New and novel coatings for medical applications

– an introduction to Physical Vapour Deposition (PVD) with examples of passive and active coatings relevant for medical applications

Lars Pleth Nielsen
Danish Technological Institute
Tribology Centre

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Outline

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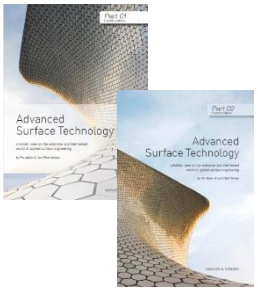
- Introduction to surface coatings
- Introduction to Physical Vapor Deposition (PVD)
  - The technique
  - Our equipment
- Examples of coatings for medical applications
  - Porous TiN for novel electrodes
  - Low friction diamond-like carbon (DLC) coating for dental applications
  - Strontium releasing coatings accelerating bone growth
- Conclusion - summary



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Tribology Centre

DANISH TECHNOLOGICAL INSTITUTE

- More than 25 years of experience in coatings
- 17 employees + 6 students
- Last year we coated 234.000 items
- 60% on production / 40 on R&D

Definition of Tribology:
It is the science and engineering of interacting surfaces in relative motion. It includes the study and application of the principles of friction, lubrication and wear.

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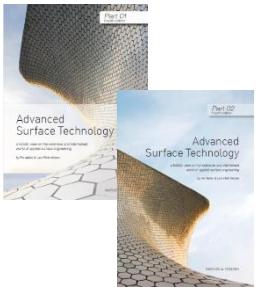

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Introduction to
surfaces coatings

Surfaces are extremely important

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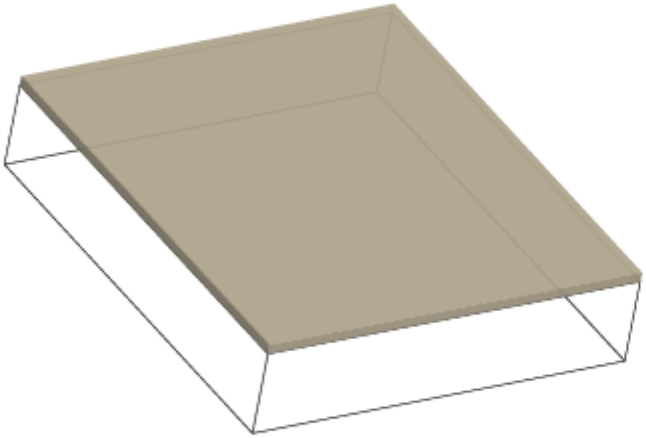

Wear resistance
Tribological properties
Magnetic properties
Electrical conductivity
Solderability/weldability
Hardness
High temperature resistance
Corrosive resistance
Biocompatibility
Hydrophobic/hydrophilic properties
Catalytic properties
Self cleaning properties
Friction properties
Color and appearance
Decorative appearance
Refractive index
Oxide formation/passivation

Surfaces are important – decorative

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Standard colors

Grey	Black	Gold	Red	Others
Stainless Steel	Anthracite	Gold 24K	Copper Rose	Dark Flat earth
Nickel	Black	Gold 18K	Bronze	Sand
Smoked Grey	Black	French Gold	Brass	Rainbow

Surfaces are important – decorative

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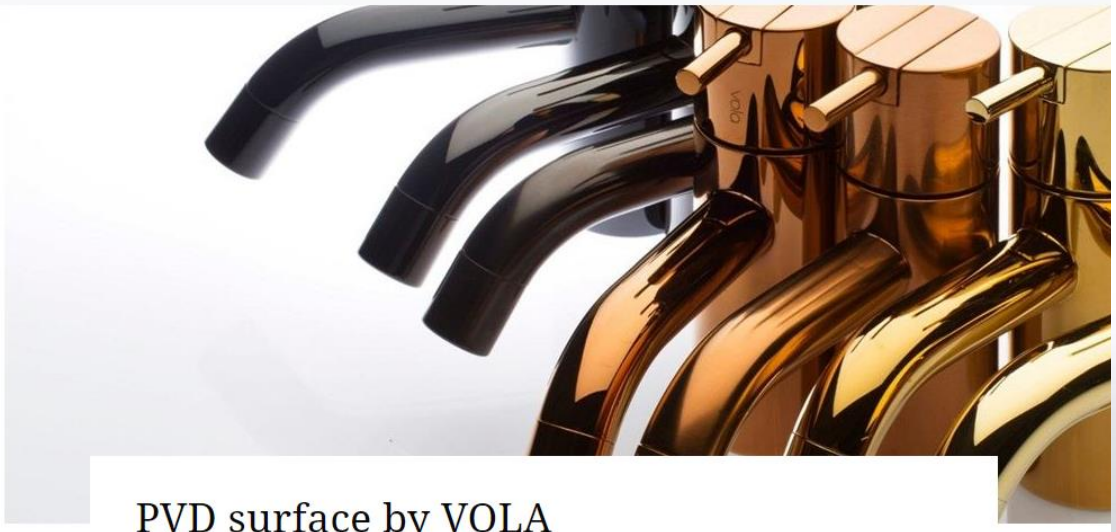

PVD surface by VOLA

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Low friction surfaces

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PRODUKTFAMILIEN DLC

Diamond Like Carbon

Diamond Like Carbon (DLC) er betegnelsen for en hård og glat kulstofoverflade - en slags mellemting mellem grafit og diamant.

LAV FRIKTION
DLC-TR er Tribologicentrets lavfriktions-PVD-belægning, som består af en funktionelt graderet belægning (FGC) af diamantlignende kulstof. Som hovedregel kan DLC-TR anvendes på metaldele, hvor der ønskes nedsat friktion. Opbygningen som FGC-belægning giver maksimal vedhæftning af DLC-belægningen på alle stål og på de fleste andre legeringer.

DLC-TR TIL BEVÆGELIGE VÆRKTØJS- OG MASKINDELE
Maskinelementer og værktøjsdele i glidende eller roterende bevægelse er næsten altid udsat for slid. Ofte er man tillige interesseret i at have så lav friktion som muligt. Disse problemer løses traditionelt med smøring enten med fedt eller med olie.

Fedtsmøring og især oliesmøring kræver at hastighed, belastning, geometri og smøremiddel er nøje afstemt efter hinanden. Når det ikke kan lade sig gøre at få alt dette til at gå op i en højere enhed er resultatet som regel for stort slid eller egentlig sammenrivning af delene.

Løsningen på dette problem kan være en overfladebelægning med DLC-TR. Denne kan enten bruges tørt eller i kombination med traditionel smøring. Når DLC-TR kombineres med olie eller fedt, sørger belægningen for smøring, når overfladernes ruhed alligevel træder gennem smørefilmen.

OLIE ELLER FEDT PÅ PLASTEMNER - IKKE MED DLC-TR
Bevægelige kerner og udstødere skal ofte smøres for ikke at rive. Denne smøring fører som regel til en vis afsmitning af smøremiddel på de færdige emner. Skal emnerne bruges til fødevarer, medicin eller som dekoration kan forurening med smøremiddel normalt ikke accepteres. Overfladebelægning med DLC-TR vil normalt løse dette problem.

BAD-DLC
IBAD-DLC er en ionstråle-assisteret udfældning af en kulstofbelægning, som især anvendes på hærdede stålværktøjer. Den samtidige ionimplantering og belægning sikrer en vis opblanding af materialerne i grænselaget mellem belægning og grundmateriale. Herved opnås en overordentlig god vedhæftning af belægningen.

FORMGIVNING AF TYNDPLADE
Ved minimalsmurt eller smørefri formgivning af tyndpladeprodukter er IBAD-DLC det helt rigtige valg. Formgivning uden eller næsten uden smøremiddel betyder ofte meget i emnernes videre forløb, fordi rester af smøremiddel som regel skal fjernes.

SMØR MED VAND
Smøring af metalkomponenter med vand er normalt ikke muligt, bl.a. på grund af vands meget lave viskositet. Hvis mindst én af glide- eller lejefladerne overfladebehandles med DLC-TR er der dog ingen risiko for rivning på komponenter eller maskindele, der kører i vand - heller ikke ved eventuel tørkørsel.

For mere information, kontakt

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www.teknologisk.dk/tribo

Hard surface - Chromenitride

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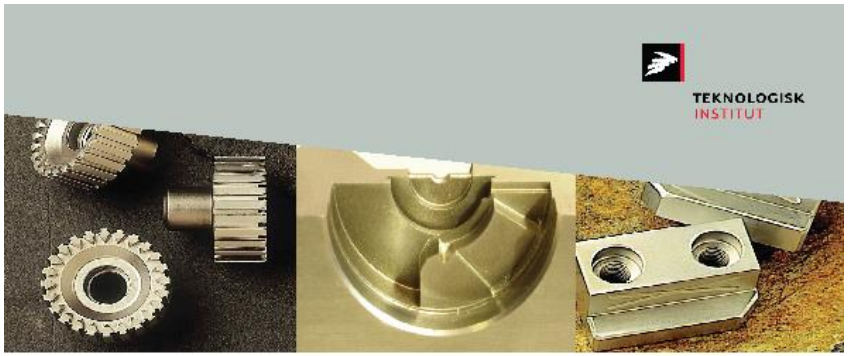

PRODUKTFAMILIEN CrN

Kromnitrid

CrN - EN FLEKSIBEL LØSNING TIL MANGE FORMÅL
Sammenlignet med mange af Tribologicentrets øvrige belægninger er CrN mindre hård, men til gengæld har CrN en lang række andre unikke fordele, som gør belægningen meget velegnet til værktøj, sliddele og maskinkomponenter. Hårdheden af CrN er ca. to gange hårdheden af traditionel hårdkrom - og så skal den ikke slibes efter belægning.

CrN er en duktil (bøjelig/elastisk) belægning og hårdheden af grundmaterialet er derfor ikke kritisk.

CrN-LT - LAVTEMPERATUR
CrN-processen kan udføres ved lav temperatur (ca. 150 °C) og Tribologicentret har udviklet en speciel skånsom proces, som efterlader overfladekvaliteten af polerede overflader intakt.

CrN-LT gør det muligt at opnå god varmeledning og høj slidstyrke, godt slip og god produktfinish på småserieforme i let bearbejdelige materialer.

Disse egenskaber gør Tribologicentrets CrN-LT til et optimalt valg på alle ståltyper (også lavt anløbne) samt på aluminium og kobberlegeringer.

SPÅNTAGNING OG FORMGIVNING AF METAL
Ved spåntagning og formgivning af metal kan CrN anvendes mod påklæbning af bløde metaller som aluminium og kobberlegeringer samt i visse tilfælde også til rustfrit stål.

CrN-SD (Super Dense)
Gennem en optimering af mikro- og nanostrukturen på kromnitridkrystallerne har Tribologicentret udviklet en særlig tæt belægning, CrN-SD, hvor krystallerne vokser på en mere kompakt måde end i de traditionelle CrN-belægninger. Herved opnås en mere hård og tæt belægning, som er særdeles velegnet til hårdt belastende processer med klæbende metaller.

SLIPBELÆGNING TIL SPRØJTESTØBEFORME
Afformning af plast er et vanskeligt problem. Grundet den meget lave polaritet har CrN og CrN-LT gode slipegenskaber over for visse plasttyper. Tribologicentret har mange års erfaring med afhjælpning af afformningsproblemer i sprøjtestøbeforme.

CrN-SS (Super Slip)
For at opnå optimeret slip i særlige vanskelige sprøjtestøbeværktøjer har vi udviklet en ny type kromnitrid, CrN-SS, der har klart forbedrede slipegenskaber over for de fleste vanskelige plasttyper. CrN-SS har endvidere vist, at det er muligt at afforme i et større temperaturområde, hvilket i højere grad tillader optimering af selve sprøjtestøbningsprocessen uden hensyntagen til afformningen. Dette betyder forbedret lønsomhed gennem reducerede cyklustider.

AFVISNING AF KALK OG ANDRE BELÆGNINGER
Den lave polaritet på Tribologicentrets CrN-belægning er årsag til, at visse stoffers tendens til at danne bindinger til overfladen reduceres. Et eksempel på denne virkning er anvendelse på dyser eller armaturer til vand, hvor CrN kan reducere kalkudfældning betydeligt.

For mere information, kontakt

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How do we select coatings?

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Ion implantation

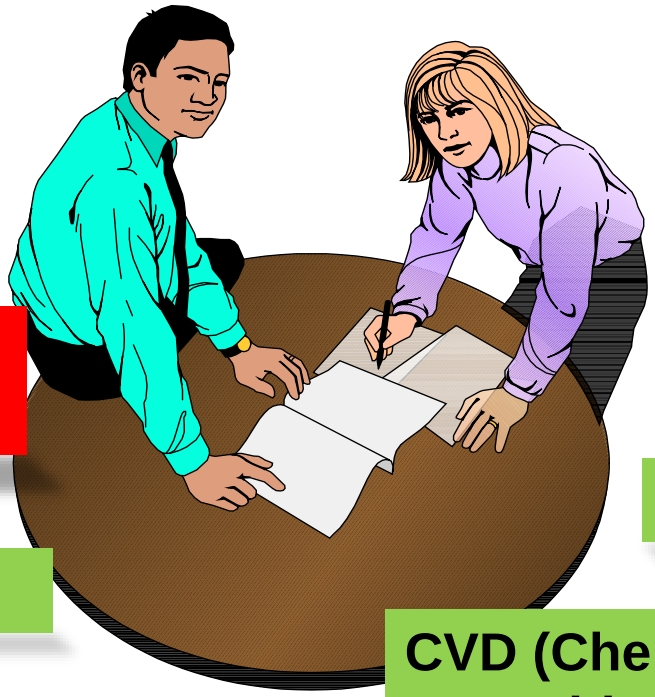
Nitriding

Electroless plating

Organic coating

Thermal sprayed coatings

Anodization



PVD (Physical Vapour Deposition)

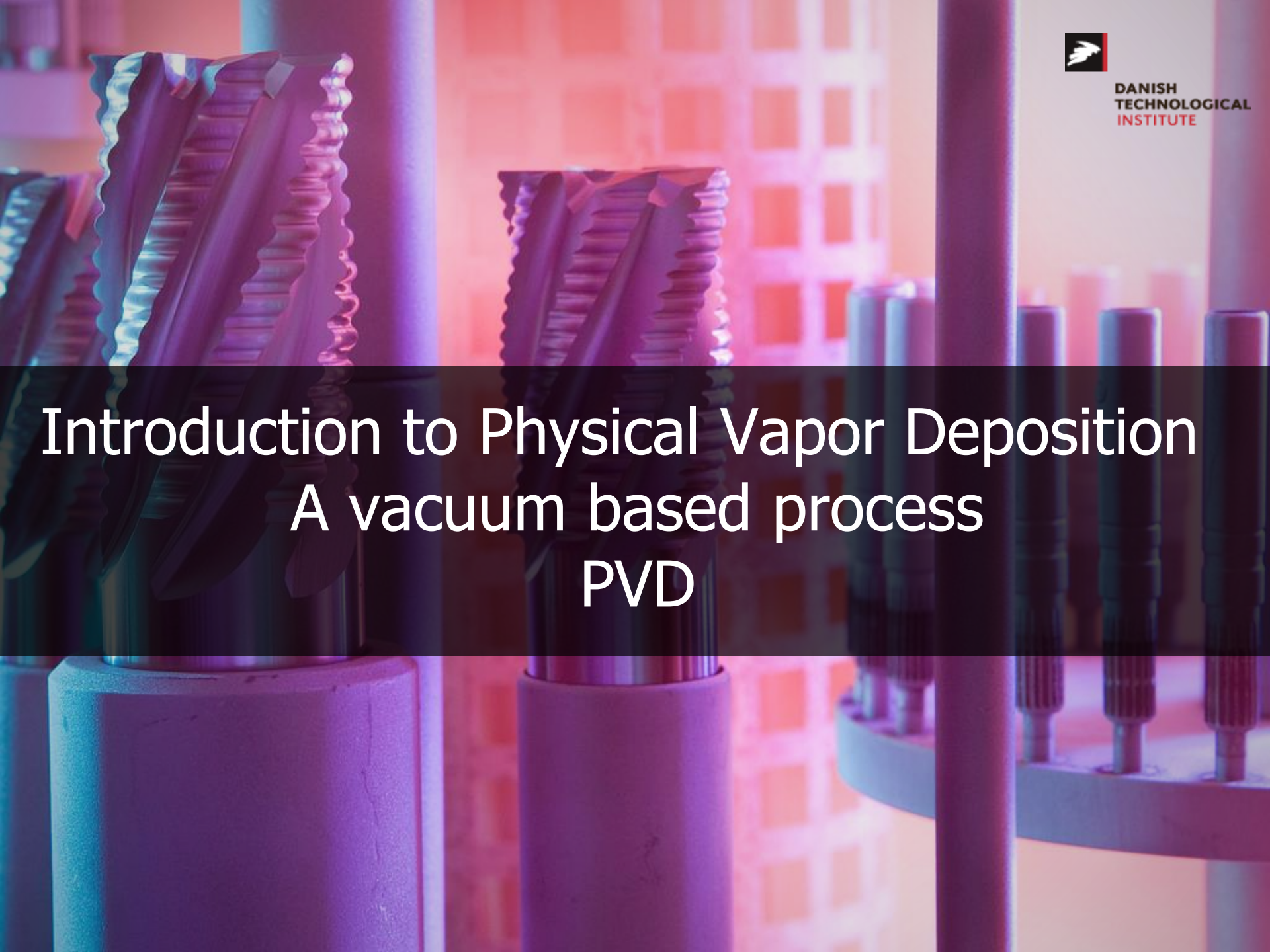
Cladding

Vitreous enameling

Electroplating

CVD (Chemical Vapour Deposition)

Hot dip galvanizing



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Introduction to Physical Vapor Deposition
A vacuum based process
PVD

Thin film processes

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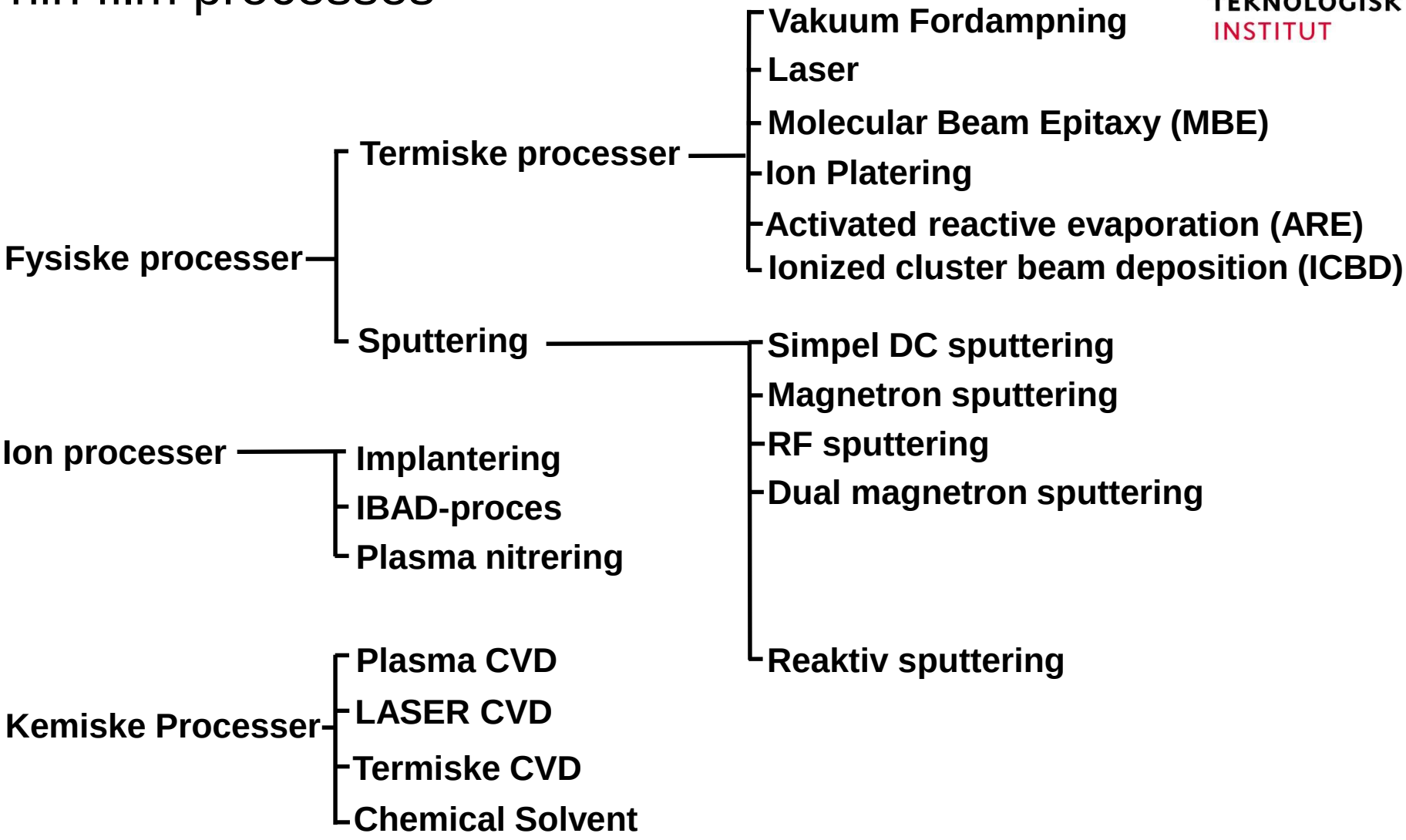

Thin film processes

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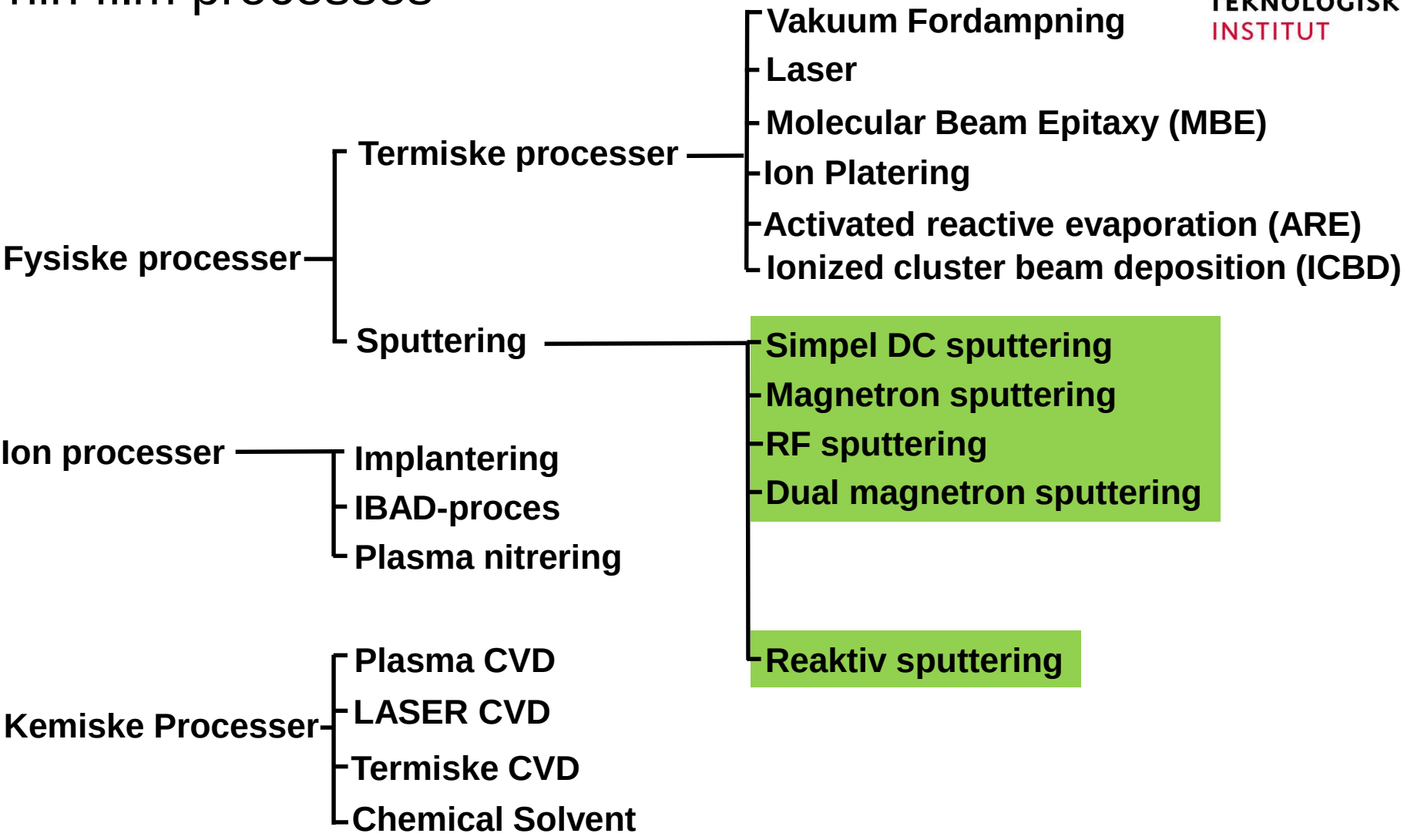

Vacuum Chamber – controlled atmosphere

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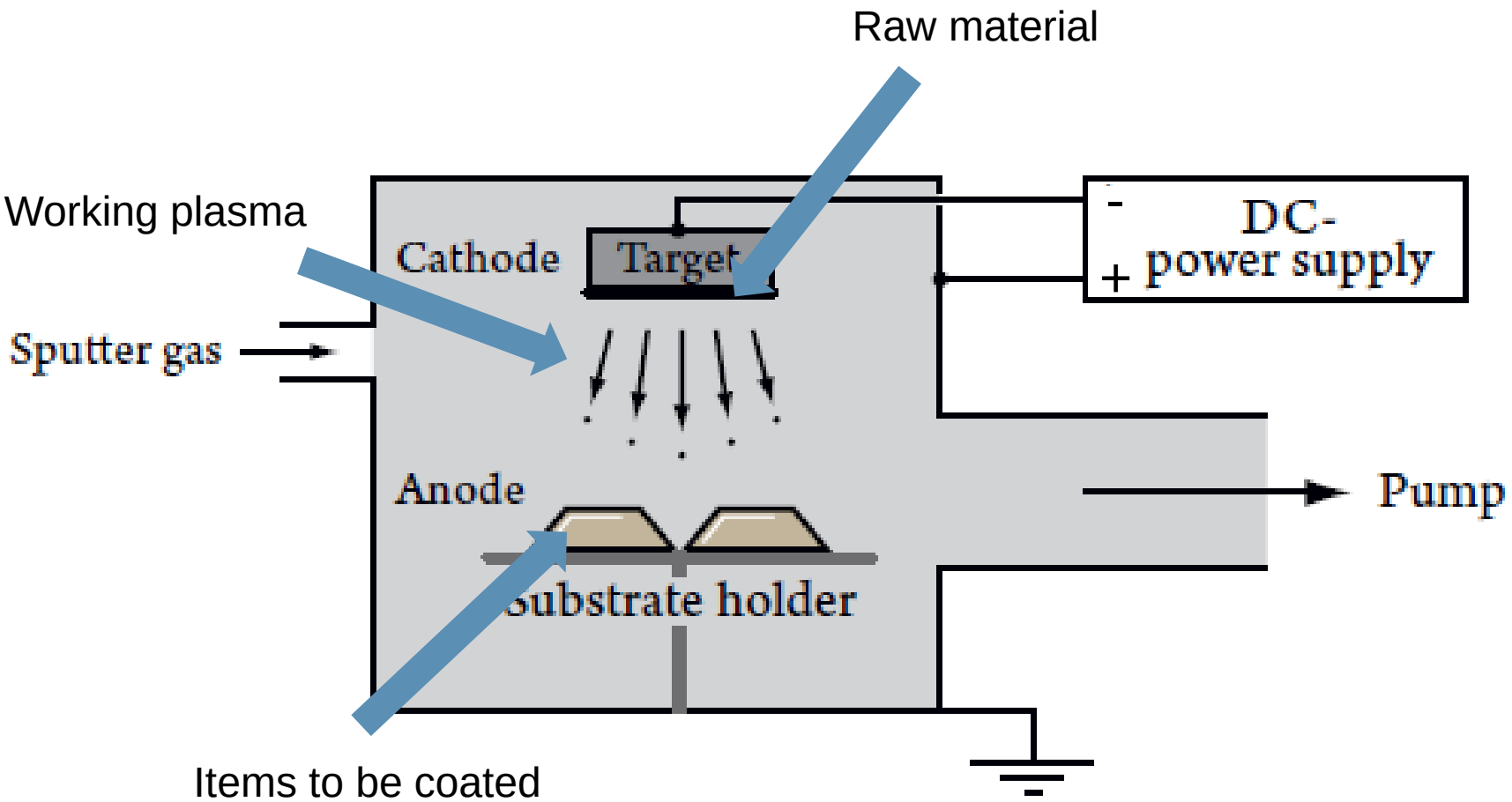

What is a plasma

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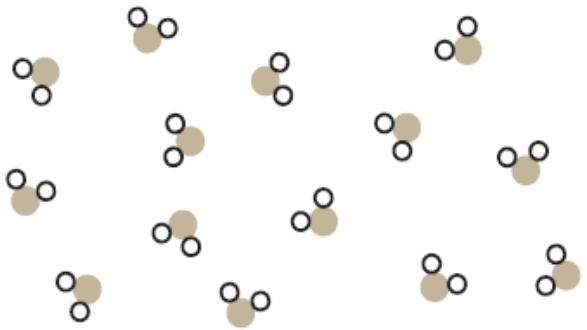
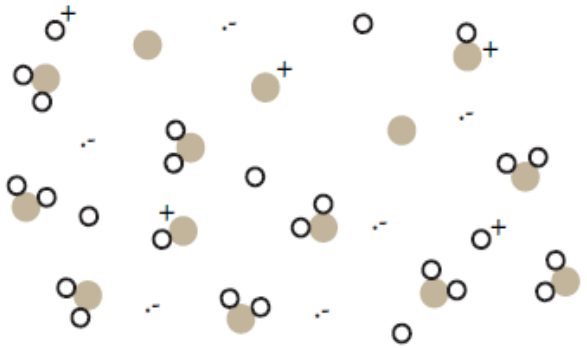


Schematic of a neutral gas.



Schematic of a plasma composed of atoms, molecules, charged atoms and molecules, as well as electrons.

Definition:

Plasma is a distinct phase of matter, separate from the traditional solids, liquids, and gases.

It is a collection of charged particles that respond strongly to electromagnetic fields, taking the form of gas-like clouds or ions. The particles in a plasma are electrically charged (formed by stripping electrons), it is frequently described as an "ionized gas“.

Plasma was first identified (as "radiant matter") by Sir William Crookes in 1879.
Sir J.J. Thomson identified the nature of the matter in 1897.
It was Irving Langmuir who assigned the term "plasma" in 1928.

Plasma is actually the most common phase of matter. Flame, lightning, interstellar nebulae, stars, and even the empty vastness of space are all examples of the plasma state of matter.

What is a plasma

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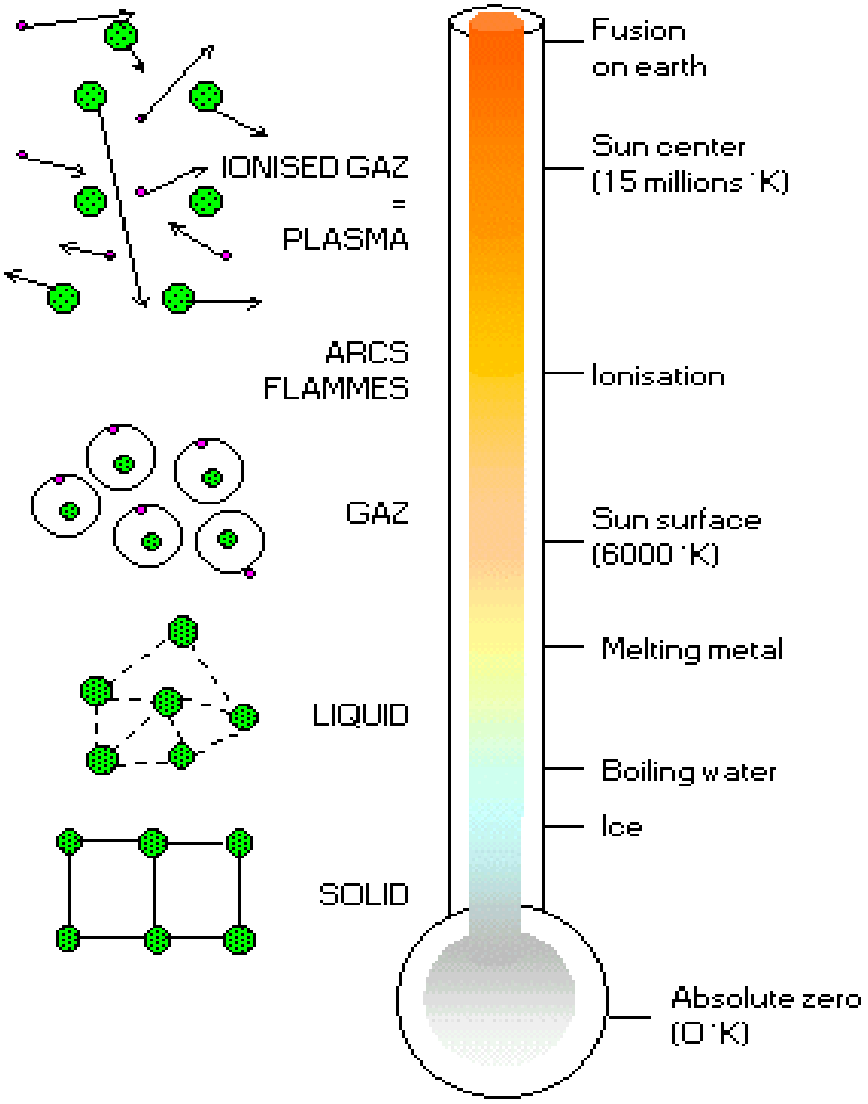

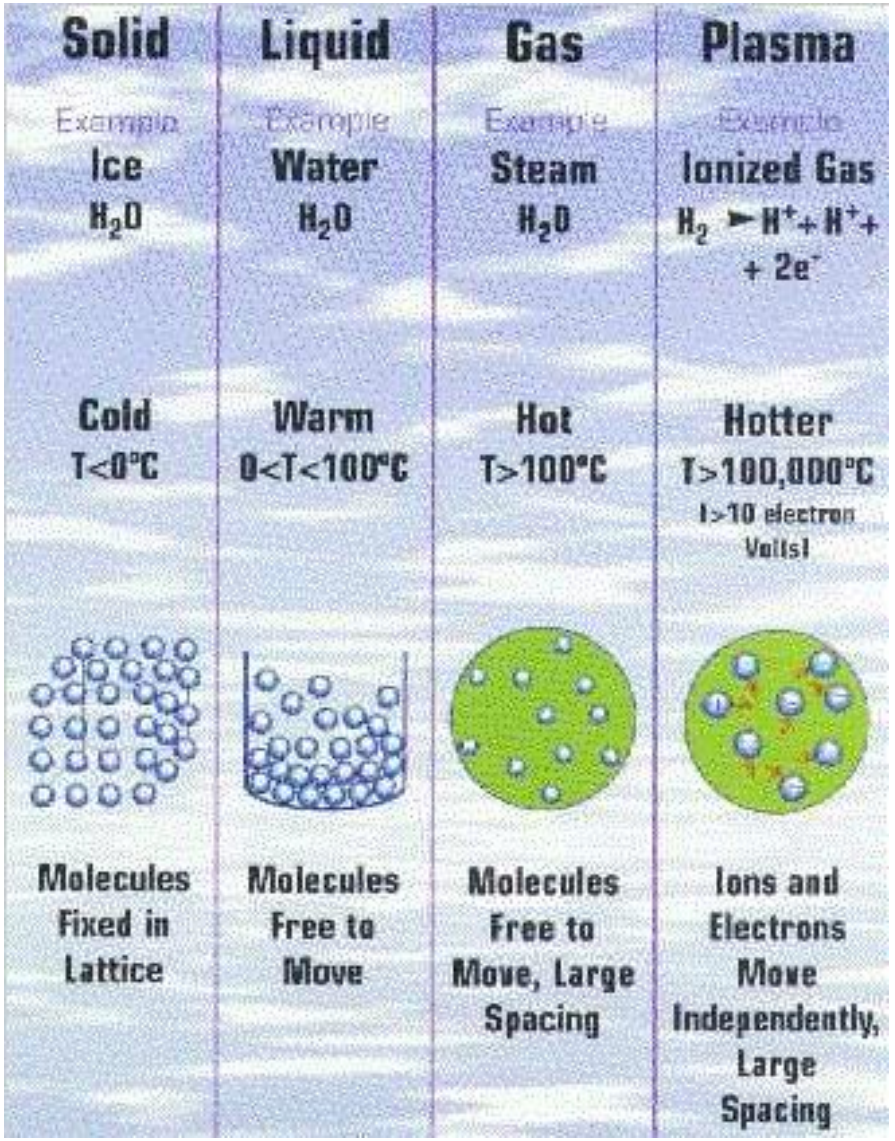

Sputtering

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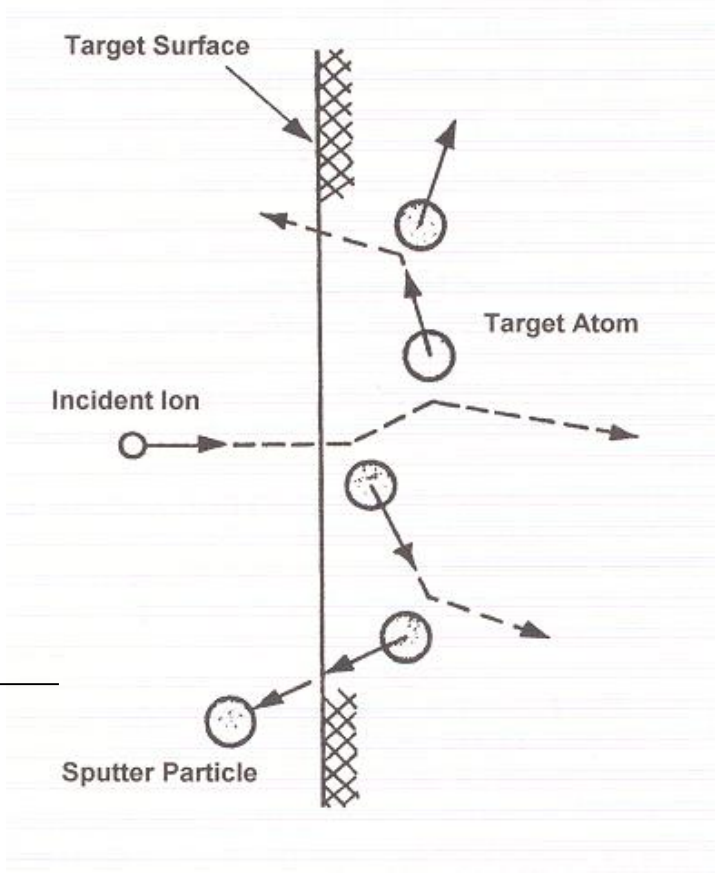
Plasma e.g. Ar^+

Cathode of the raw material



$$\text{Sputter yield} = \frac{\text{Atoms removed}}{\text{Incoming ions}}$$

Sputter yield

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$$\text{Sputter yield} = \frac{\text{Atoms coming out}}{\text{Atoms coming in}}$$

What does it depends on?

- Type of incoming atoms (the plasma)
- Type of atoms in the target
- Incoming energy
- Incoming angle

Sputter yield; Ar versus Ne

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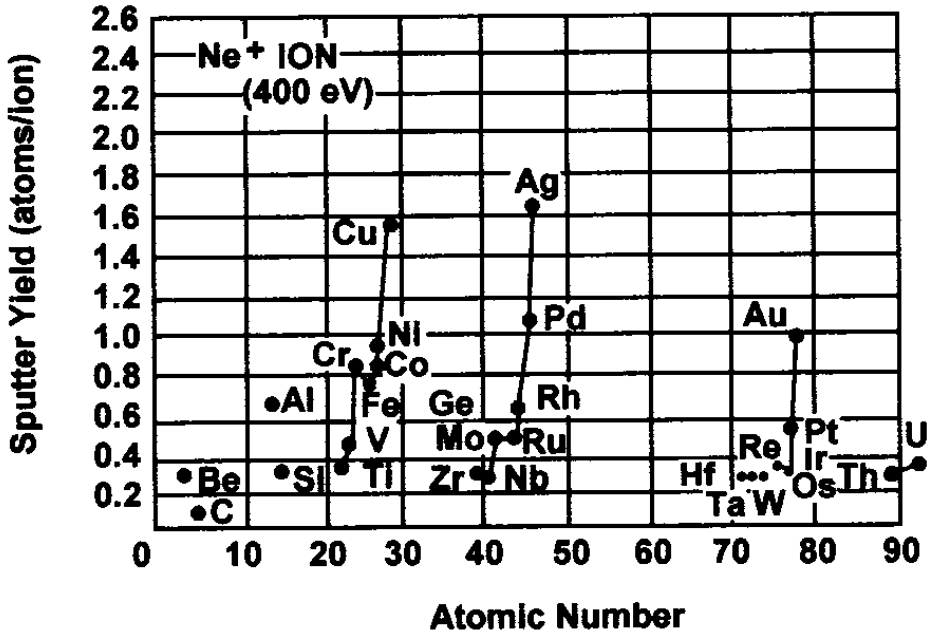

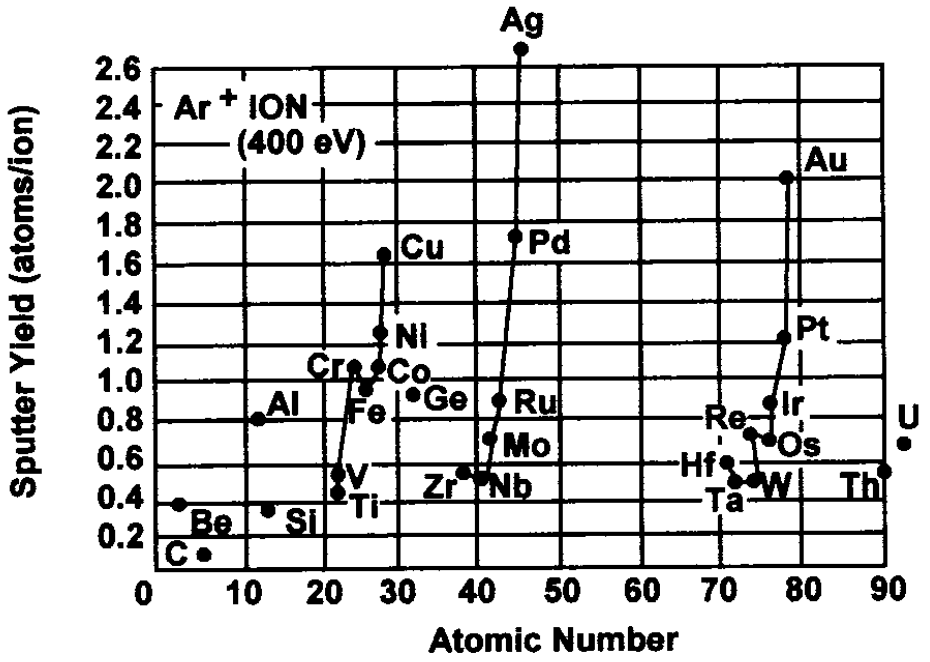

Op in mass (Ne to Ar) -> Sputter yield increases

Morphology of films
vs. energy of bombarding ions

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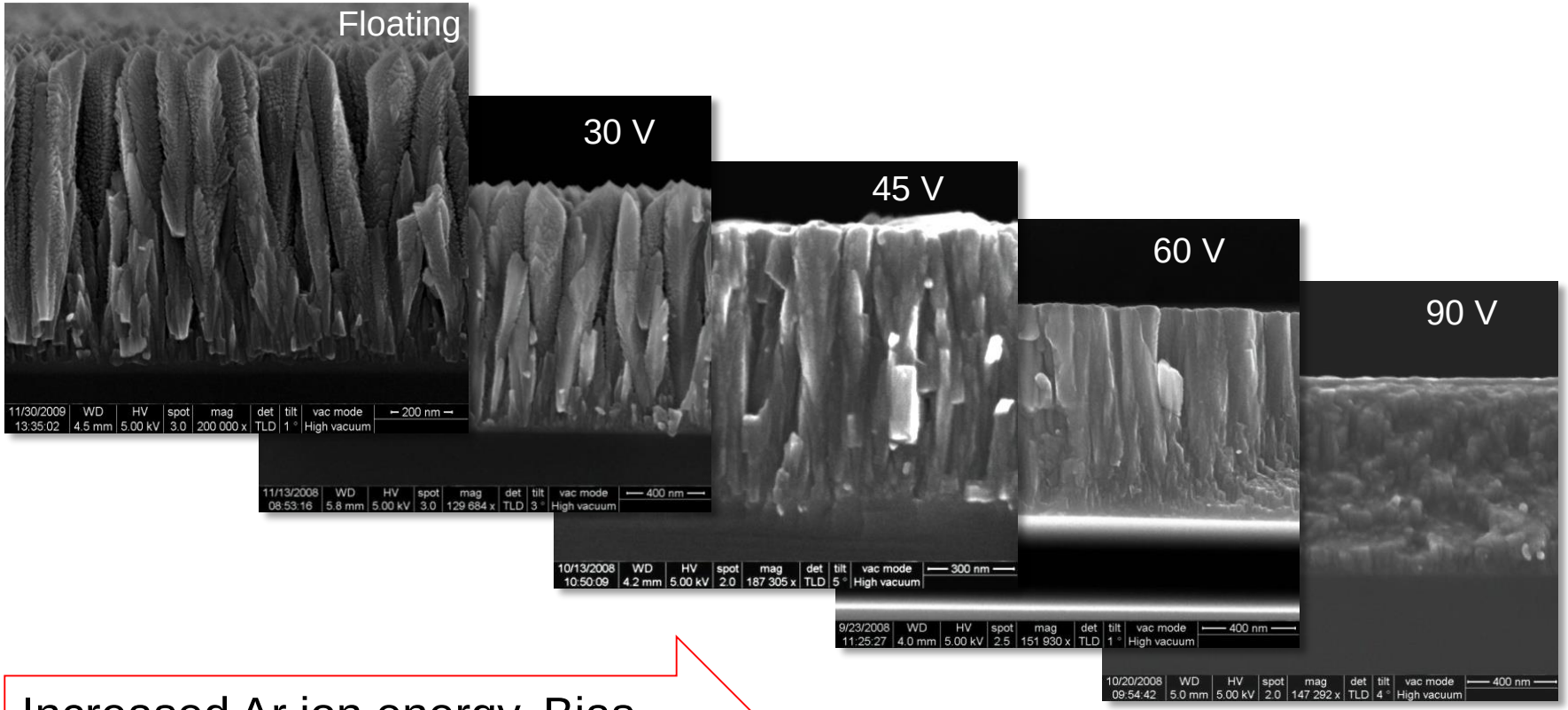

Increased Ar ion energy, Bias

TiO_2 morphology

5 x PVD units

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- 4×Targets:
- DC sputtering
- Bias: DC or RF supply and samples
- 6 towers

- 4×Targets:
- DC or pulsed-DC
- Bias DC, MF or RF
- 6 towers

- 6×Targets:
- DC or pulsed-DC
- Bias: DC, MF or RF
- HiPIMS
- Bias: HiPIMS
- 6 towers

- 4×Targets
- DC sputtering
- Bias: DC, MF
- 10 towers

5 x PVD

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- 4×Targets:
- DC sputtering
- Bias: DC or RF and samples
- 6 towers

- 4×Targets
- DC sputtering
- Bias: DC, MF
- 10 towers

Vacuum Chamber

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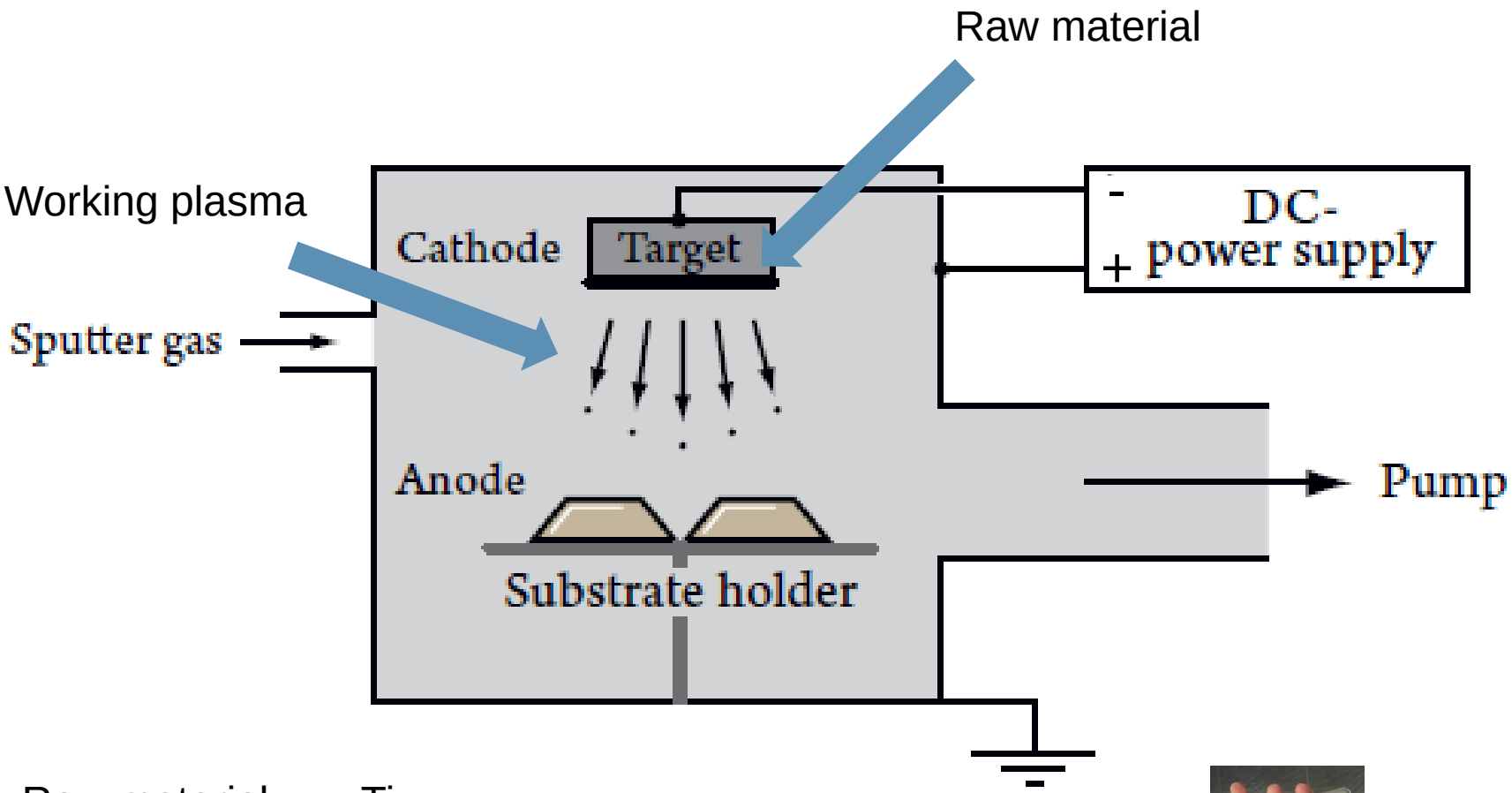

Raw material: Ti
Working plasma: Ar

Coating Ti metal

Raw material: Ti
Working plasma: Ar/N_2

Coating TiN ceramic nitride

Vacuum Chamber

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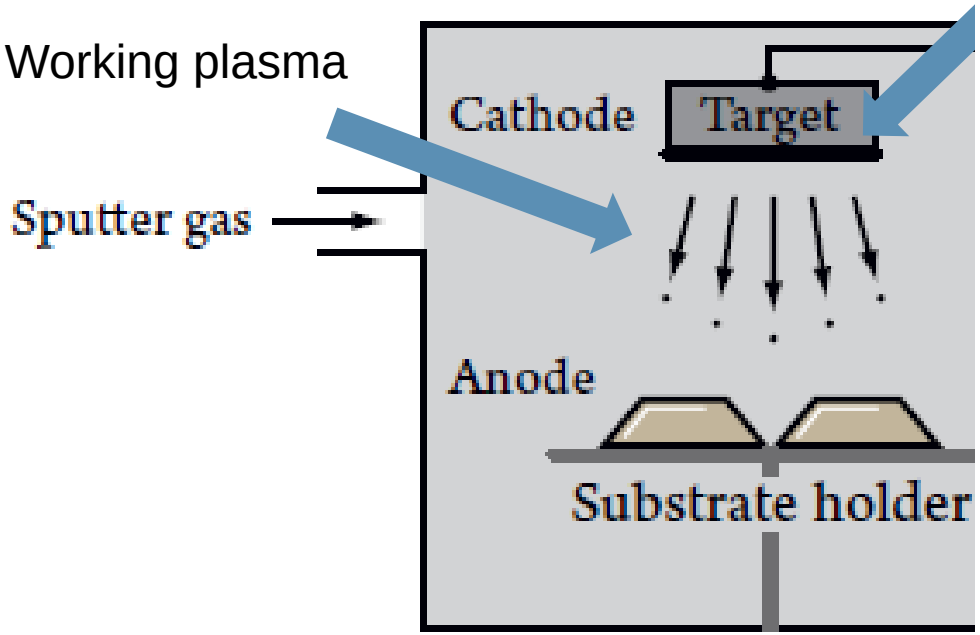
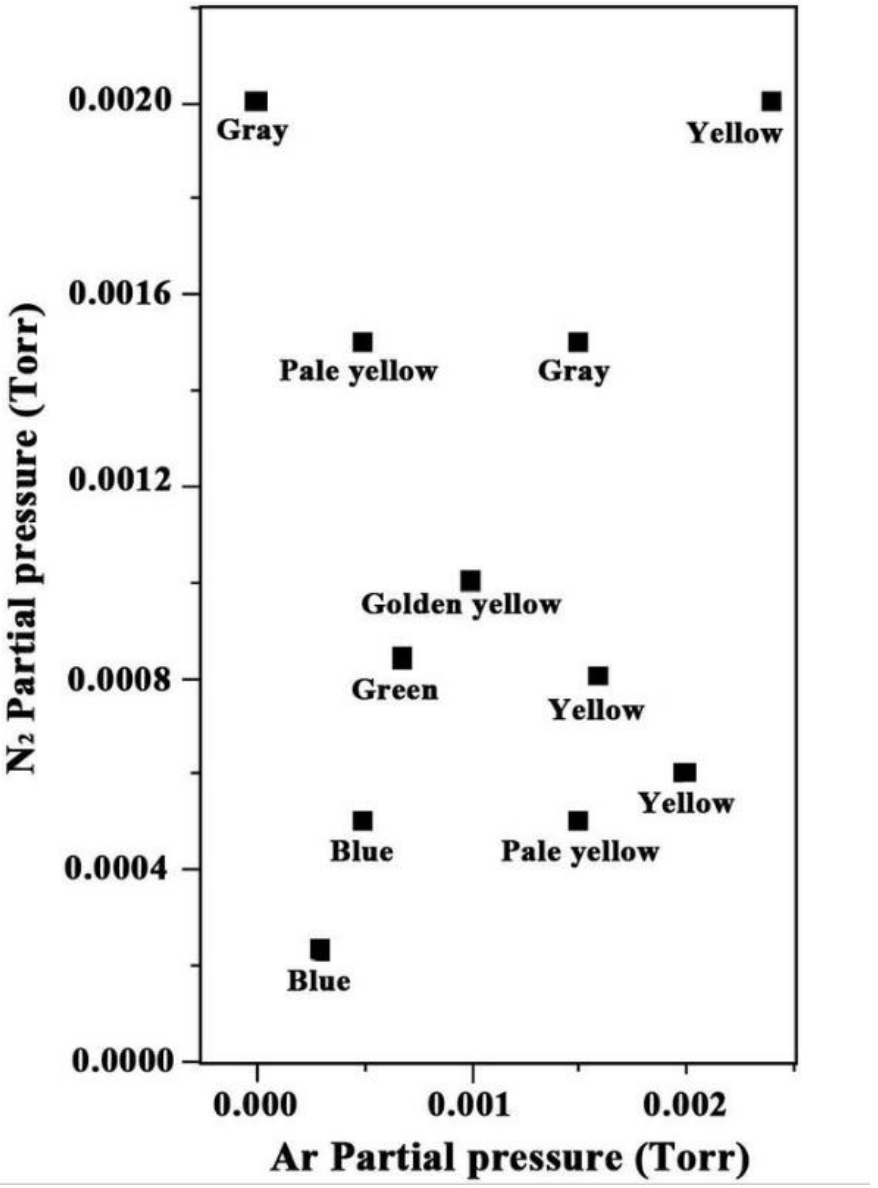


Raw material: Ti
Working plasma: Ar

Raw material: Ti
Working plasma: Ar/N_2



Sputter TiAlN

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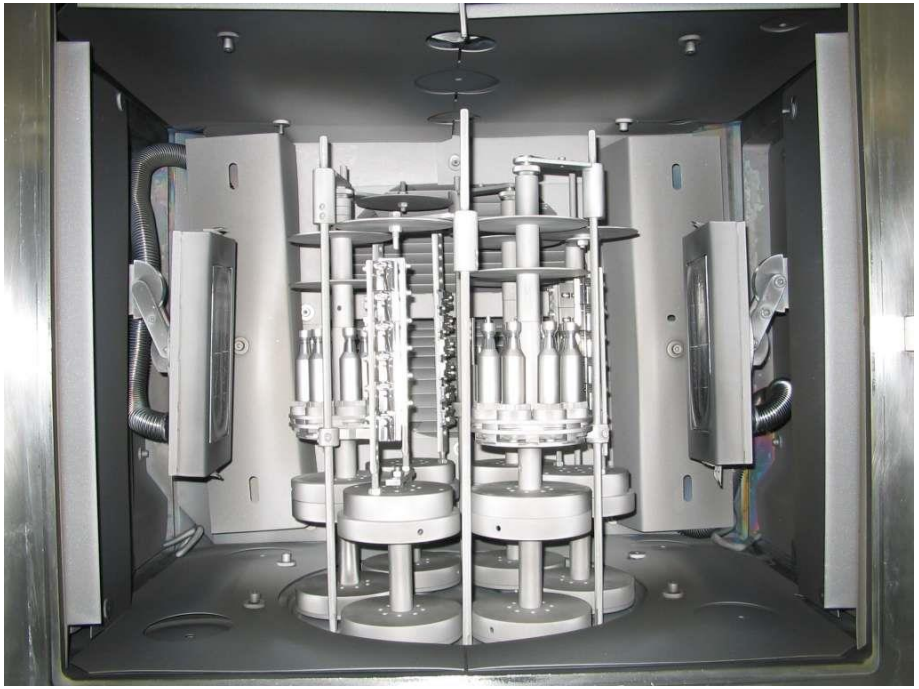

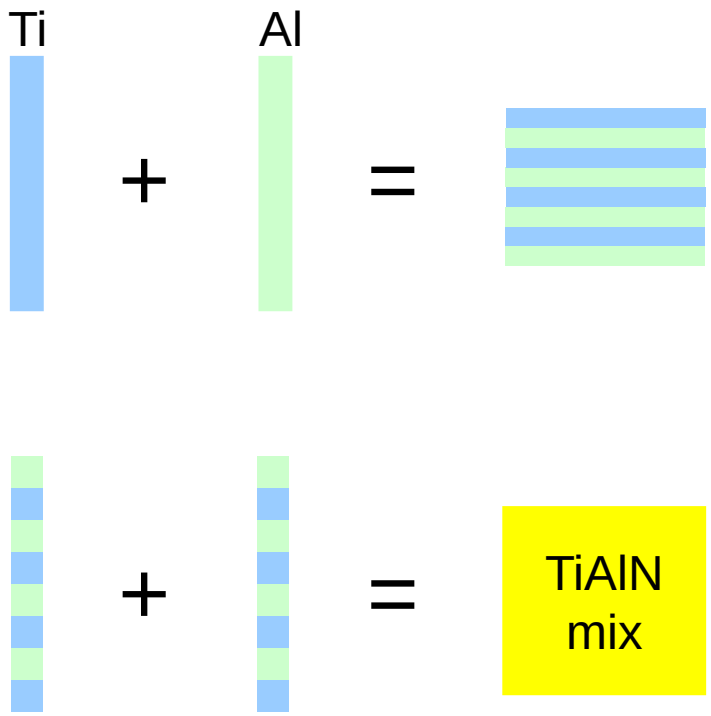

TiN – this is an old workhorse

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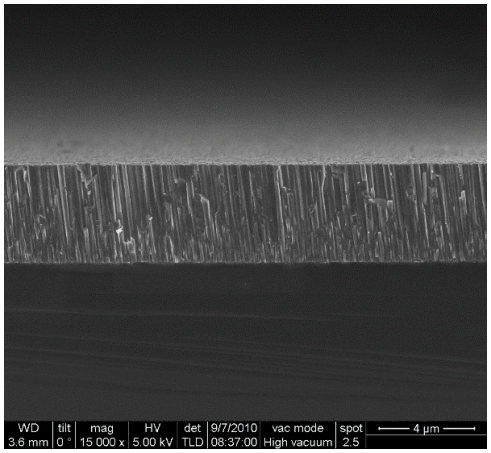

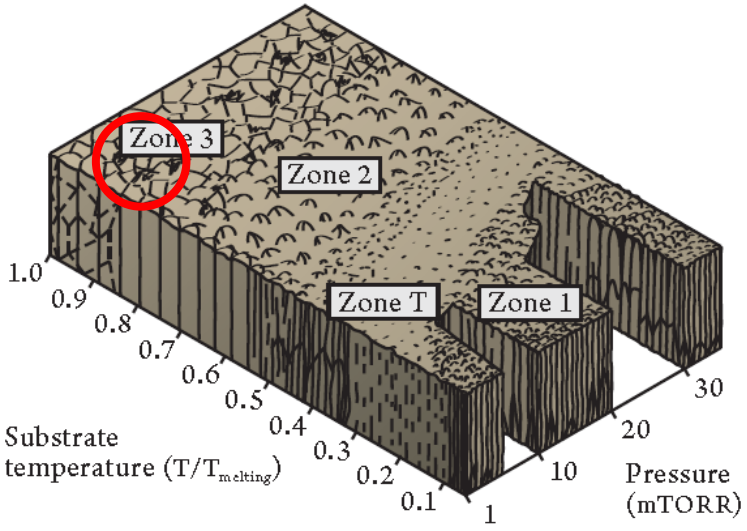

Zone 1	Coarse columnar growth morphology characterized by low mobility. This area is characterized by a high degree of intergranular porosity, which is linked to the low mobility. This region is not significantly affected by changes in the process pressure, since the sum of the kinetic energy (Maxwell distribution linked to the acceleration voltage) and the thermal energy (temperature) is simply too low to enable sufficient mobility.
Zone T	The T-zone is an intermediate region where the obtained morphology is dependent on the sputtering pressure, which in turn has a major impact on the obtained morphology. By lowering the pressure the ion energy increases and the morphology becomes denser.
Zone 2	Coarse columnar type growth and significantly more dense than Zone 1. Ceramic PVD-coatings are often of the Zone 2 type, when the substrate temperature is insufficient to produce Zone 3 morphology.
Zone 3	Equi-axial grown crystals. This area is characterized by the fact that the thermal energy is high enough to approach equilibrium growth independent of the pressure. Nucleation and growth is progressing according to minimization of Gibbs free energy, i.e. preferring low surface energy.

TiN; an old workhorse for tools

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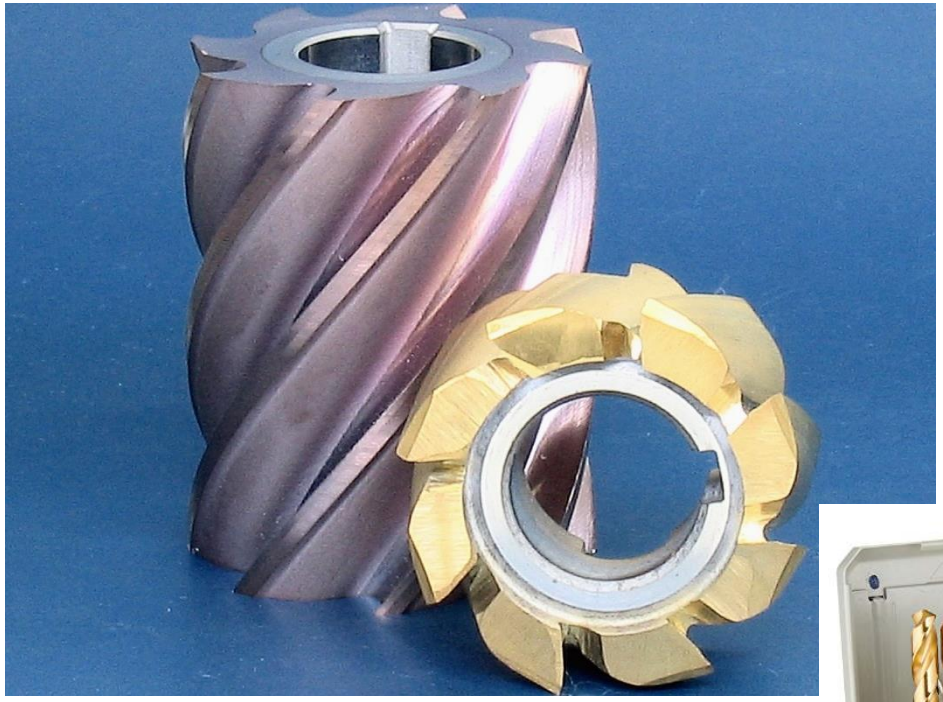

TiN – this is an old workhorse

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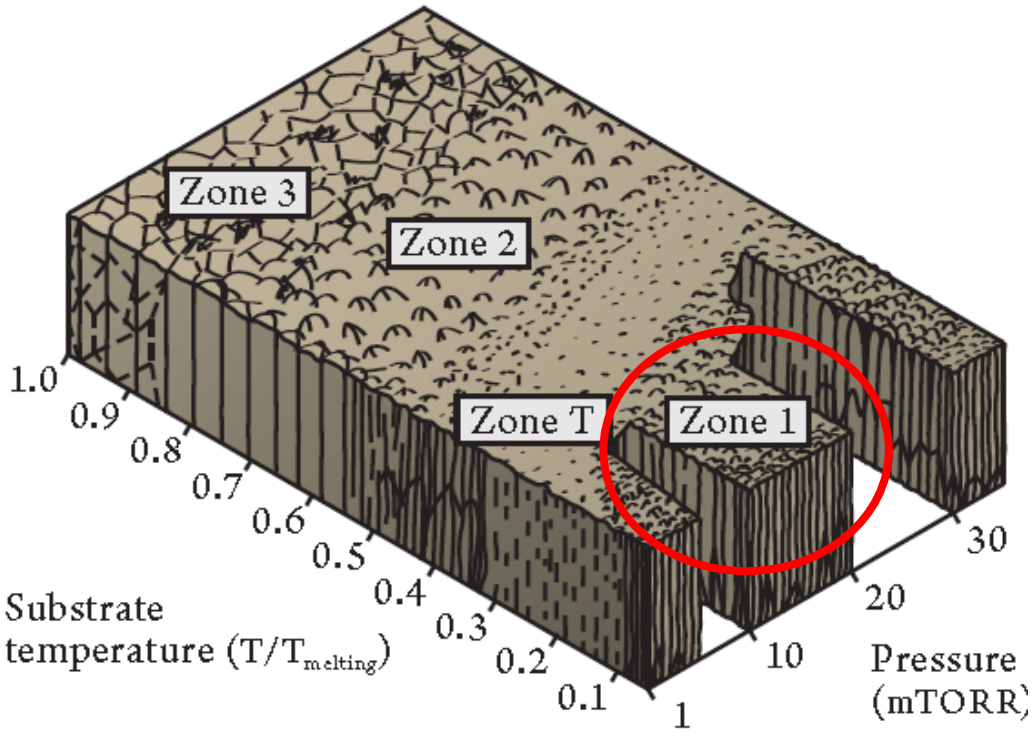
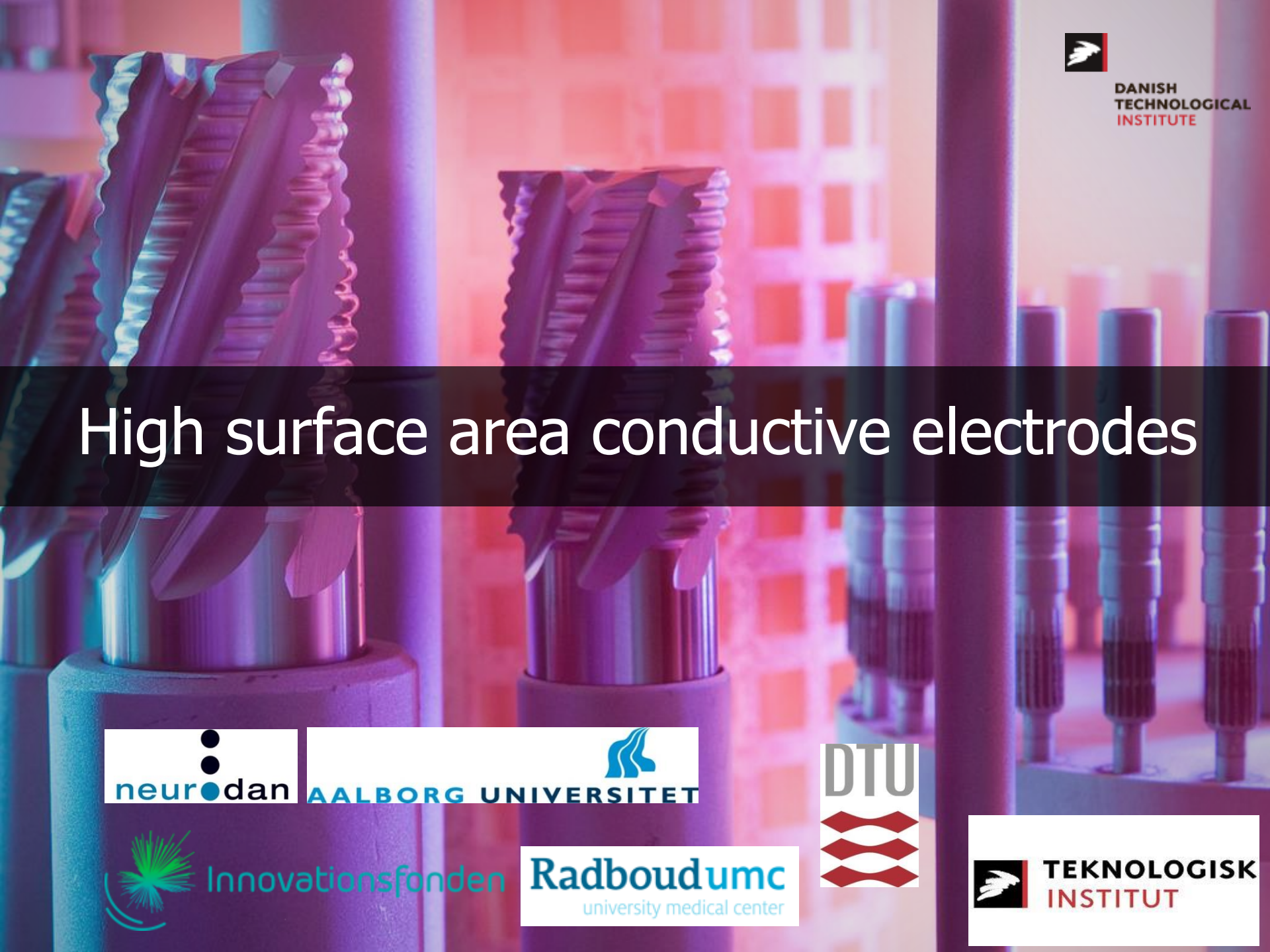


Zone 1	Coarse columnar growth morphology characterized by low mobility. This area is characterized by a high degree of intergranular porosity, which is linked to the low mobility. This region is not significantly affected by changes in the process pressure, since the sum of the kinetic energy (Maxwell distribution linked to the acceleration voltage) and the thermal energy (temperature) is simply too low to enable sufficient mobility.
Zone T	The T-zone is an intermediate region where the obtained morphology is dependent on the sputtering pressure, which in turn has a major impact on the obtained morphology. By lowering the pressure the ion energy increases and the morphology becomes denser.
Zone 2	Coarse columnar type growth and significantly more dense than Zone 1. Ceramic PVD-coatings are often of the Zone 2 type, when the substrate temperature is insufficient to produce Zone 3 morphology.
Zone 3	Equi-axial grown crystals. This area is characterized by the fact that the thermal energy is high enough to approach equilibrium growth independent of the pressure. Nucleation and growth is progressing according to minimization of Gibbs free energy, i.e. preferring low surface energy.


DANISH
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High surface area conductive electrodes
neurodan
AALBORG UNIVERSITET
DTU
Innovationsfonden
Radboudumc
university medical center
TEKNOLOGISK
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High surface area – conductive electrodes

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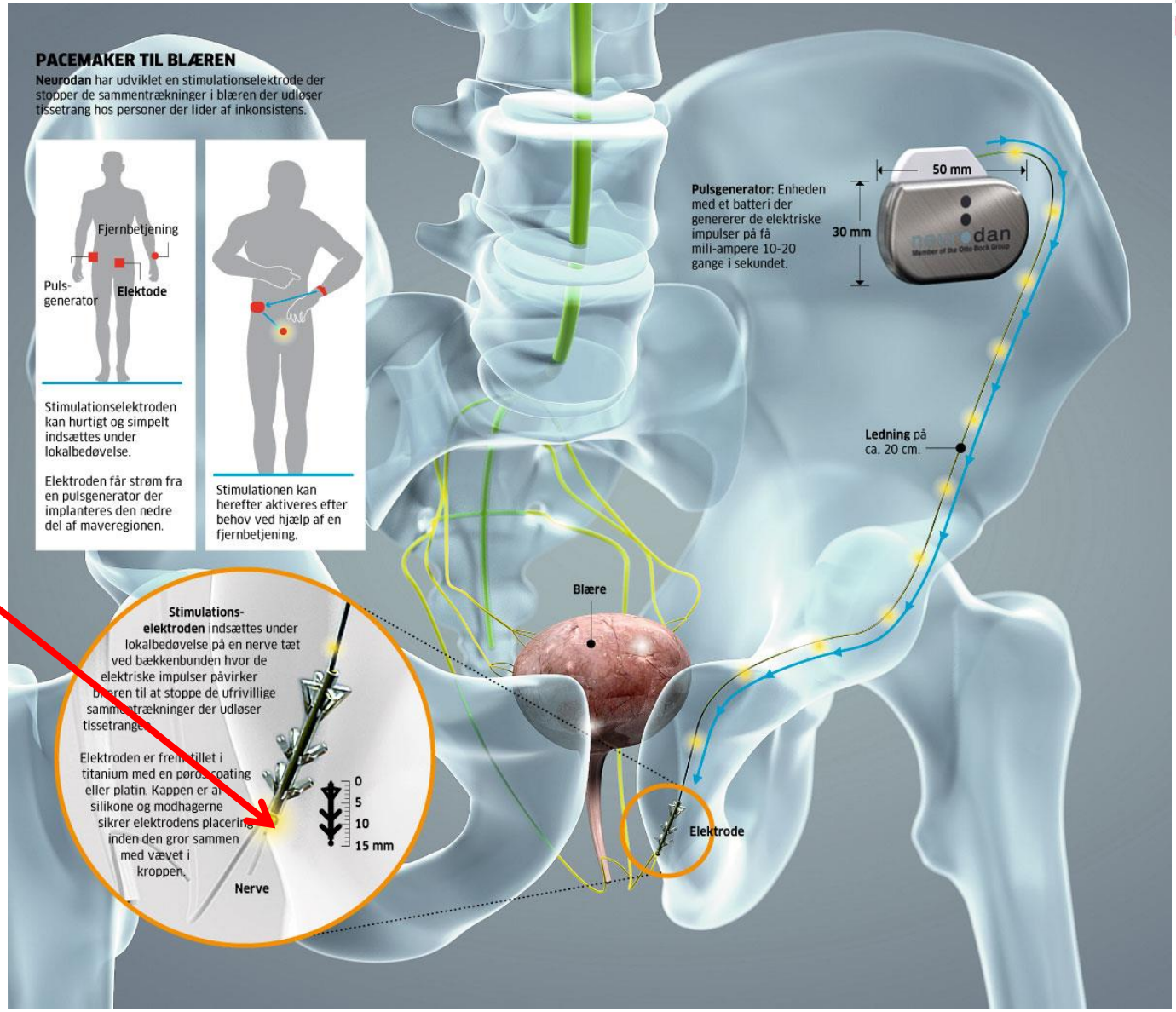

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High surface area – conductive electrodes

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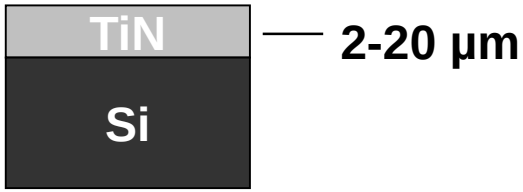
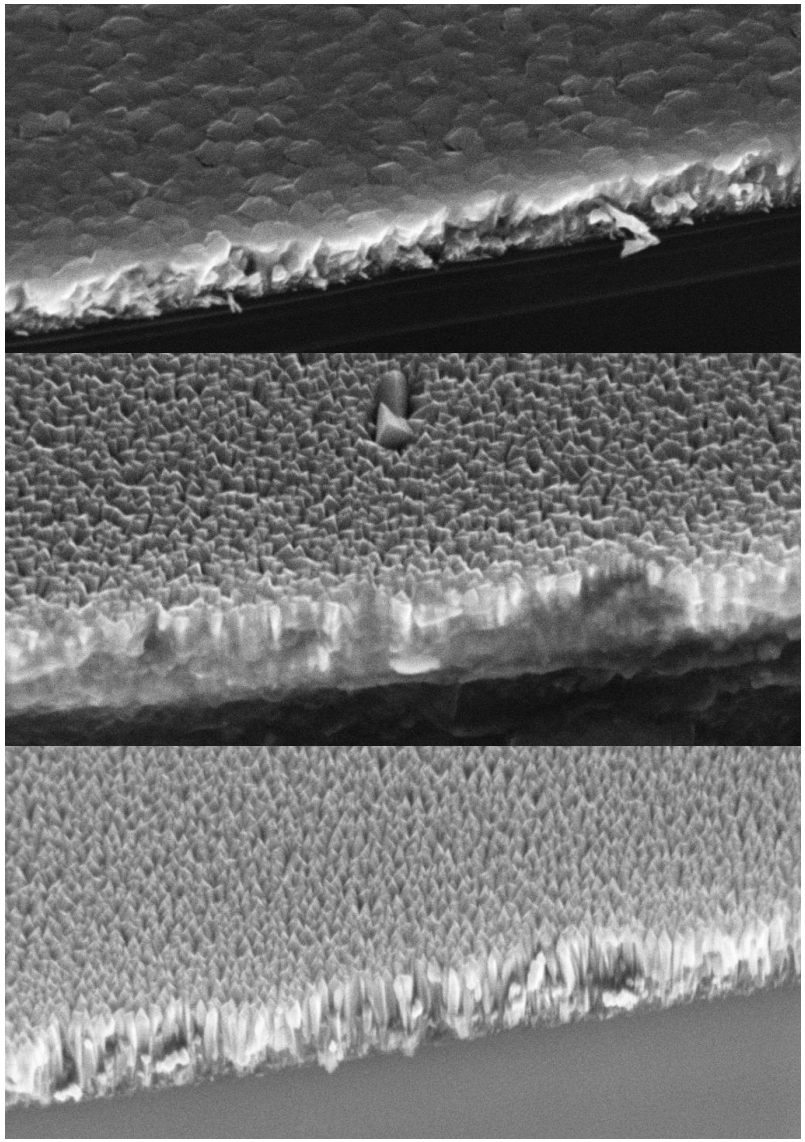


”Changing deposition parameters can have a large effect on the coating morphology”



neurodan
Member of the Otto Bock Group

High surface area – conductive electrodes

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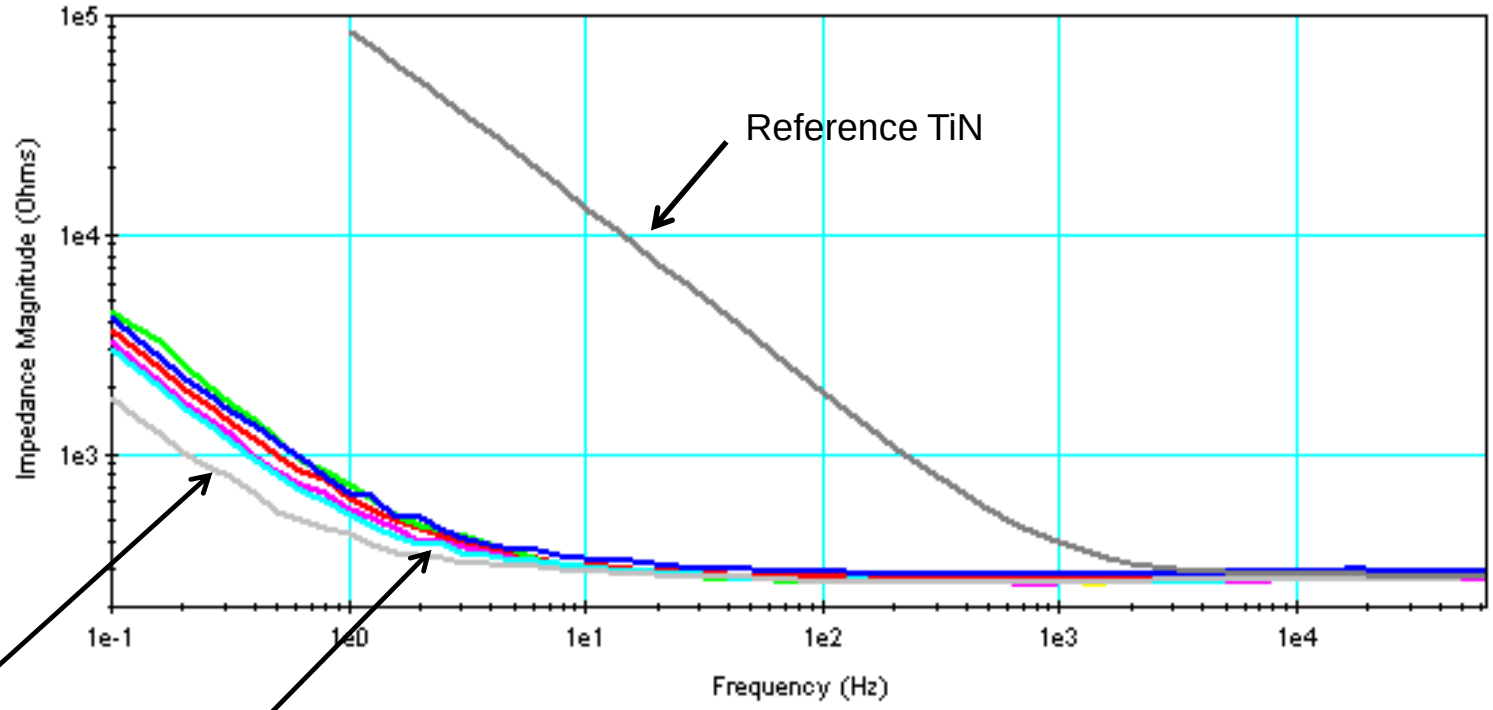

Highest N content

Thicker TiN coating (~x2)

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Member of the Otto Bock Group

High surface area – conductive electrodes

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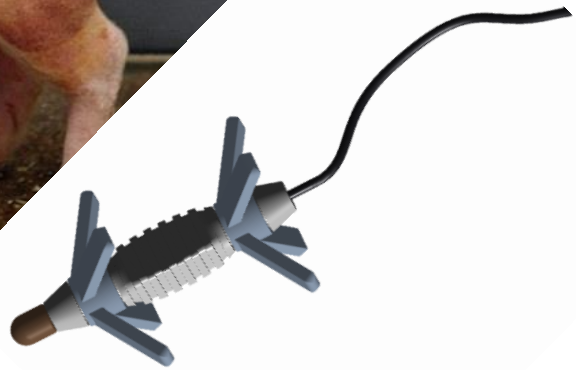

Göttingen minipig

High surface area – conductive electrodes

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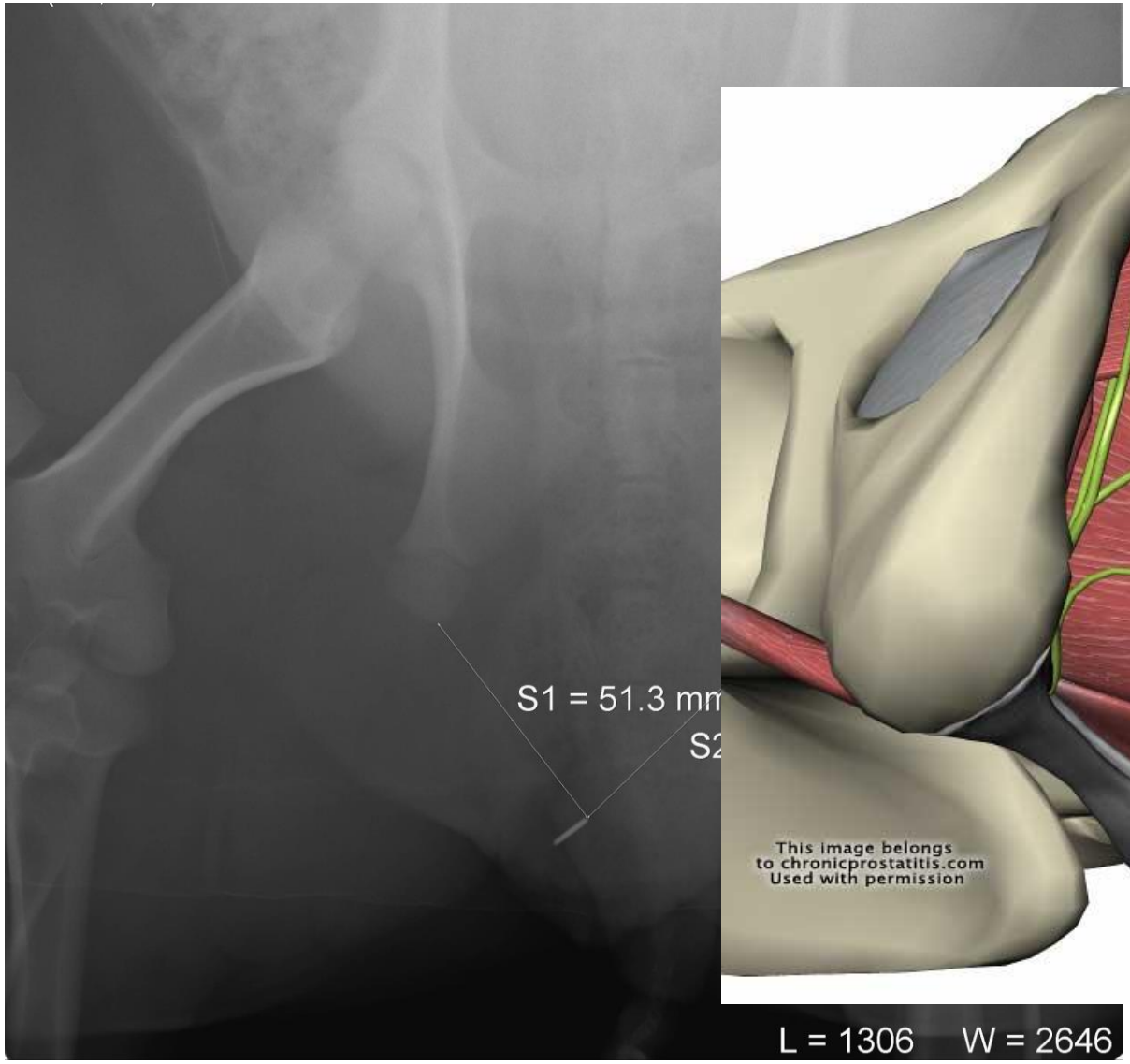

High surface area – conductive electrodes

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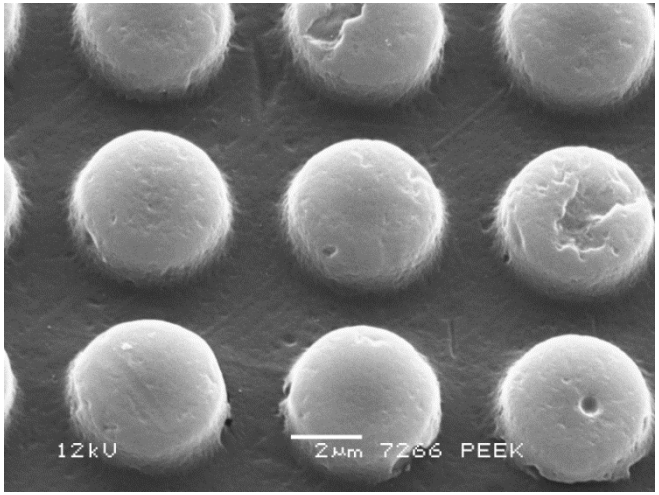
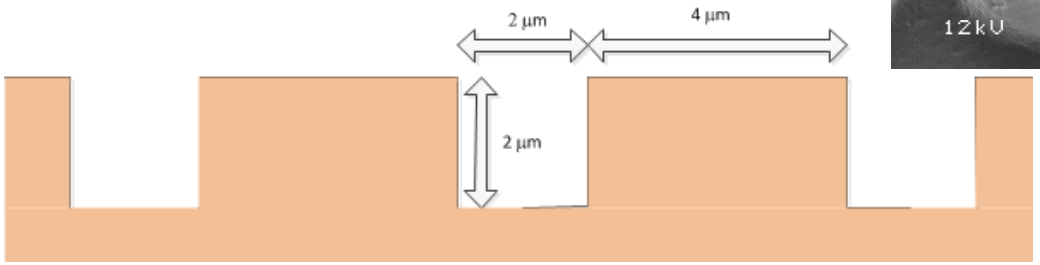
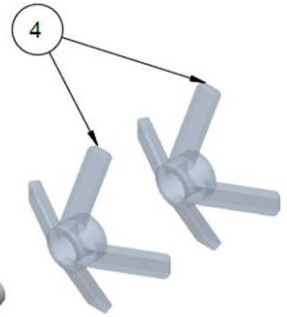
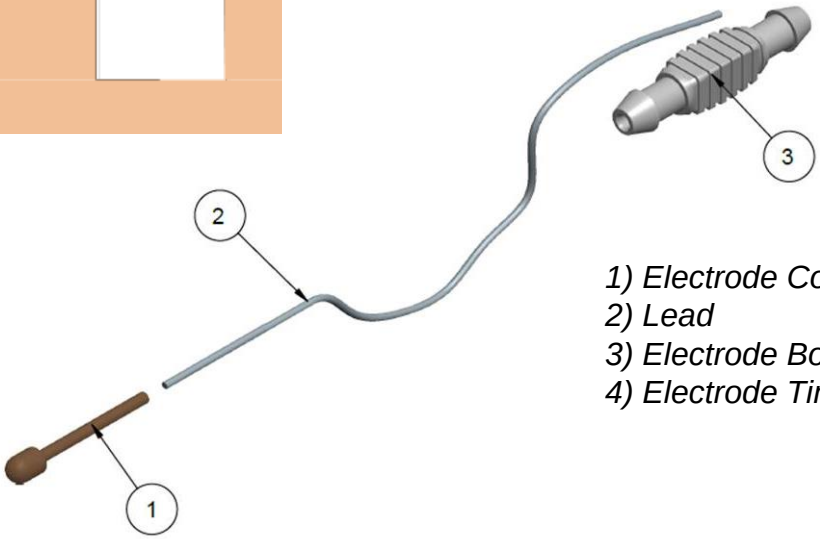
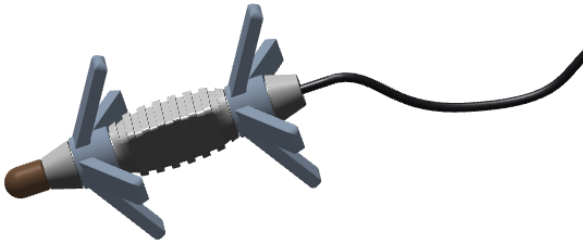
Microstructures on the PEEK body and the silicone tines to accelerate and improve tissue integration











1) Electrode Contact
2) Lead
3) Electrode Body
4) Electrode Tines.

High surface area – conductive electrodes

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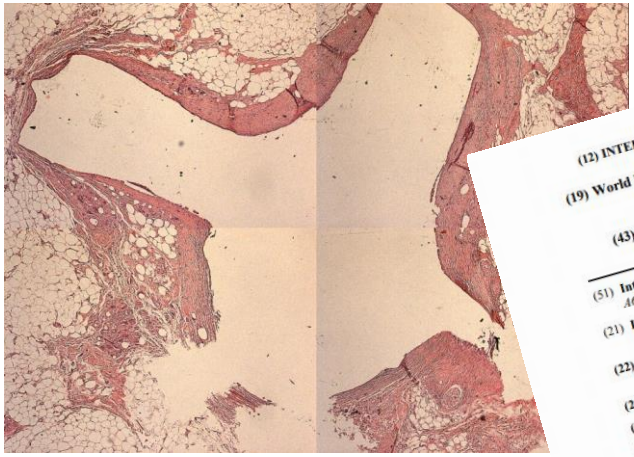

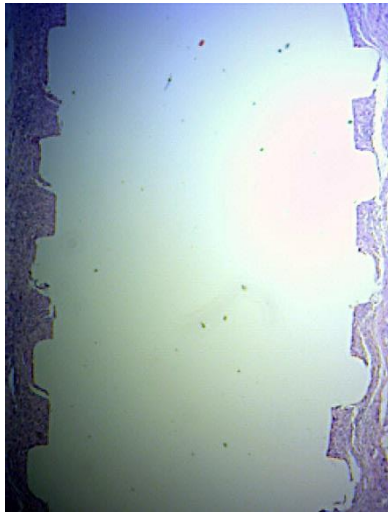

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[Continued on next page]

(54) Title: TREATMENT OF PELVIC DISORDERS BY ELECTRICAL NERVE STIMULATION

(57) Abstract: A system for the treatment of urge incontinence by electrical stimulation of pudendal nerve afferents (108; 110) of a living being (102) includes an implantable electrode (116) configured to be placed at a portion of a pudendal nerve or its branches, and a pulse generator (118) configured to provide a sequence of electrical pulses to the electrode (116) in order to achieve the electrical stimulation. The pulse generator (118) is configured to enter an active mode when a control signal is provided, and to enter a dormant mode when the sequence of electrical pulses has been completed. A wake-up circuit causes the pulse generator (118) to exit the dormant mode and to enter the active mode. A power source of the system is configured such that a voltage is maintained at the wake-up circuit in the dormant mode of the pulse generator (118), and no voltage is maintained at the pulse generator in the dormant mode.


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Low-friction coatings for dental applications

Low frinction DLC to dental applicaiton

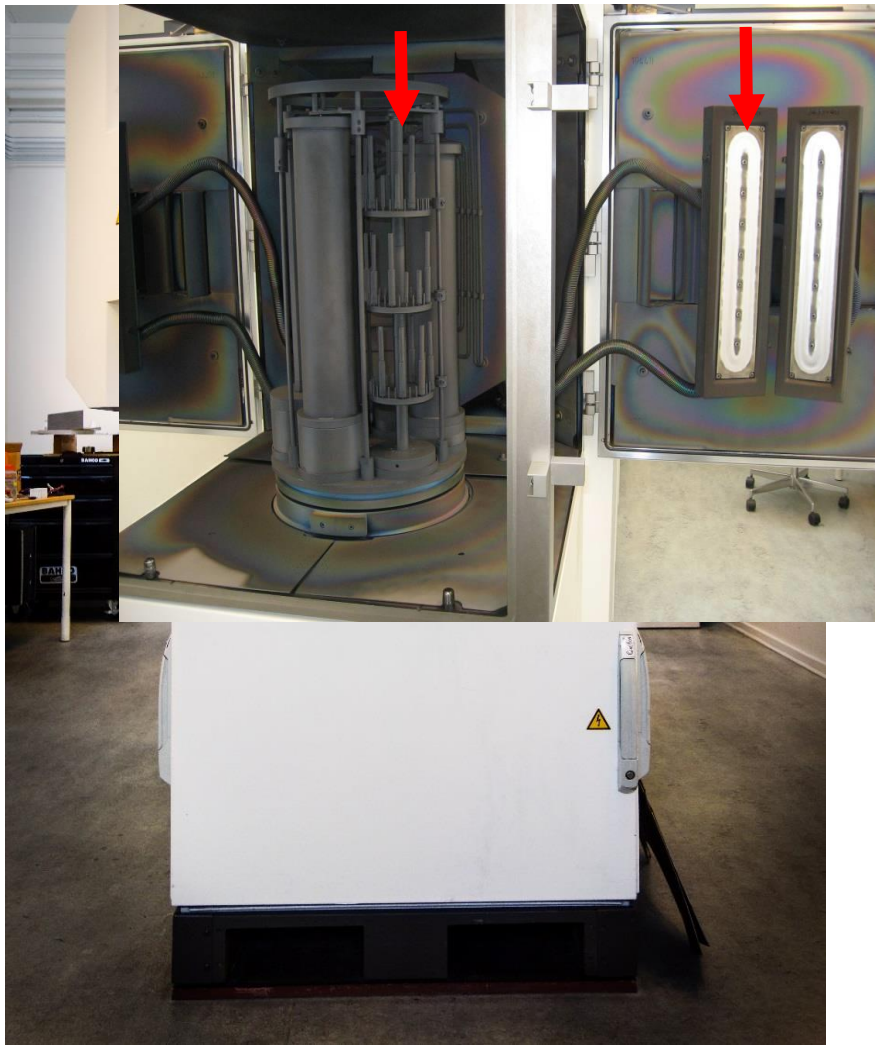
TEKNOLOGISK

Reactive DC magnetron sputtering

Deposition parameters:

- Cr to make CrN adhesion layer
- C to make a low-friction top layer

- Ar/N_2
- Deposition temperature ~180-200 °C
- Thickness ~ 3 µm incl. adhesion layer



Low frinction DLC to dental applicaiton

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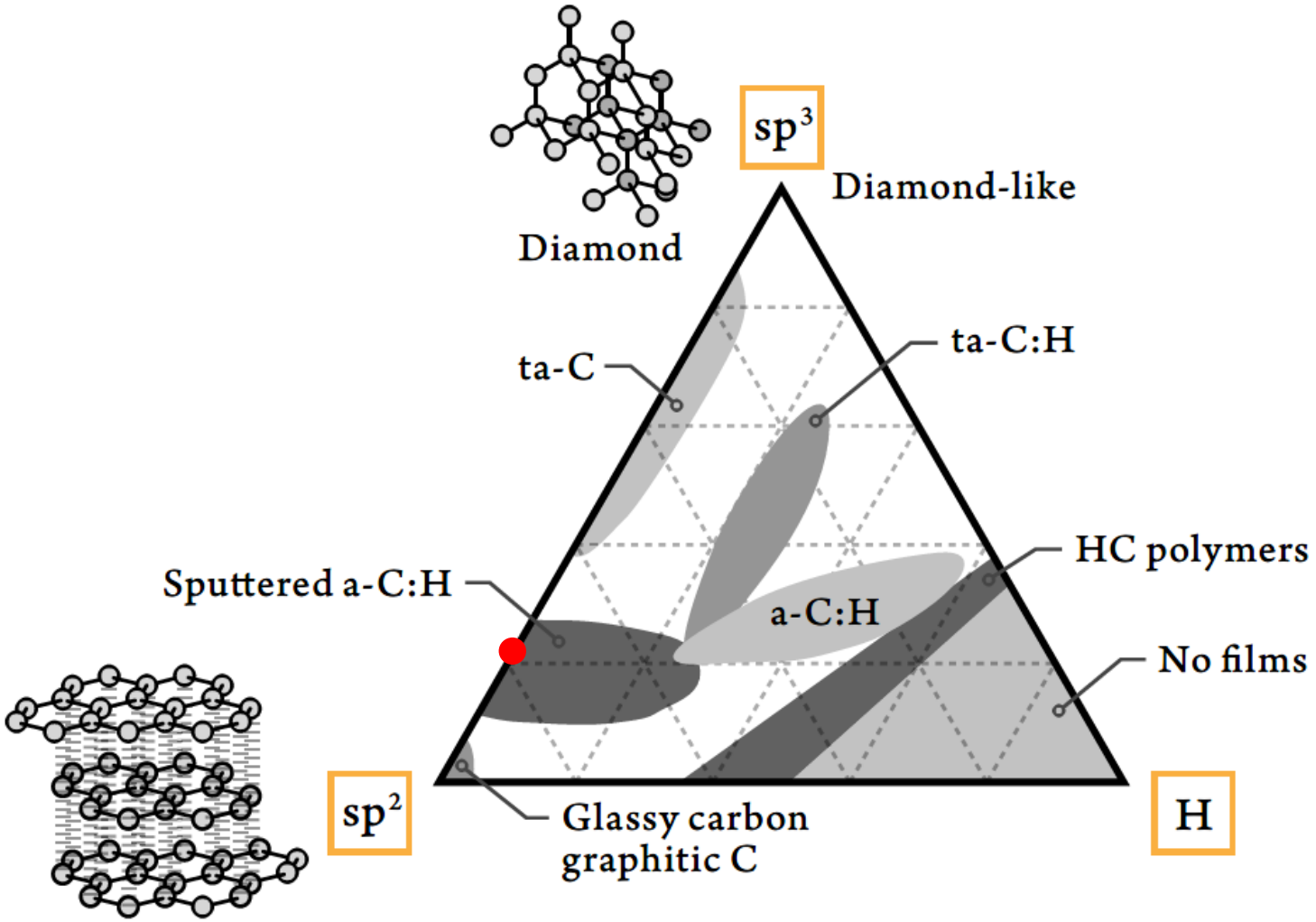

Robertson 2002

Dental implants

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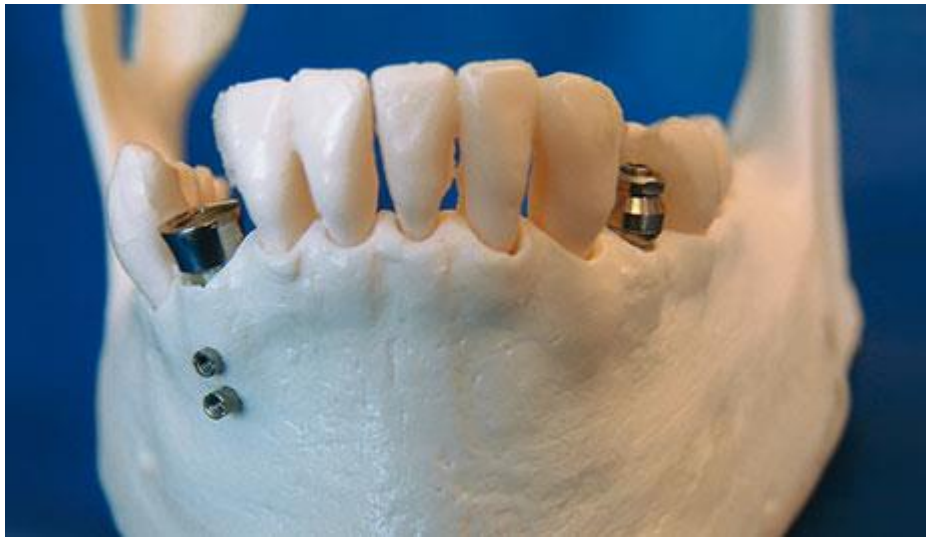
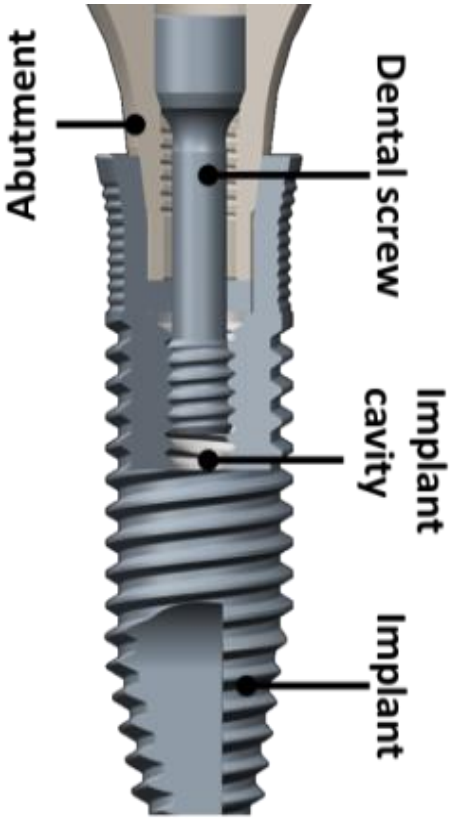
~10 million patients per year



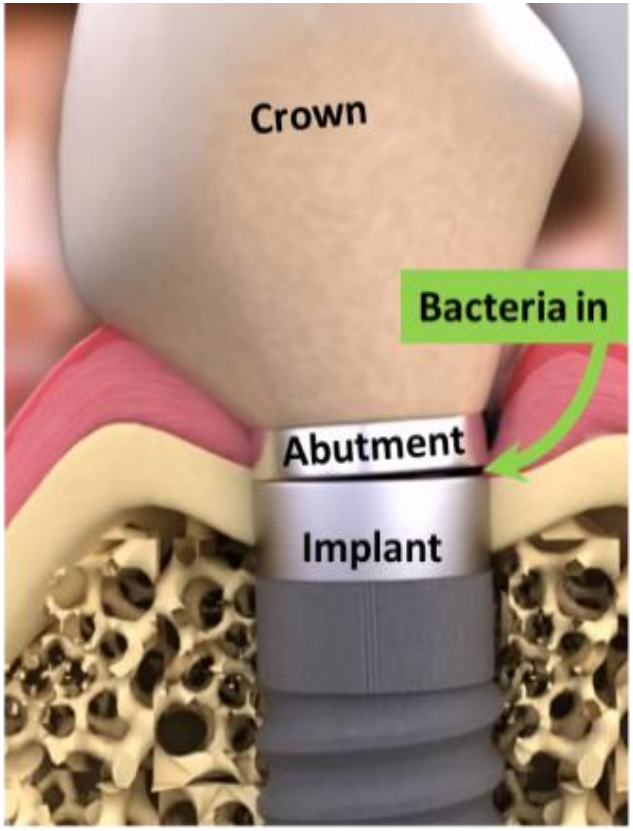


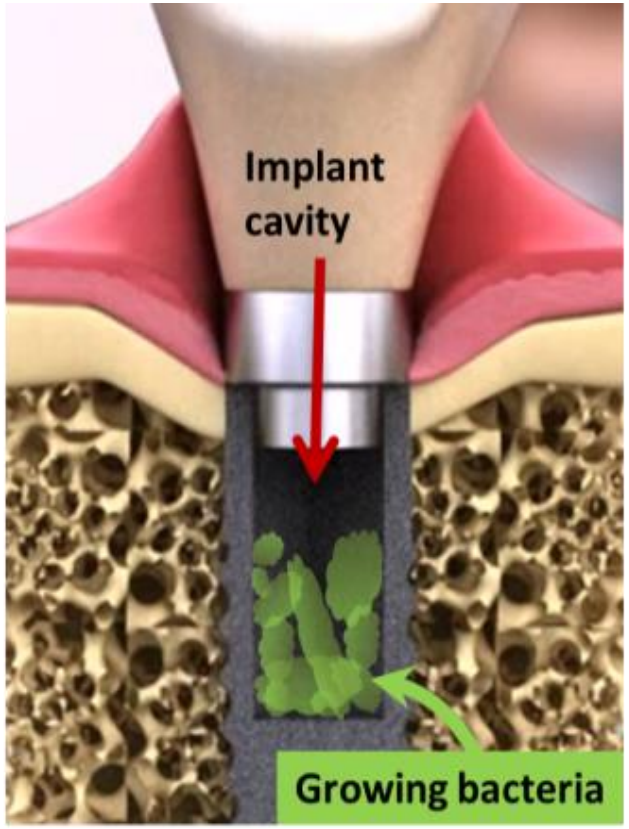
Dental implants

TEKNOLOGISK INSTITUT

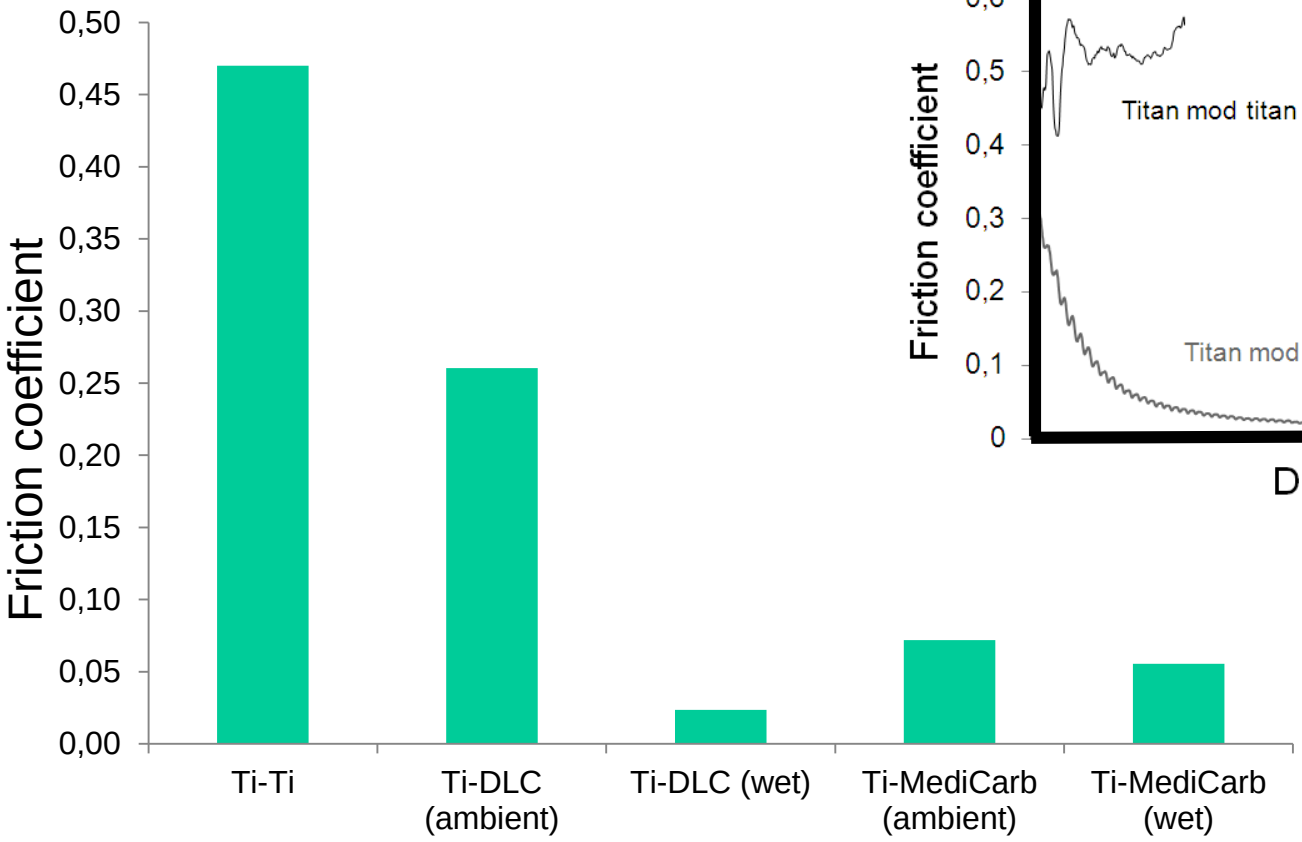
~10 million patients per year

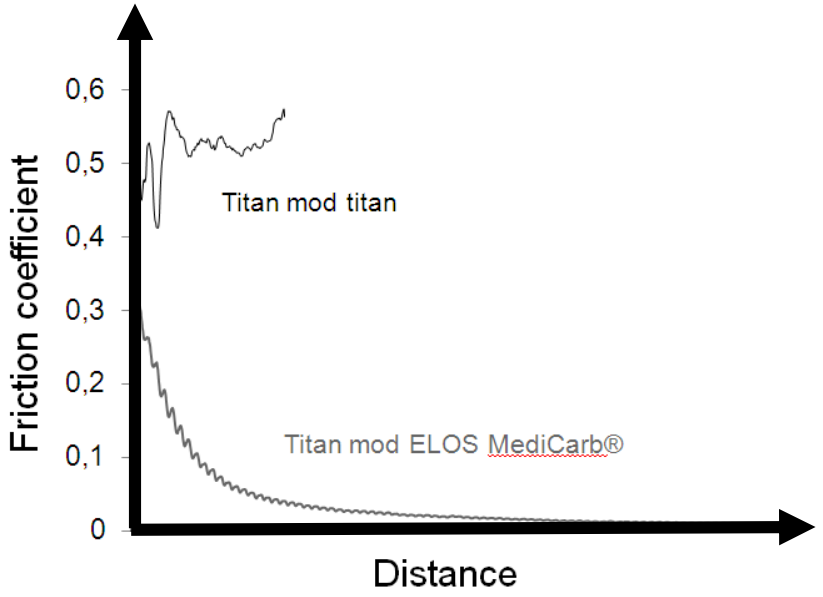
~10 % experience issues with peri-implantitis (bacteria,





- Bad mouth smell





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ELOS MEDTECH

Danmarks Tekniske Universitet DTU

Titanium against titanium is very bad

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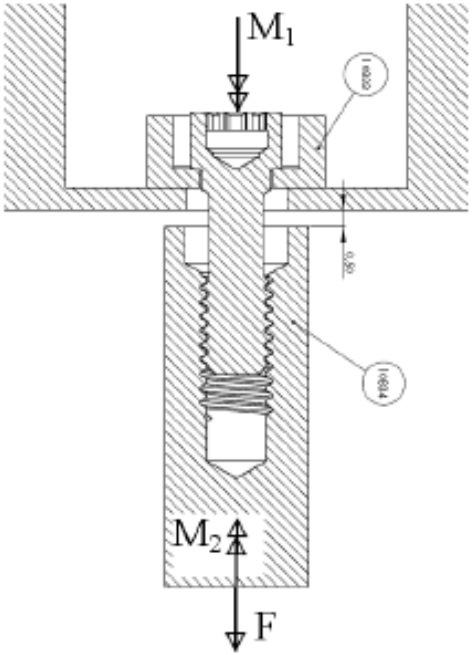
Applied a fixed Torque [M_1]



F should be as high as possible for fixed Torque [M_1]

Coatings

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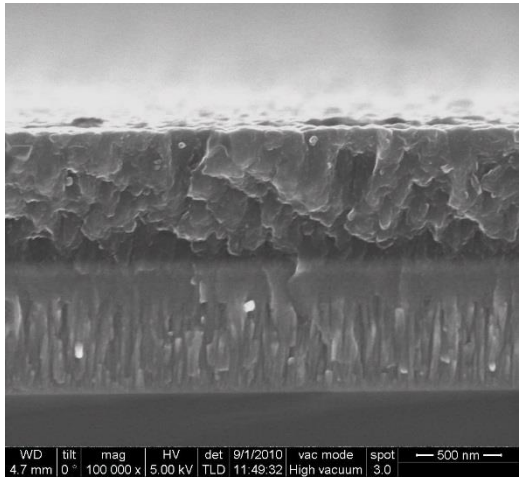
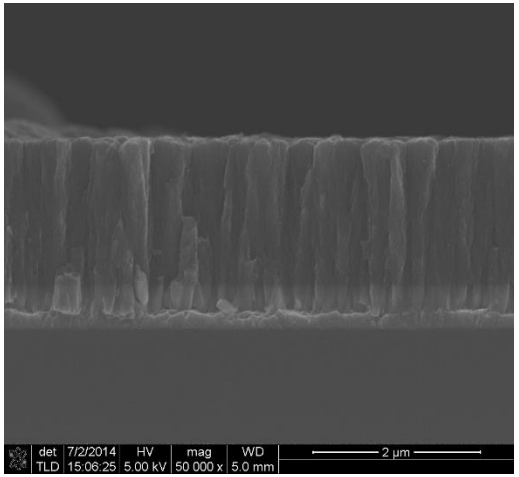
a-C toplayer
CrN adhesion
Titanium substrate



a-C toplayer
Ti adhesion
Titanium substrate



Hydrogen free – no hydrogen embrittlement

Coatings

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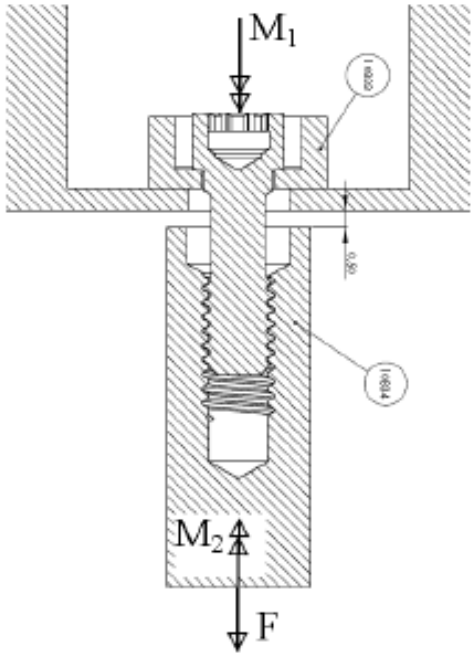
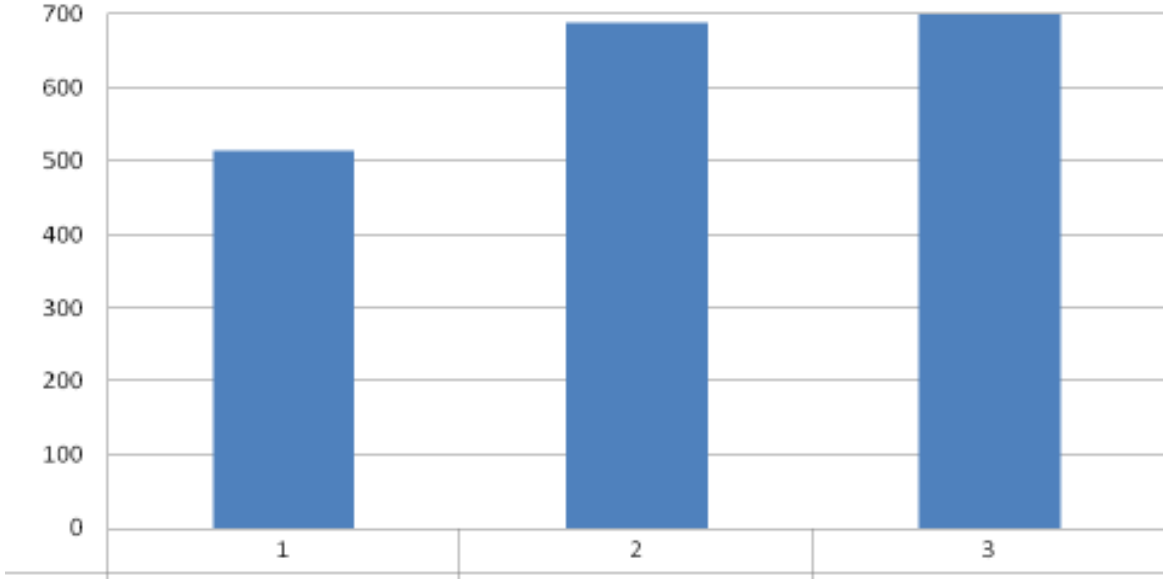
Applied a fixed Torque [M_1]



F should be as high as possible for fixed Torque [M_1]

Average of; 1st, 2nd and third tightening



Tighting force – fixed momentum

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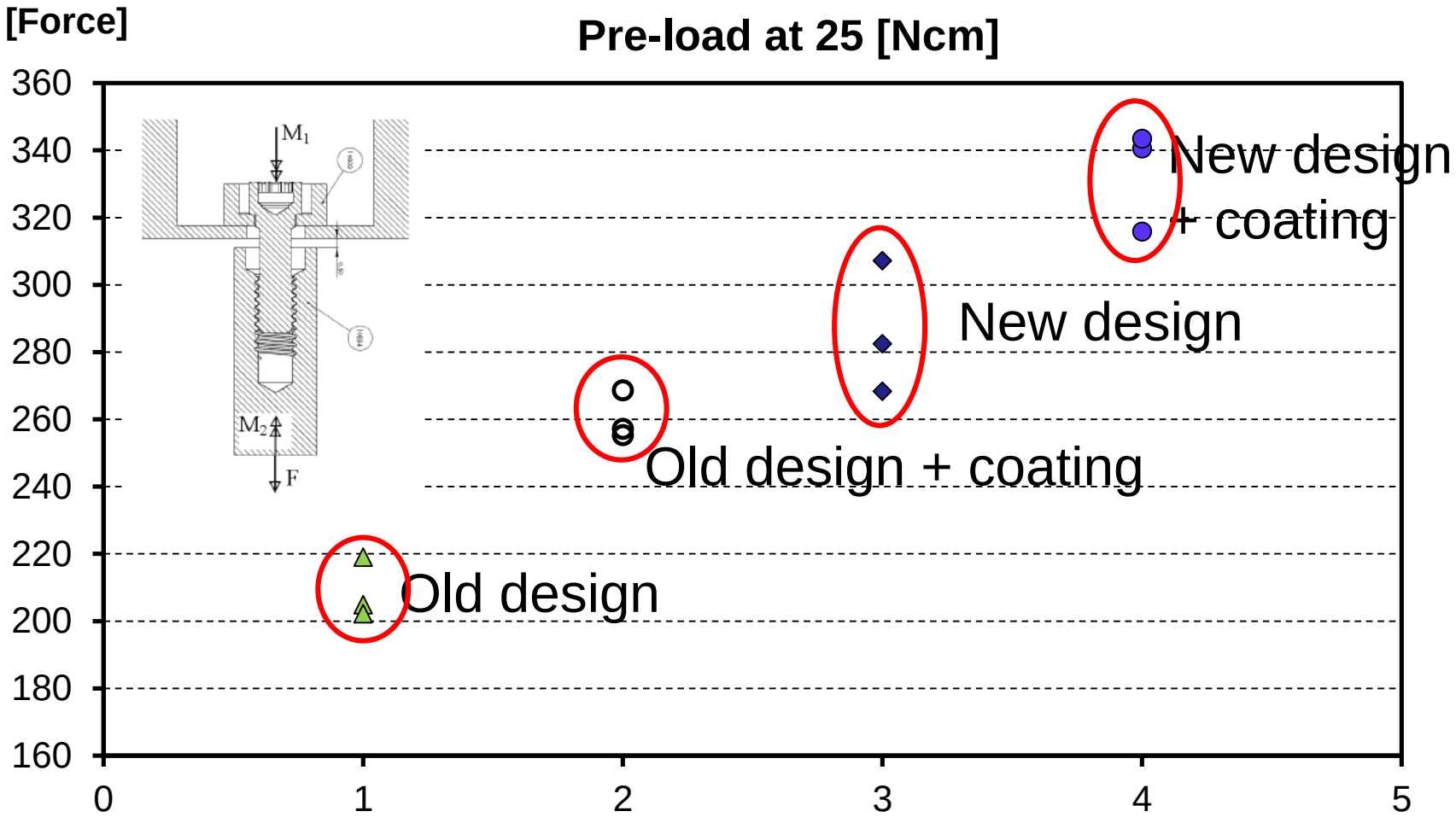

Low-friction drills

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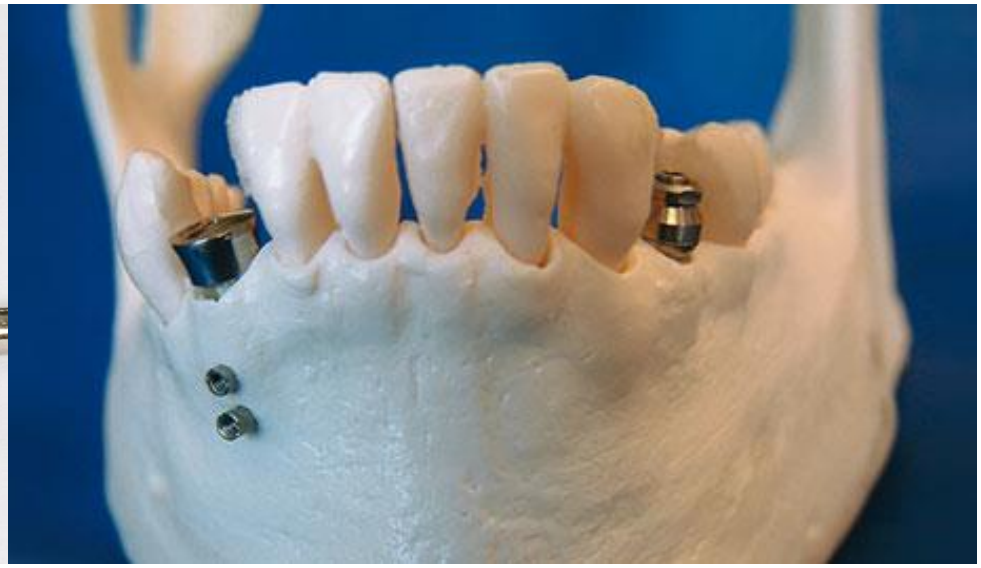

ELOS MEDTECH

Low-friction drills

TEKNOLOGISK INSTITUT

ELOS MEDTECH

DANISH TECHNOLOGICAL INSTITUTE

Sr-Ti coating for accelerated bone growth

The Danish National Advanced Technology Foundation

ELOS MEDTECH

MEDIZINISCHE UNIVERSITÄT INNSBRUCK

iNANO

Why Sr?

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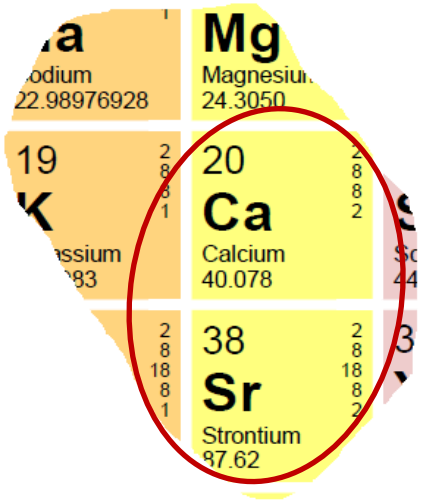
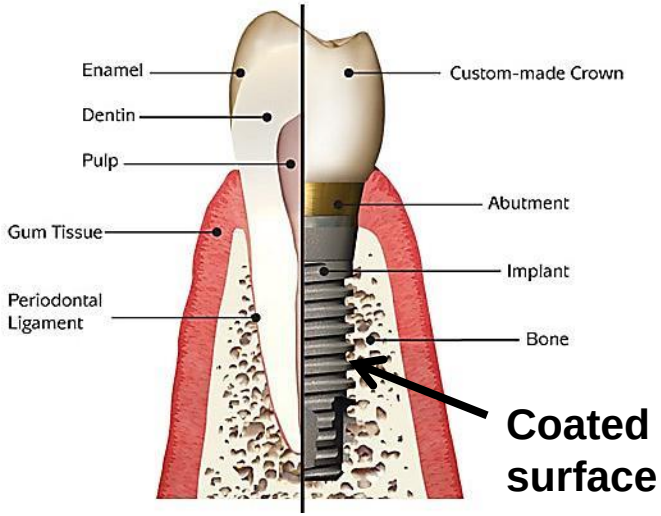
- Substitutes for Ca in mineral phase of bone
- Stimulates bone formation
- Reduces bone resorption
- Shown anti-inflammatoric effect
- Sr is used via strontium ranelate as treatment for osteoporosis
- Sr in the surface of an implant gives local effect where needed





Beneficial effects for accelerated and increased implant osseointegration. General healing times are 2-4 month before placement of the prostheses

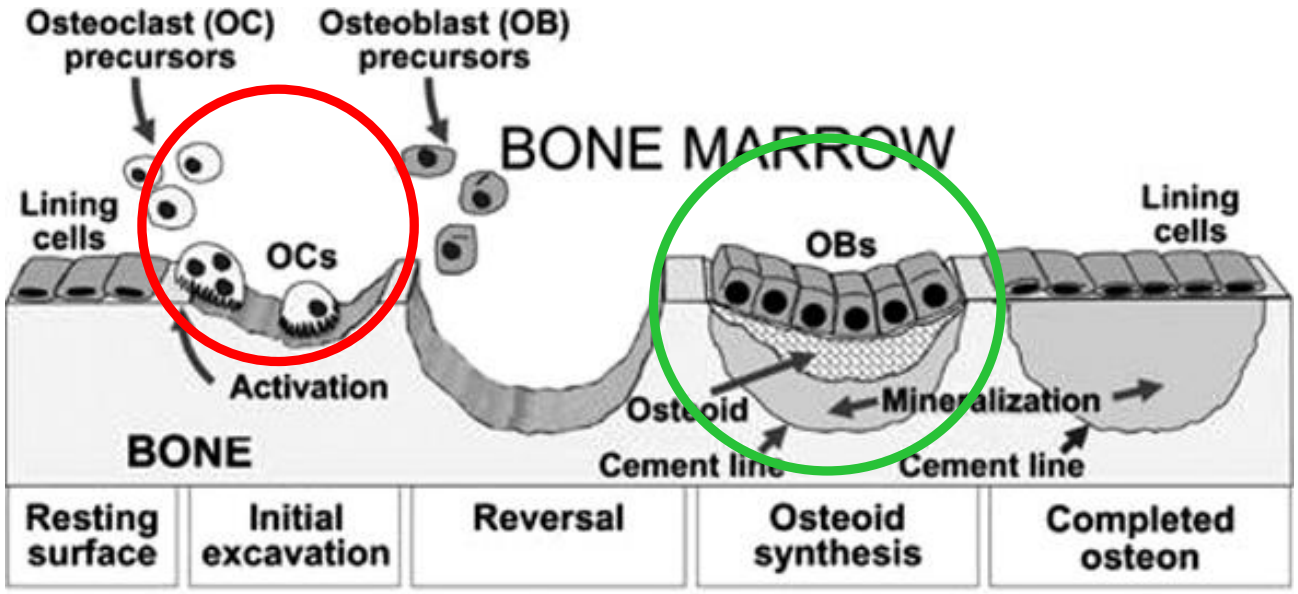
Park, J.-W. et al.; *Acta Biomaterialia* (2010) **6(7)**; pp. 2843.
Roy, M. M. et al.; *J. Biomed. Mat. Res. Part A* (2012) **100(9)**; pp. 2450.
Xin, Y. et al.; *ACS Nano* (2009) **3(10)**; pp. 3228.

The dynamic structure of bone

DANISH TECHNOLOGICAL INSTITUTE

Constant remodeling – ”new” skeleton every 10 years

Sr → ”negative” effect on OC - ”positive” effect on OB



Riggs and Parfitt;
J. of Bone and Mineral Research, 2005

A sufficient amount of Sr must be released from the implant surface to stimulate the formation of bone around the implant

No delamination of particles from the surface can be accepted

Coating synthesis

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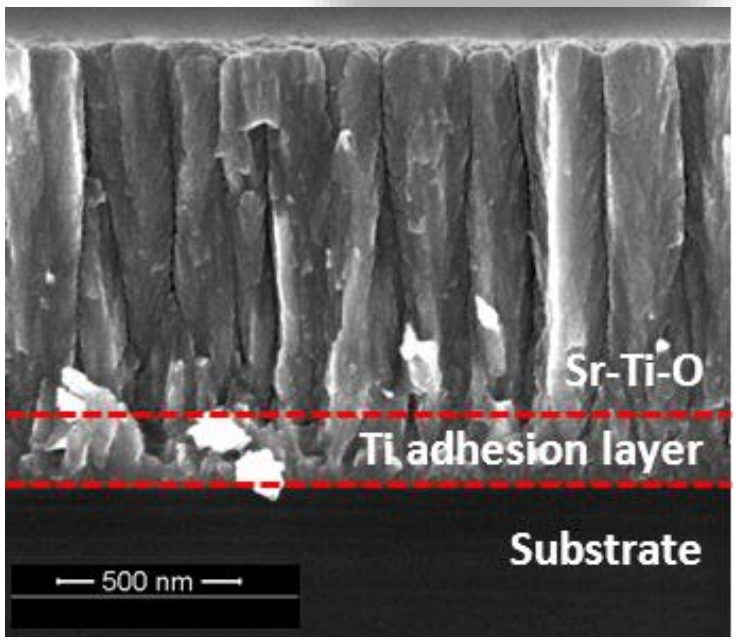
- DC magnetron sputtering
- Industrial coating unit (CC 800/9 SinOx)
- Deposition parameters
  - Co-sputtering of Ti target (Grade 1) and Ti-Sr-O target (99.9%), 88mm x 500mm
  - Sr content 0 - 10 at.%
  - Argon sputtering gas
  - Deposition temperature ~ 100°C
  - Pressure ~ 0.4 – 2.4 Pa
  - Deposition rate ~ 240 nm/hr
  - Substrates (rotating): Silicon wafer, titanium implants, steel, …



Coating characterization

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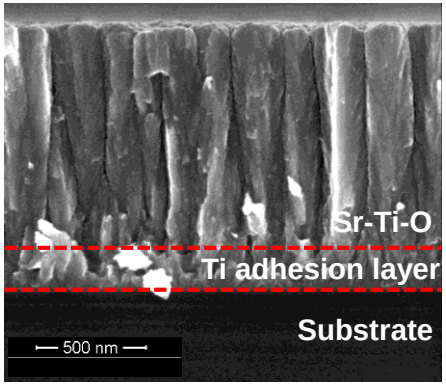
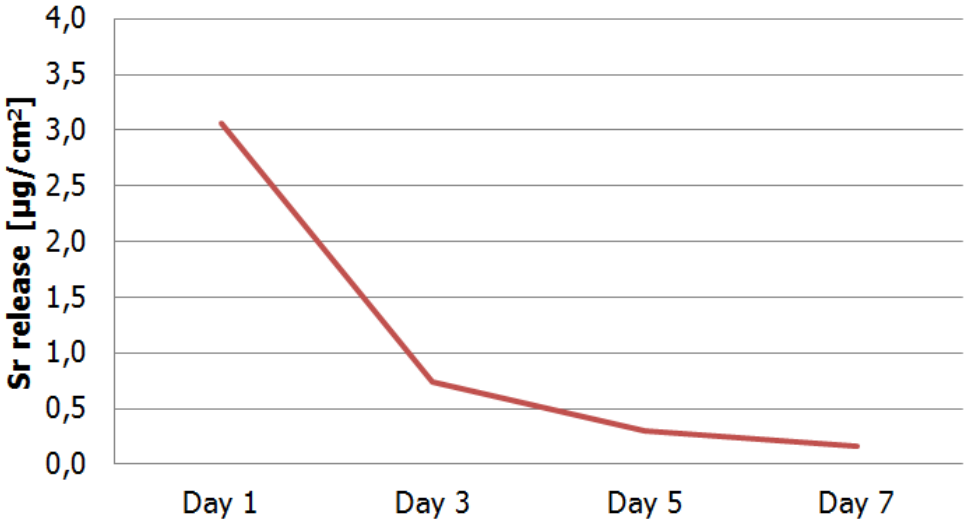
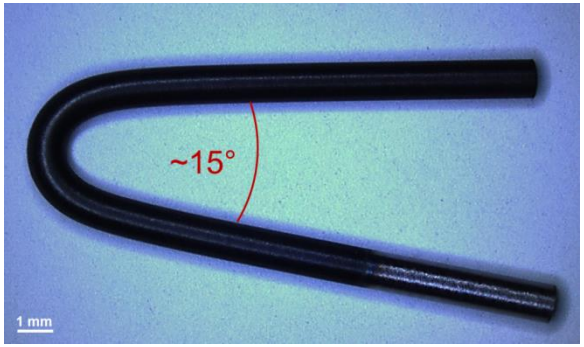


- Morphology (SEM), chemical composition (XPS, RBS), crystal structure (XRD)

- Sr release: wash-out in PBS solution + inductive coupled plasma optical emission spectroscopy (ICP-OES)





- Bending test of Sr-Ti-O coated Ti rod – no delamination
- Adhesion tests by screwing implants into polyoxymethyl (POM)

Sr release vs. Sr concentration

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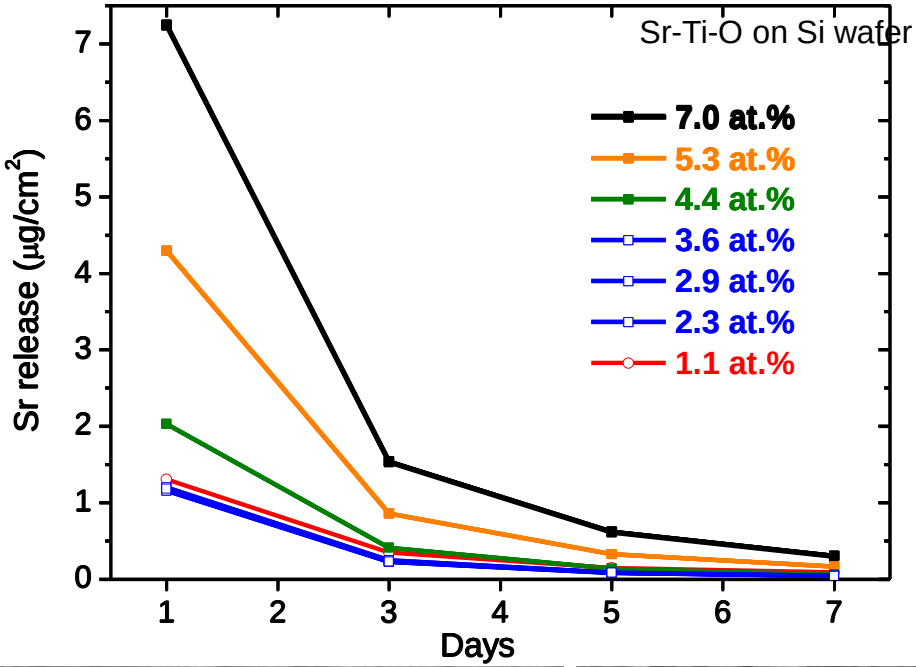

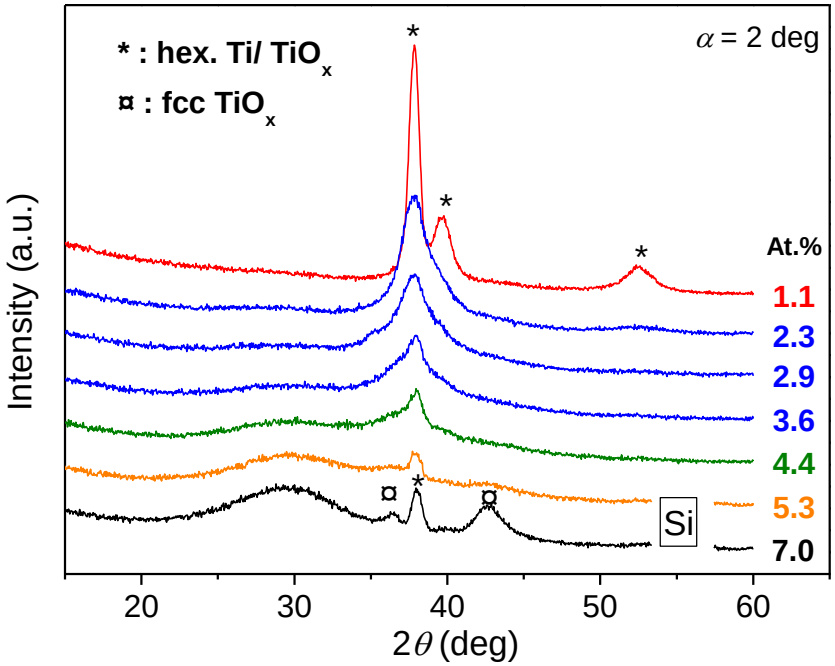

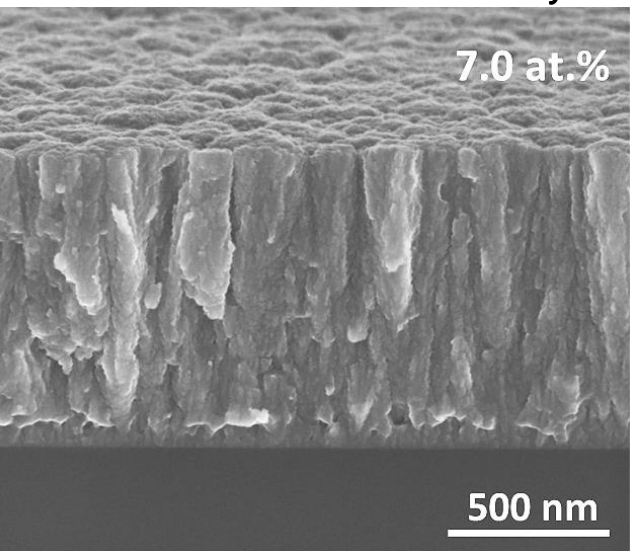

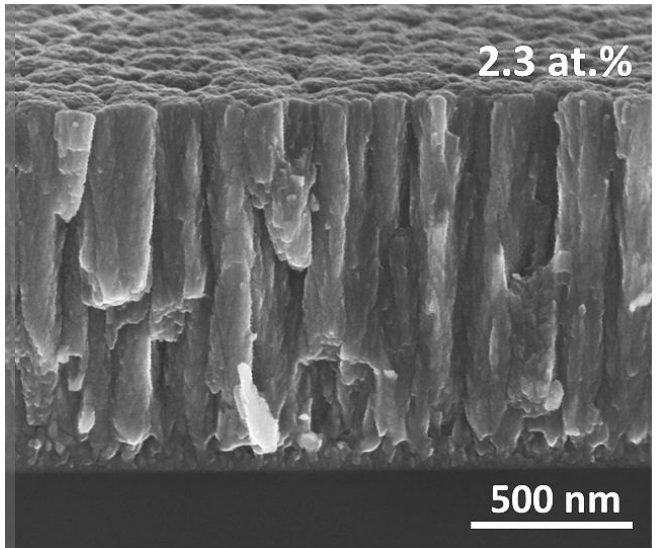

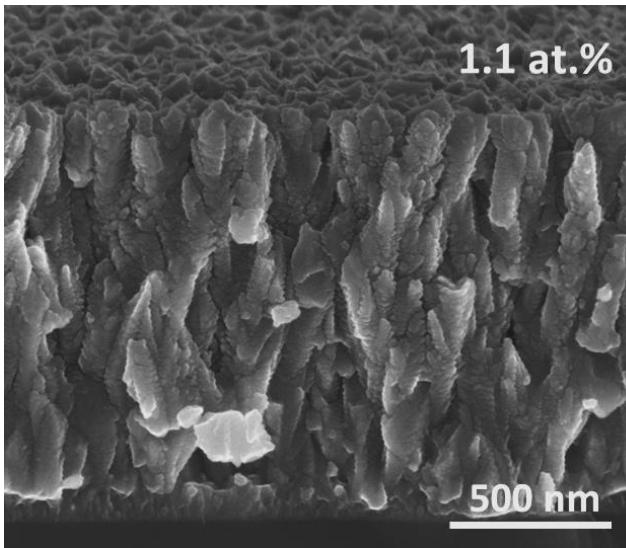

Sr release vs. coating thickness

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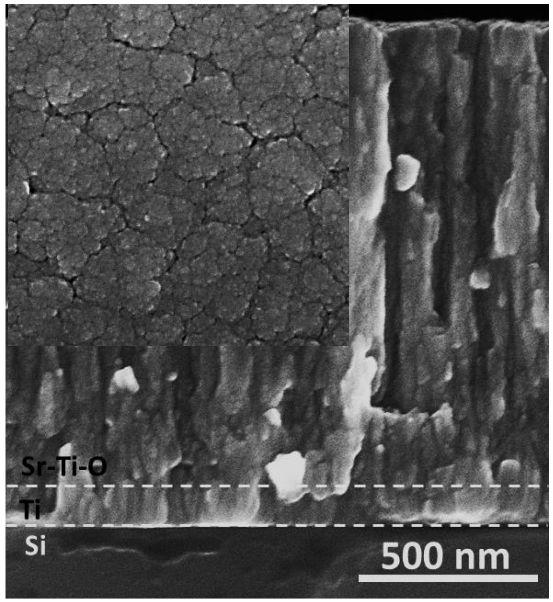

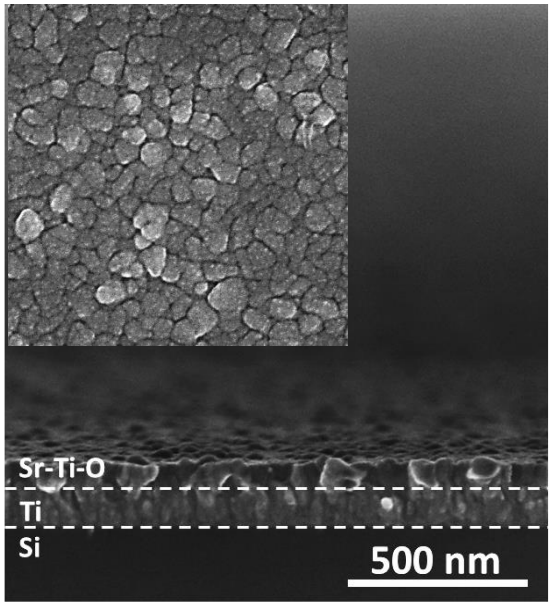

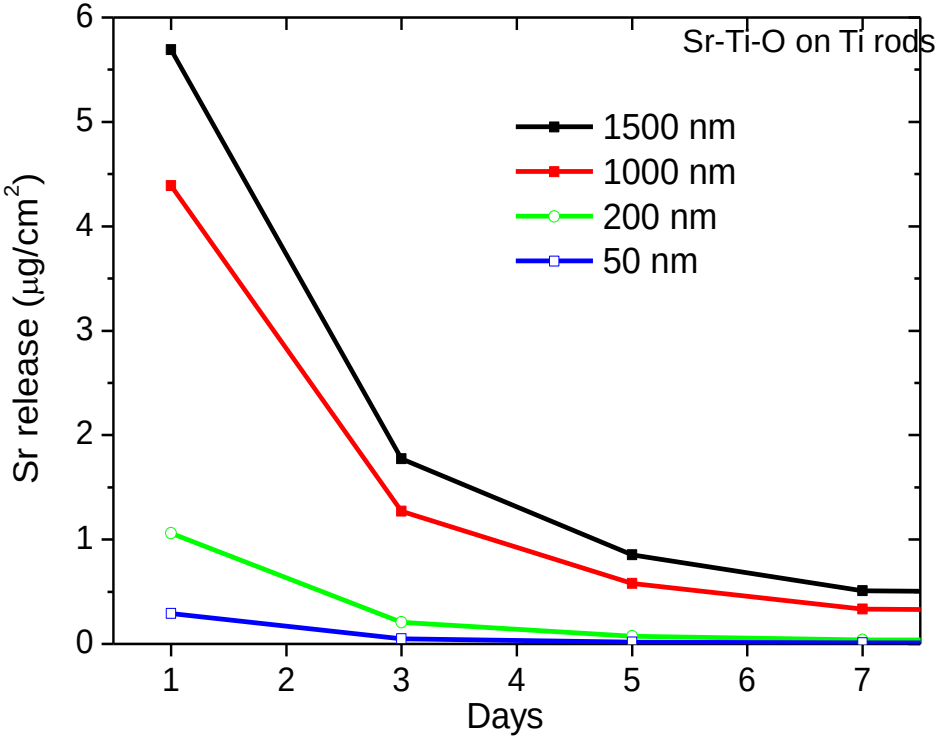

- At.%: Sr 4.4; Ti 35.6; O 60.0
- Pressure 1.1 Pa
- Increased Sr release vs. thickness
- Thickness + porosity → larger effective area

Sr release vs. pressure

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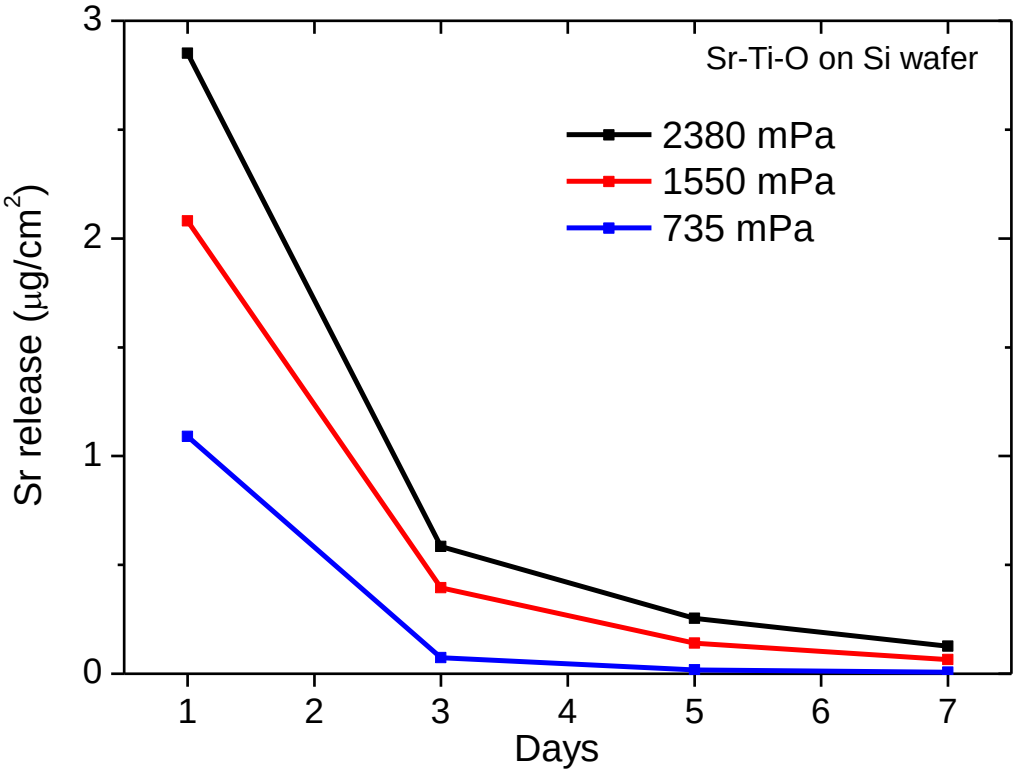
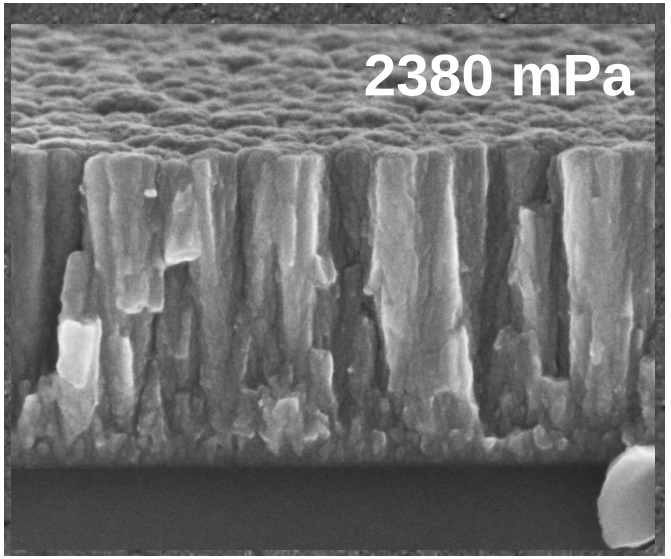
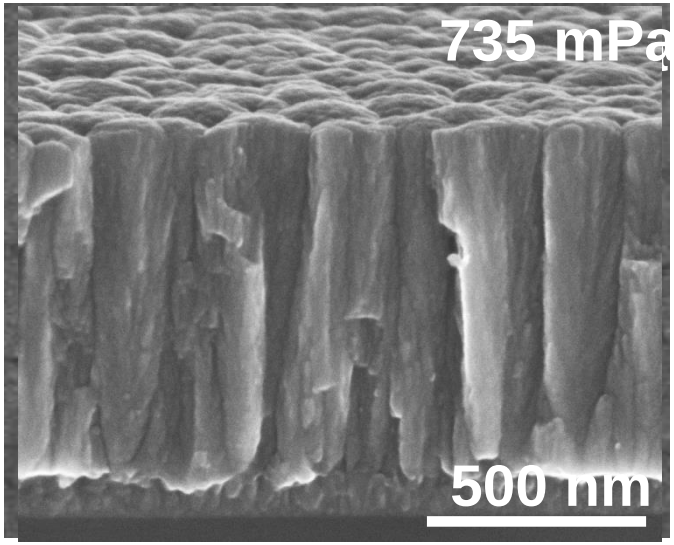
- At.%: ~ Sr 4.5; Ti 37; O 58.5
- Thickness 650 – 900 nm (high pressure → lower thickness due to gas collisions)
- Increased porosity (more/thinner columns) with higher pressure







Sr-Ti-O coatings

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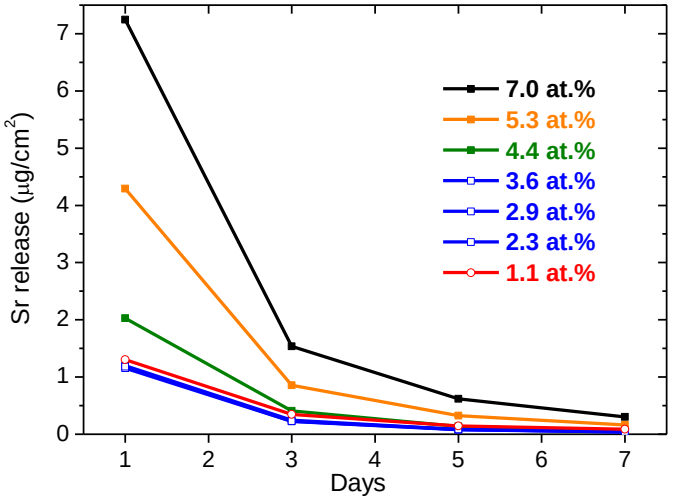
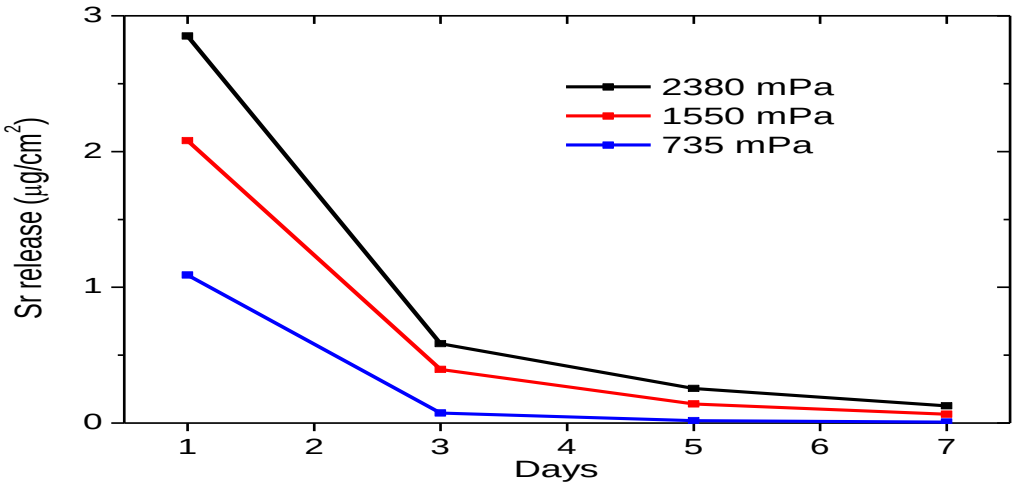
- Sr-release profile can be tailored by deposition parameters
- Coating thickness, deposition pressuer, Sr concentration





Do Sr-Ti-O coatings improve implant osseointegration?

In vivo model

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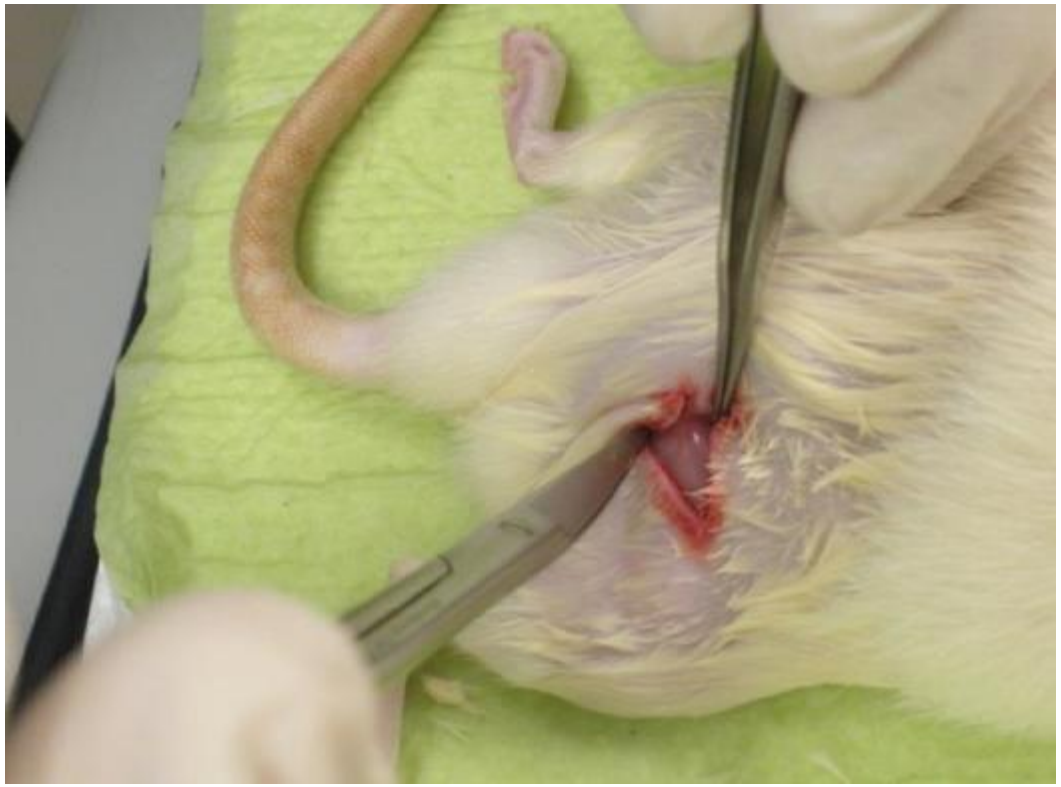

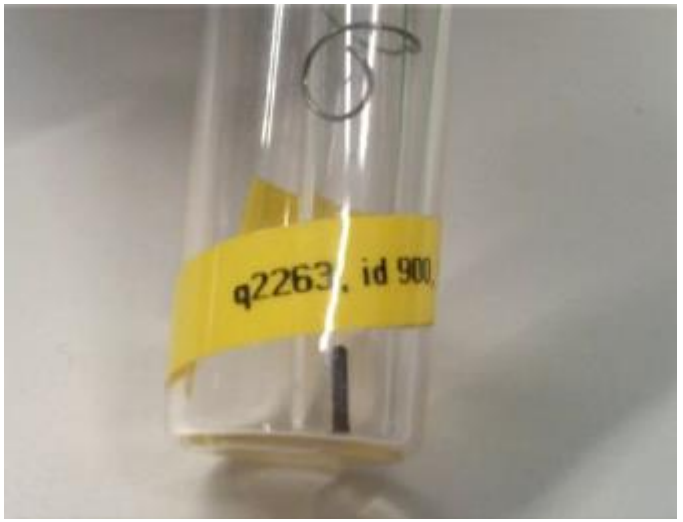

In vivo model

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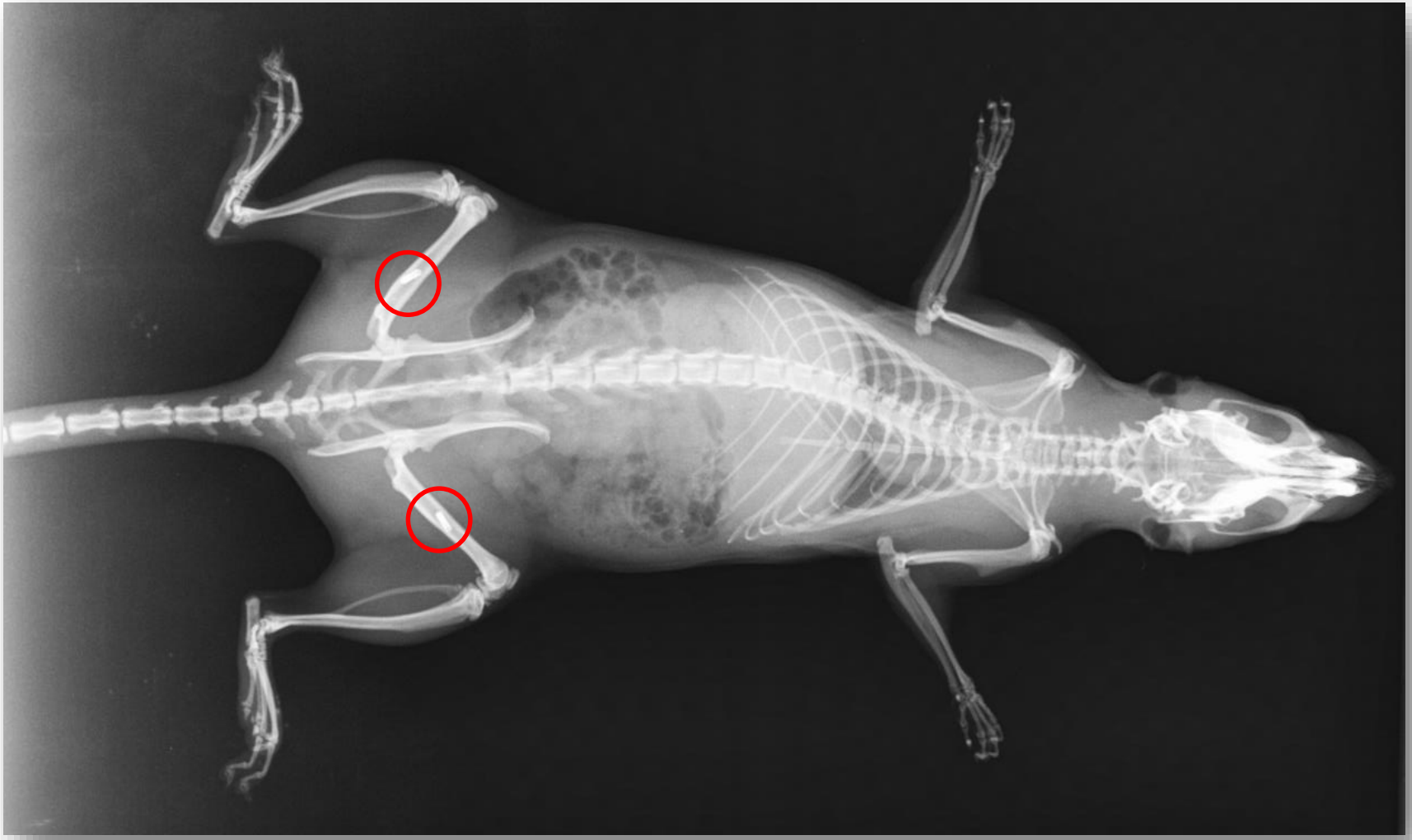

In vivo model

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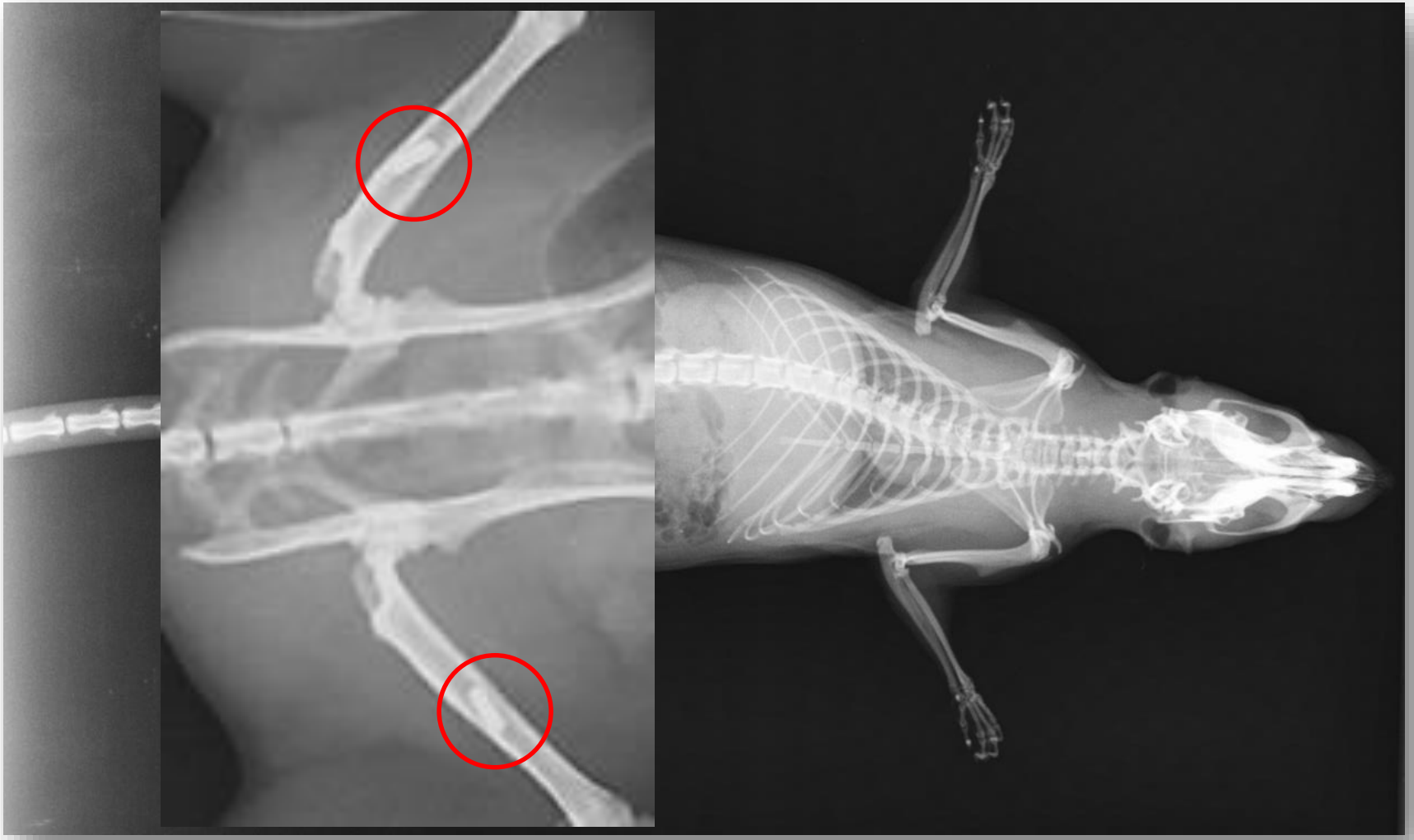

Tested in rats and rabbits

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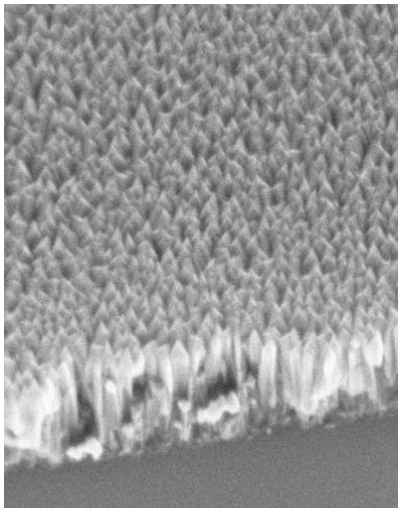
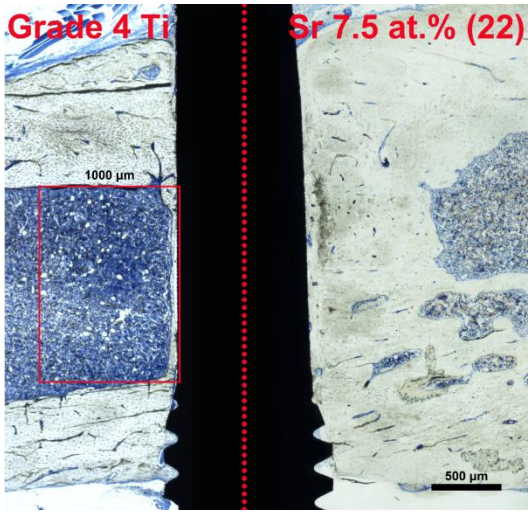
(54) **Strontium based coating for body implants**

Conclusion - summary

- An introduction to surface coatings
- An introduction to Physical Vapor Deposition (PVD)

- Examples of coatings for medical applications
  - Porous TiN for novel electrodes
  - Low friction diamond-like carbon (DLC) coating for dental applications
  - Strontium releasing coatings accelerating bone growth





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We would like to invite you to collaborate with us

In the Bioneca COST action we are already cooperation with:

- University of Malta
- University of Eastern Finland

In a privous COST action:

- Nicolaus Copernicus University

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