Centre for Advanced
Hybrid Materials

Ultrafine grained titanium and magnesium alloys for permanent and bioresorbable medical implants

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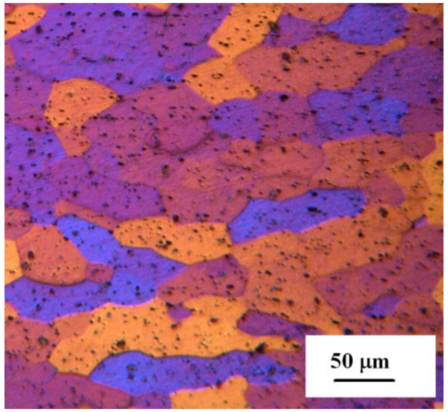
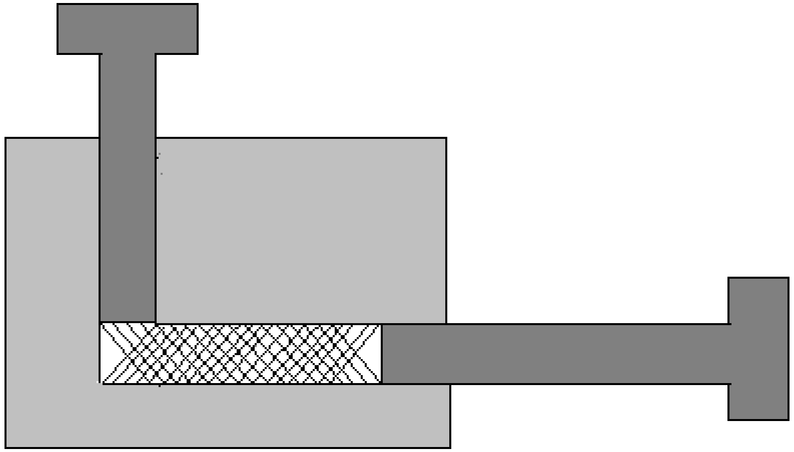
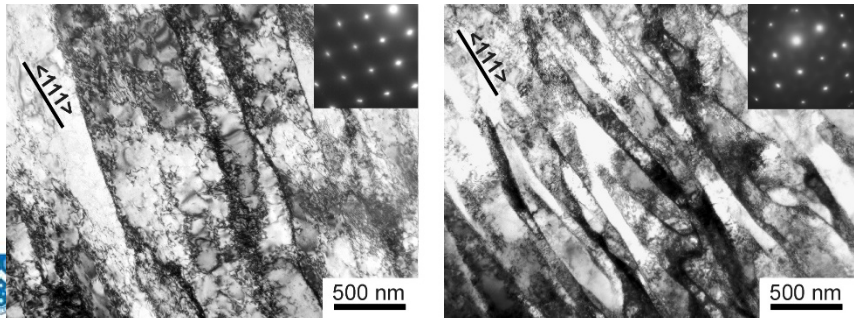
Aims of Research

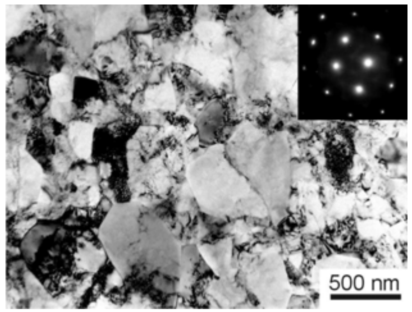
- Determine the effect of grain refinement by severe plastic deformation on mechanical properties and biocompatibility of titanium and magnesium alloys
- Identify the effect of grain refinement on surface properties (roughness) of the materials with polished and modified surfaces
- Assess the effect of surface modification on mechanical properties and cell response of titanium and magnesium alloys with different structure

MOST POPULAR TECHNIQUE: EQUAL CHANNEL ANGULAR PRESSING (ECAP)



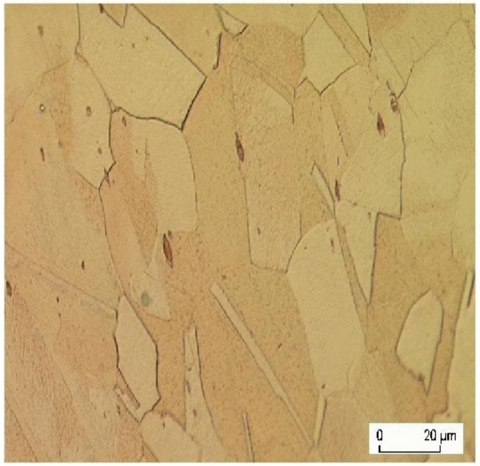


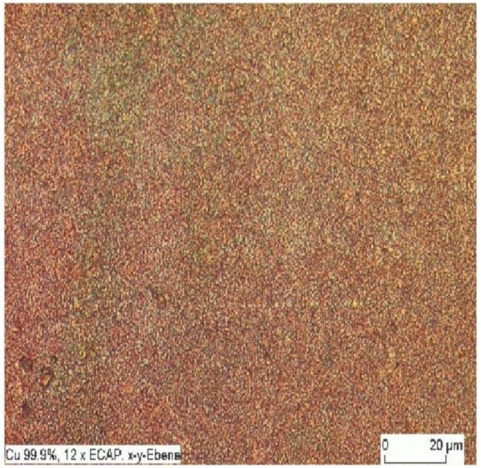


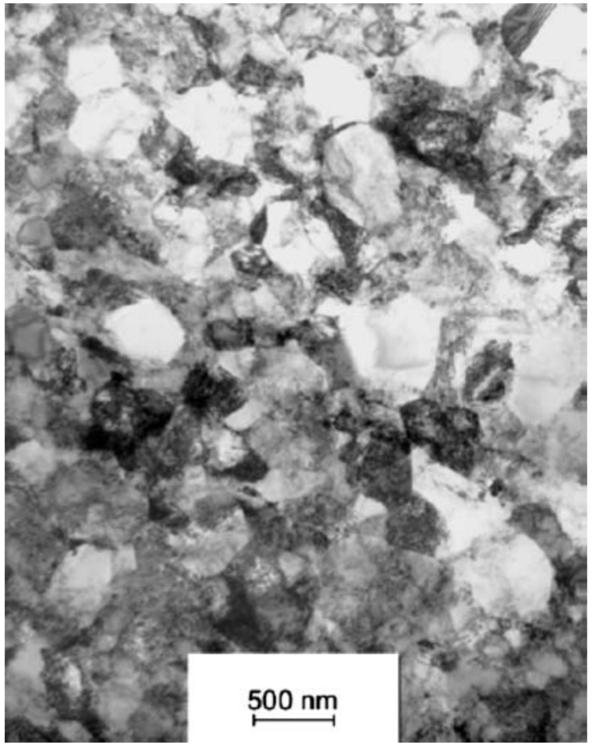


Copper

AFTER

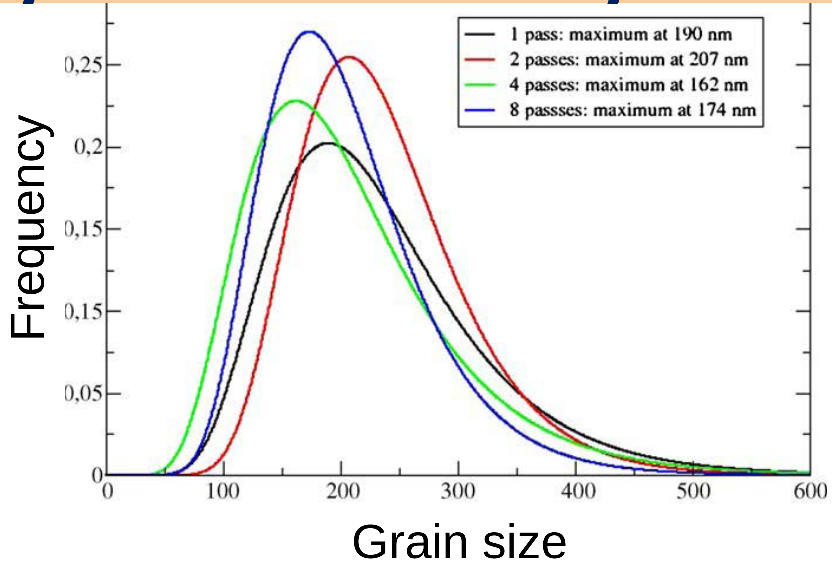


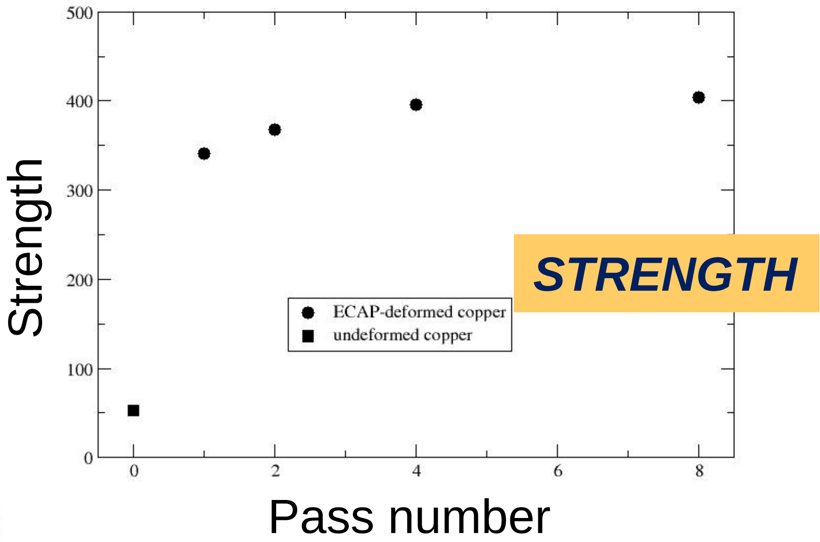


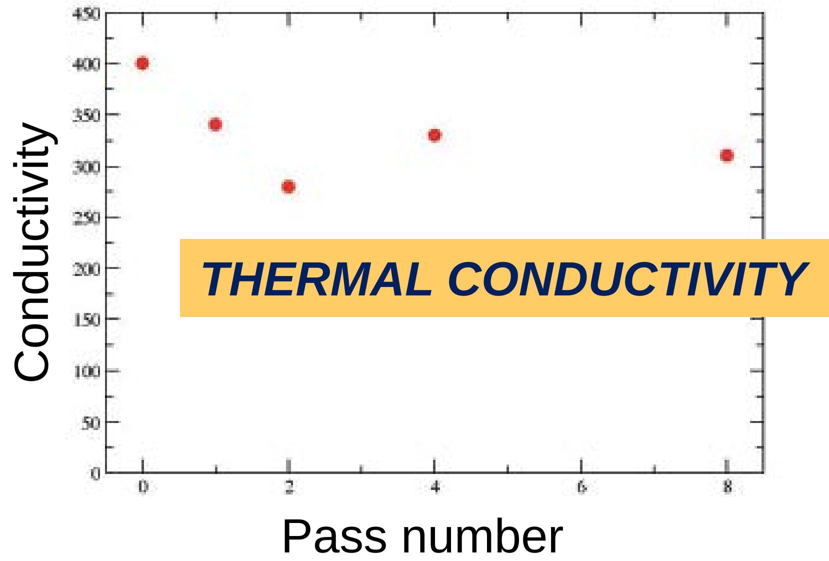


BEFORE

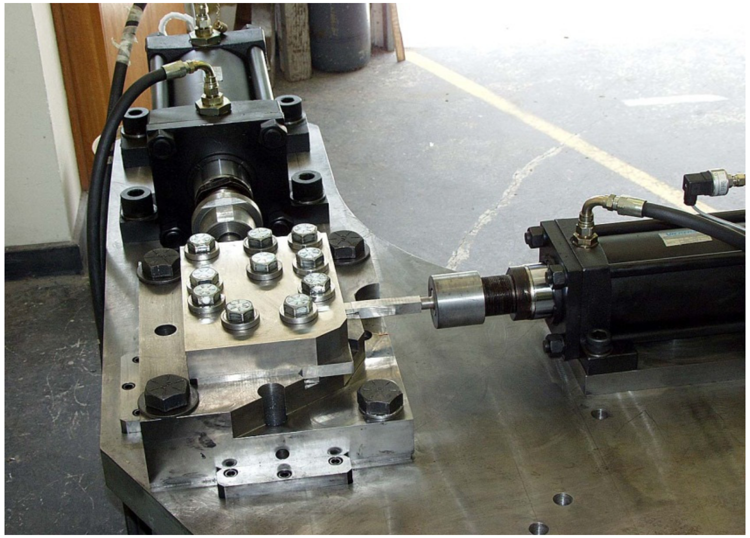
Property profile of ECAP-processed Cu

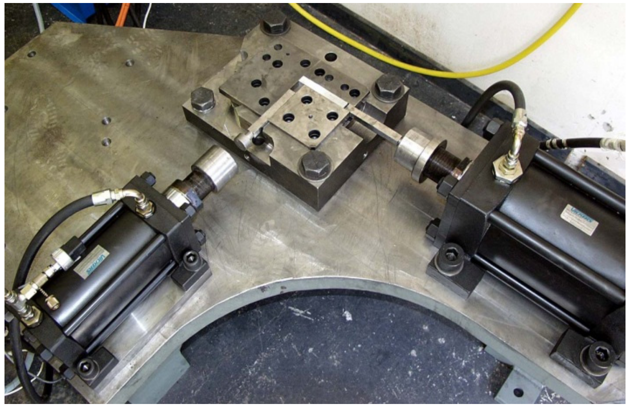


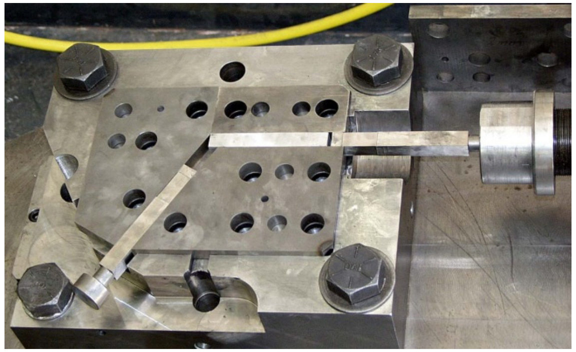


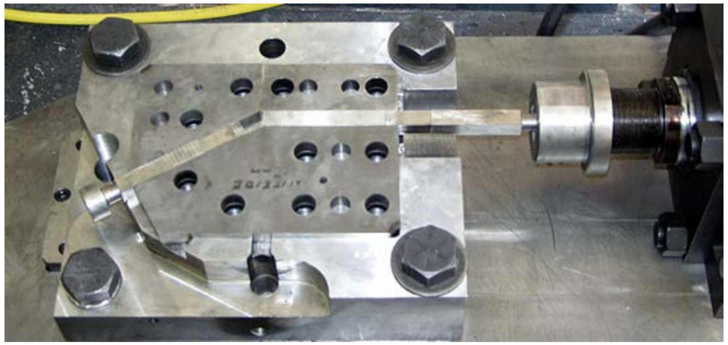


ECAP EQUIPMENT



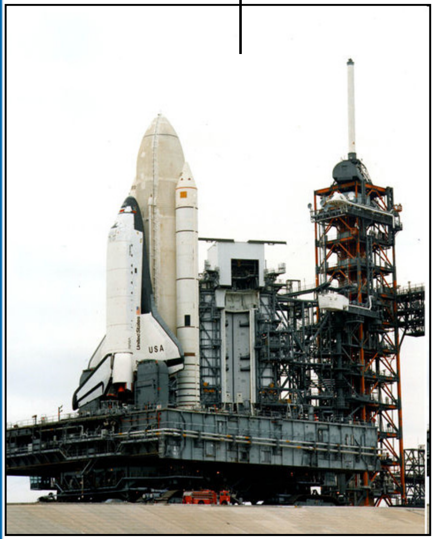




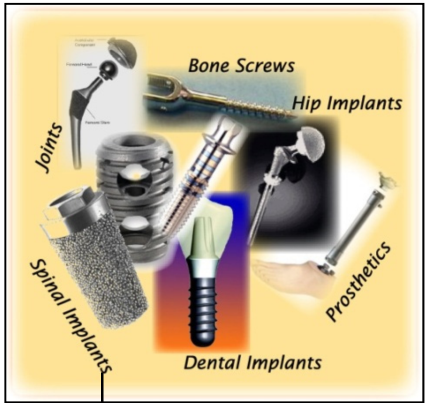


Possible marketplace applications of SPD processing

Transportation




- Lighter weight
- Longer lifetime
- Higher performance
- New products

Biomedical

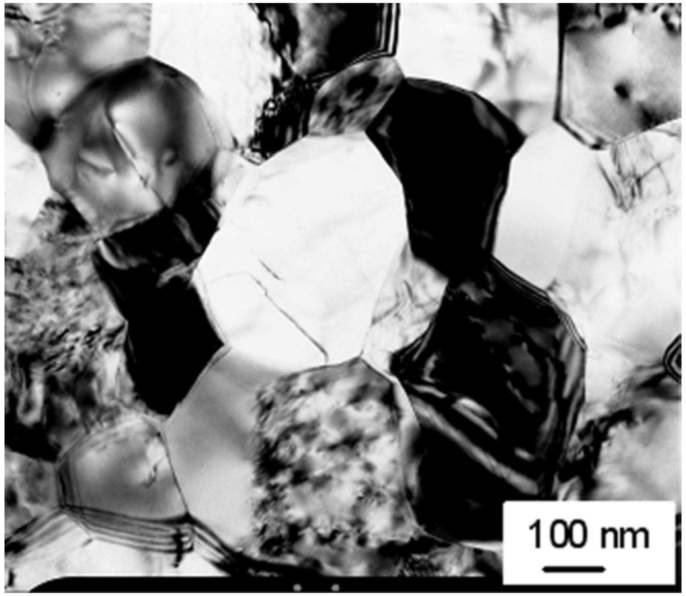

Aerospace

Sports

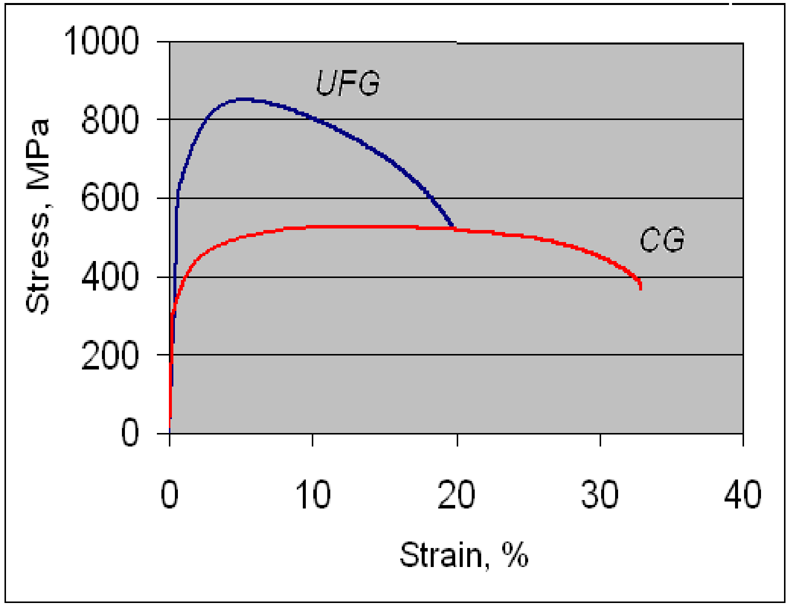
Courtesy: Dr Terry Lowe, Manhattan Scientifics, Inc.

Enhanced Strength and Biocompatibility of Titanium (Permanent Implant Applications)

Grain refinement and mechanical property improvement of Grade 2 CP Titanium



Grain structure of ECAP-modified titanium



Tensile curves of coarse-grained (CG) and ultrafine-grained (UFG) titanium

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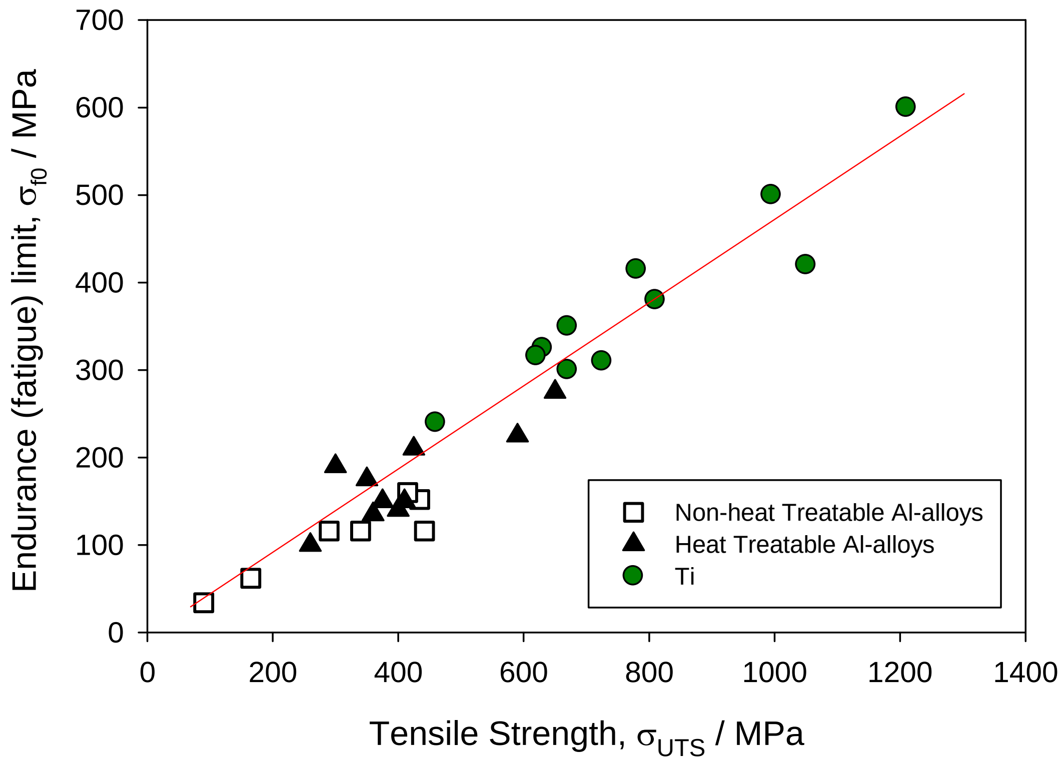
Correlation between fatigue strength and UTS



Estrin & Vinogradov, Intl. J. Fatigue 2010

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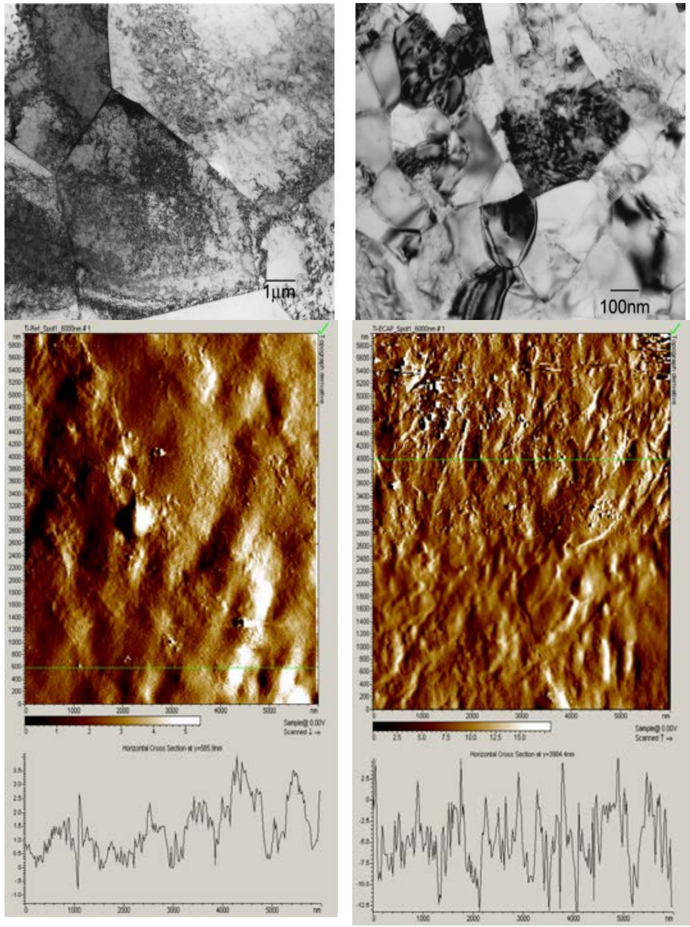
Surface topography



Coarse-Grained **UFG**

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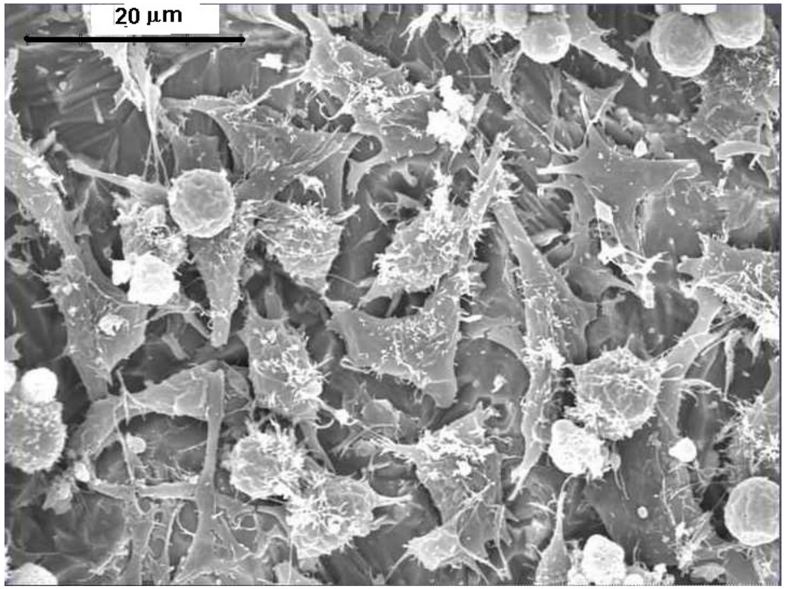
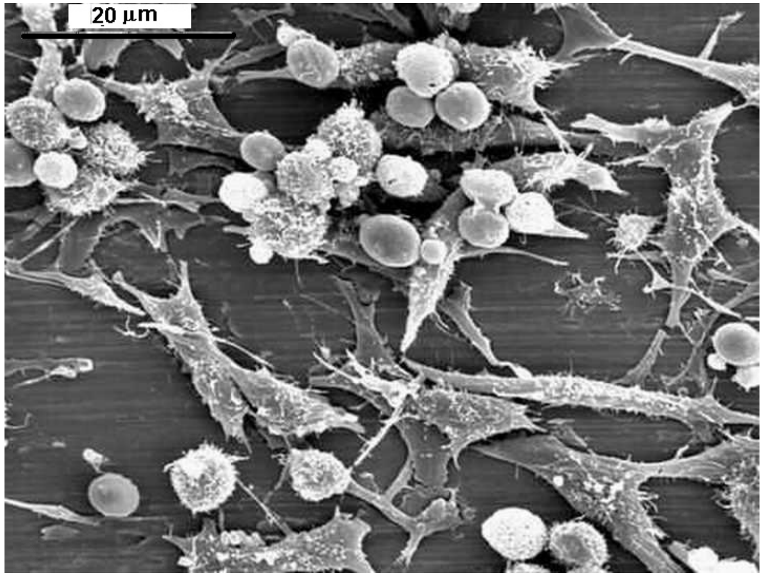
Proliferation of fibroblast cells on Ti CP4



UFG



CG

I. Semenova, R. Valiev, H. Rack, T. Lowe et al., 2008

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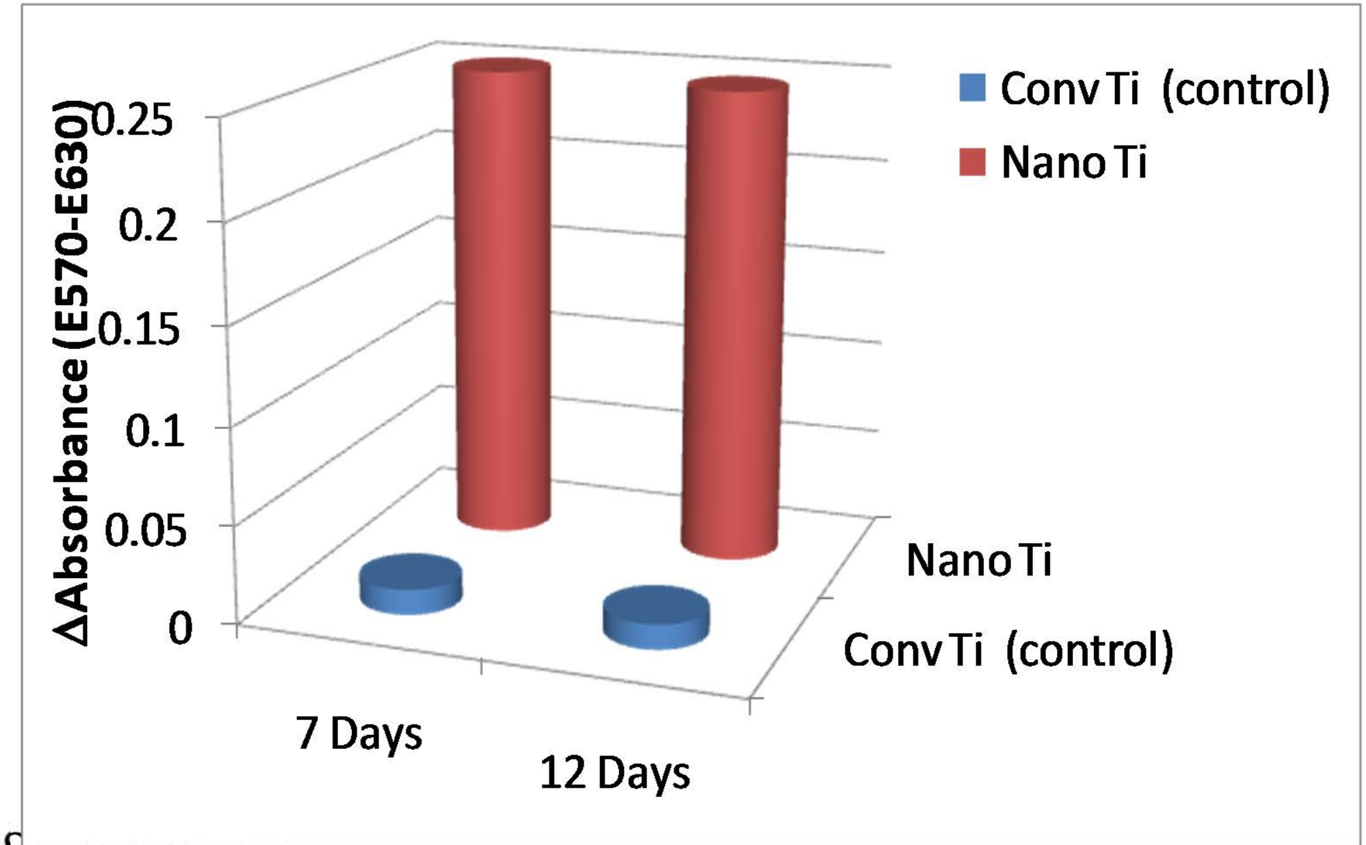
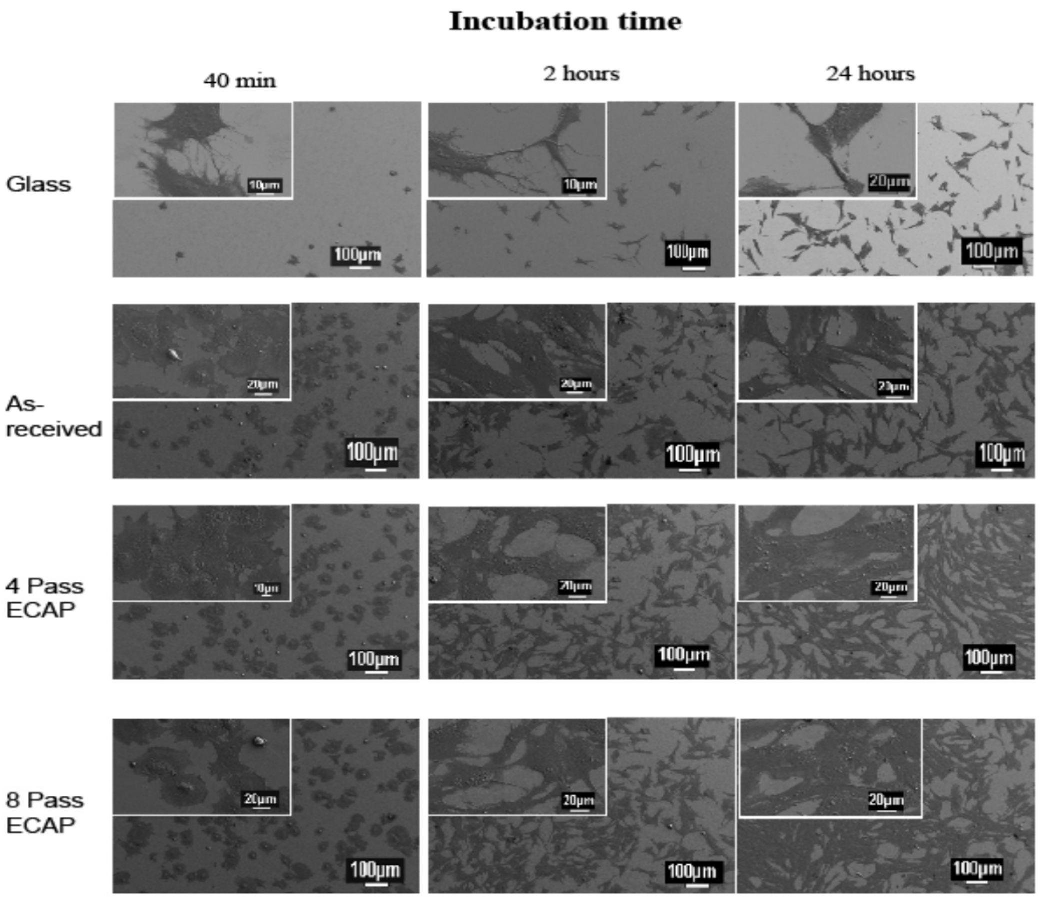
Nanostructured Ti Shows Distinctly Greater Preosteoblastic Cell Growth in vitro

MC3T3-E1 cells from mice embryos



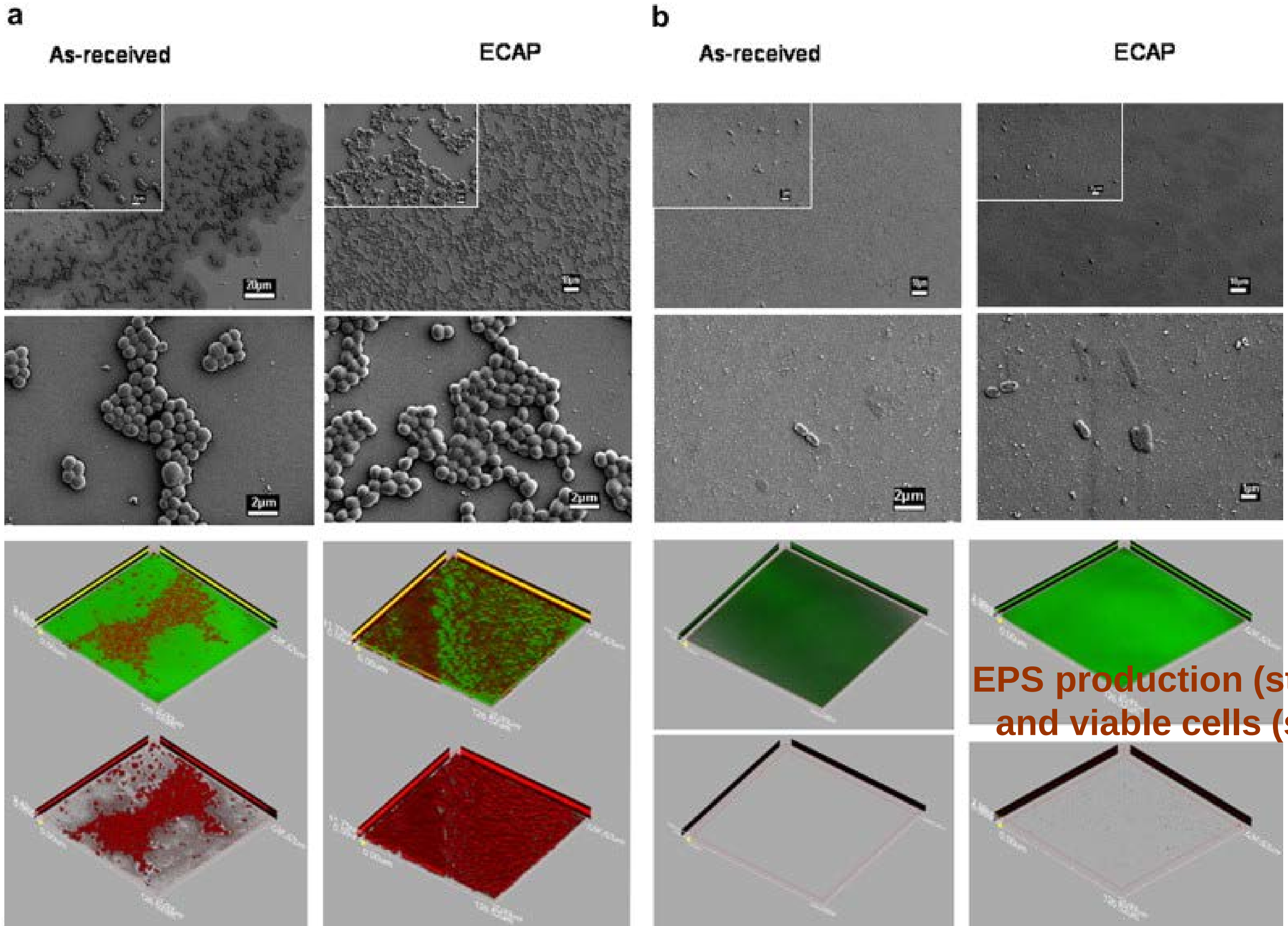
Y. Estrin et al. J. Biomed. Materials Res., 2008

Stem cell attachment and growth

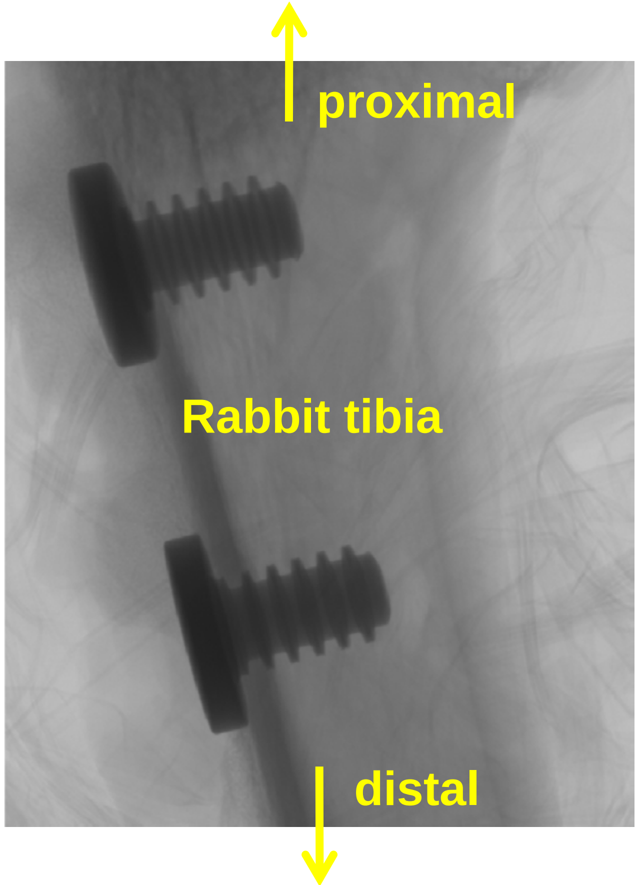


Y. Estrin et al., Acta Biomater. (2010))

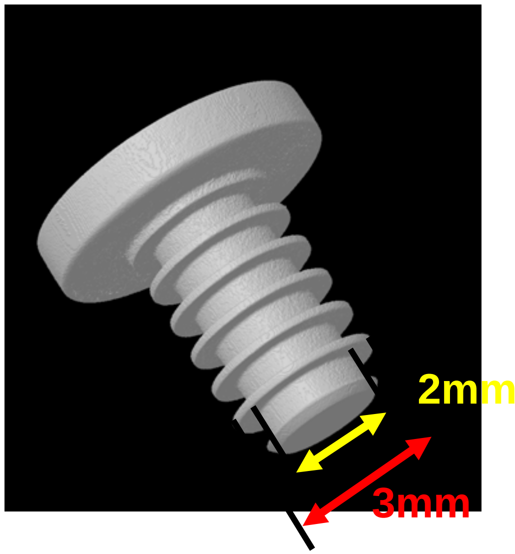
Bacteria adhesion: S. aureus (left) and P. aeruginosa (right)



Animal test

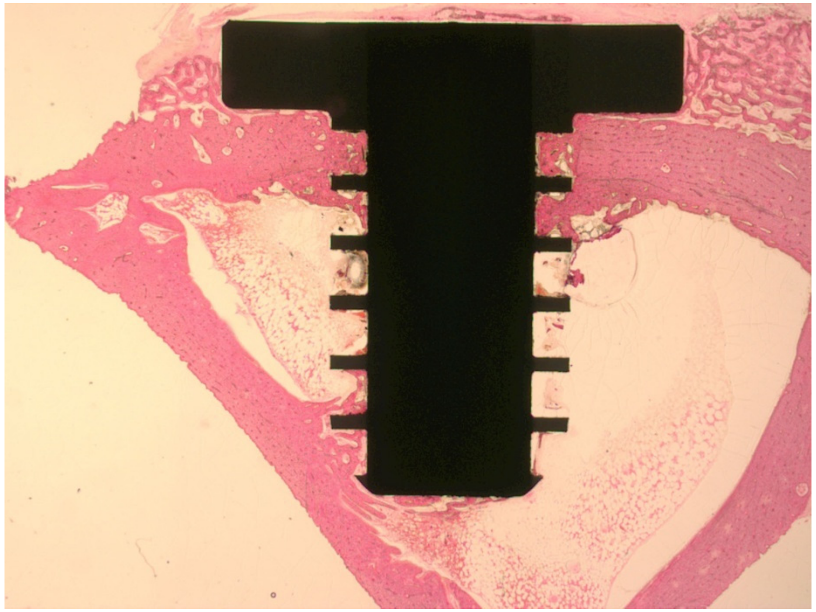


4 week implantation
Rabbit tibia

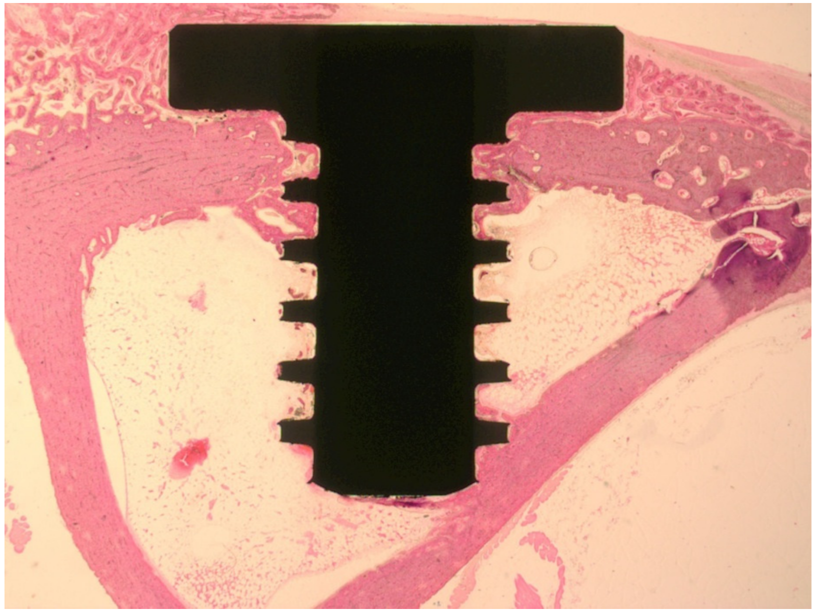


Ji-Hoon Jo, Hyon-Ee Kim, SNU

2D image (Histology)



Conventional Ti



ECAP Ti

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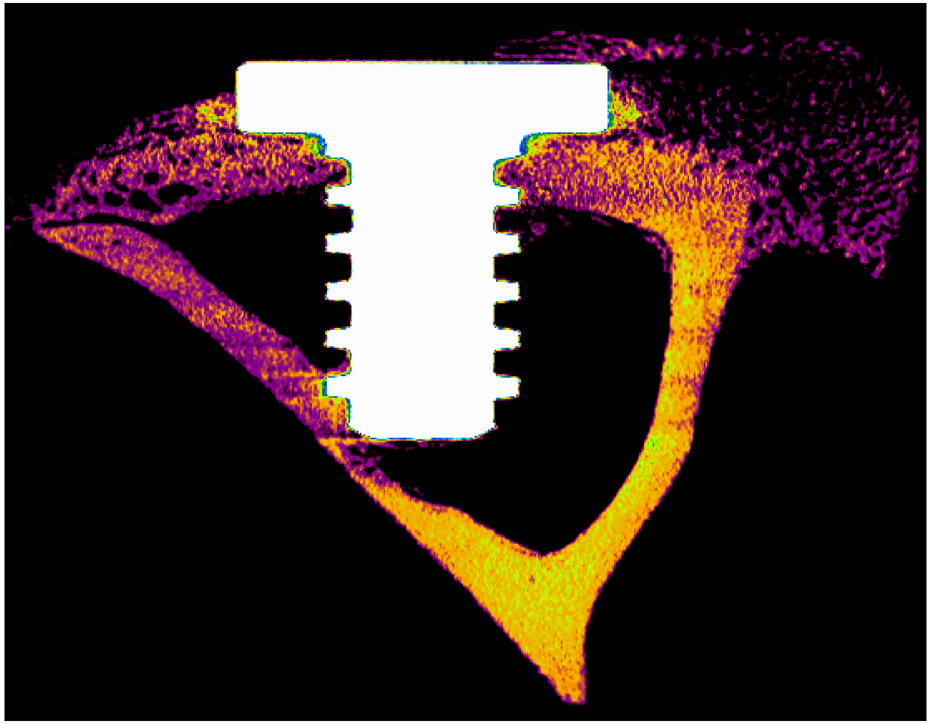
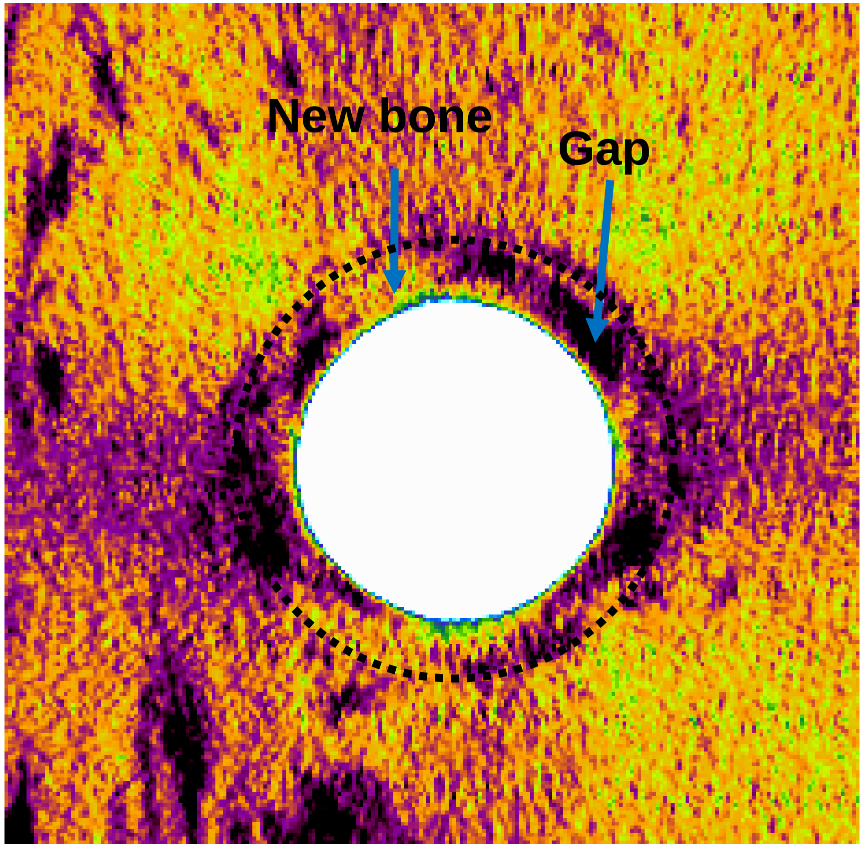
2D image (Micro CT)





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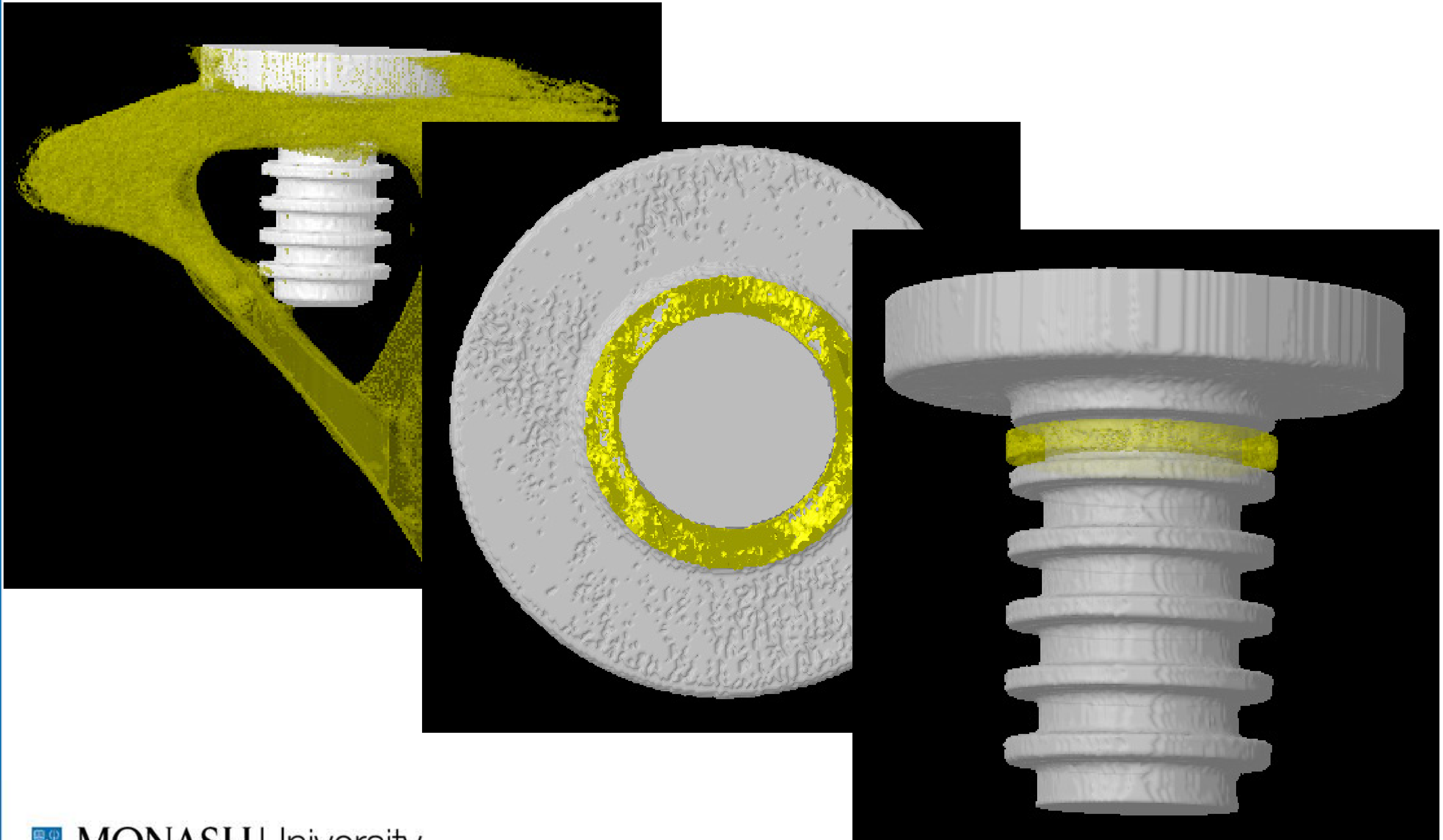
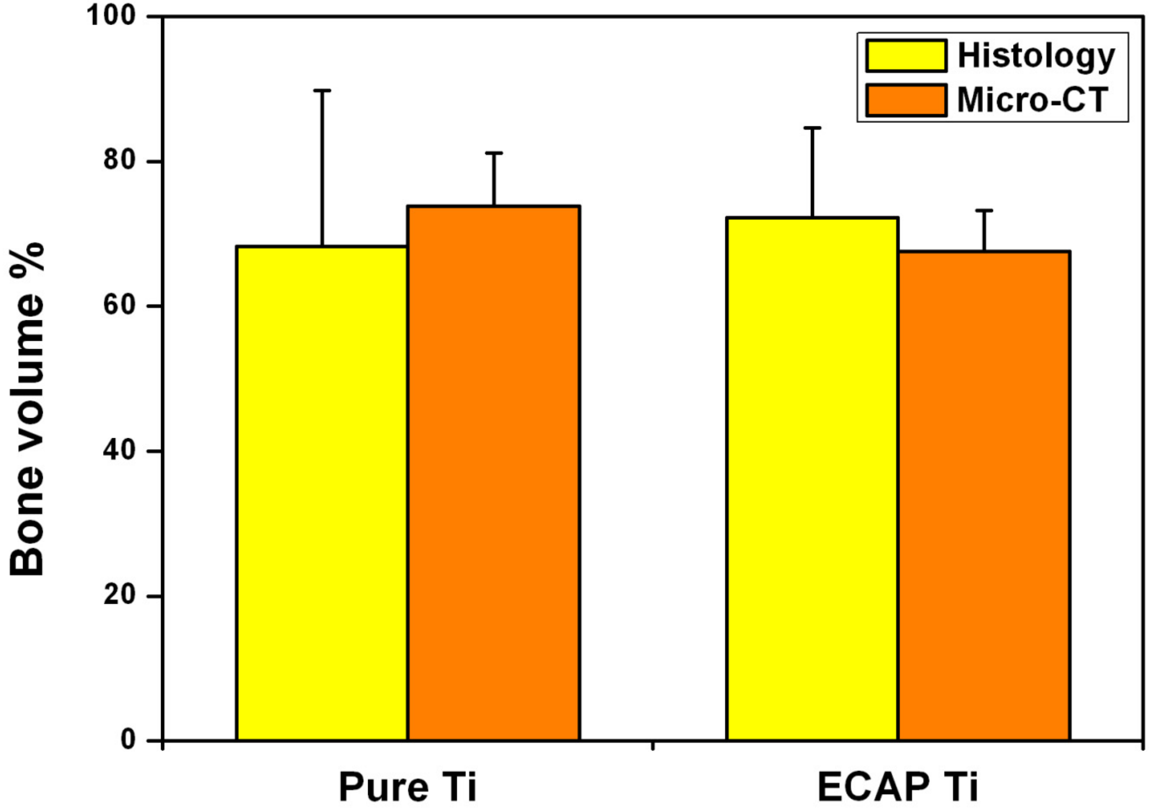
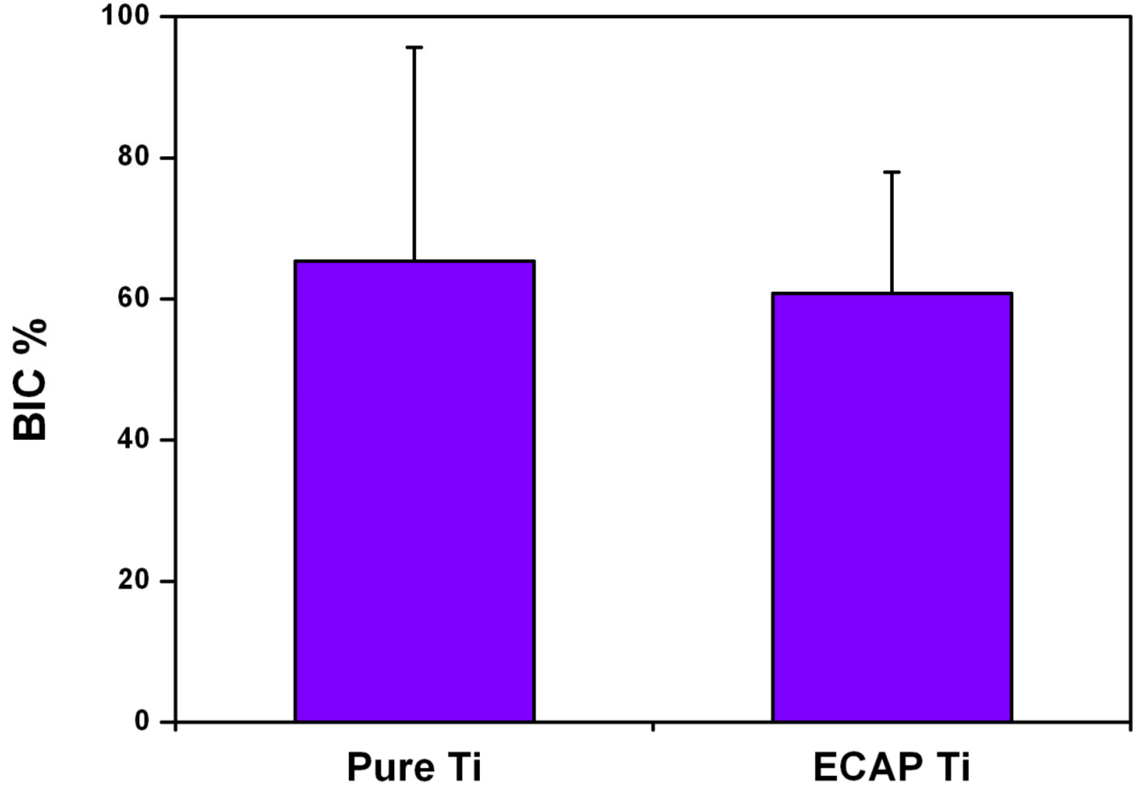
3D image (Bone in Volume of Interest)



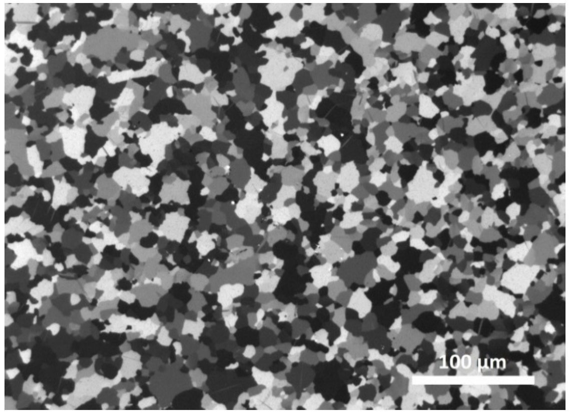
Bone volume %


Bone volume %
100
80
60
40
20
0
Histology
Micro-CT
Pure Ti
ECAP Ti

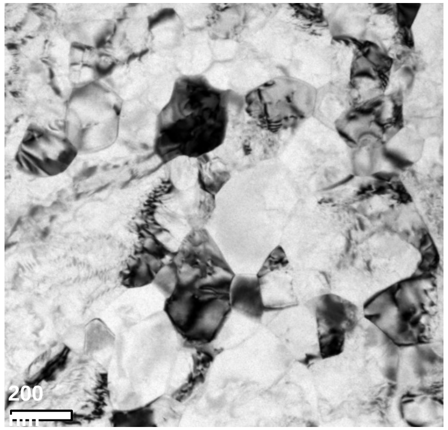
BIC (Bone-to-Implant contact) %



Microstructure



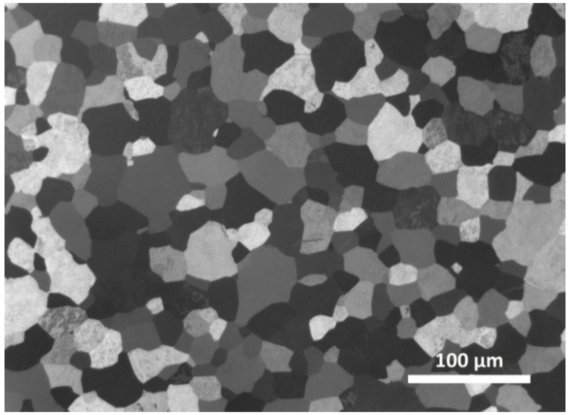
Gr2AsR



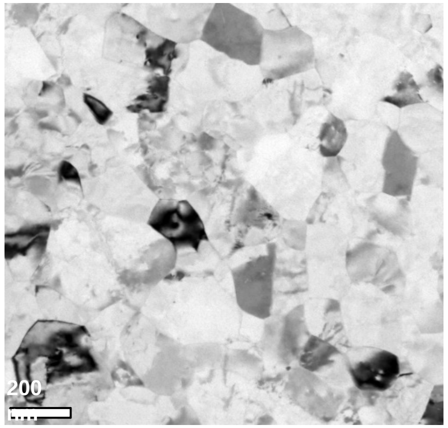
Gr2ECAP (I)

Average grain size

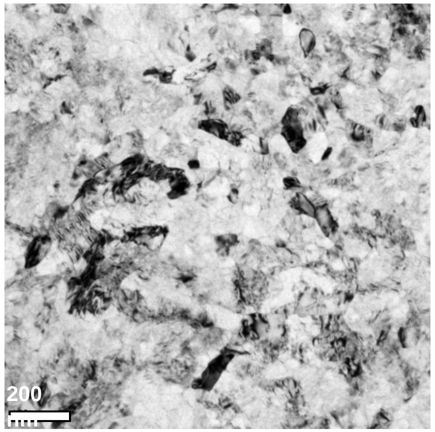
Condition	Grain size, µm
Gr2AsR	20
Gr4AsR	30
Gr2ECAP (I)	0.25
Gr4ECAP (I)	0.23
Gr4ECAP (II)	0.1



Gr4AsR



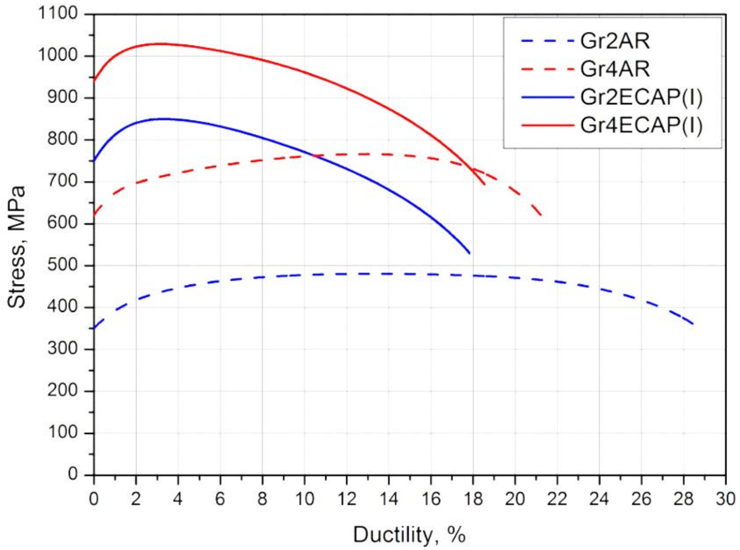
Gr4ECAP (I)



Gr4ECAP (II)

Mechanical properties

Condition	Average grain size, µm	UTS*, MPa	YS*, MPa	Elongation**, %
Gr2AR	20	480	350	29
Gr2ECAP (I)	0.25	850	750	18
Gr4AR	30	765	620	21.5
Gr4ECAP (I)	0.23	1030	940	18.5
Gr4ECAP (II)	0.1	1275	1130	13
Ti-6Al-4VAR^	3-4 (α-grains)^{x}	940-970	840-900	16-20
Statistical error does not exceed: * 5-7MPa ** 1-2%; Statistical analysis is not applicable to Ti-6Al-4V data				



Increase in strength by **70-80 %**

Alex Medvedev, PhD thesis, 2016 Monash University

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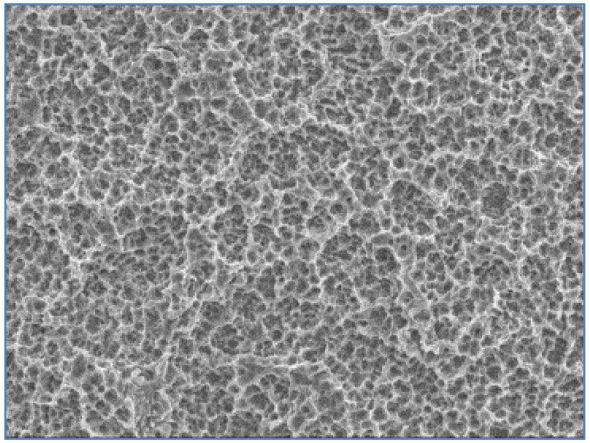
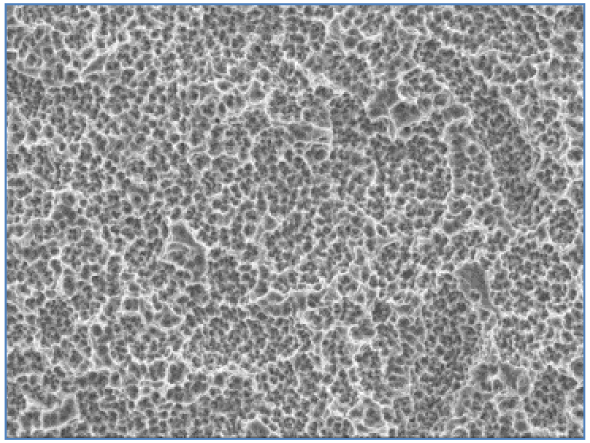
Surface after SLA treatment



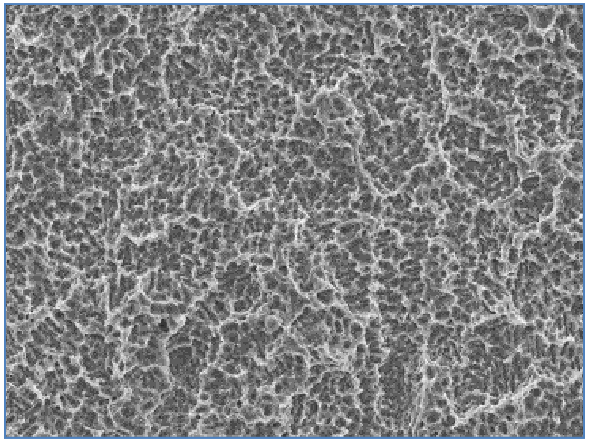
Gr2AsR



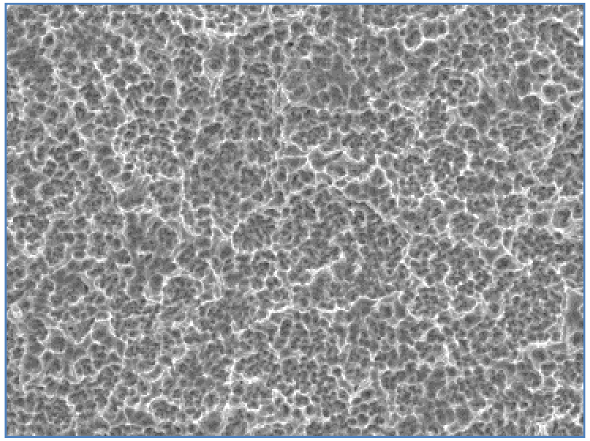
Gr2ECAP(I)

Magnification
x1,000

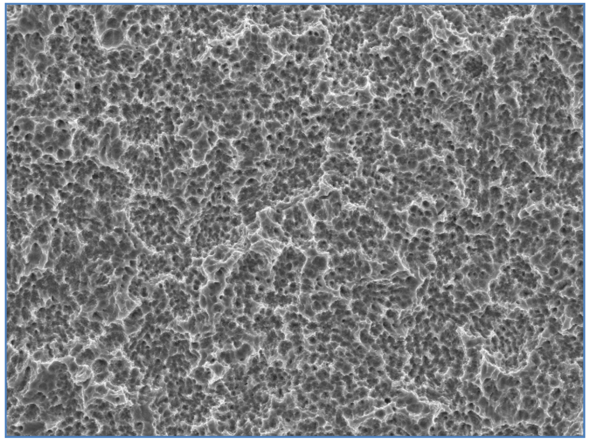
20 µm



Gr4AsR

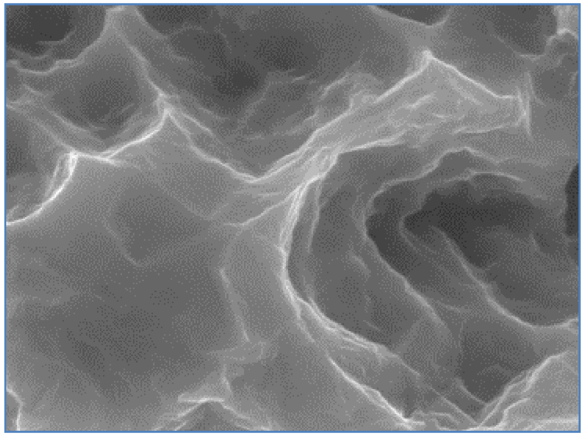


Gr4ECAP(I)

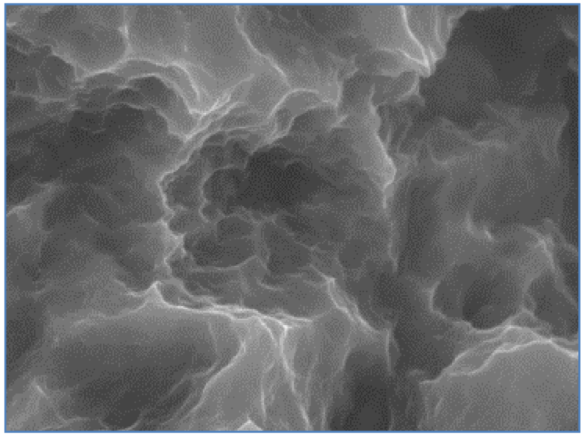


Gr4ECAP(II)

Surface after SLA treatment



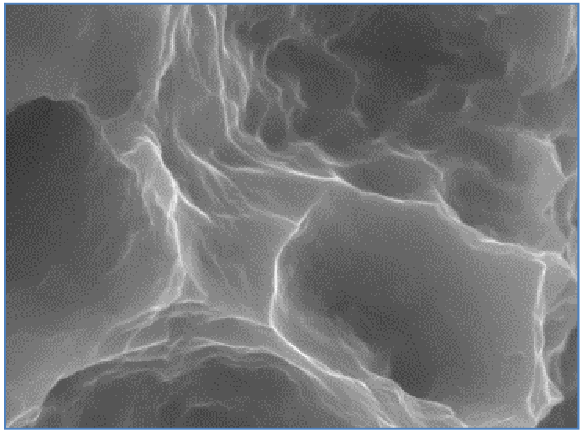
Gr2AsR



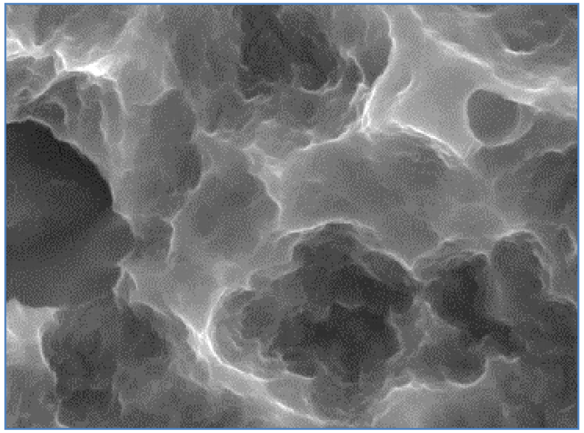
Gr2ECAP(I)

Magnification
x25,000

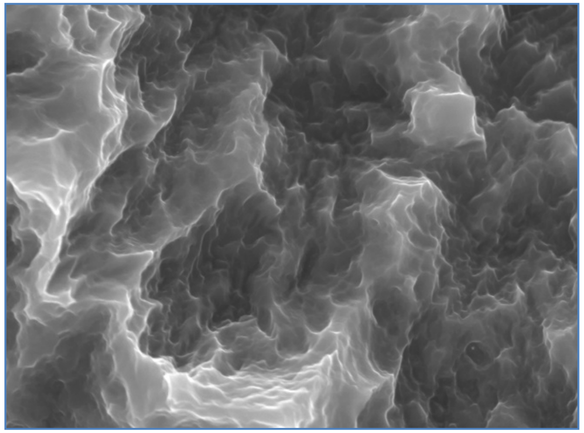
1 µm



Gr4AsR



Gr4ECAP(I)

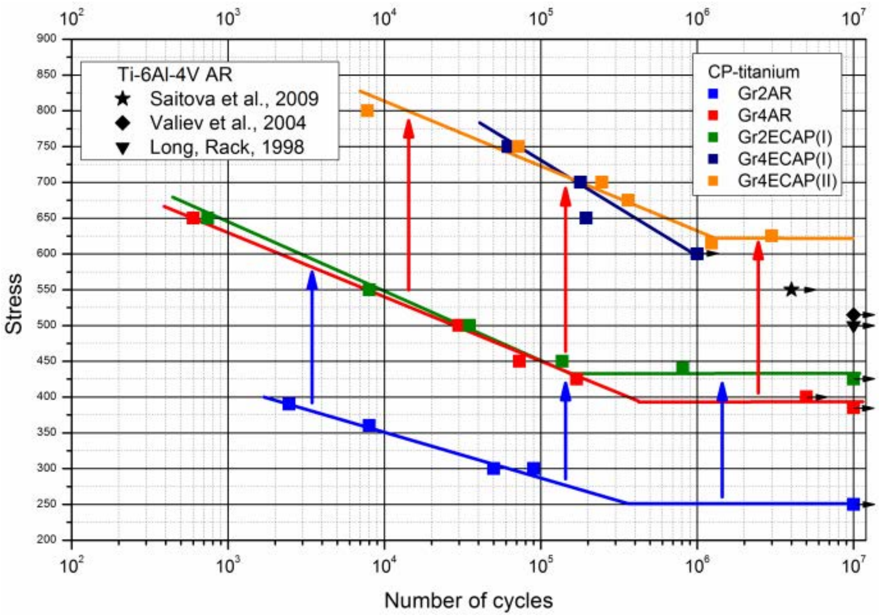


Gr4ECAP(II)

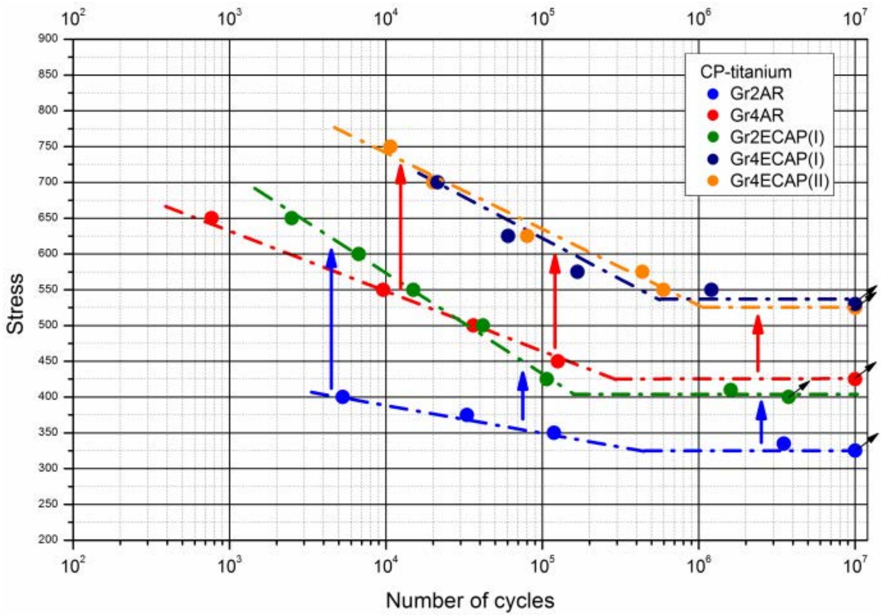
Fatigue properties

T = 25°C, Air, R = -1

Polished surface



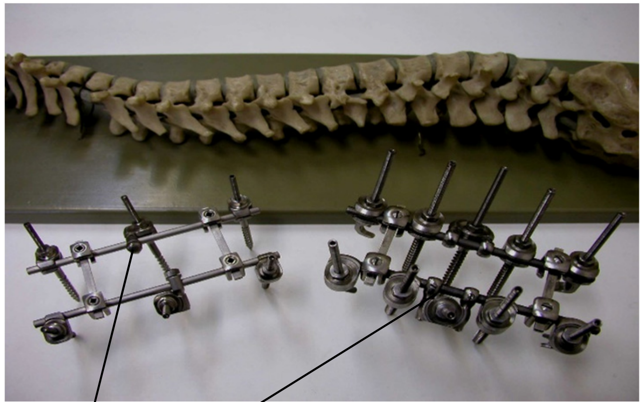
SLA-treated surface



Arrows ⬆ indicate an increase of fatigue life of titanium after ECAP-processing compared to as-received coarse-grained counterparts

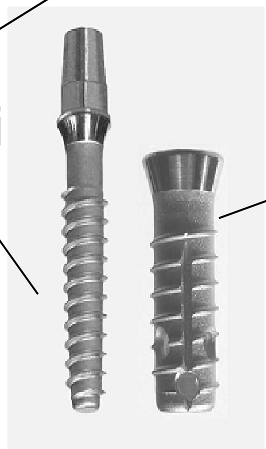
Alex Medvedev, PhD thesis, 2016 Monash University

Surgical and dental implants from ultrafine-grained Ti

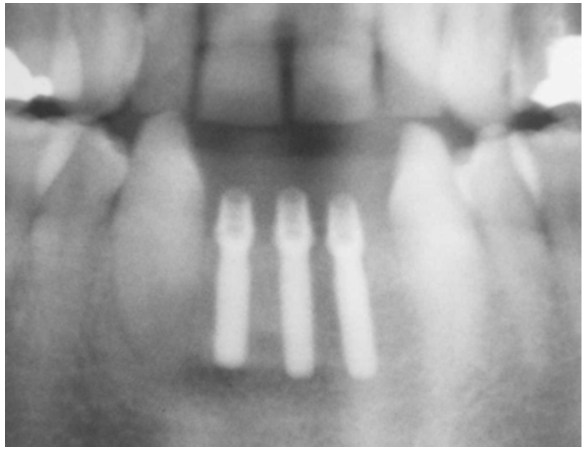


Development of a new lightweight structure for spine fixation

“nano” Ti



Usual Ti



[Timplant Co. , *www.timplant.cz*]

From I. Semenova et al., TMS 2008 Annual Meeting, New Orleans, USA

Mg as an Implant Material

Light
High Specific Strength

Bioactive

Elastic modulus

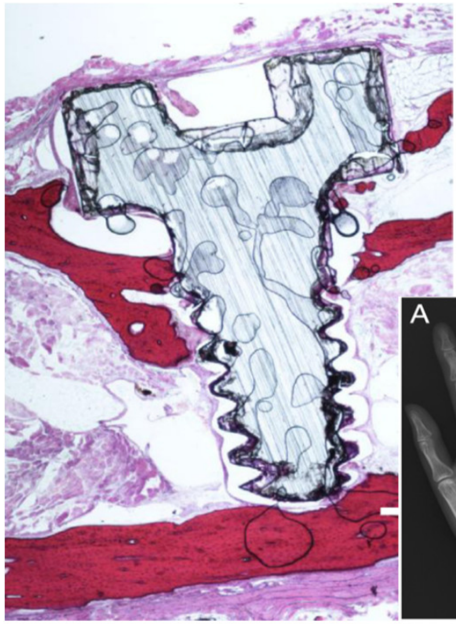
Biodegradable

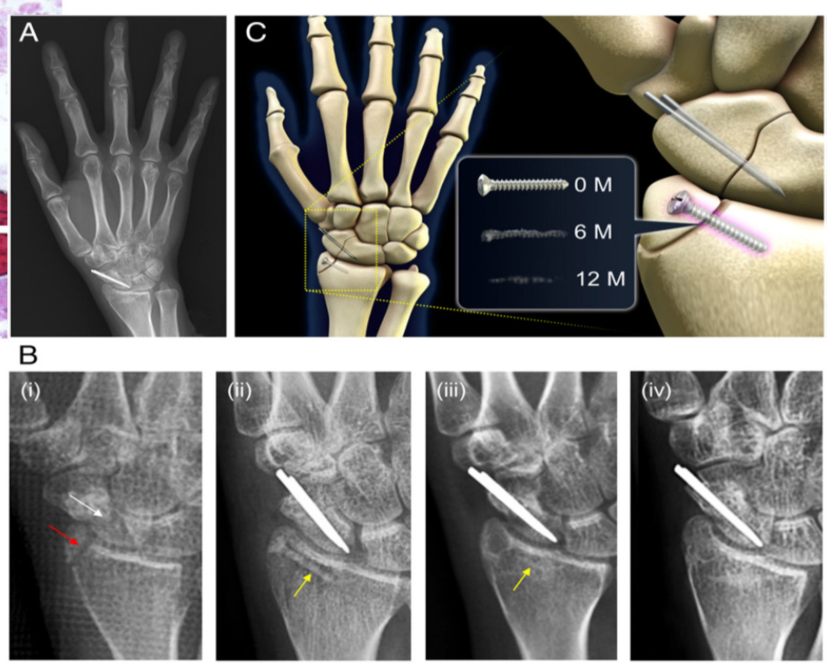
Corrosion
(Strength)

Alloying..
Coating..

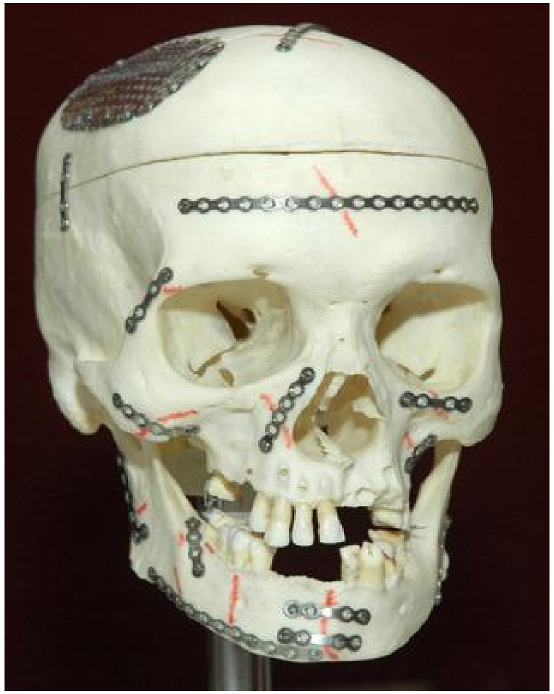
$$Mg + 2\,H_2O = Mg(OH)_2\,(s) + 2\,H_2\,(g)$$

Mg-based bone implants





Possible applications of Mg



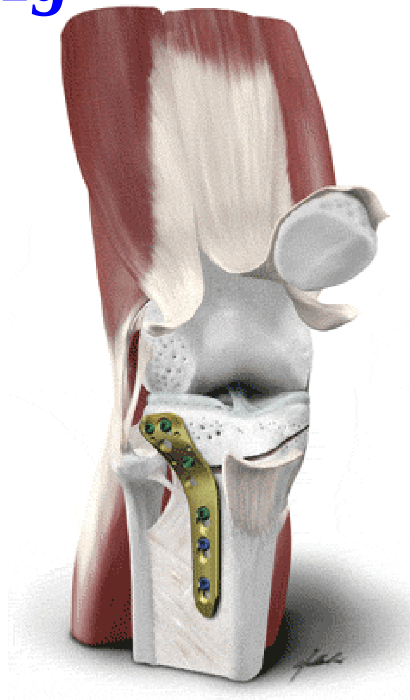
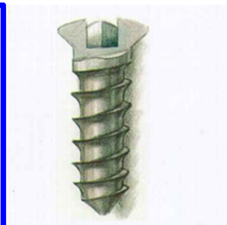


Fig. 4.1 **Four hole, 2 mm miniplate**



ig. 4.2 **Miniscrew, 2 mm**

Courtesy Prof. Hyoun-Ee Kim

Magnesium

Mg : Corrosion

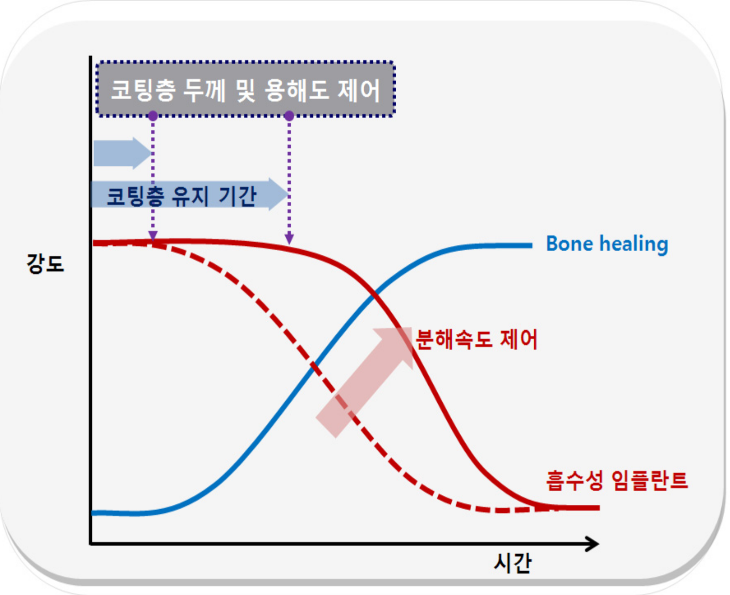
$$Mg + 2H_2O = Mg(OH)_2\,(s) + 2H_2\,(g)$$

Table 8.2 The standard EMF series

Metal ⟶ Metal Ion	Standard Electrode Potential (V)	
Au ⟶ Au^{3+}	+1.50	↑
Pt ⟶ Pt^{2+}	+1.2	
Pd ⟶ Pd^{2+}	+0.99	
Ag ⟶ Ag^{+}	+0.80	
Cu ⟶ Cu^{2+}	+0.34	
H ⟶ H^{+}	0	Increasingly inert
Ni ⟶ Ni^{2+}	−0.25	
Co ⟶ Co^{2+}	−0.28	Increasingly active
Fe ⟶ Fe^{2+}	−0.44	
Cr ⟶ Cr^{2+}	−0.74	
Al ⟶ Al^{3+}	−1.66	
Ti ⟶ Ti^{3+}	−2.00	
Mg ⟶ Mg^{2+}	−2.36	↓

Alloying

Coating



Courtesy Prof. Hyoun-Ee Kim

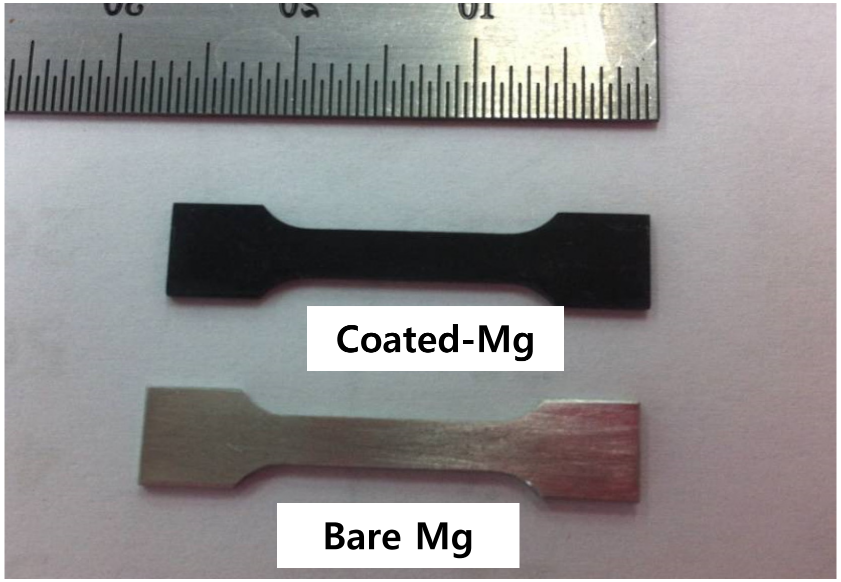
Alloying element	Abbreviation letter
Aluminum	A
Bismuth	B
Copper	C
Cadmium	D
Rare earth metals	E
Iron	F
Thorium	H
Zirconium	K
Lithium	L
Manganese	M
Nickel	N
Lead	P
Silver	Q
Chromium	R
Silicon	S
Tin	T
Yttrium	W
Antimony	Y
Zinc	Z

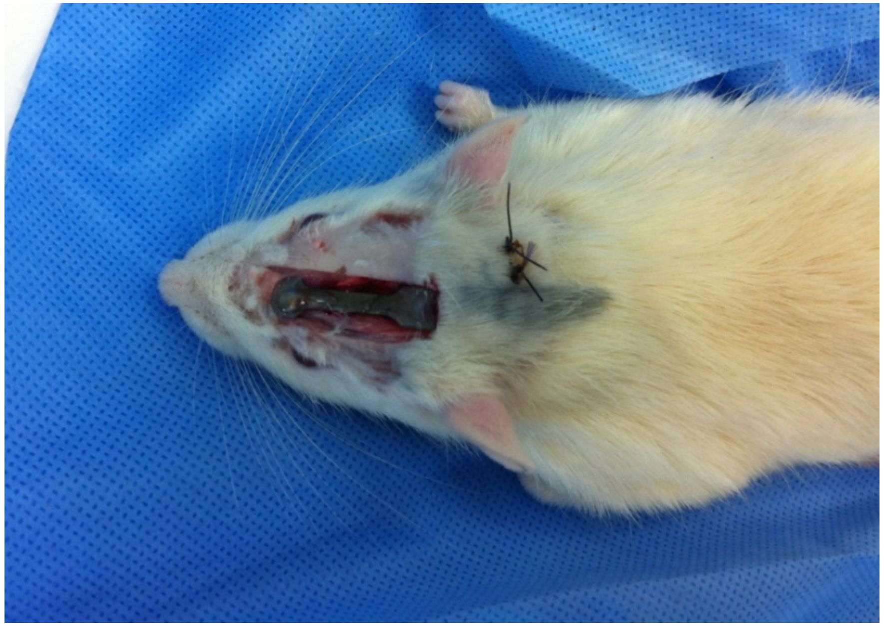
AZ31, AZ91 . .

ZK60, ZM21. .

WE43

Strength retention

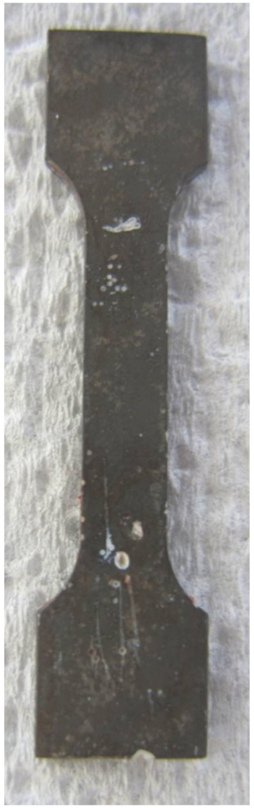


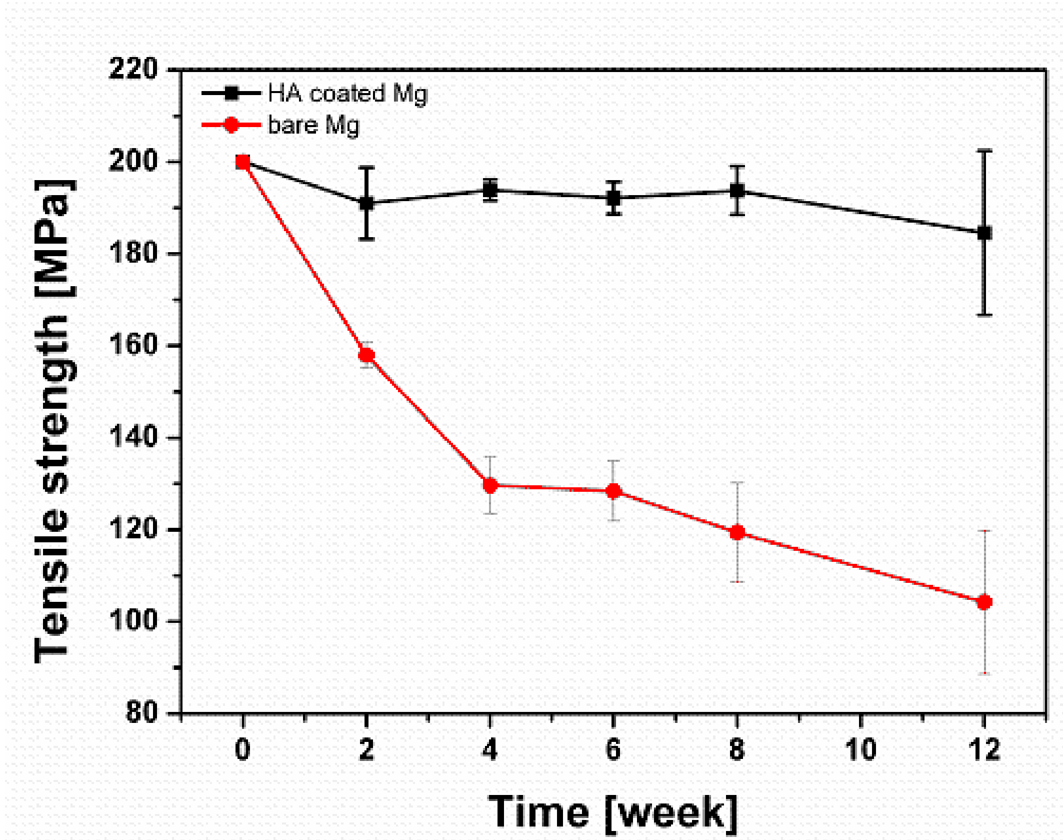


Hyoun-Ee Kim, SNU
Calvarias of Rat for ***up to 12 weeks***

Bare

HA coated

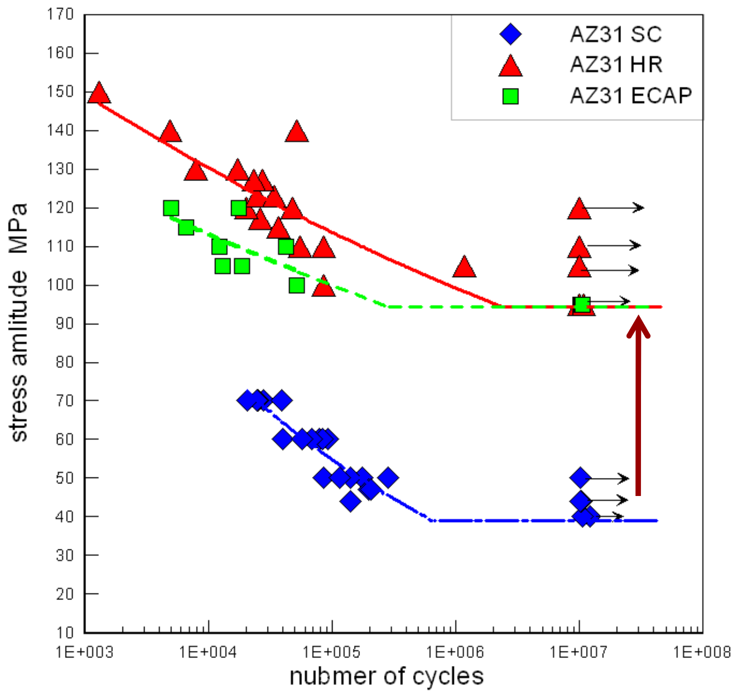


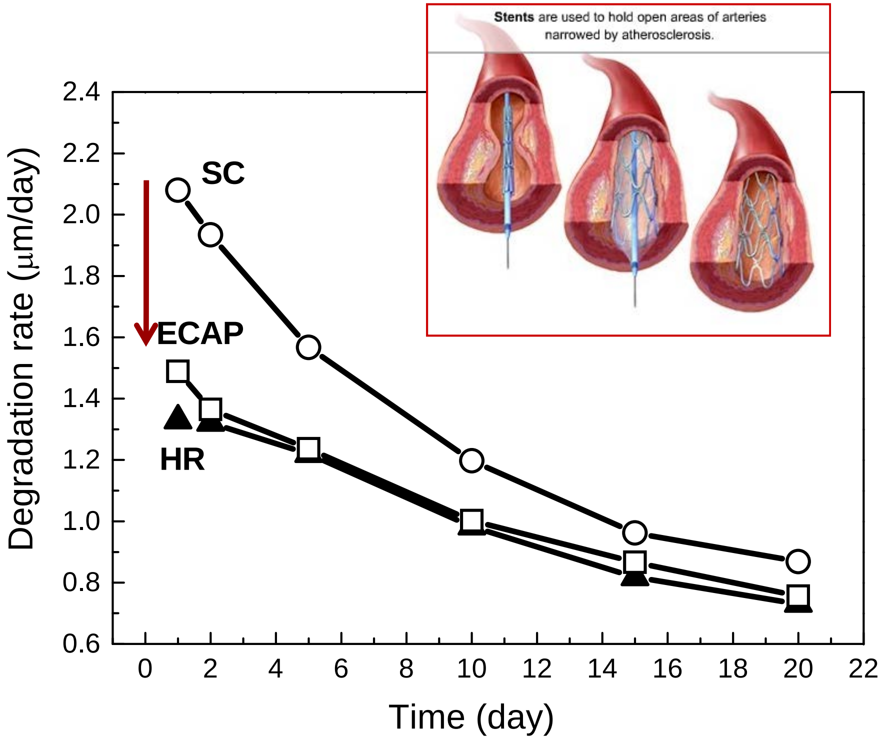


6 weeks

8 weeks

Mg for temporary implants?





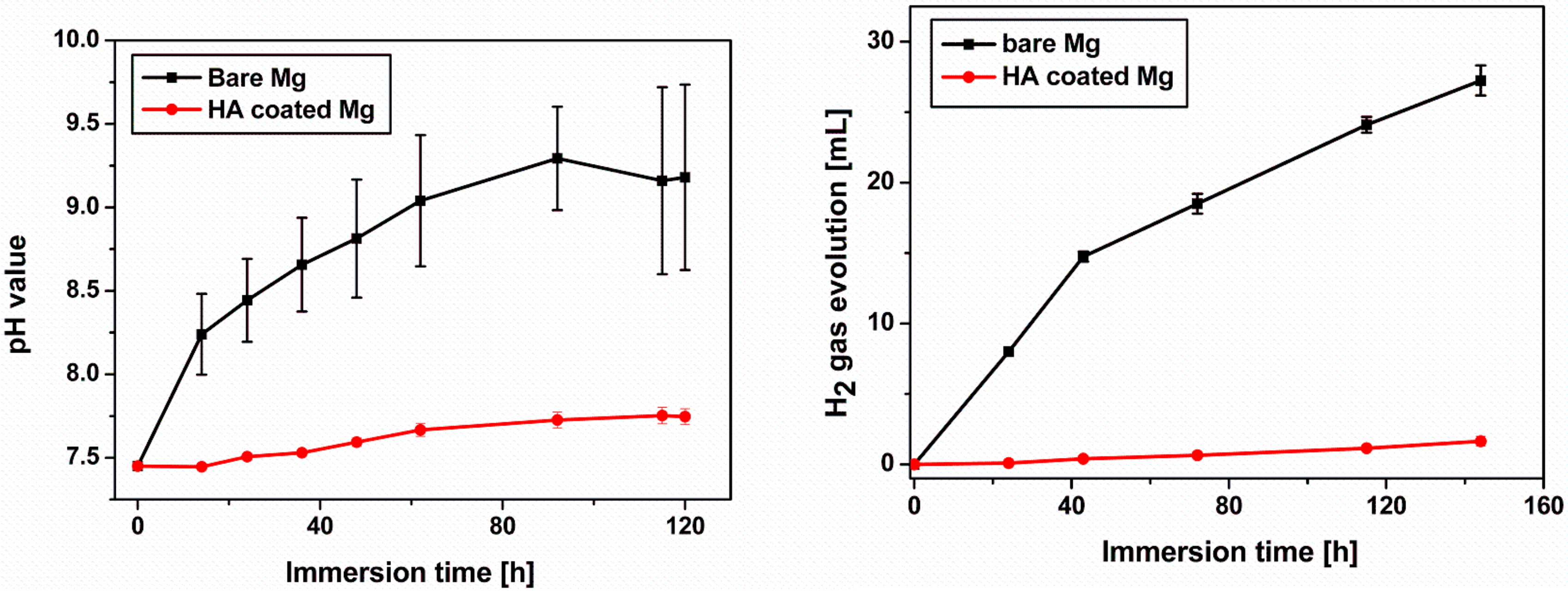
Fatigue strength

Corrosion in Hank's solution

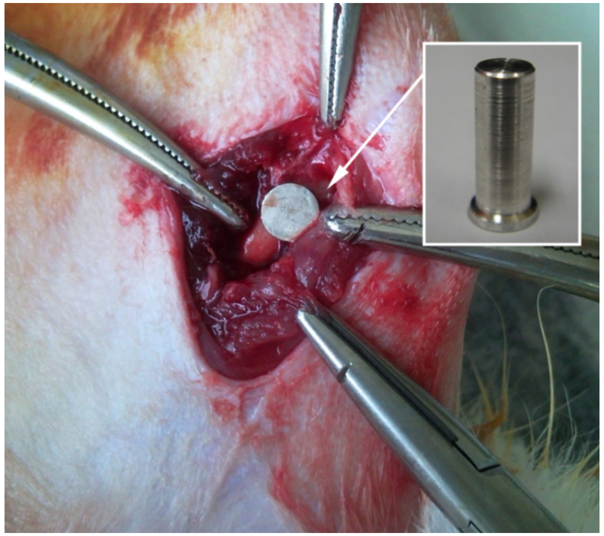
Wang, Song, Estrin, Zuberova, Adv. Eng. Mater. 9 (11), 967-972 (2007)

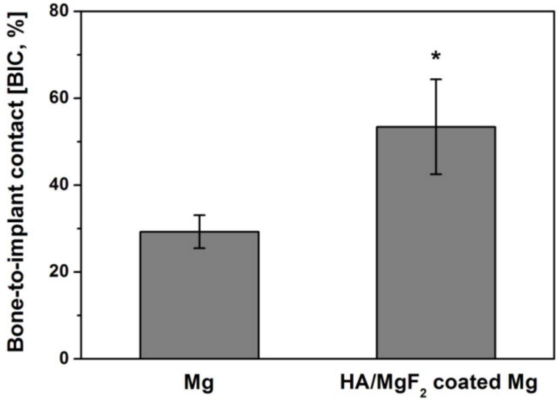
Research at Seoul National University, Prof. Hyoun-Ee Kim

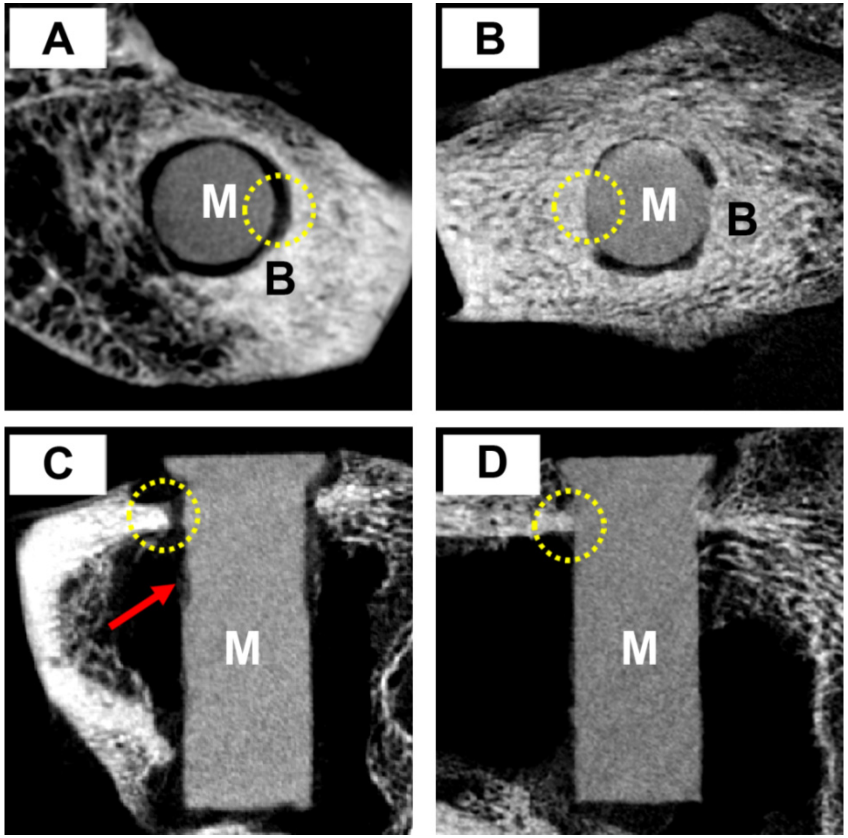
Corrosion resistance of Mg – Role of coating



Research at Seoul National University, Prof. Hyoun-Ee Kim







Rabbit femoral shaft, 4 weeks

Magnesium alloys

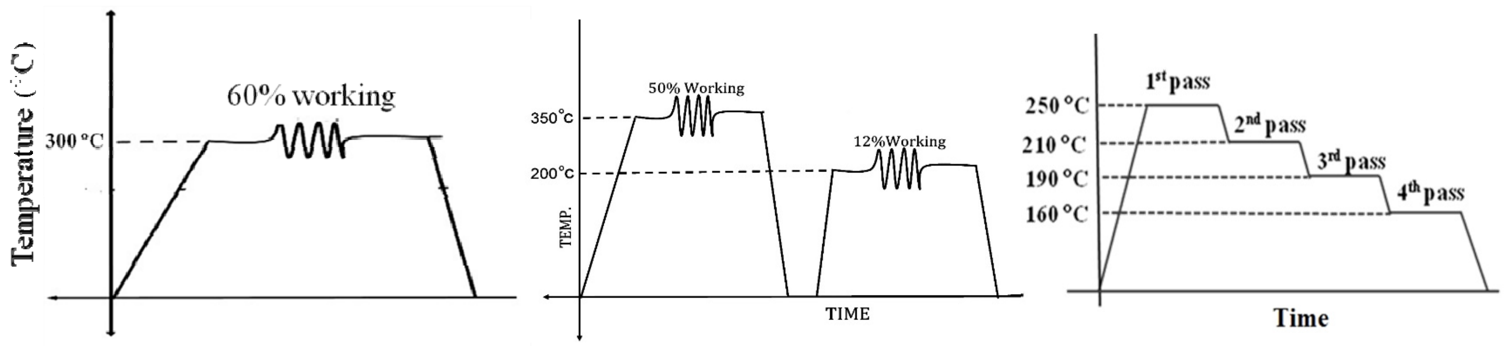
- *Mg-RE (WE43)*
- *Mg-Ca*
- *Mg-Zn-Ca*
- *Mg-Li-Ca*

Methods of SPD

- *High pressure torsion (HPT)*
- *Equal Channel Angular Pressing (ECAP)*
- *Multiaxial Deformation (MD)*
- *Rotary swaging (RS)*

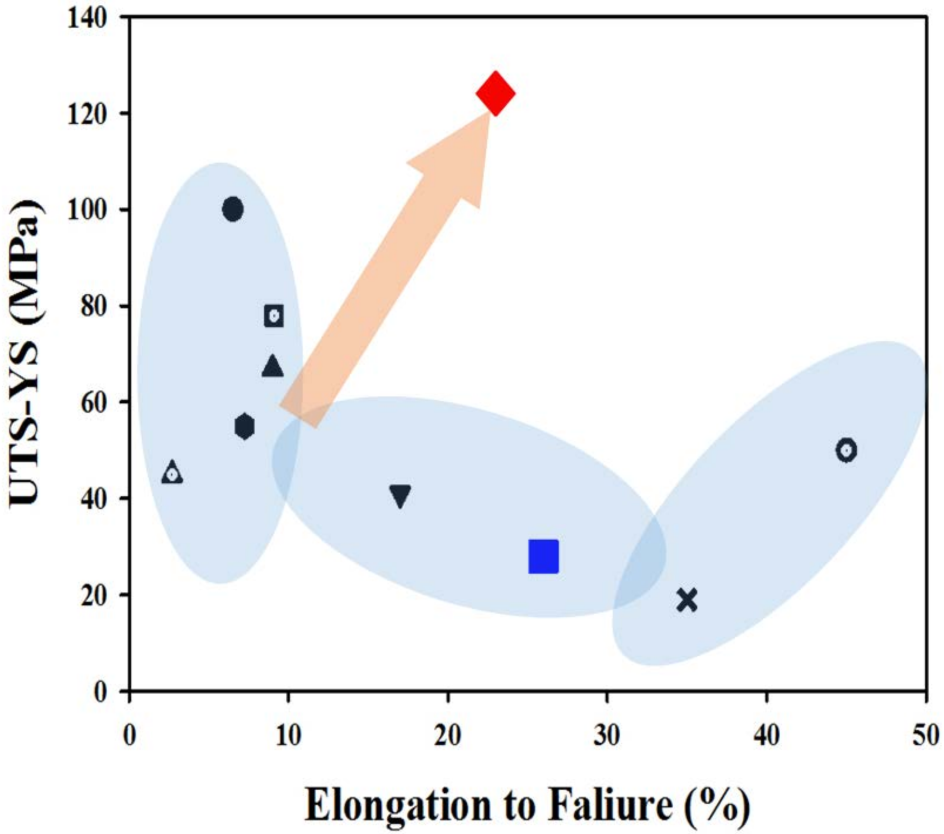
Estrin, Dobatkin, Lukyanova, Anisimova (work in progress)

LX41: Processing schedule

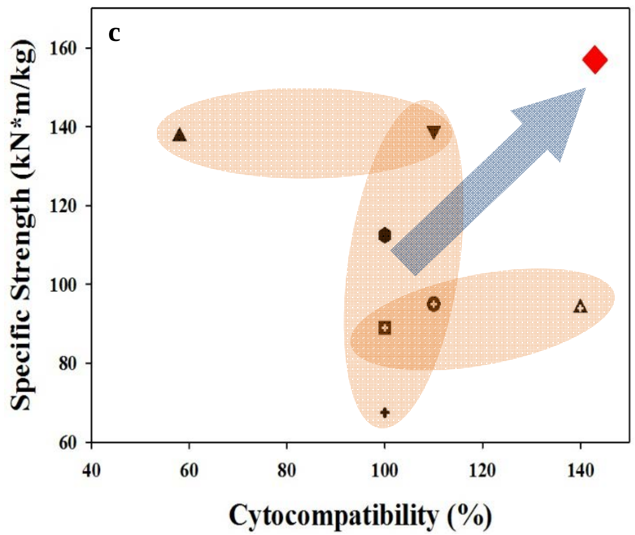


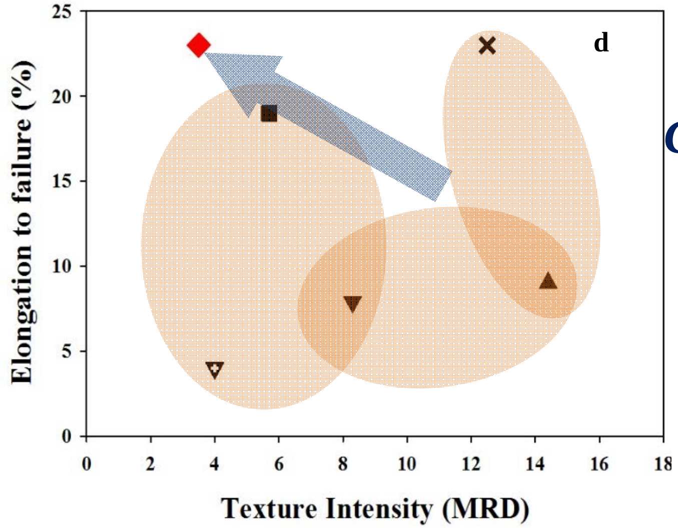
Saurabh Nene, PhD Thesis 2016
(Monash-IITB)

Property profile of alloy LX41 after rolling and annealing

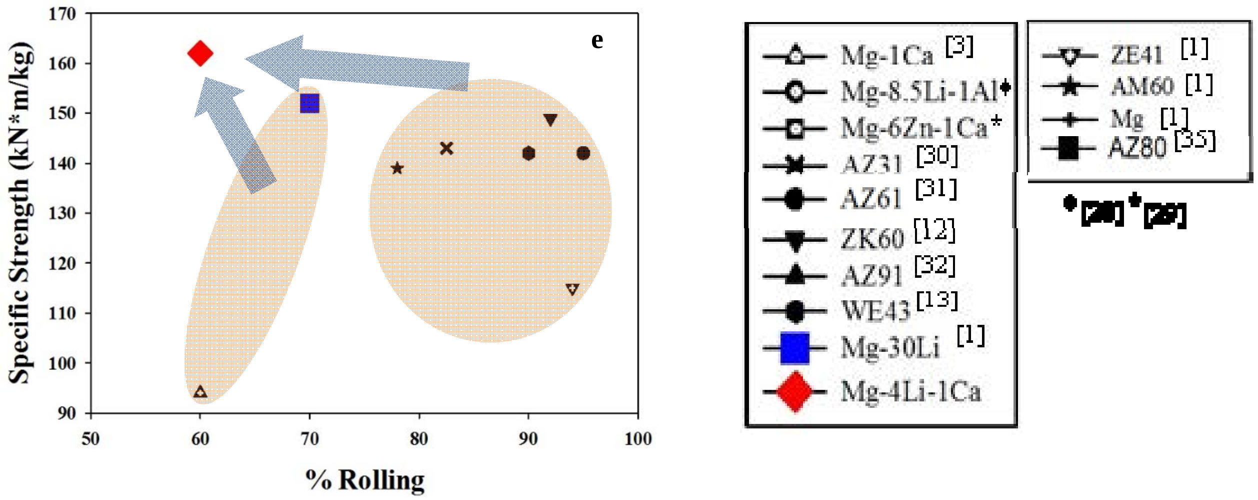


Estrin et al., Mater. Lett. 173, 252 (2016))
(Materials Today, April 2016)

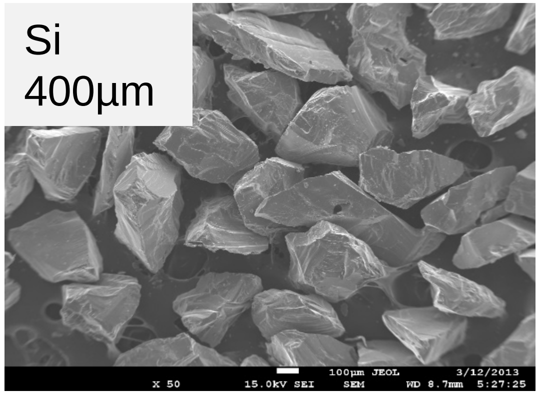


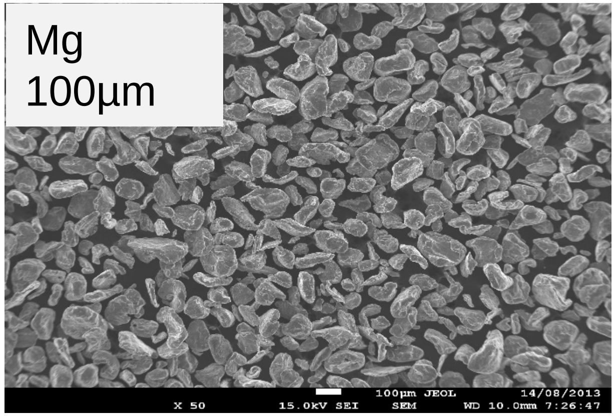


Specific Strength, Cytocompatibility and Texture Intensity

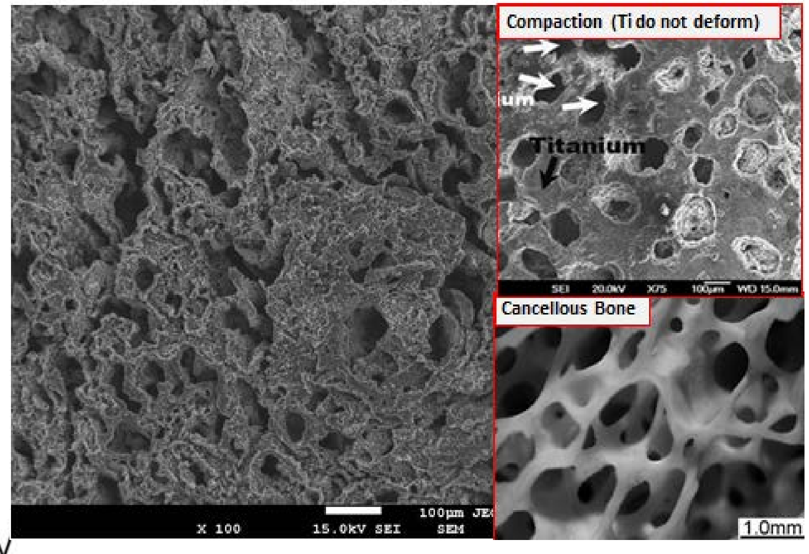


Obtaining porous Ti and Ti/Mg





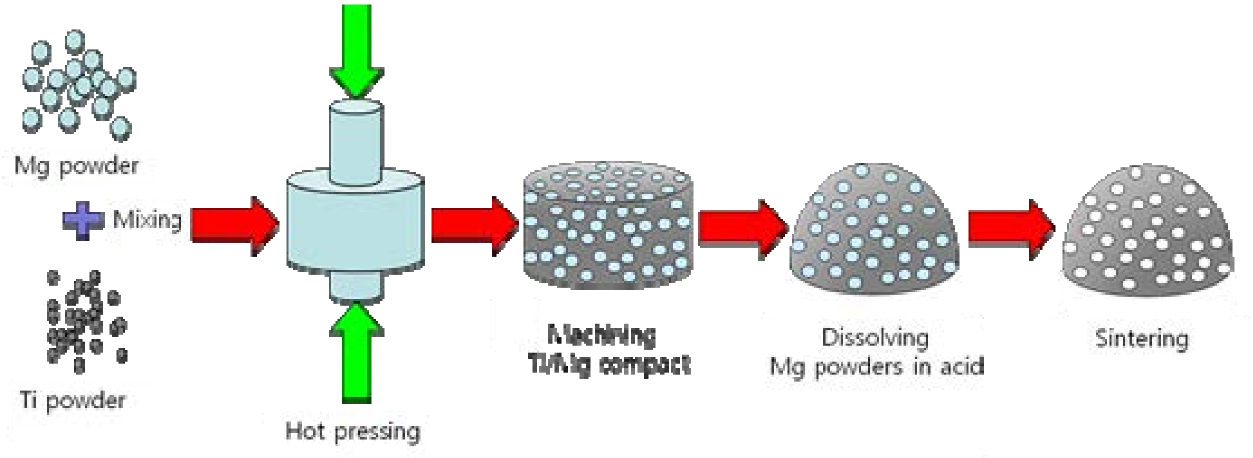
Ti40Mg20Si40 after leaching

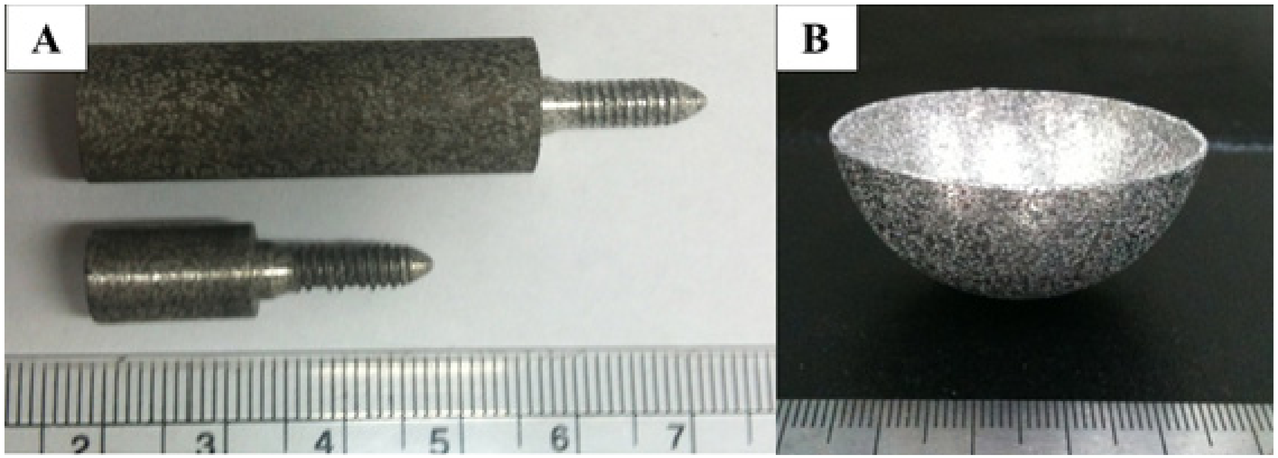


Y. Qi, R. Lapovok, Y. Estrin, 2014

MONASH University

Porous titanium for medical implants





S.W. Kim et al.,"Fabrication of porous titanium scaffold with controlled porous structure and net-shape using magnesium as spacer", Mater. Sc. Eng.: C 33, 2808 (2013).

GENERAL CONCLUSION:

There is a lot of scope for research and development in the burgeoning area of medical implants – both permanent and bioresorbable

Thank you for your attention!

yuri.estrin@monash.edu