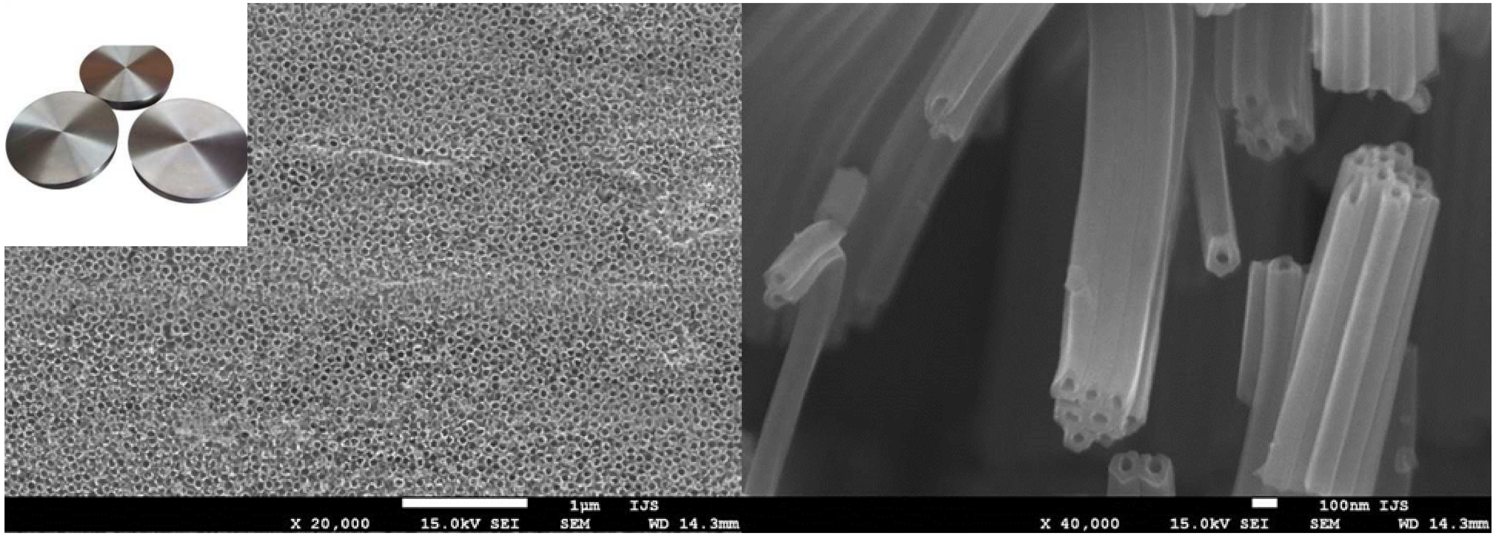
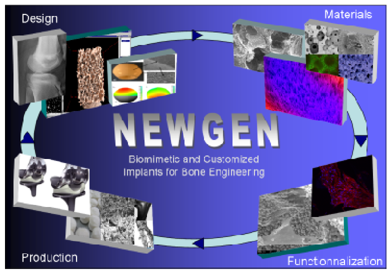
Synthesis

- Anodization of Ti-disc ⟶ TiO_2 porous structure



- Annealing at 500°C and 1200°C

anatase rutile



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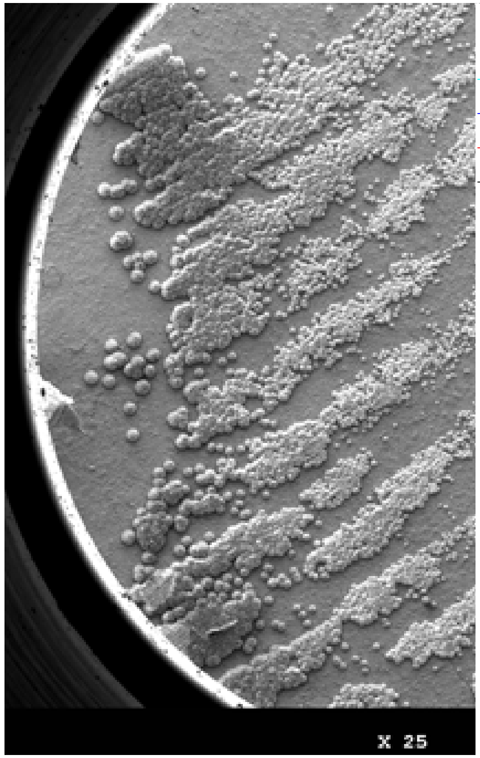
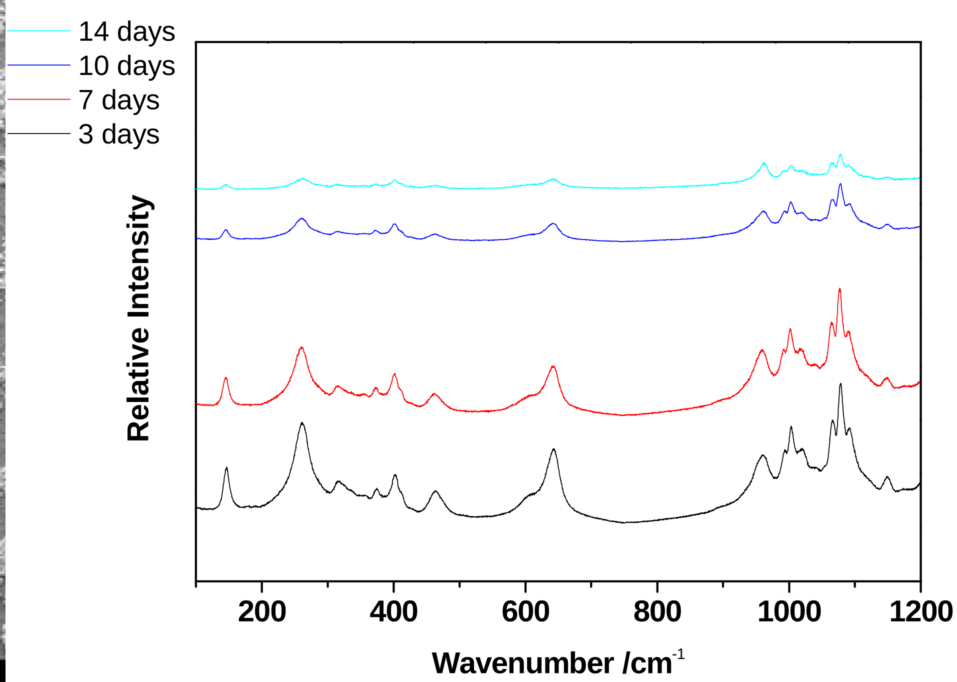
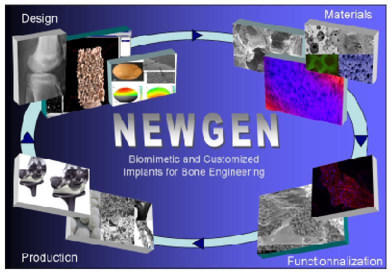
CaP deposition-SG





Sol-gel ZrO_2+10%Y_2O_3+CaP -14 days in MCS solution, MCS change every day



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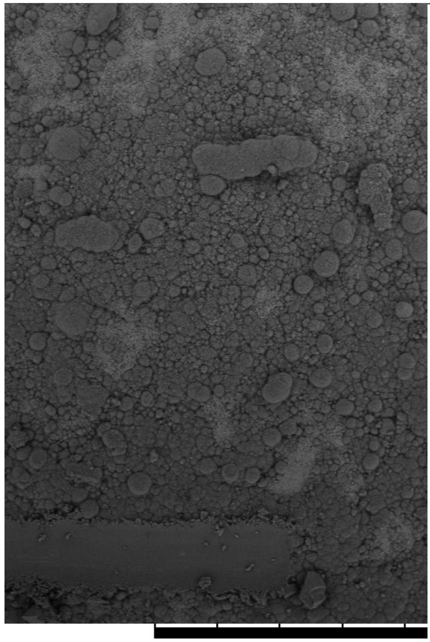
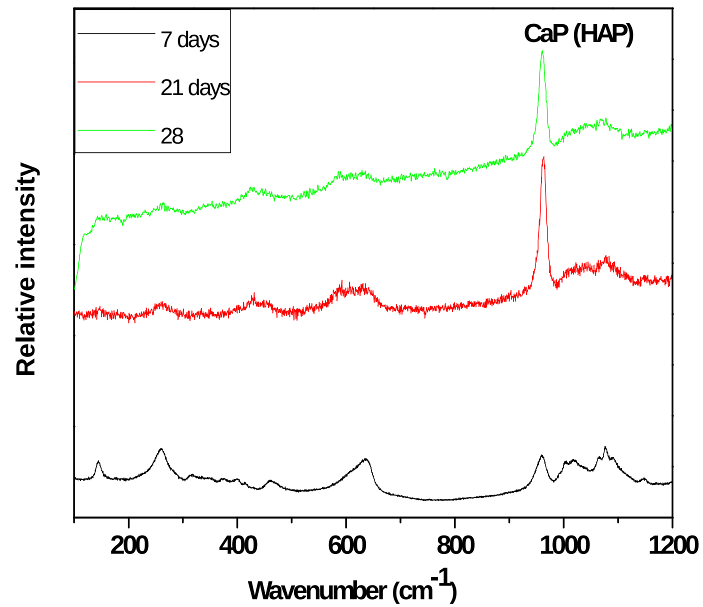
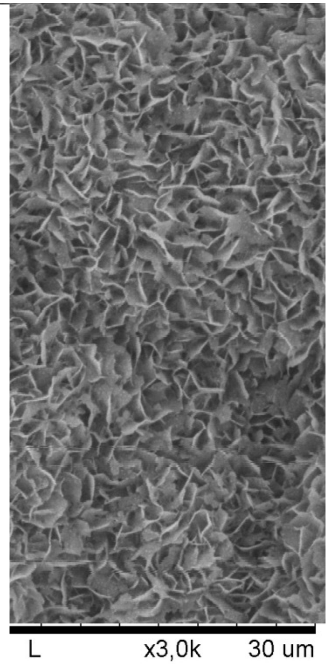
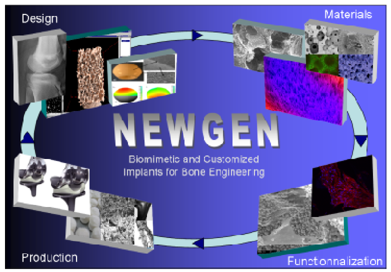
CaP deposition-MH







MH ZrO_2 + 10% Y_2O_3 +30% Al_2O_3+ CaP – 28 days in MCS solution, MCS change every day



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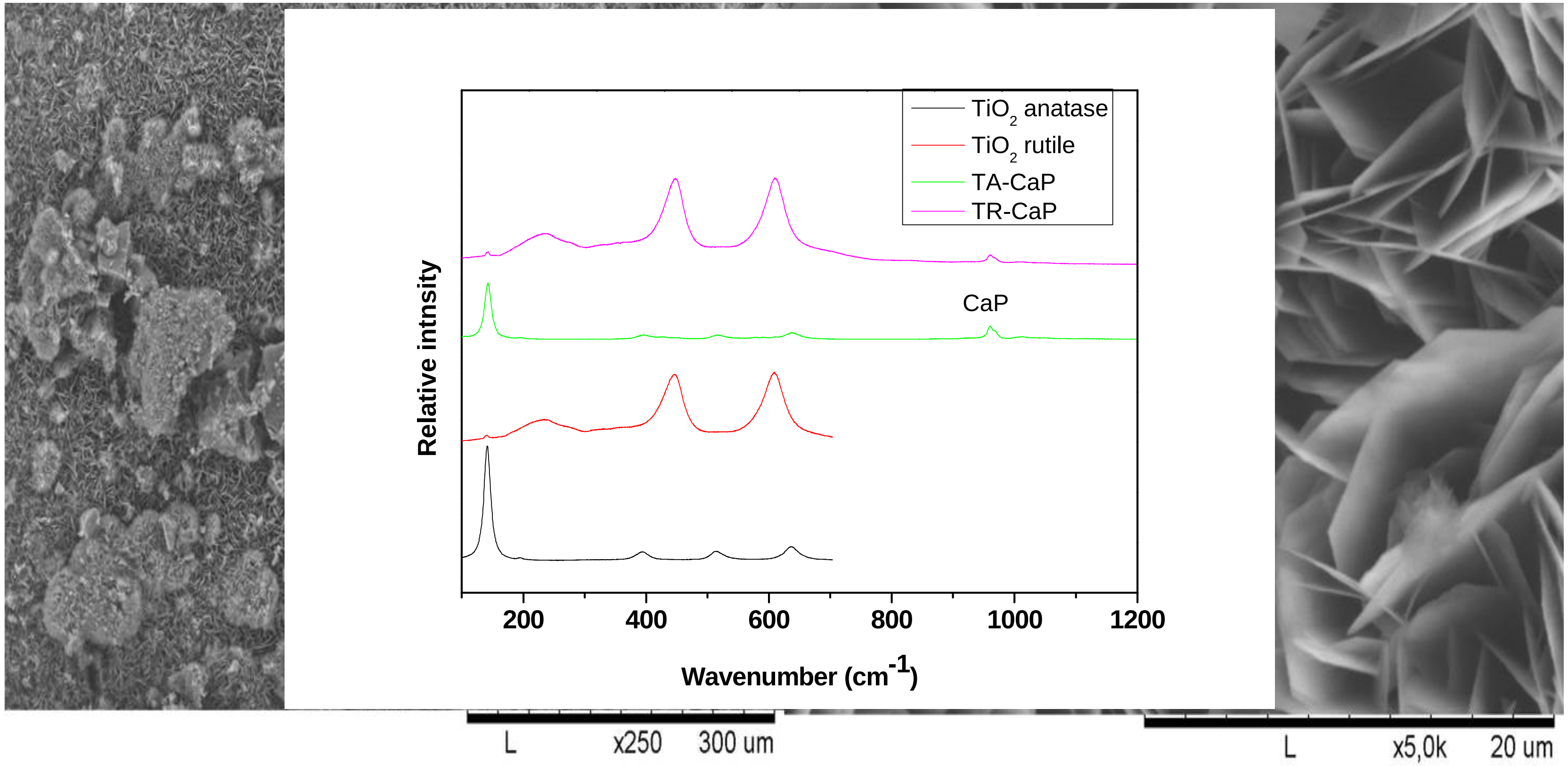
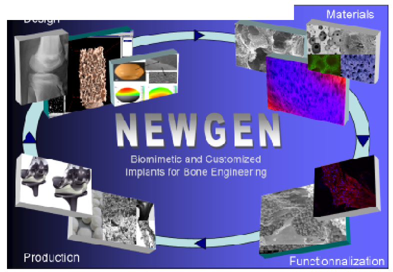
CaP deposition-TiO_2 NT



TiO_2 nanotube arrays+immersing in MCS CaP solution for 28 days



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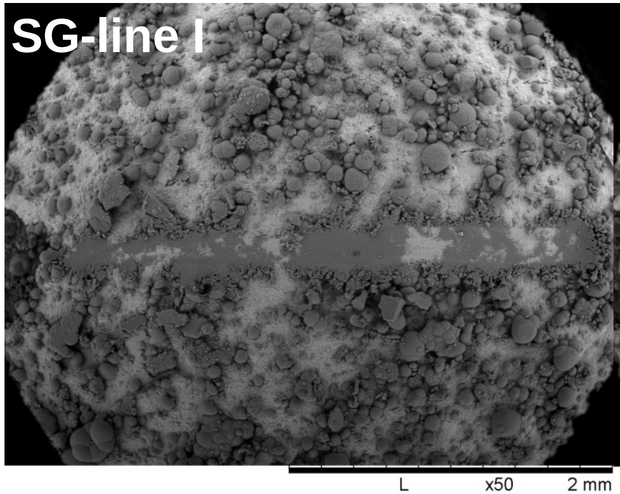
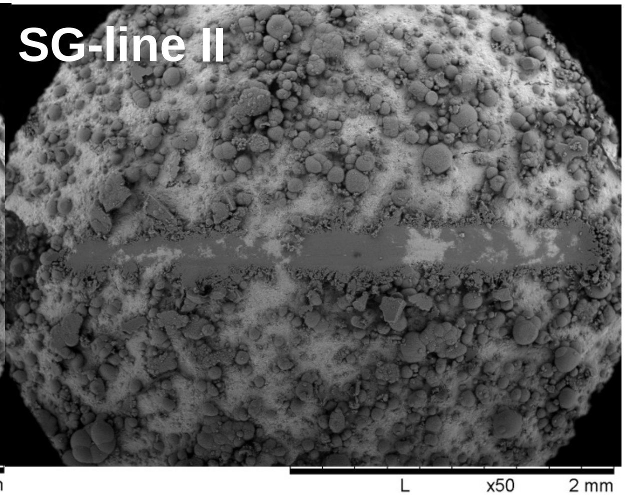
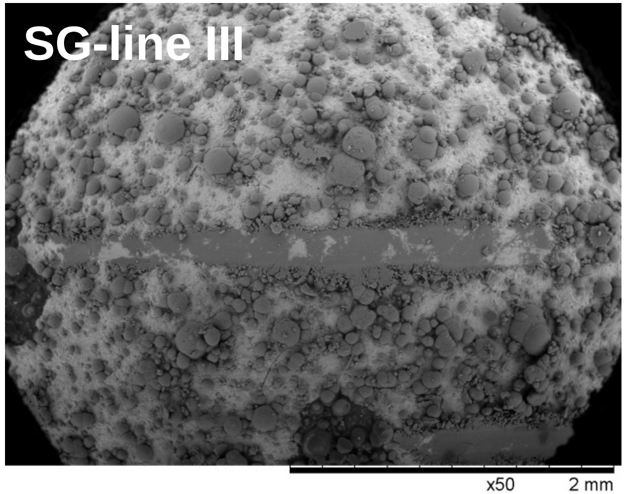
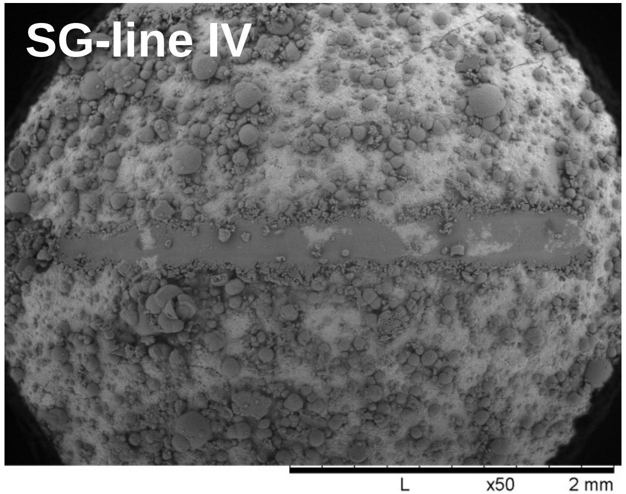
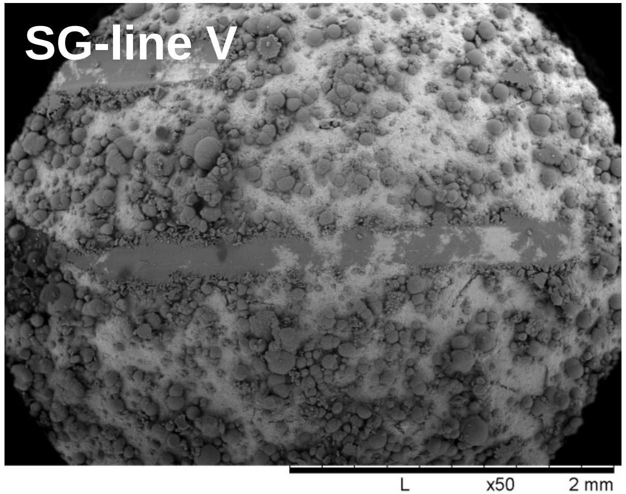
Results
Mechanical characterization-linear scratch test











cost
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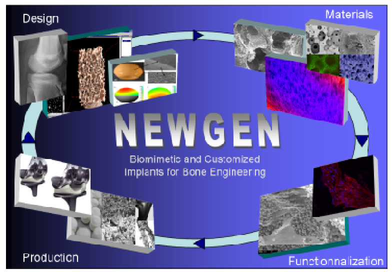

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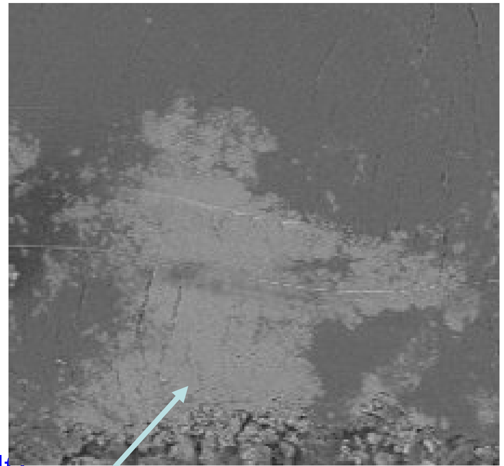
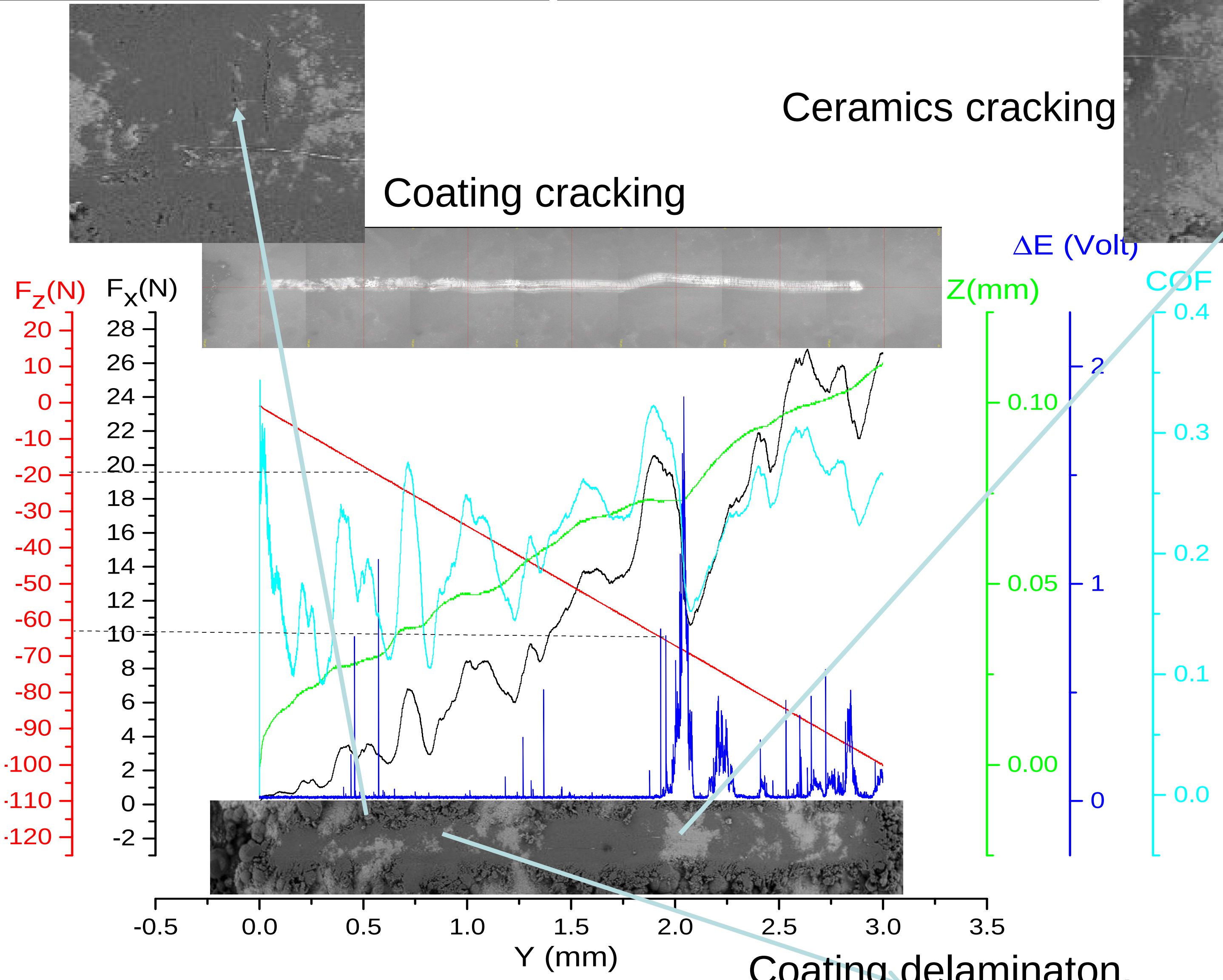
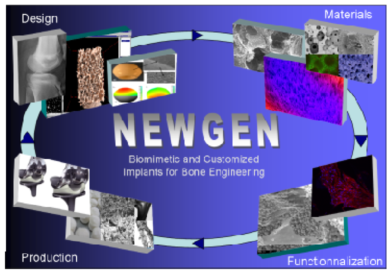
Mechanical characterization-SG sample





SG sample, line II load 1-100 N, 120 s, 3 mm



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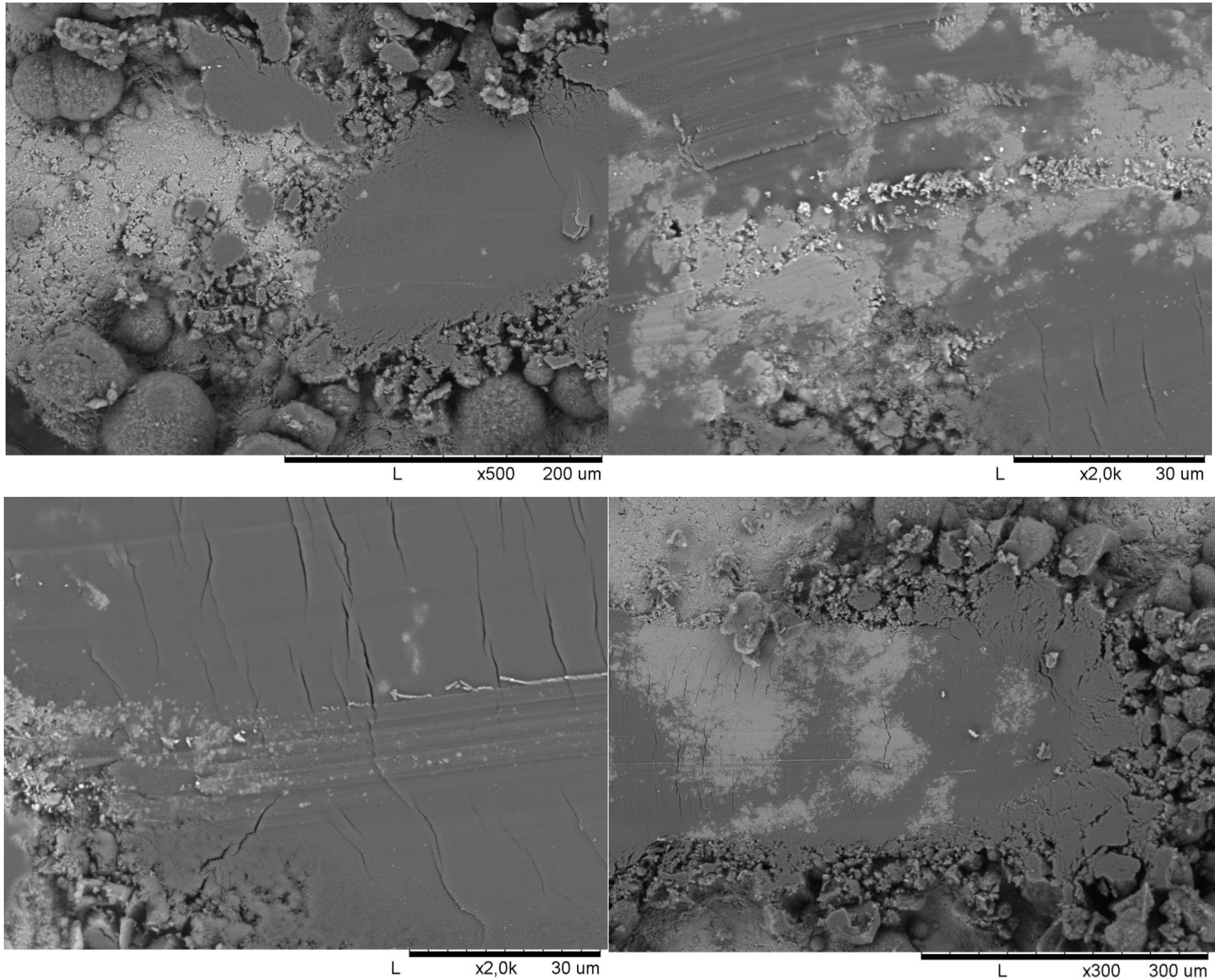
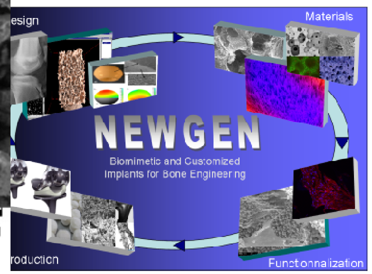
Mechanical characterization-SG ample





Mechanical characterization-SG

	Coatings failure (Lc_1, N)	Ceramic failure (Lc_2, N)
1	20	50
2	22	55
3	17	42
4	16	48
5	20	40
6	20	60
7	17	45
8	18	50
9	18	45
10	20	50
11	18	65
12	15	55
13	20	45
14	18	40
Mean value	**18**	**49**
error	**2**	**7**

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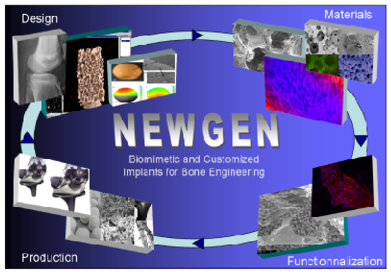

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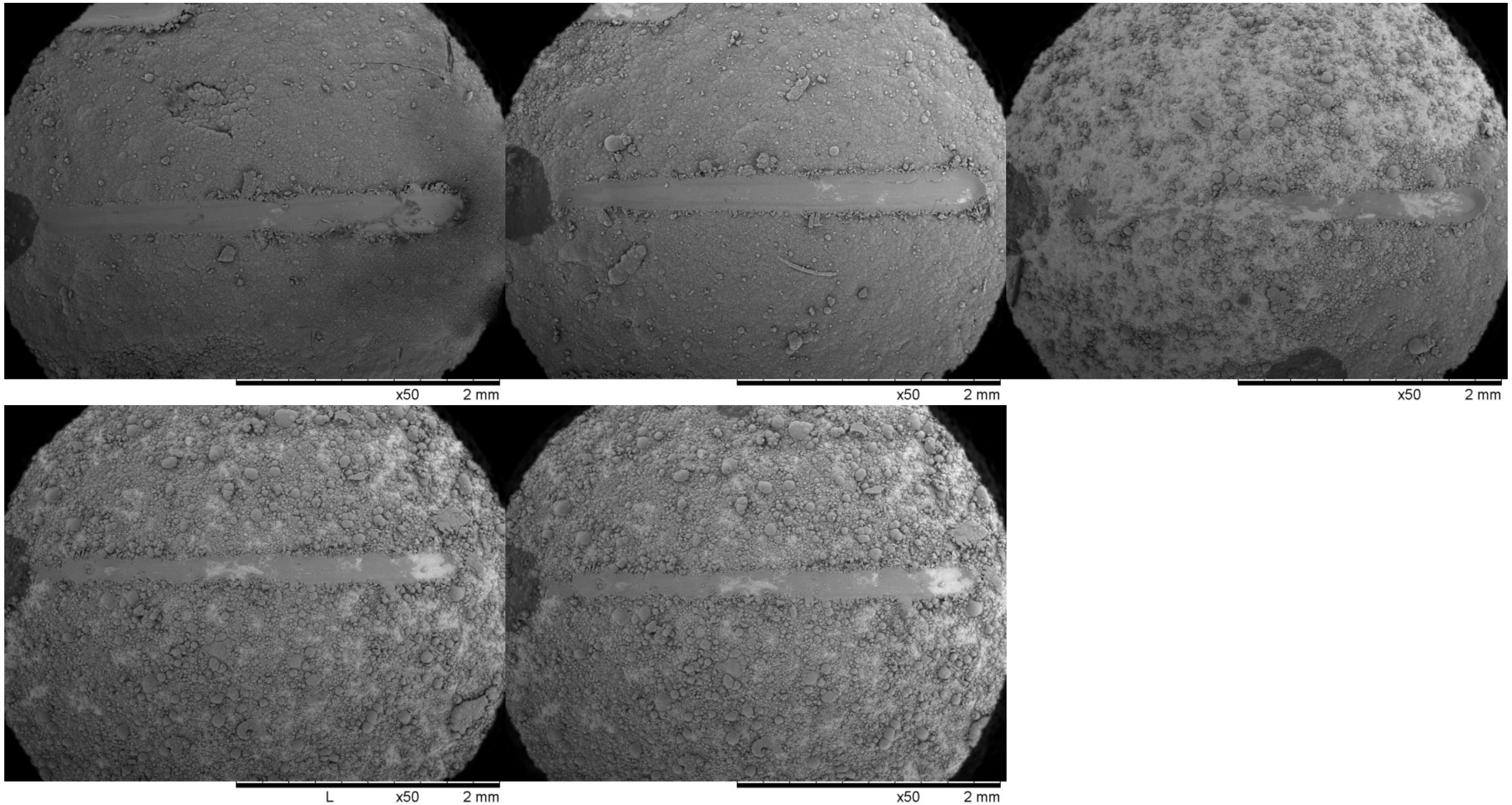
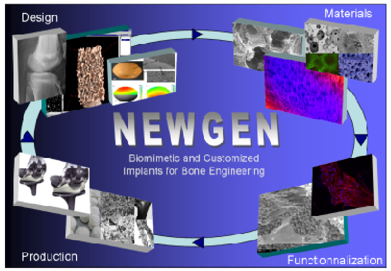
Mechanical characterization-MH samples





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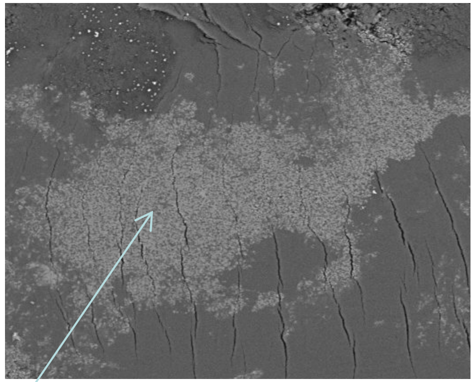
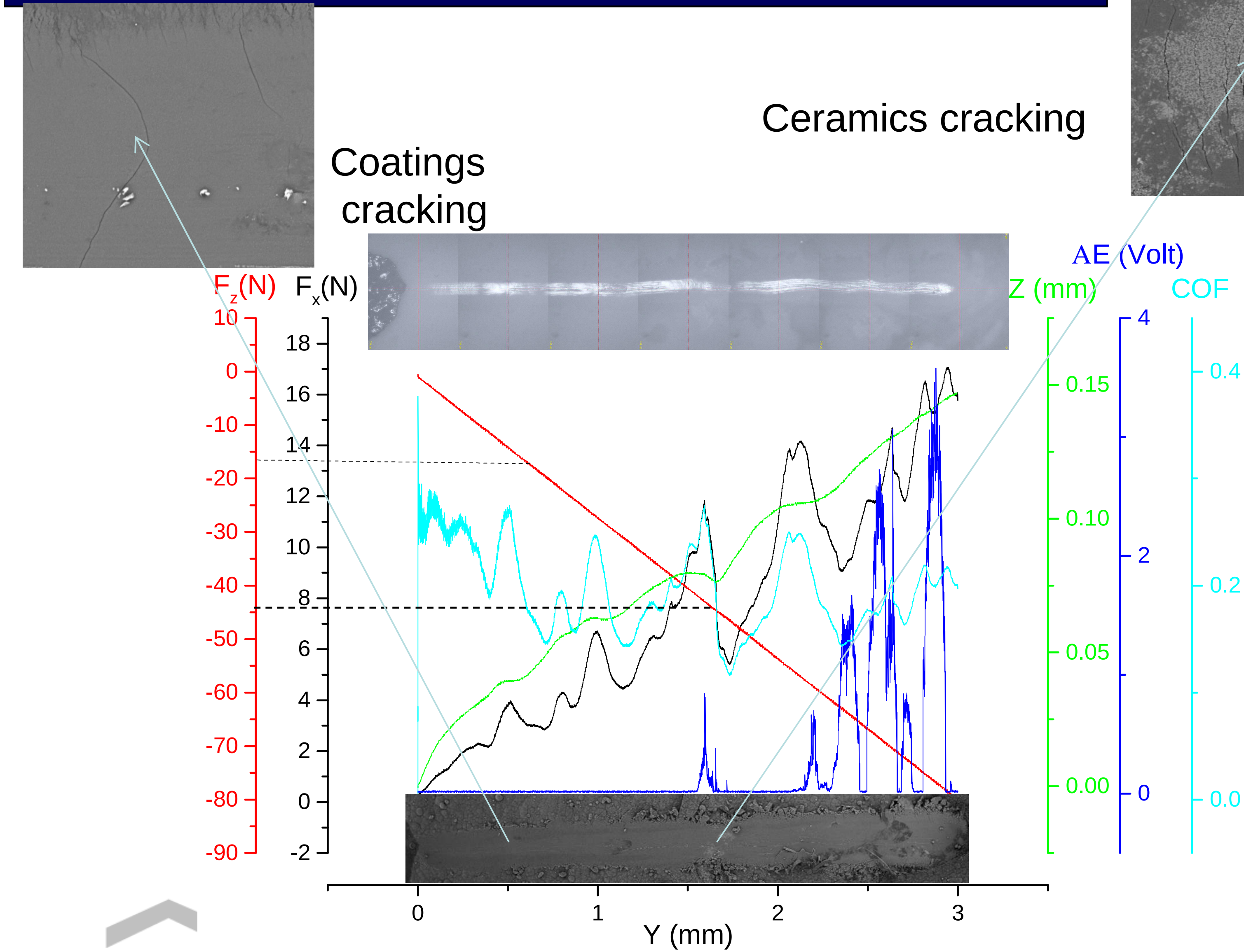
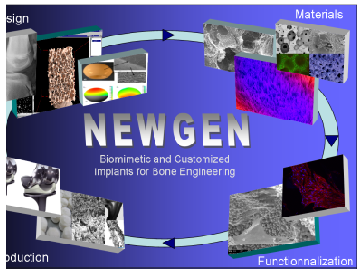
Mechanical characterization-MH sample





line IV, load 1-80 N, 120 s, 3 mm



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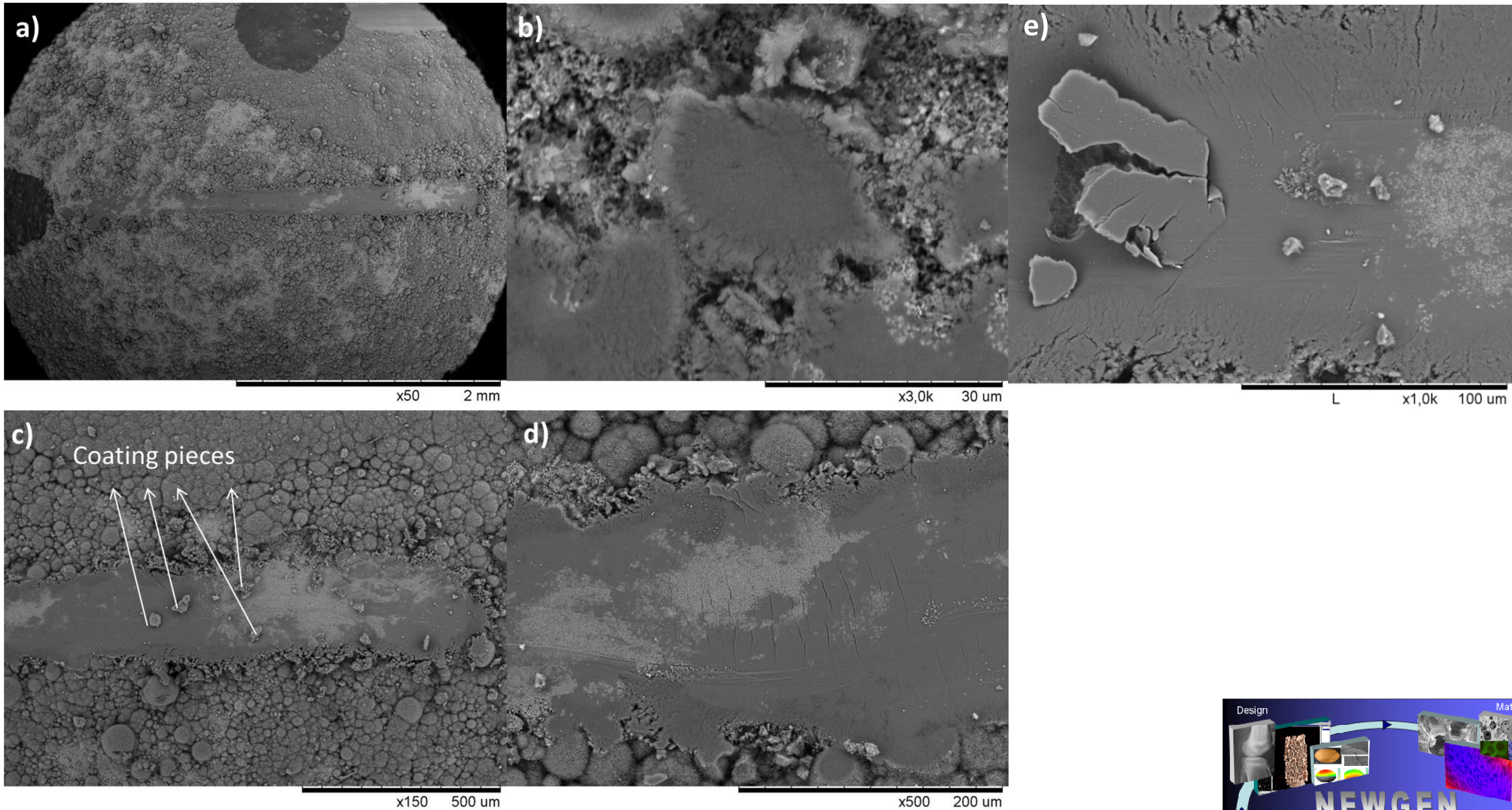
Mechanical characterization-MH sample



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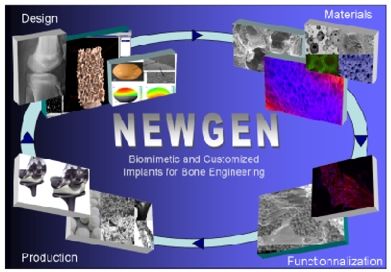

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Mechanical characterization-MH sample

	Coatings failure (Lc_1, N)	Ceramic failure (Lc_2, N)
1	20	52
2	18	32
3	15	42
4	16	50
5	18	42
6	15	32
7	15	42
Mean value	**17**	35
error	**2**	38
	Mean value	**41**
	error	**7**

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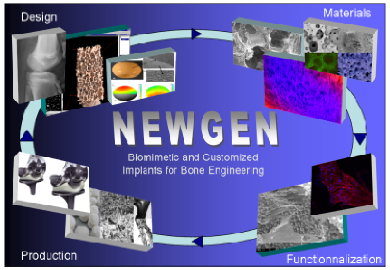

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Mechanical characterization- ceramic sample

Mechanical properties of β-TCP coating and biomimetic CaP coating on zirconia.

Coating	Scratch test critical load-L_c (N)	Tensile strength test (MPa)	Implantation and explantation of the coated implant from the artificial bone Remaining mass of the coating (%)
β-TCP coating	97 ± 9	52.3 ± 3.8	92 ± 7
Biomimetic CaP coating	5.3 ± 0.6	2.6 ± 0.4	36 ± 9

M. Stefanic et al. / Journal of the European Ceramic Society 33 (2013) 3455–3465

	SG samples	MH samples
Coating failure	18±2 N	17±2 N
Zirconia failure	49±7N	41±7 N

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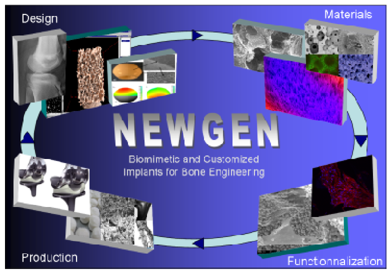

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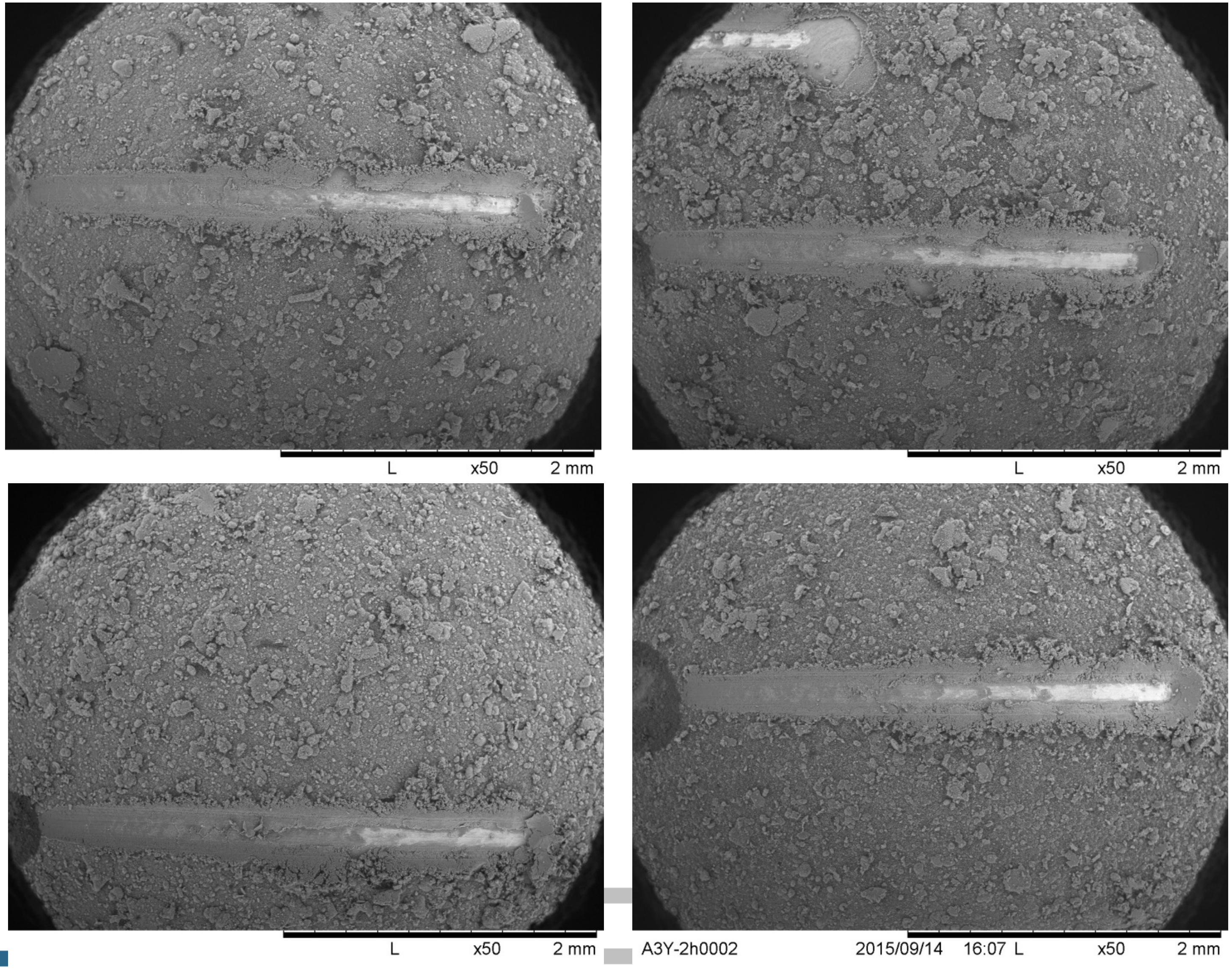
Mechanical characterization-TA samples



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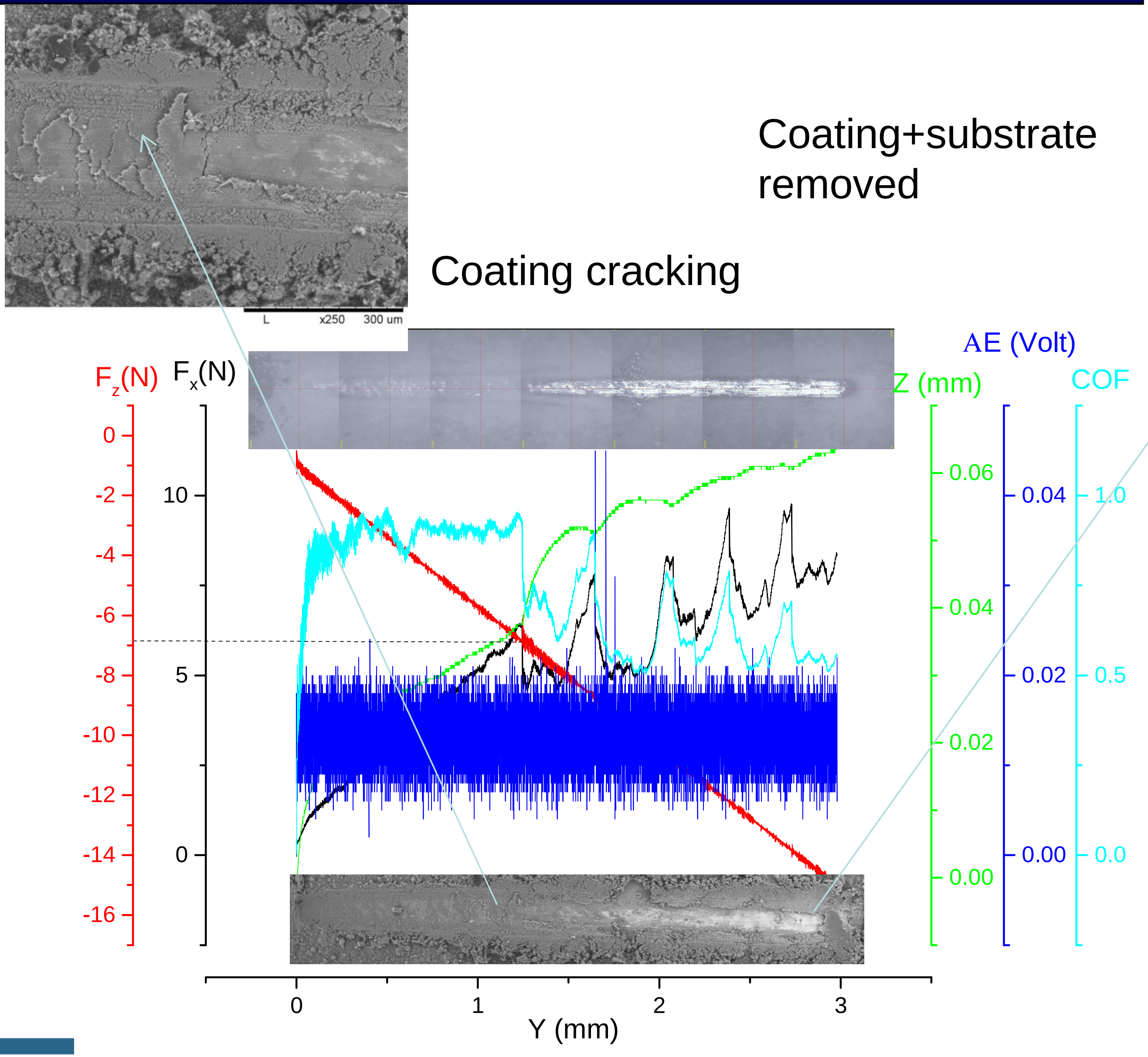
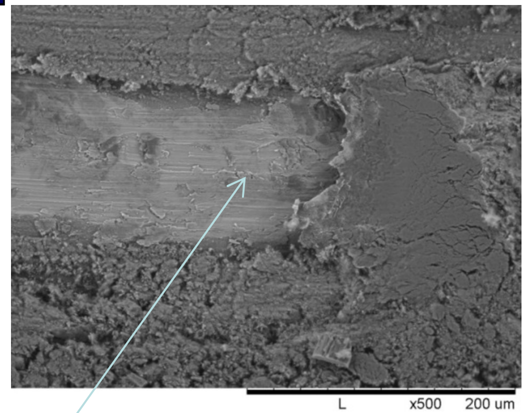
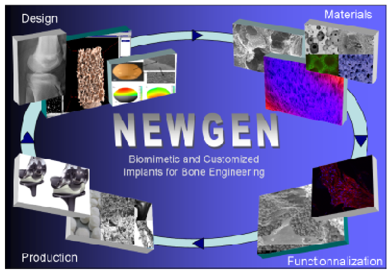
Mechanical characterization-TA samples







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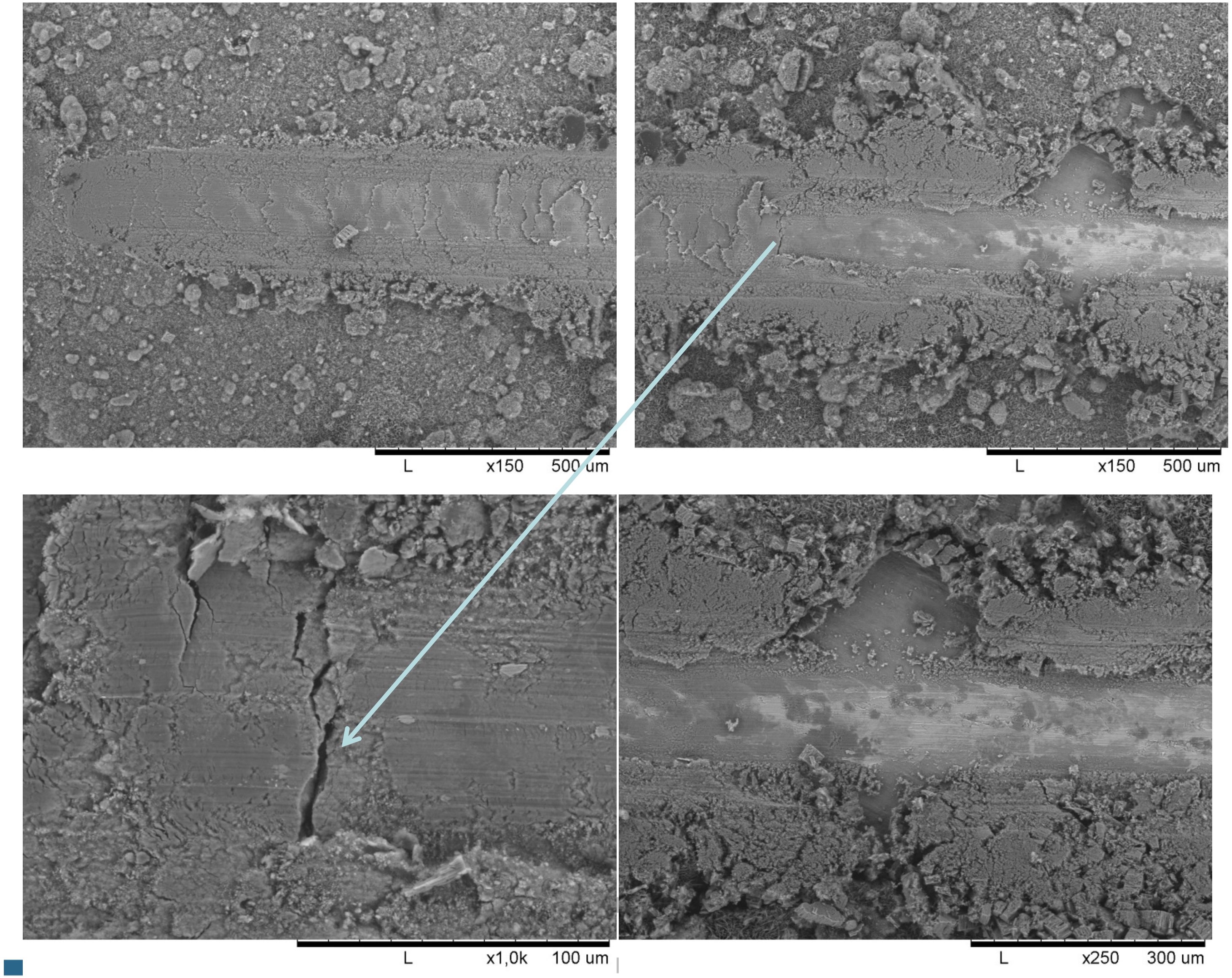
Mechanical characterization-TA samples



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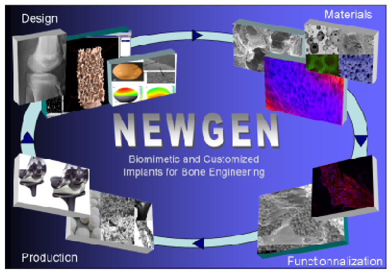
Mechanical characterization-TA samples

	Coating and nanotubes failure and delamianation (Lc, N)
1	6,6
2	6,2
3	7,2
4	6
5	6,2
6	6,2
7	5
8	6,2
9	4,8
10	5,2
11	5,70
12	4,40
13	5,2
14	4,7
15	5
Mean value	**5,6**
error	**0,8**



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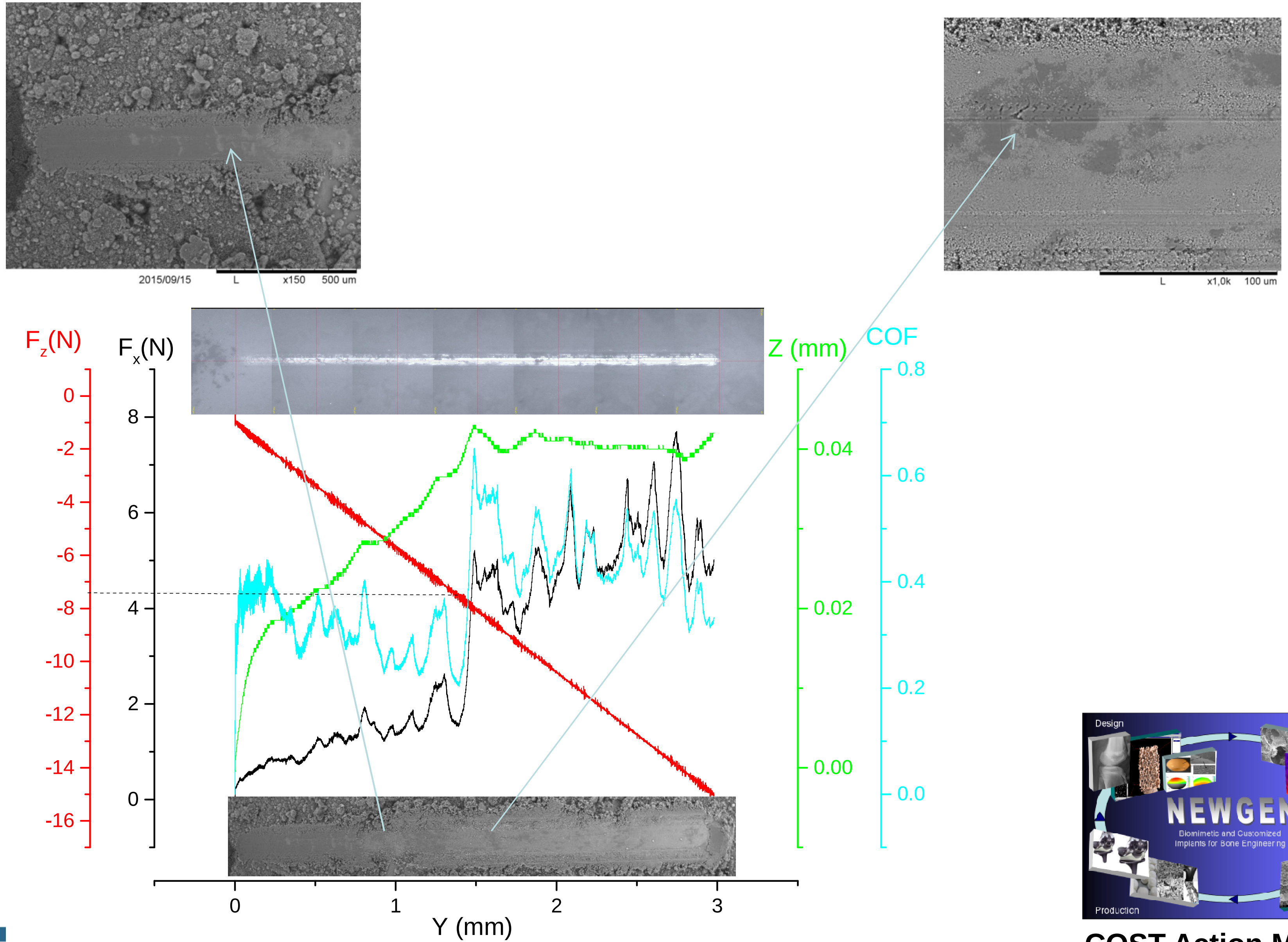
Mechanical characterization-TR samples



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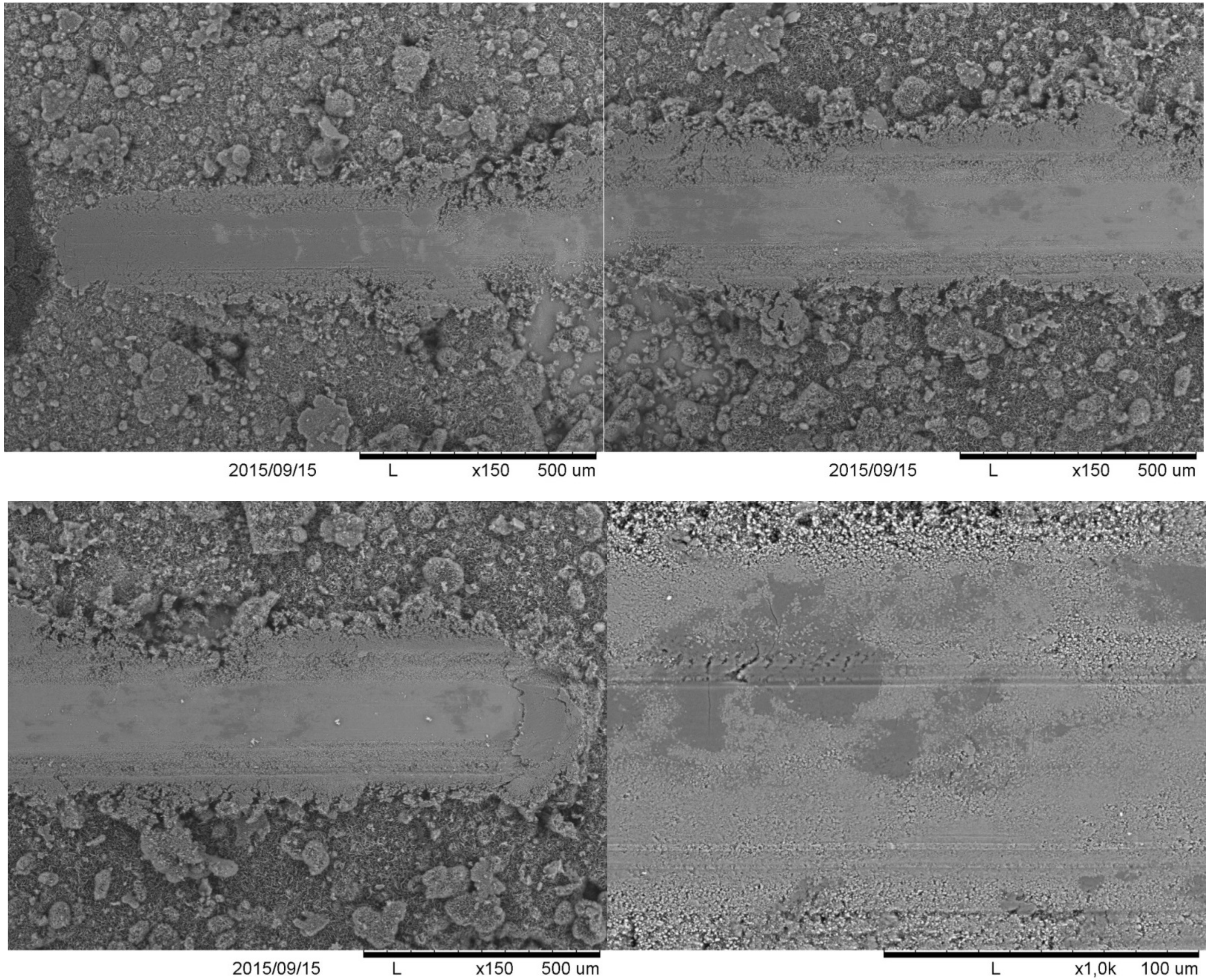
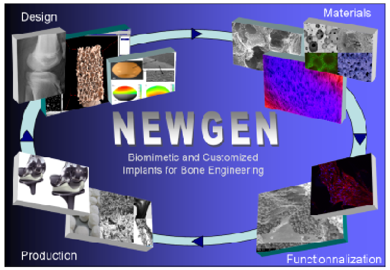
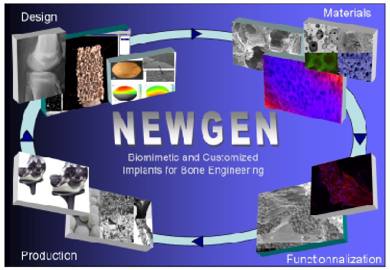
Mechanical characterization-TR samples



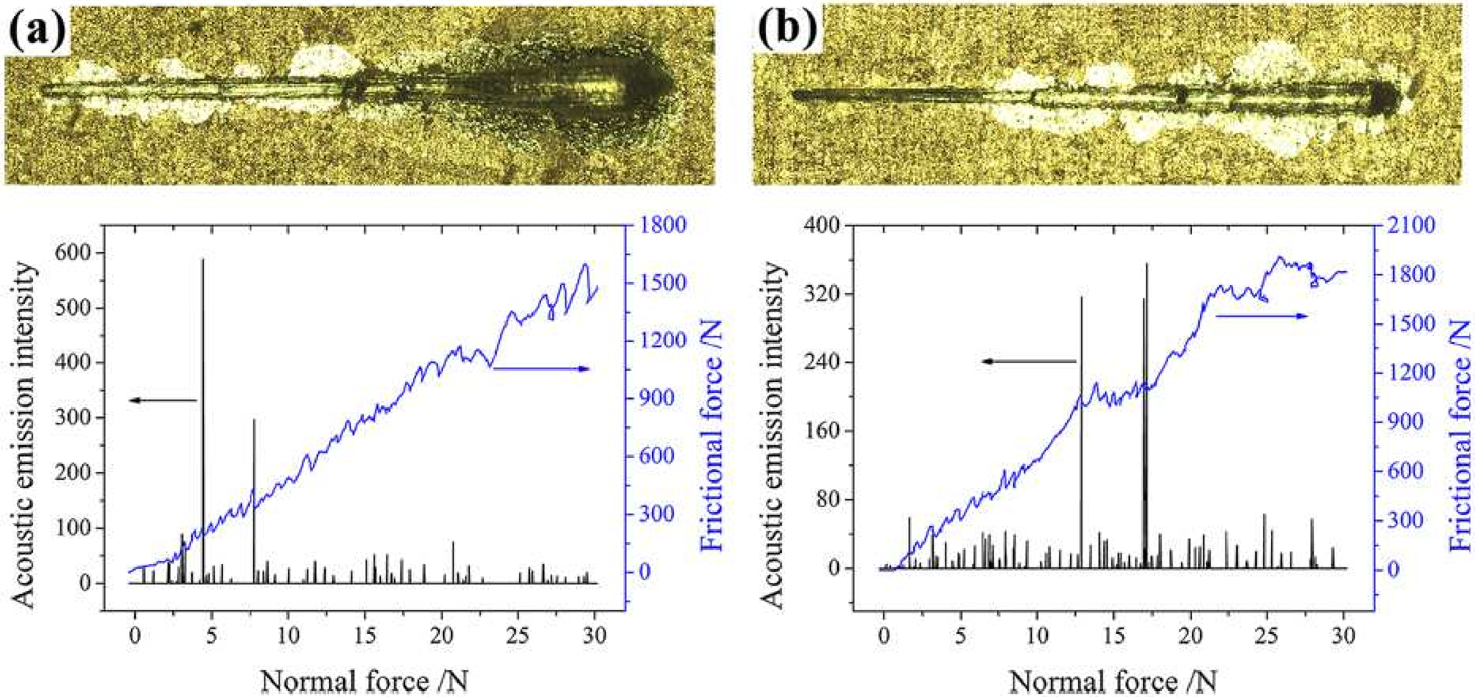


Mechanical characterization-TR samples

	Coating and nanotubes failure and delamianation (Lc, N)
1	7,1
2	6,6
3	5,2
4	7,1
5	4,8
6	7,5
7	5,1
8	10,3
9	6,7
10	6,1
11	5,5
12	7,8
13	7,5
14	7,6
Mean value	7
error	1



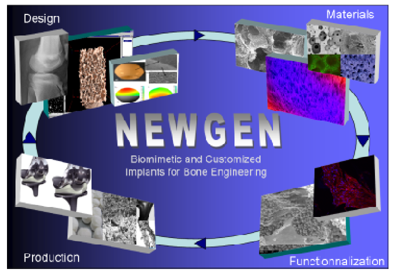
Mechanical characterization-TR samples



TiO_2 nanotubes
4N load failure

TiO_2 nanotubes adhesion enhance by oxide compact layer, 13 N load failure

D. Yu et al. ACS Appl. Mater. Interfaces 2014, 6, 8001−8005



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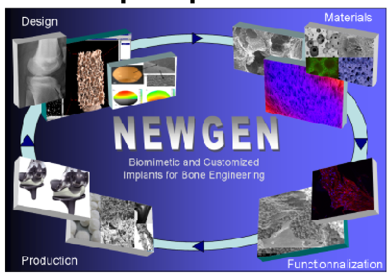
Conclusion

- The scratch test results obtained on coated porous ZrO_2 (SG-CaP MH-CaP) and TiO_2 nanotube arrays (TA-CaP, TR-CaP) provide us very important information about adhesion of calcium phosphate coated on the surfaces of all substrate.

- As we saw our results are similar to previous published results, the coating obtained by biomimetic procedure on ceramics have critical load value (L_c) around three times higher

- Also TA nanotubes+CaP have around 50% higher value and TR nanotubes+CaP more than three time higher that non coated nanotubes

- Obtained results during this STSM project will be used for further developments, processes optimization and improvements of proposed biomimetic material for application as a bone implants



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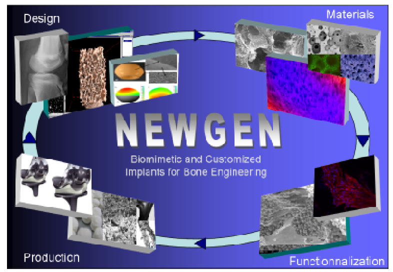

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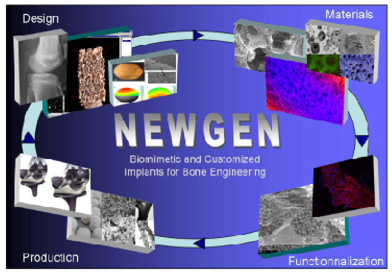
Thank you for the attention



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