

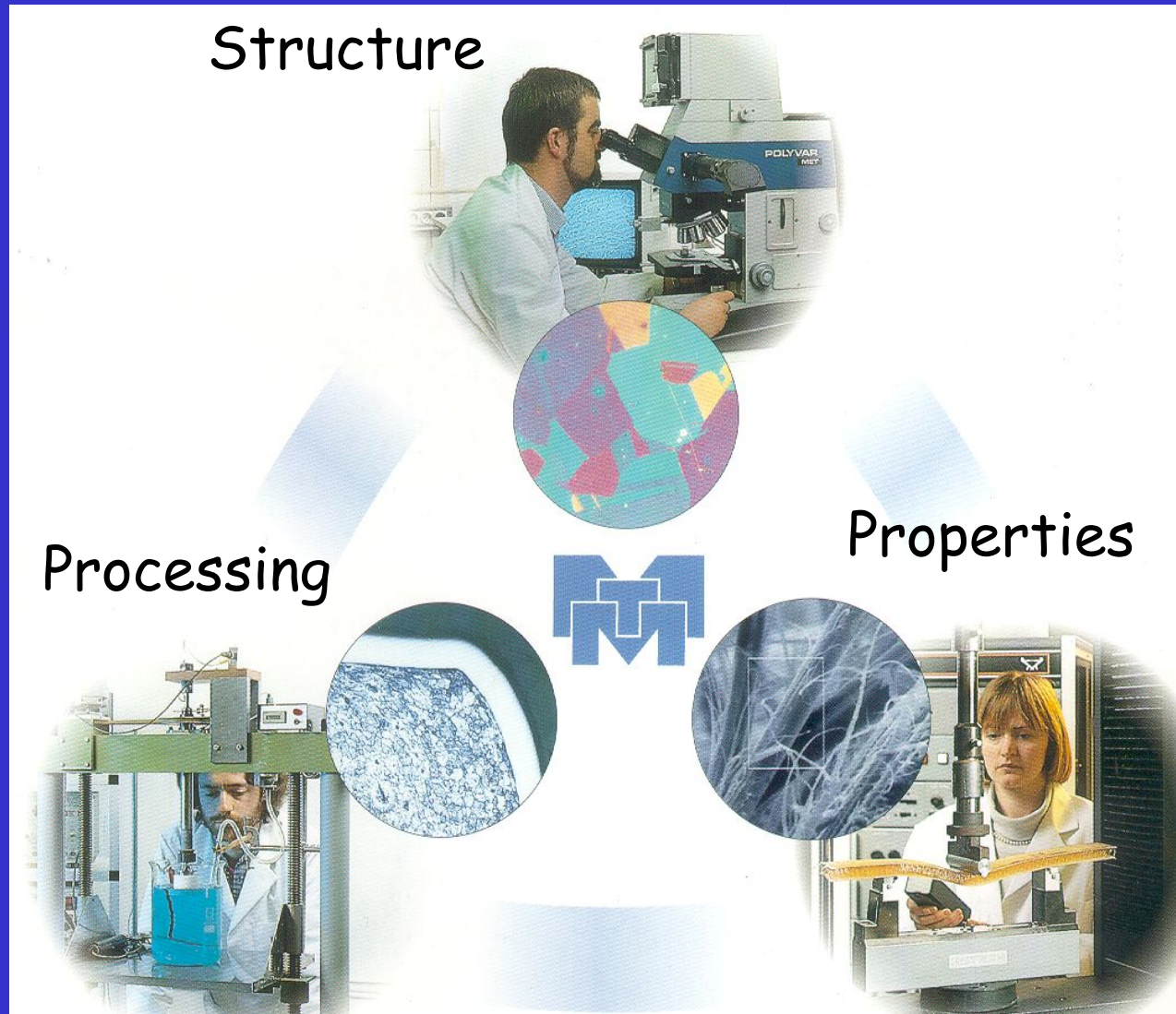


**Research on ceramics**  
**Department of Metallurgy and Materials Engineering**



[www.mtm.kuleuven.be](http://www.mtm.kuleuven.be)

# The MTM triangle



# Ceramics research group

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## ZAP (Professors):

Prof. Omer Van der Biest  
Prof. Jef Vleugels

## ATP (Technical support):

Joop Vandeursen  
Wout Veulemans  
Olivier Van Roey  
Mohammed Abid

## Postdoctoral Researchers:

Dr. Bernd Baufeld  
Dr. Shuigen Huang  
Dr. Kim Vanmeensel  
Dr. Songlin Ran  
Dr. Bram Neirinck

## Visiting scientists:

## PhD students:

Tina Mattheys  
Swarnakar Akhilesh Kumar  
Li Zhang  
Khuram Shahzad

## PhD students:

Annabel Braem  
Olivier Malek  
Ezhil Jothinathan

# Activities ceramics research group

## Processing of ceramics

- Powder synthesis (sol-gel, carbo- and borothermal reduction)
- Powder metallurgical shaping
- Colloidal shaping by electrophoretic deposition (EPD)

## Microstructural analysis and functional properties

- Sintering (pressureless, hot pressing, microwave, SPS)
- Microstructural analysis (SEM, EPMA, XRD, TEM)
- Mechanical properties (hardness, toughness, strength, etc.)
- Elastic and damping properties at room and elevated temperature

## Modelling

- Chemical compatibility
- Electrophoretic deposition (EPD)
- Functionally graded materials (FGM)
- Damping

# Ceramic Materials under investigation

## Composites

### Glass-ceramics

Bariumaluminosilicate (BAS)

Magnesiumaluminosilicate (MAS)

### Ceramic matrix composites

Matrix:  $\text{Si}_3\text{N}_4$ ,  $\text{ZrO}_2$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{ZrB}_2$ ,  $\text{TiB}_2$

Additive:  $\text{Al}_2\text{O}_3$ ,  $\text{TiB}_2$ ,  $\text{TiN}$ ,  $\text{TiC}$ ,  $\text{TiCN}$ ,

$\text{WC}$ ,  $\text{NbC}$ ,  $\text{ZrC}$ ,  $\text{HfC}$ ,  $\text{HfTiC}$ ,  $\text{ZrN}$ ,

$\text{B}_4\text{C}$ ,  $\text{SiC}$ , etc.

### Cermets

$\text{WC-Co}$ ,  $\text{TiCN}$ -based

$\text{NbC}$ -based

## Reinforced ceramics

### Fibre reinforced

Borosilicate, MAS & BMAS  
with  $\text{SiC}$  fibres

### Platelet reinforced

Sialon,  $\text{Al}_2\text{O}_3$ , mullite  
with  $\text{Al}_2\text{O}_3$  platelets

## Monoliths

### Oxides

Mullite,  $\text{Al}_2\text{O}_3$

Y-TZP & Ce-TZP

Mixed stabiliser  $\text{ZrO}_2$

### Non-oxides

$\text{SiAlON}$ ,  $\text{Si}_3\text{N}_4$

$\text{SiC}$ ,  $\text{TiB}_2$ ,  $\text{TiN}$ ,  $\text{TiCN}$ ,

$\text{WC}$ ,  $\text{ZrB}_2$ ,  $\text{B}_4\text{C}$ , etc

## Graded Materials

### Functionally graded (FGM)

$\text{ZrO}_2/\text{Al}_2\text{O}_3$  &  $\text{ZrO}_2/\text{WC}$

$\text{WC-Co}/\text{WC-Co}$

$\text{TiCN}$ -based/ $\text{WC-Co}$

Ce-TZP/Y-TZP

### Laminates

$\text{SiC}$  / graphite &  $\text{SiC}$  / porous  $\text{SiC}$

### Coatings

Metal /  $\text{ZrO}_2$

$\text{WC-Co}$  / steel

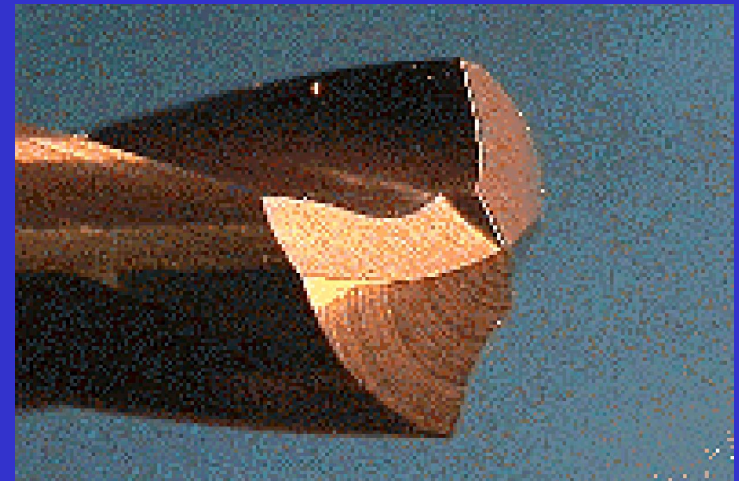
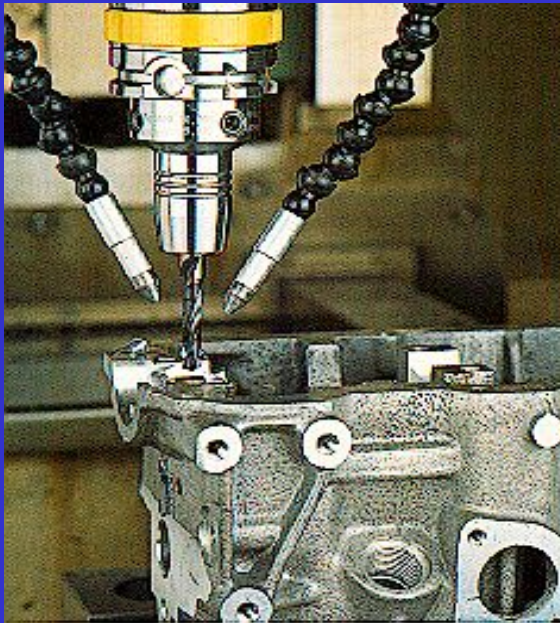
# Research Topics

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- Processing and characterisation of **functionally graded materials (FGM)**
- Colloidal processing by means of **electrophoretic deposition (DC & AC-EPD)**
- Development and characterisation of **ceramic, CMC's and cermets**
- Modelling and application of **field assisted sintering (FAST, SPS, PECS)**
- Investigation of **elastic and damping properties** of materials
- Cutting tool development and **chemical compatibility** assessment
- **Nanomaterials and nanocomposites** (biomaterials, photovoltaics, batteries)
- Processing of **Porous materials** (ceramics, glass & metals)

# Cutting tool development and chemical compatibility studies

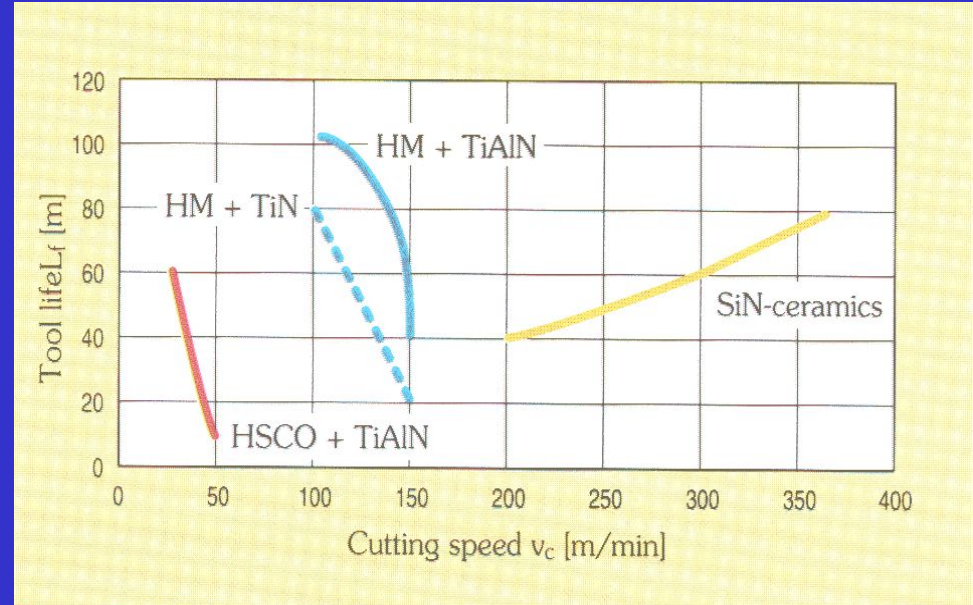
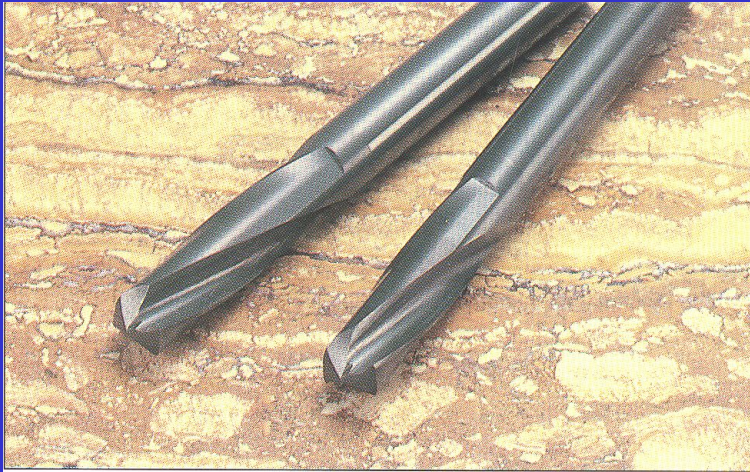
DEVELOPMENT OF NEW CUTTING MATERIALS, TOOLS, MACHINE CONCEPTS  
AND TECHNOLOGIES FOR DRY HIGH SPEED CUTTING



- New composites
- Chemical wear assesment
- Gradient materials

# Cutting tool development

## Dry machining of cast iron with siliconitride tools



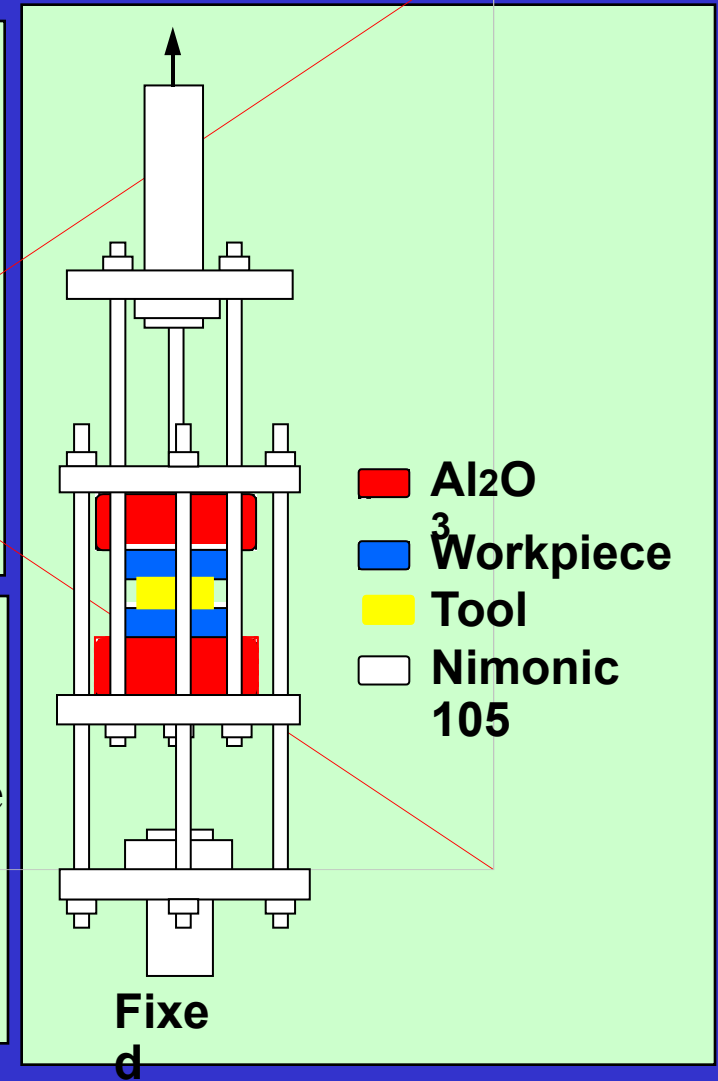
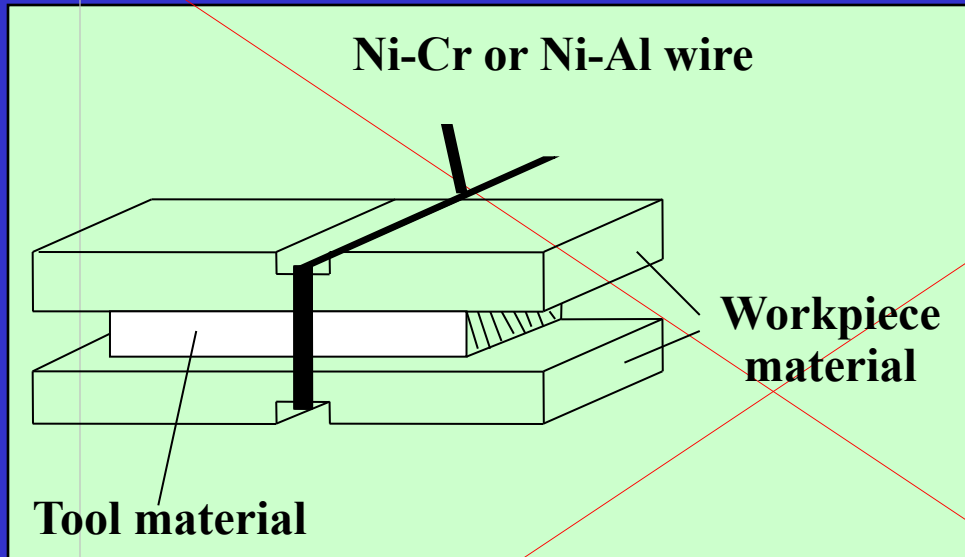
## Dry machining of cast iron with ceramic composite tools



Dry drilling of cast iron at 450 m/min

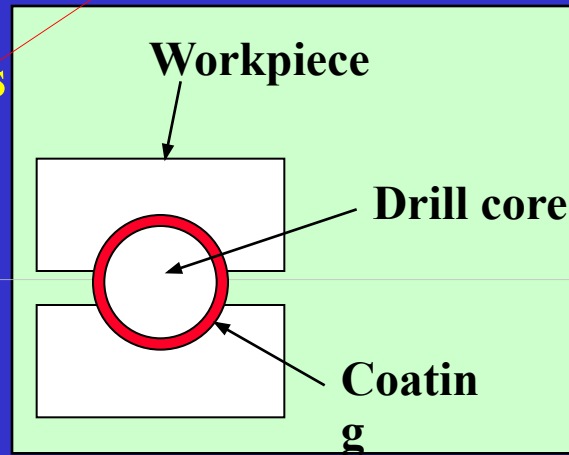


# Chemical compatibility assessment



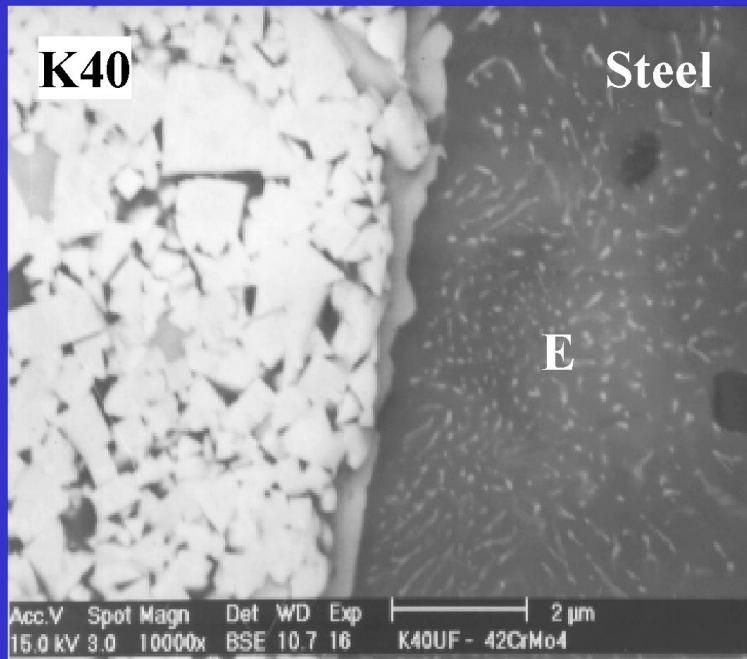
## Experimental parameters

Temperature  
Holding time  
Mechanical load

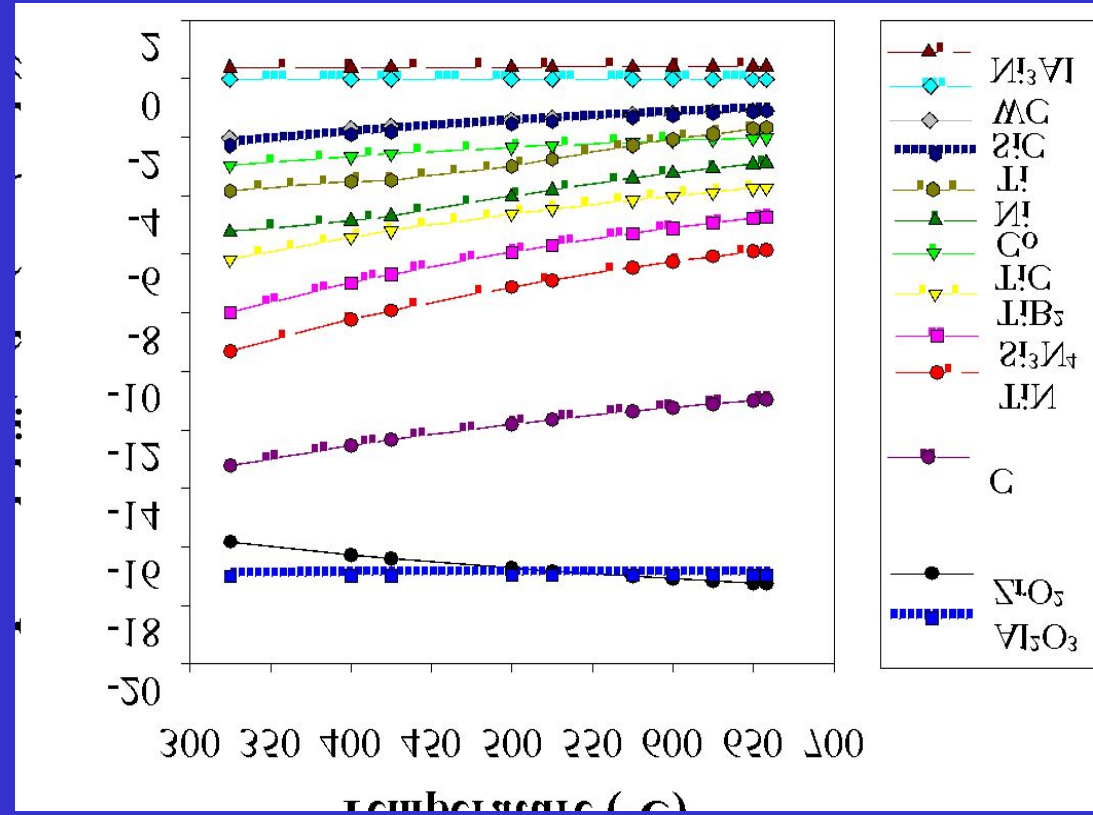


# Chemical compatibility assessment

## Interaction couples

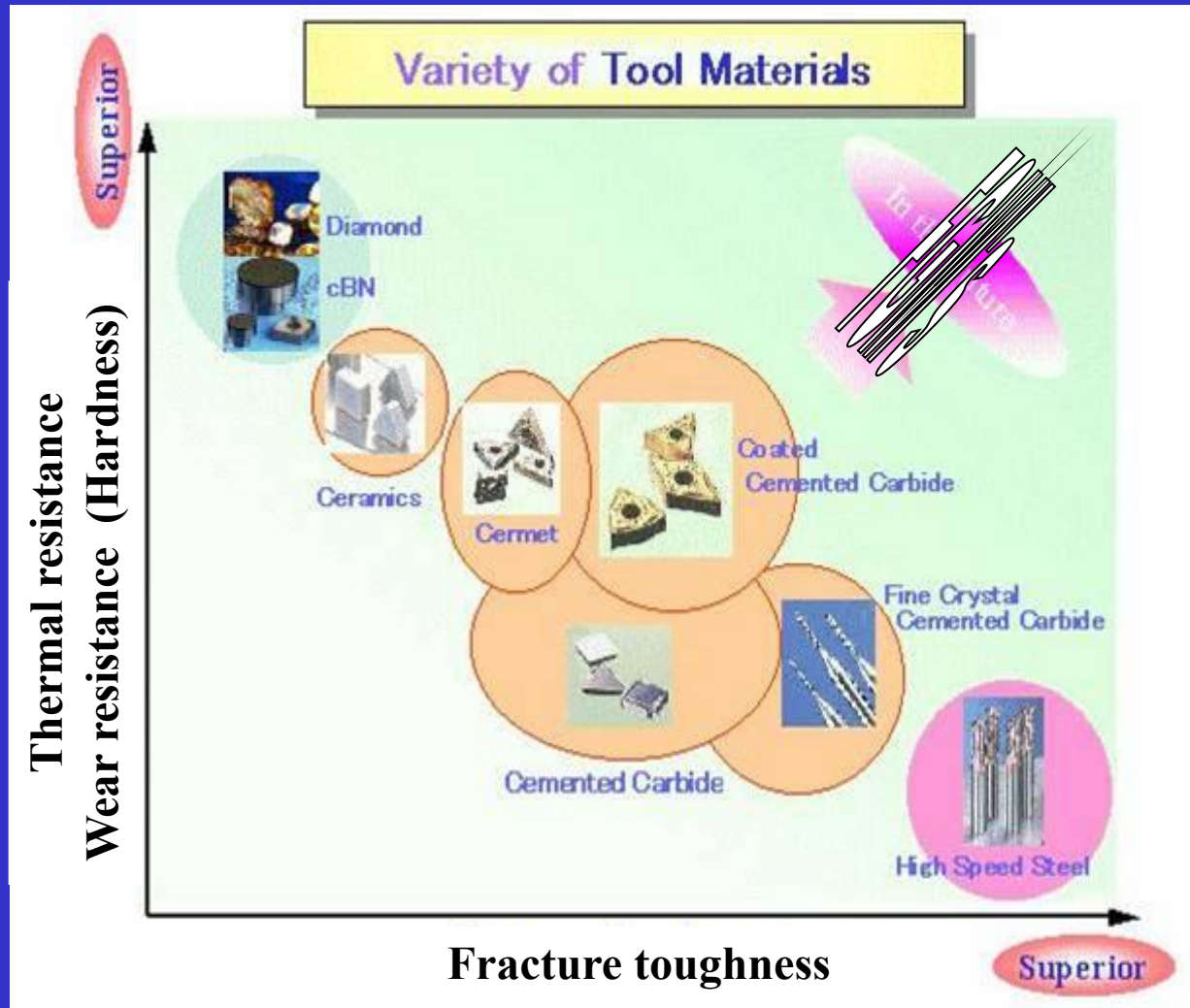


## Equilibrium solubility calculations



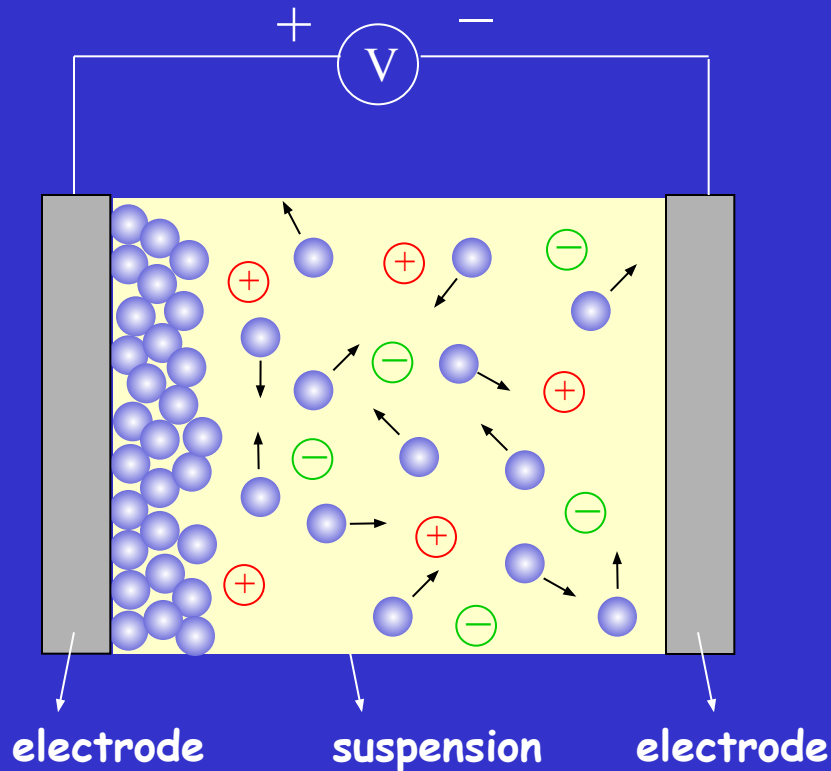
# Functionally graded materials (FGM)

To combine irreconcilable properties in the same component by engineering a gradient in composition and concomitant properties



# Electrophoretic deposition (EPD)

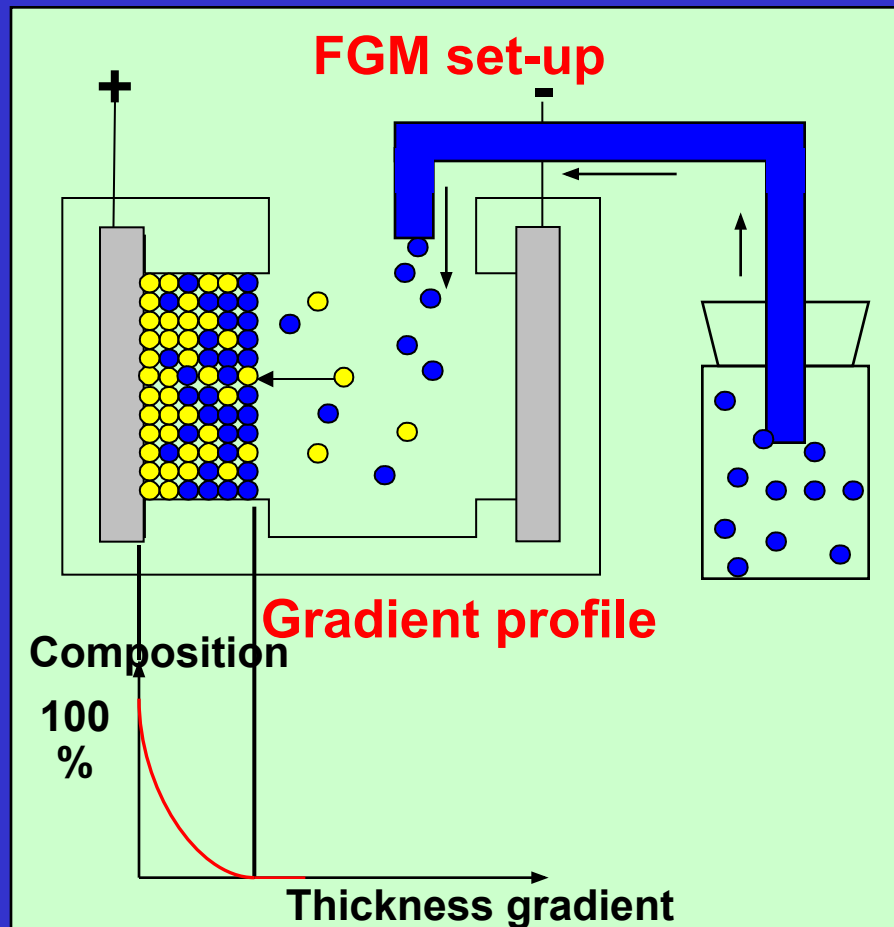
Colloidal processing technique in an electric field



- Charged particles
- ⊕ Cations
- ⊖ Anions

- Particles are charged by interaction with the solvent and additives
- Charged particles move under the influence of an applied electric field (**electrophoresis**)
- Particles form a growing deposit on the deposition electrode (**deposition**)

# Electrophoretic deposition (EPD) of FGM



## Applications:

- Gradient materials
- Laminates
- Coatings (nm-mm)
- Infiltration
- Textured materials
- Save processing of nanopowders

**Graded Tribological Materials Formed by Electrophoresis**

# EPD of FGM and coatings

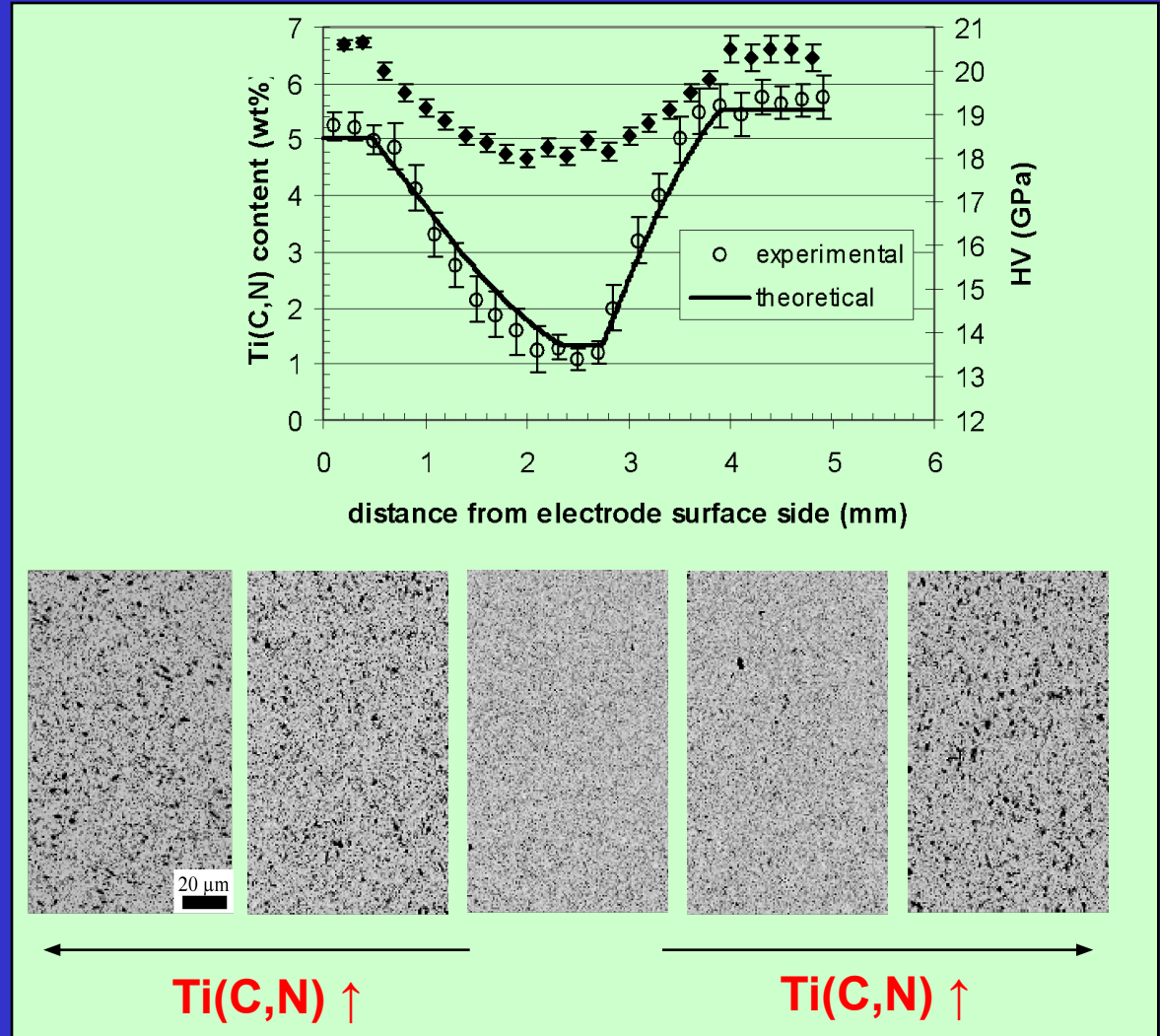
Cutting tool inserts : WC-Co-Ti(C,N)/ WC-Co/ WC-Co-Ti(C,N)



WC-Co-Ti(C,N)

WC-Co

WC-Co-Ti(C,N)



# EPD of FGM and coatings



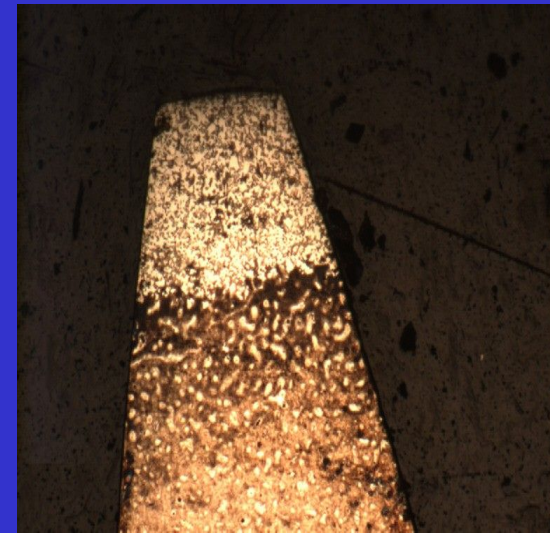
HSS substrate

EPD-coated

Sintered

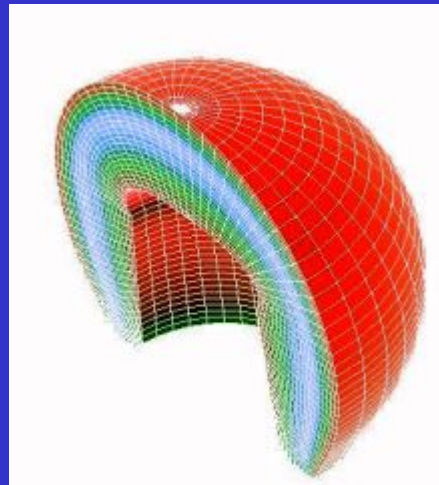
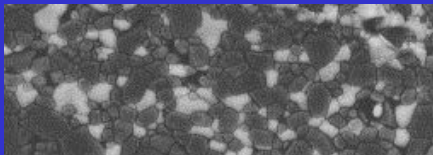
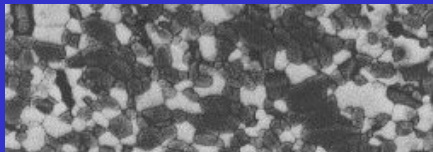
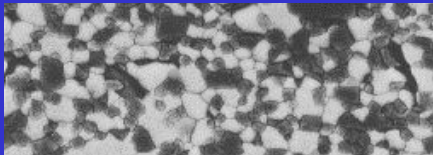
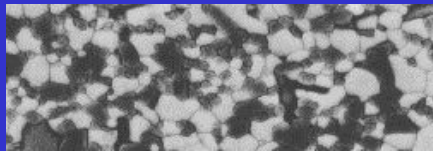
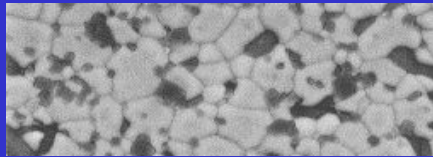
Final machined

HSS taps with carbide coating



# EPD of FGM

## Increasing the Performance of Total Hip Replacement Prostheses through Functionally Graded Material Innovation and Design



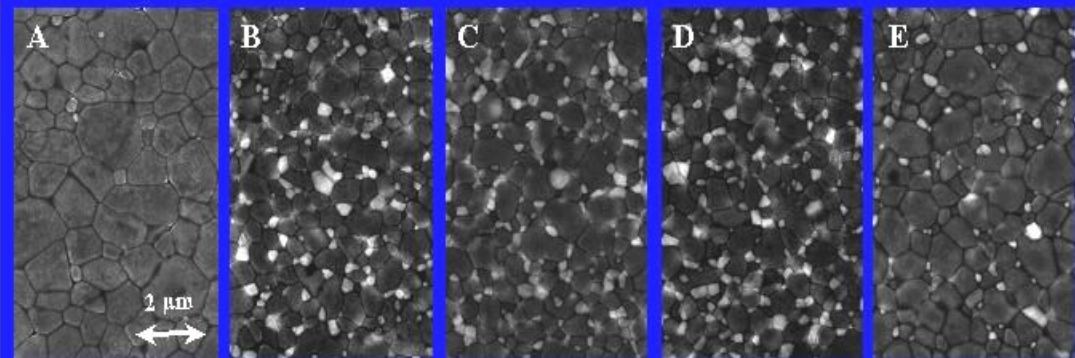
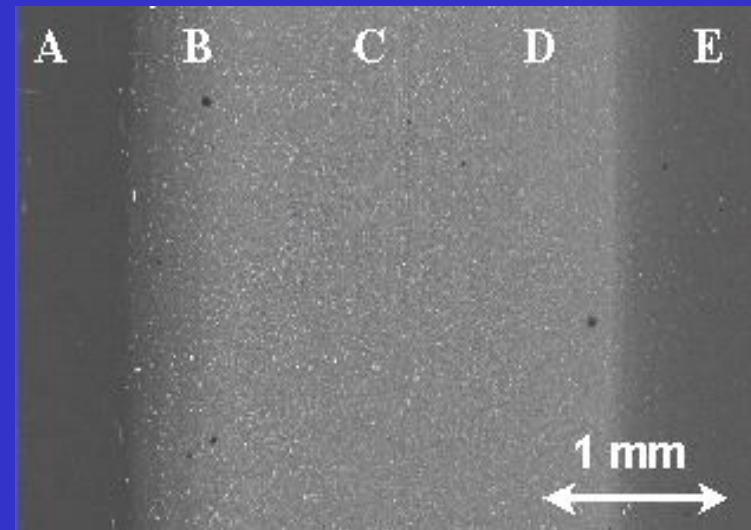
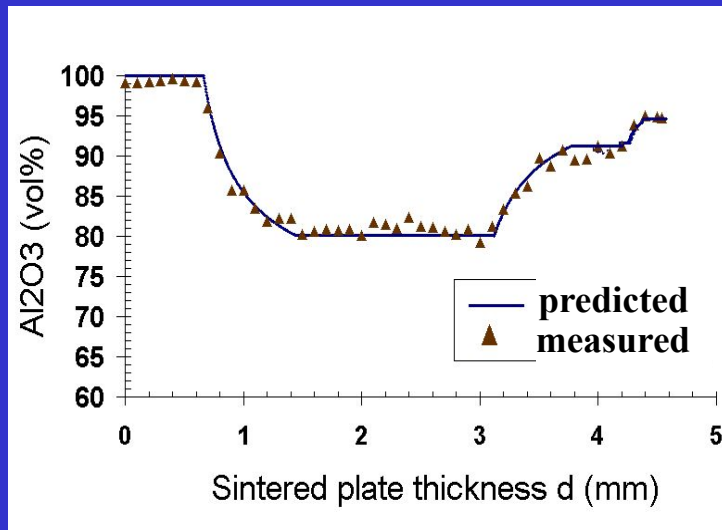
### Gradient in composition resulting in:

- Gradient in properties
- Residual thermal stresses
- improved strength and wear resistance

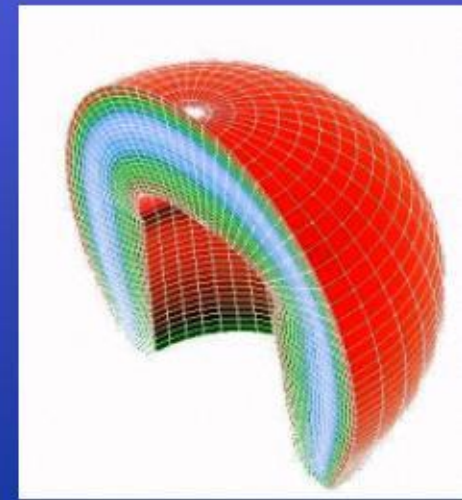
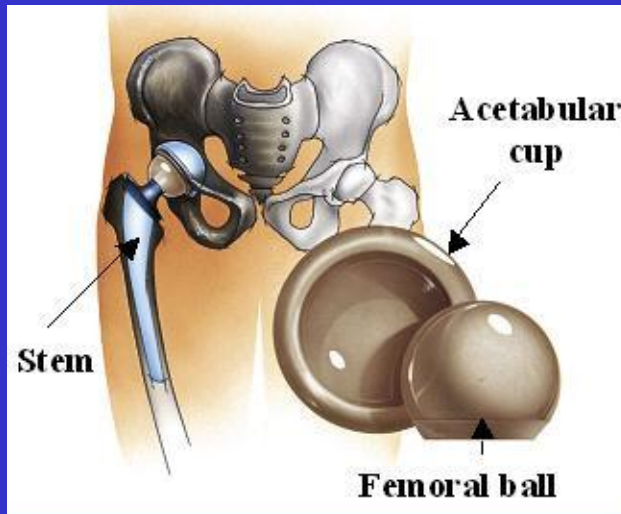


# EPD of plate shaped FGM

## Symmetrical $\text{Al}_2\text{O}_3/\text{Al}_2\text{O}_3\text{-ZrO}_2/\text{Al}_2\text{O}_3$ FGM

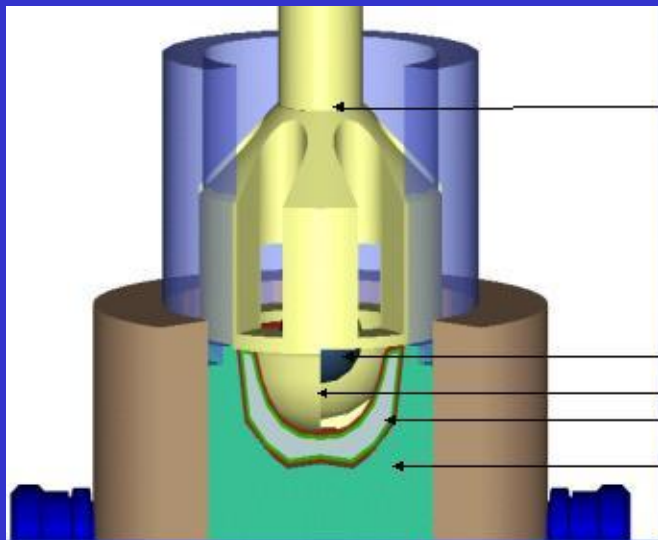


# EPD of complex shaped FGM



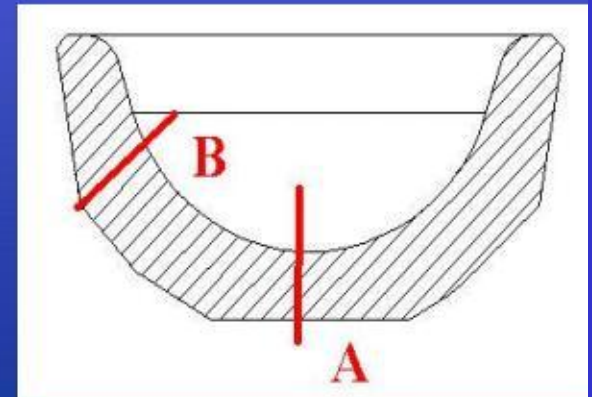
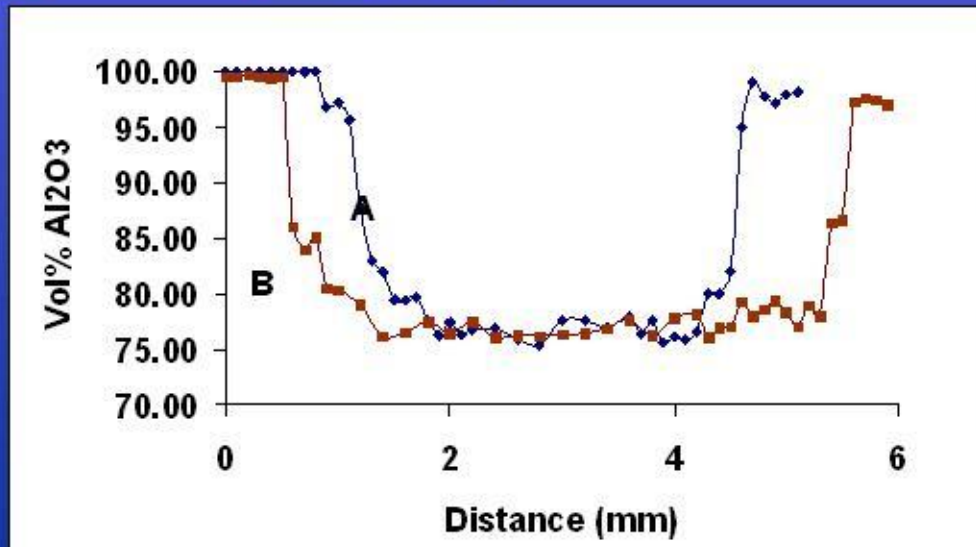
**Near-net-shape processing (max + 100  $\mu\text{m}$ )**

# EPD of complex shaped FGM

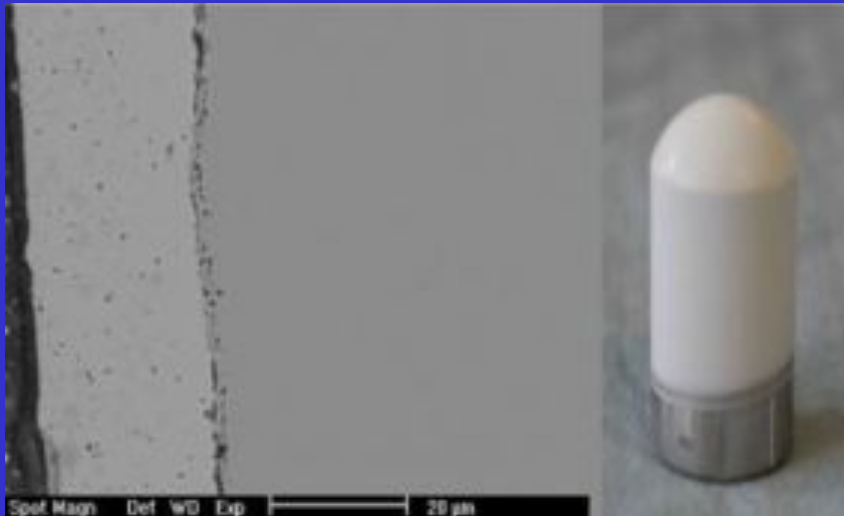


Counter electrode holder

Counter electrode  
Isolating cap  
Deposit  
Deposition electrode

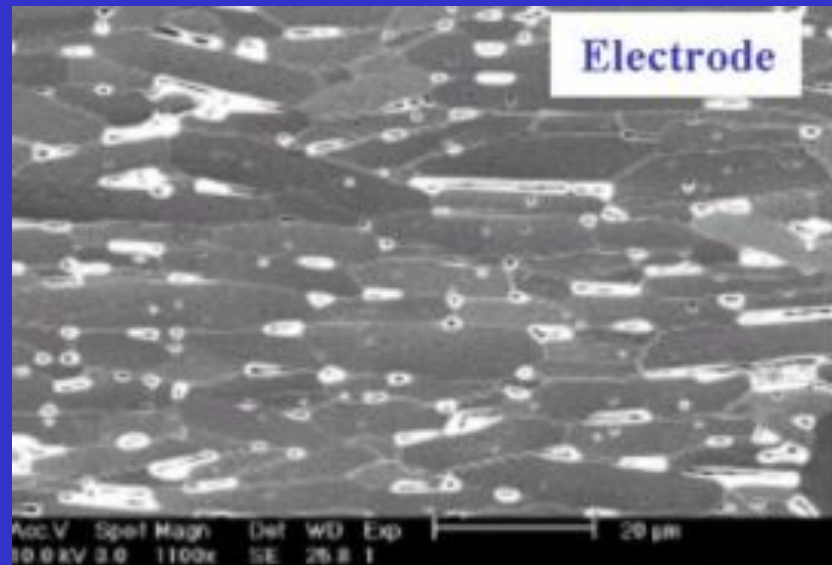


# EPD of coatings



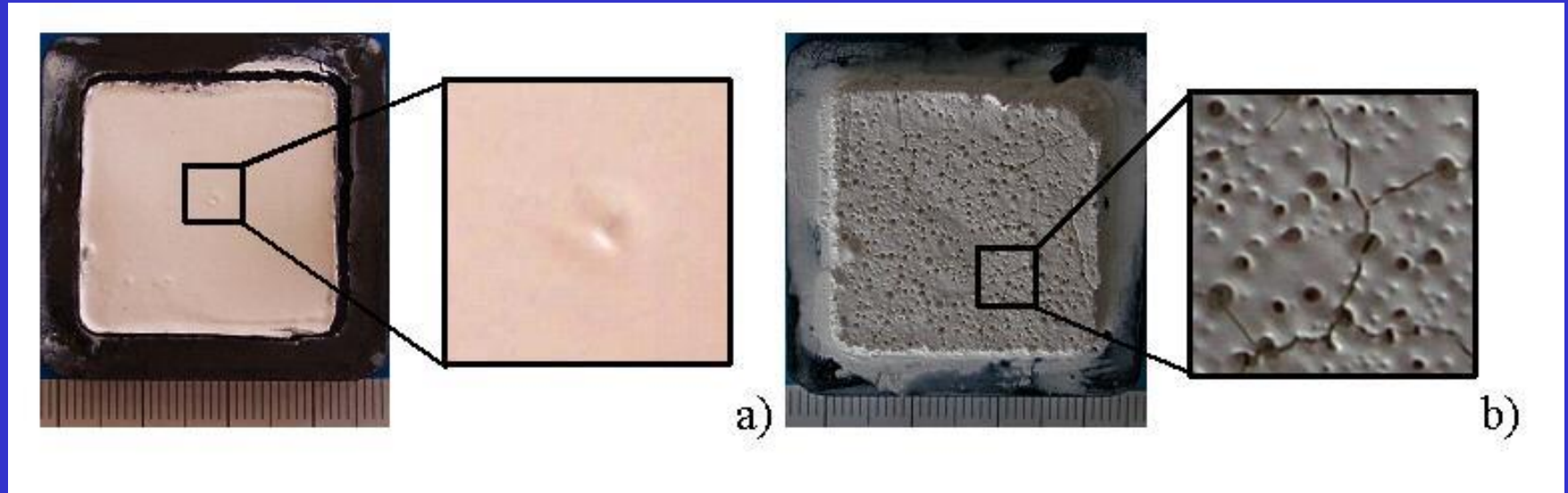
ZrO<sub>2</sub> coating on metal substrates

Texturing of materials



# Fundamentals of AC electrophoretic deposition (AC-EPD)

Aqueous electrophoretic deposition in asymmetric AC electric fields

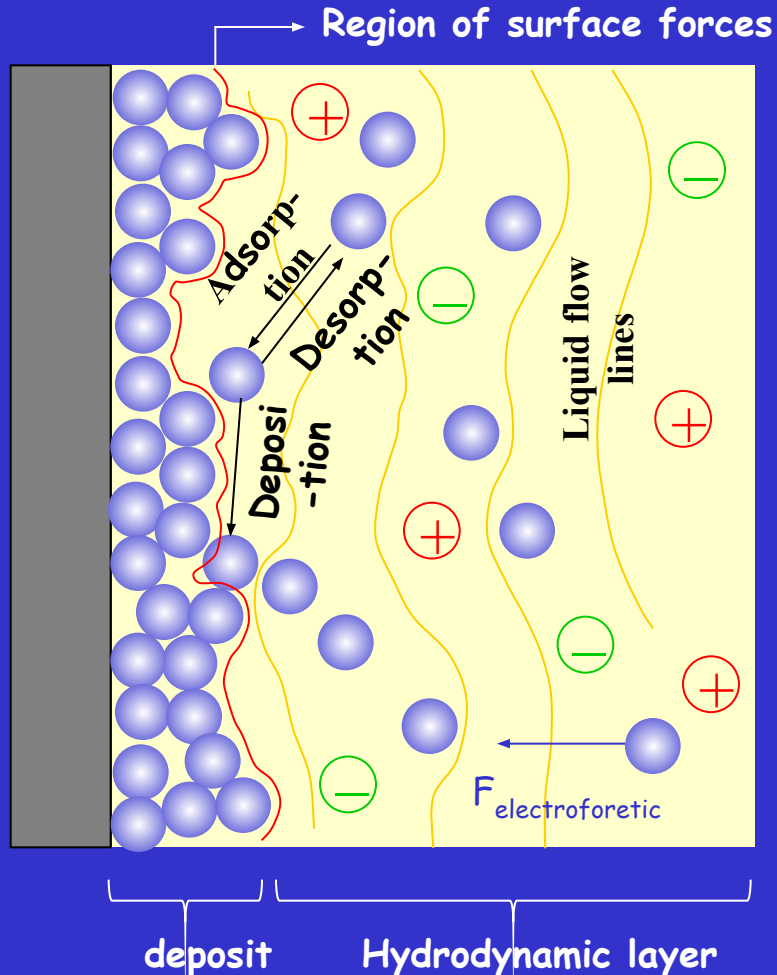


Alumina powder deposit formed by unbalanced AC (a) and DC (b) electric fields from a water-based suspension

**How does this work ?**

# Basic science on EPD

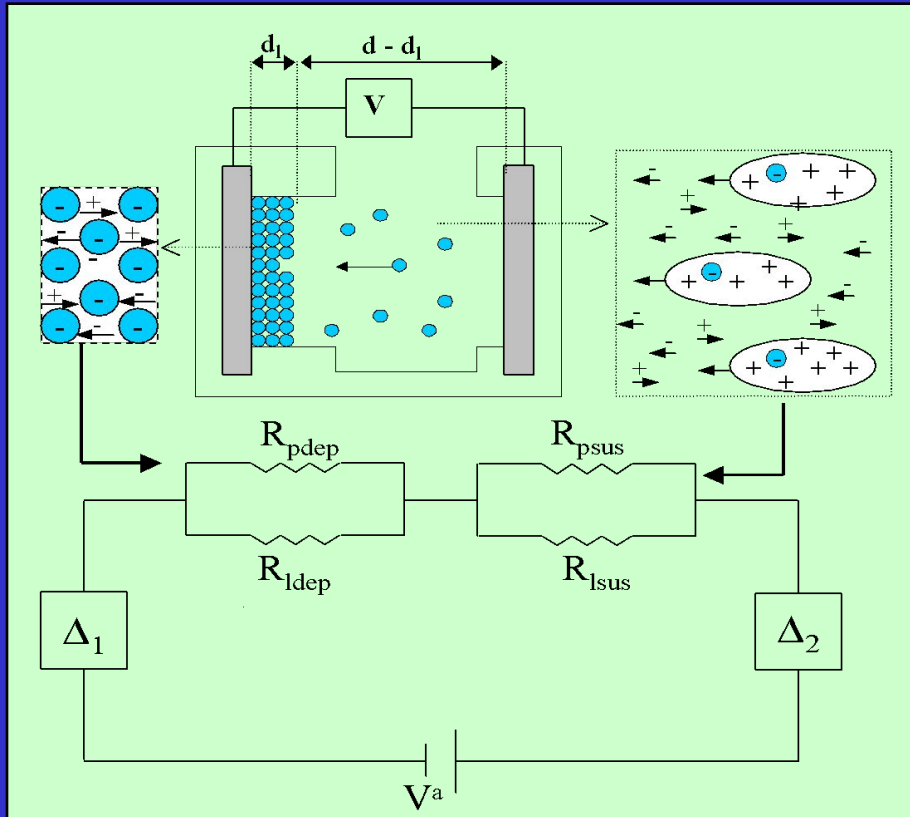
## Electrophoretic forming of functionally graded materials and coatings



- Suspension stability studies
- Charging mechanisms and particle-additive interactions
- Electrophoretic mobility and zeta potential measurements
- Study of the deposition process
- AFM of particle-electrode interaction
- Electrochemical reactions
- Fluid dynamic interactions during EPD
- Modelling of the EPD kinetics

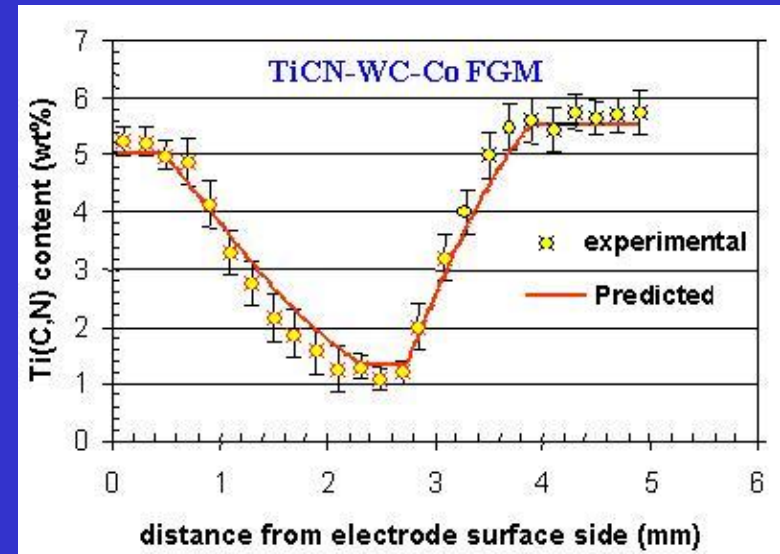
**GOA-TBA** 2005-2008 K.U.Leuven

# Modelling of EPD



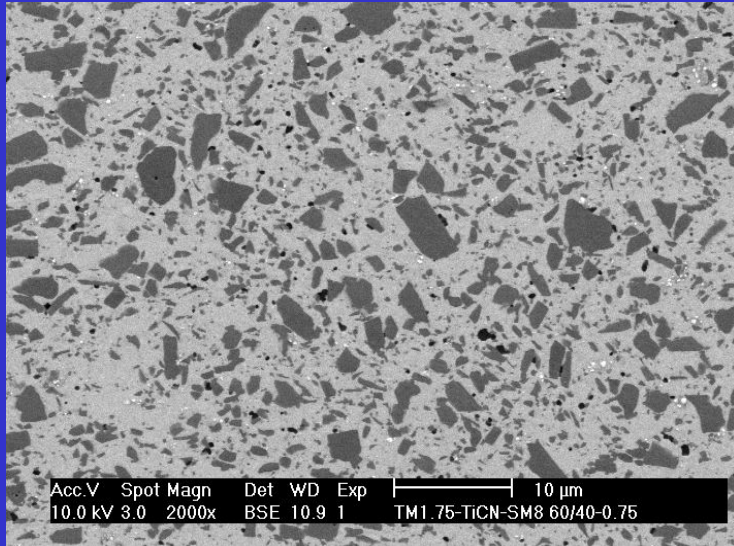
The currents and voltages during EPD are calculated from the equivalent electric circuit shown

To calculate the composition gradient in the FGM material from the starting composition of the suspensions, the EPD operating parameters and the powder-specific EPD characteristics.

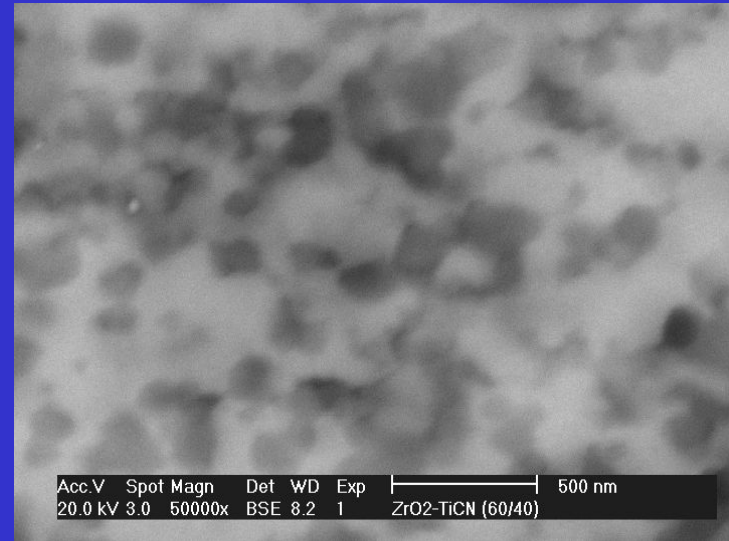


# Nanomaterials and Nanocomposites

Conventional composite



Nanocomposite < 100 nm !



Colloidal processing of nanopowders

Shaping of Coatings Composites Gradient materials

Densification with limited grain growth

Characterisation: microstructural physical mechanical

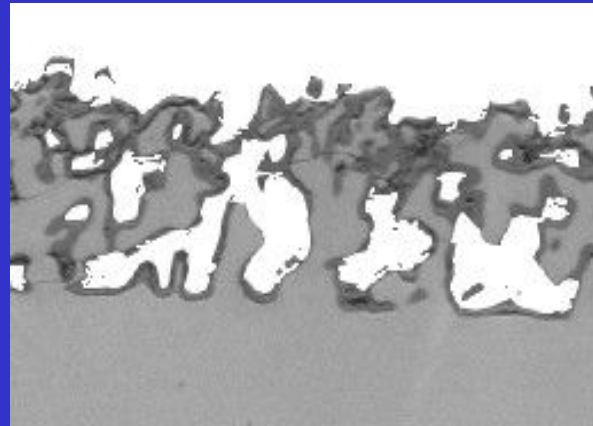


GOA 2008-2011 K.U.Leuven



# Development of biocompatible coatings

Multifunctional bioresorbable biocompatible coatings with biofilm inhibition and optimal implant fixation



6<sup>th</sup> Framework Project

**Meddelcoat**

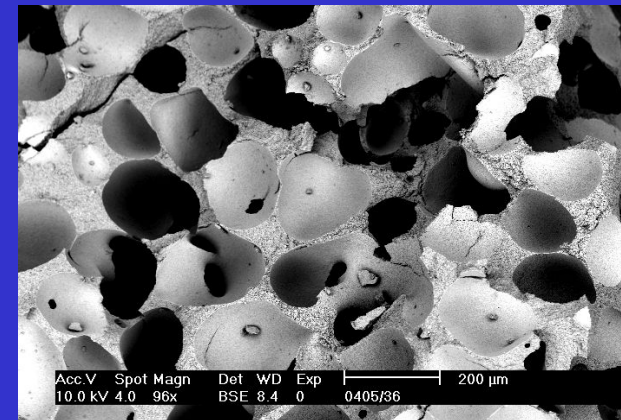
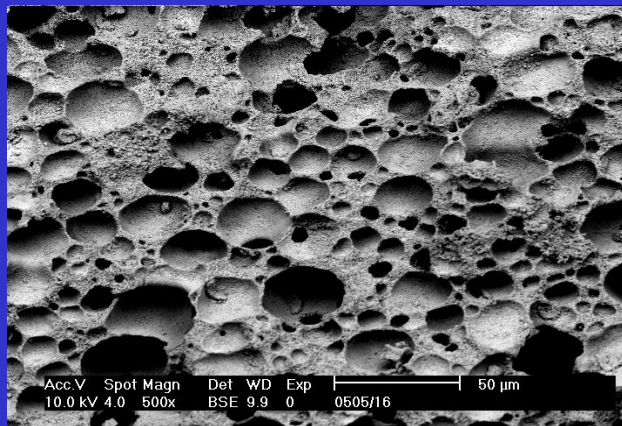
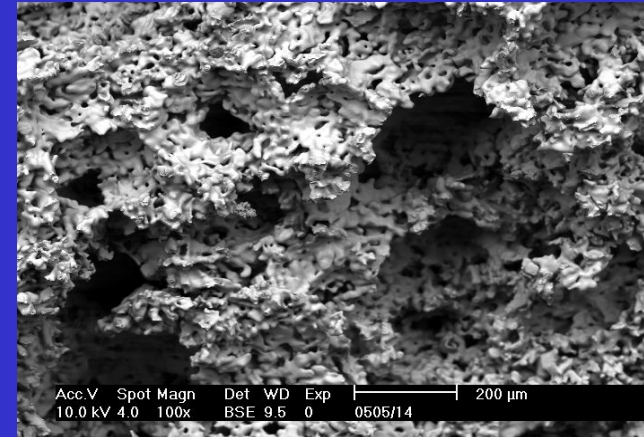
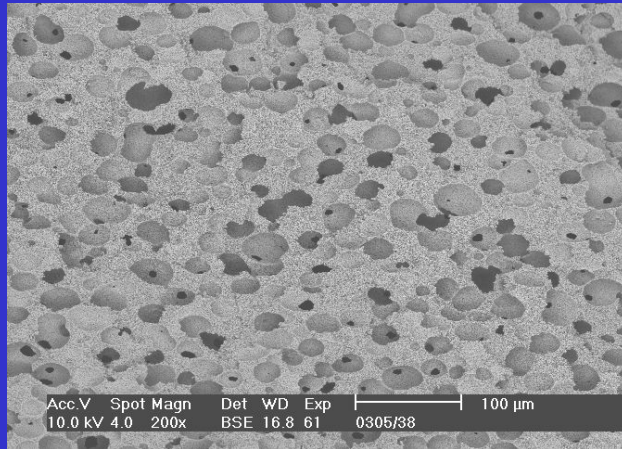
2006-2010

Project Coordinator:

K.U.Leuven

[www.meddelcoat.eu](http://www.meddelcoat.eu)

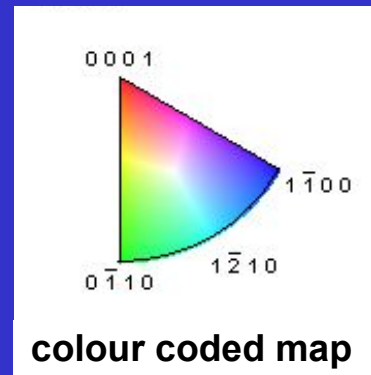
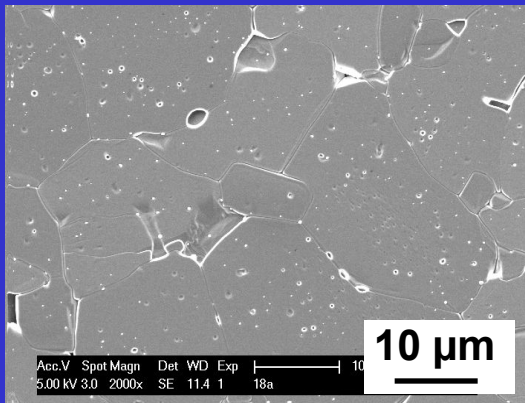
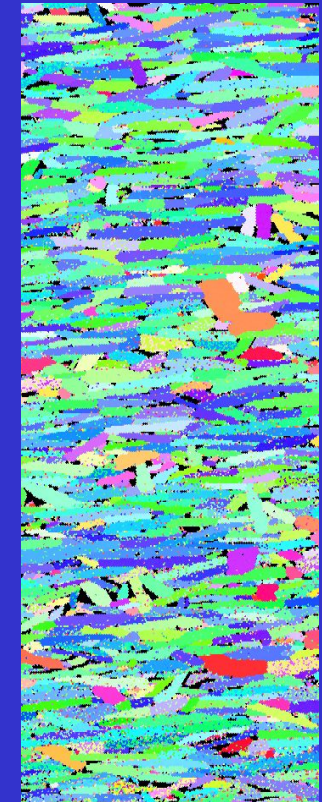
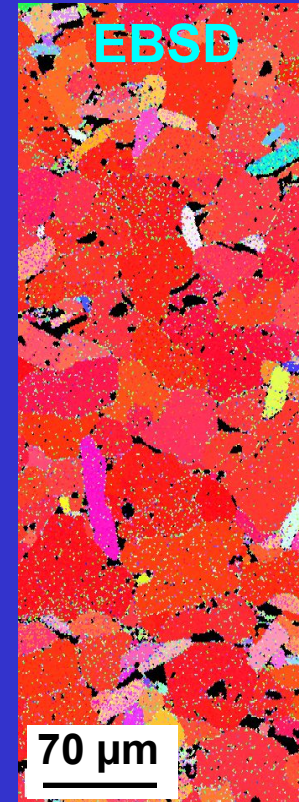
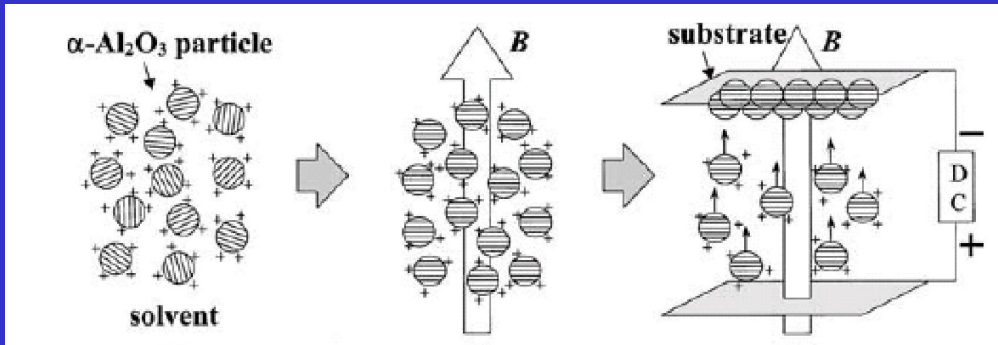
# Development of Porous Materials



Development of porous glass, ceramic and metal structures and coatings

# Development of textured materials by EPD

## Processing of materials using a strong magnetic field



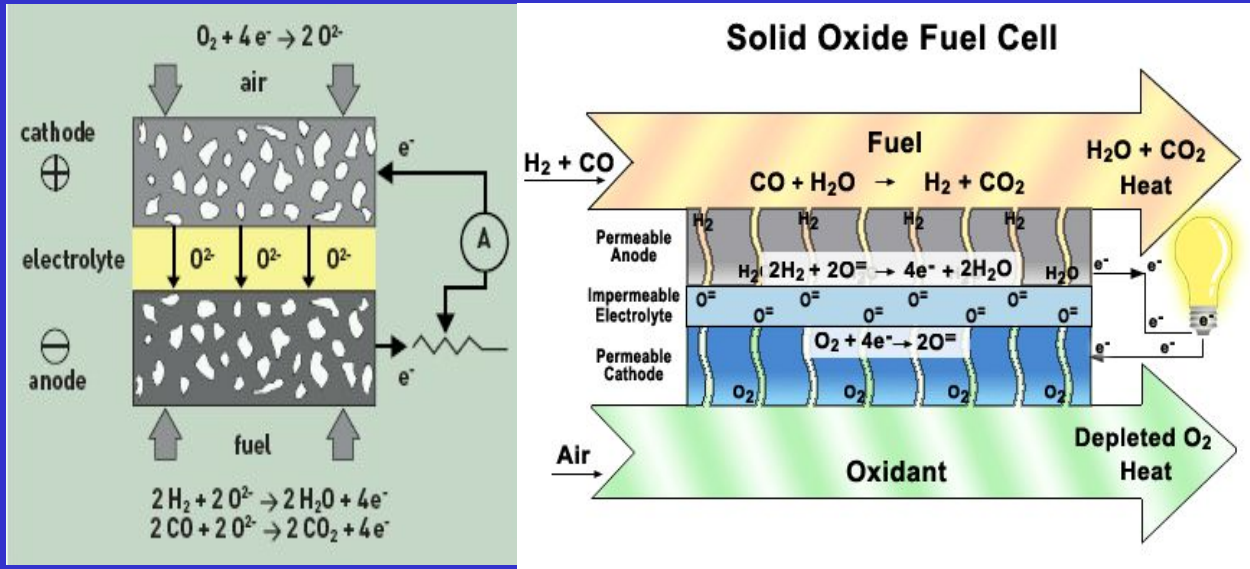
**PROMAG**  
2007-2010

Project Coordinator  
K.U.Leuven

Plane parallel and perpendicular to electrode

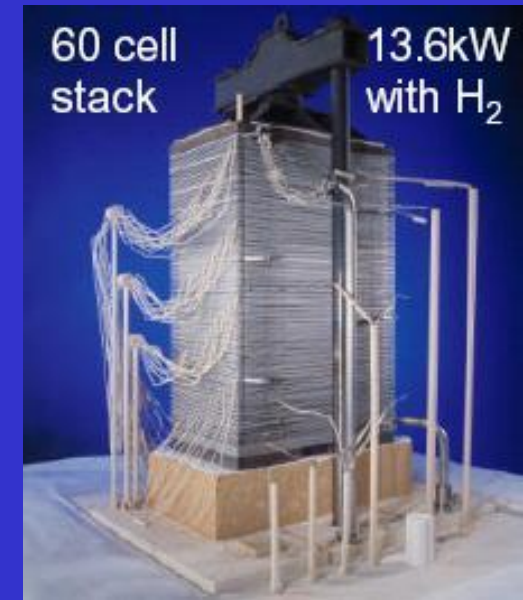
# EPD of SOFC

## Novel Materials for Silicate-Based Fuel Cells



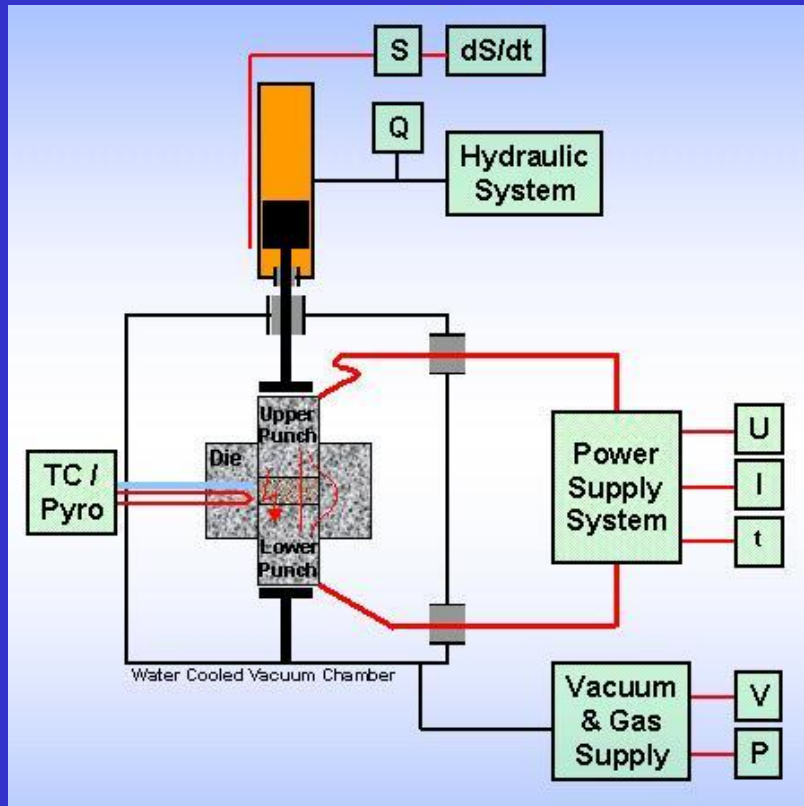
## Processing of Solid Oxide Fuel Cells

- Nanopowder synthesis
- Colloidal processing of half cells
- Sintering of half cells



# Field Assisted Sintering Technology (FAST)

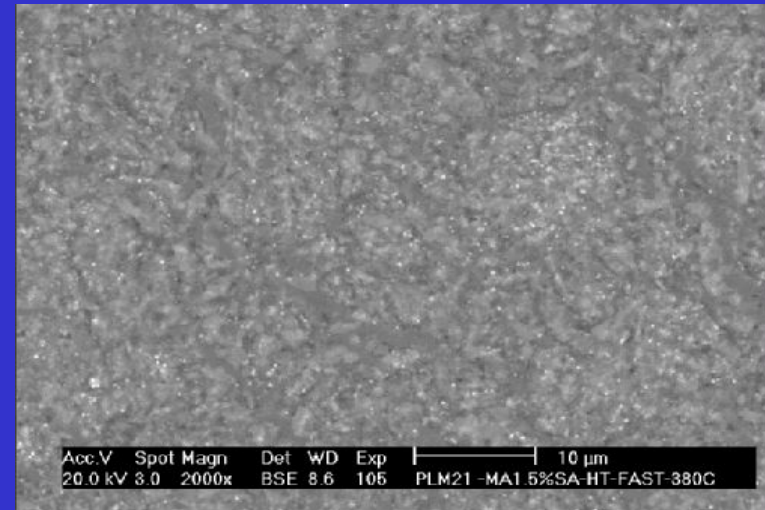
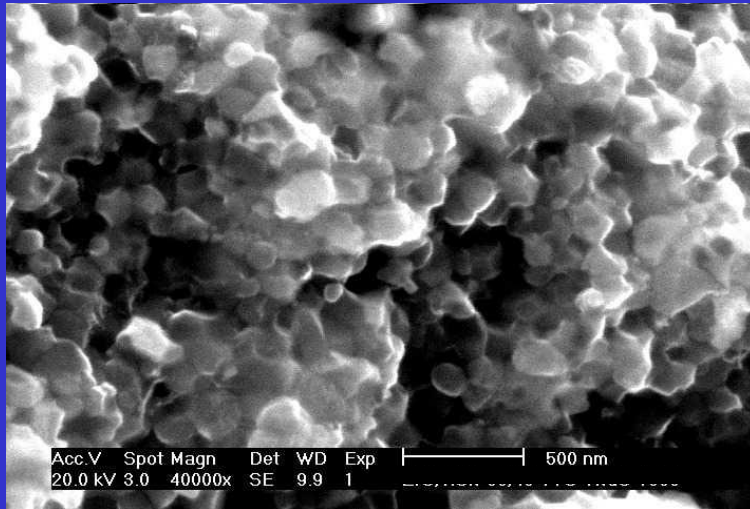
Field assisted sintering technology for the densification of nanostructured powders and fabrication of functionally graded materials



- Technology development
- Experimentation
- Thermo-electrical modelling
- Thermo-electrical-mechanical modelling
- Superplastic deformation

# Field Assisted Sintering Technology (FAST)

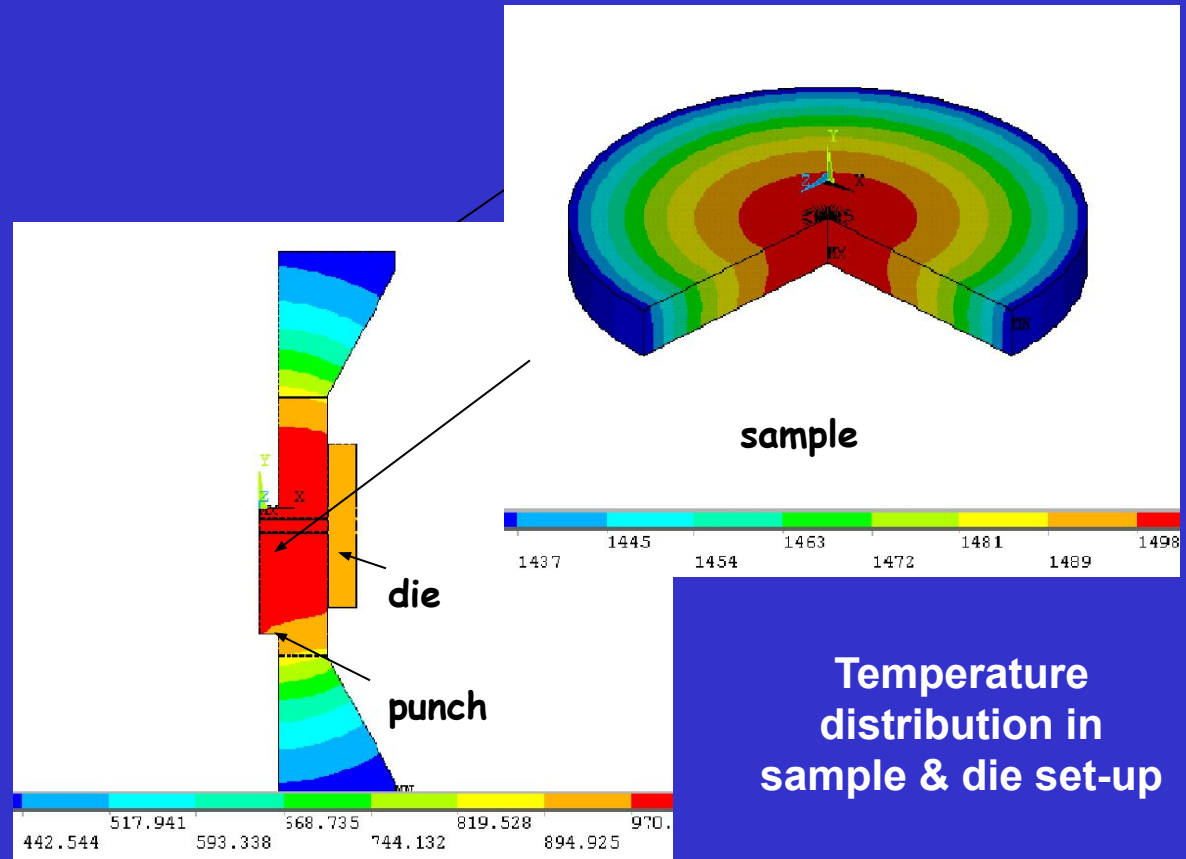
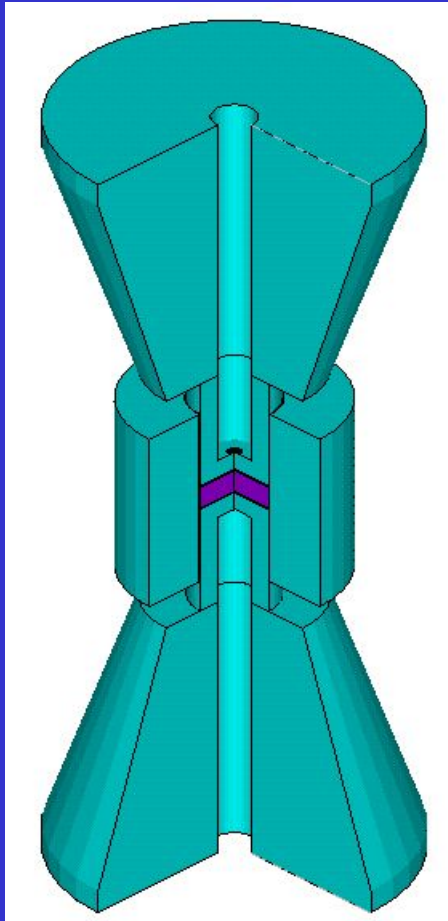
**Ceramic and ceramic-metal nanocomposites (cermets) fabricated from nanopowders**



**Nanostructured aluminium based alloys** from rapid solidification or mechanical alloying

# Field Assisted Sintering Technology (FAST)

## FE-modelling of the temperature distribution during FAST



# High temperature equipment

## SPS



### Equipment Properties:

pulsed electric current: 0 – 8000 A  
pulse/pause time combinations:

0 - 255 ms

force: max. 250 kN

heating rate: up to 1000°C/min

heating cycle duration: 10 -30 min  
(incl. heating-cooling)

max temperature > 2200°C

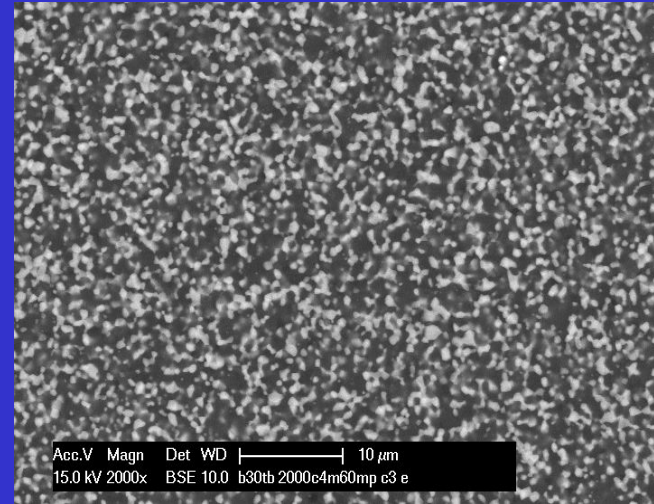
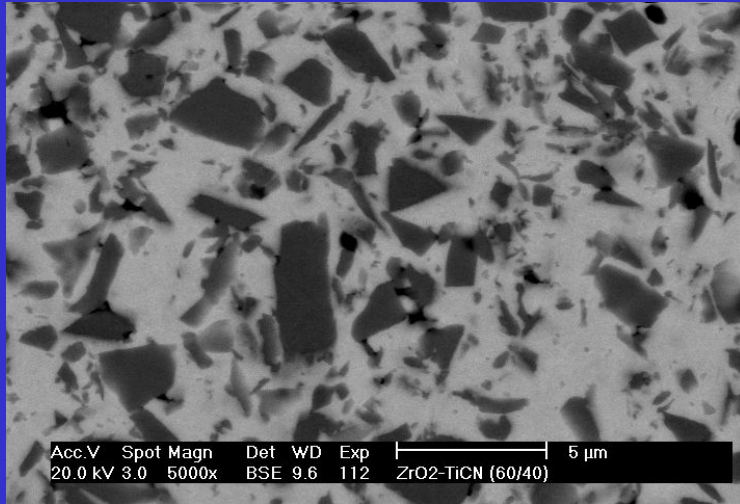
min controllable temperature = 150°C

**Materials :** Al-alloys, intermetallics, steel, ZnSe, ITO, borides, carbides, nitrides, Cu<sub>3</sub>Sn, oxides, electroceramics, BaTiO<sub>3</sub>, cermets, cemented carbides, ceramic composites, tungsten, etc.



# Electro-conductive ceramic composites

For electrical discharge machining (EDM) and wear applications

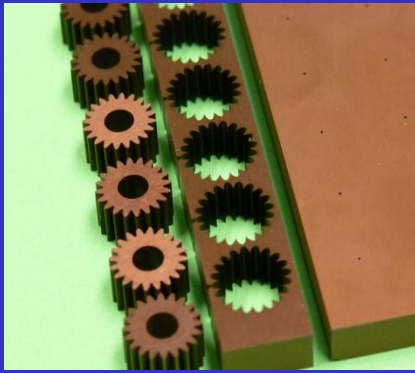


<http://www.mtm.kuleuven.be/Research/GBOU-IWT/spark/index.html>

<http://www.moncerat.org>

# Development of ceramic composites

## Electrical discharge machined new composites



**Gears**



**Extrusion die insert**



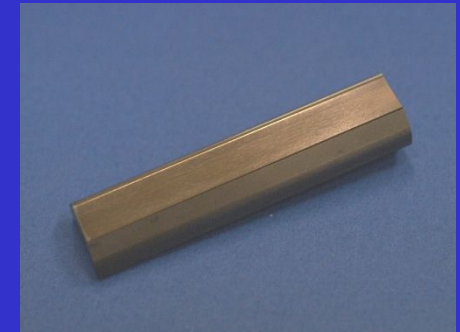
**Attritor disc**



**Lens mould insert**



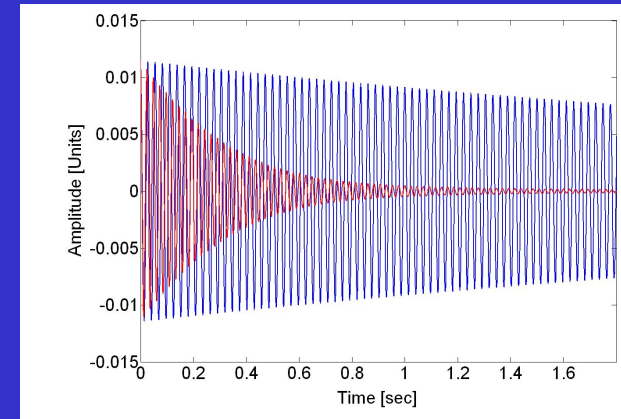
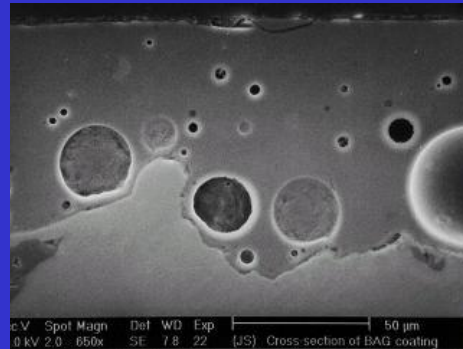
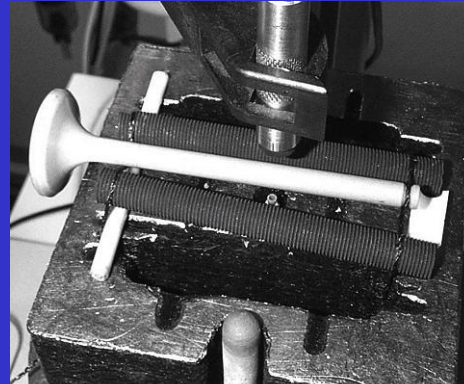
**Injection moulding tool**



**Fine blanking tool**

# Elastic and internal friction properties of materials

## Measuring of resonance frequency and damping

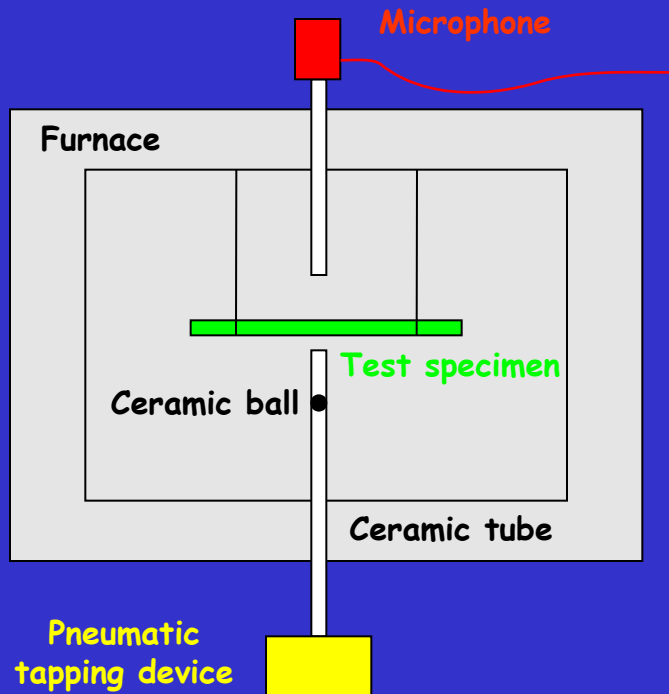


**Impulse  
Excitation  
Technique  
(IET)**

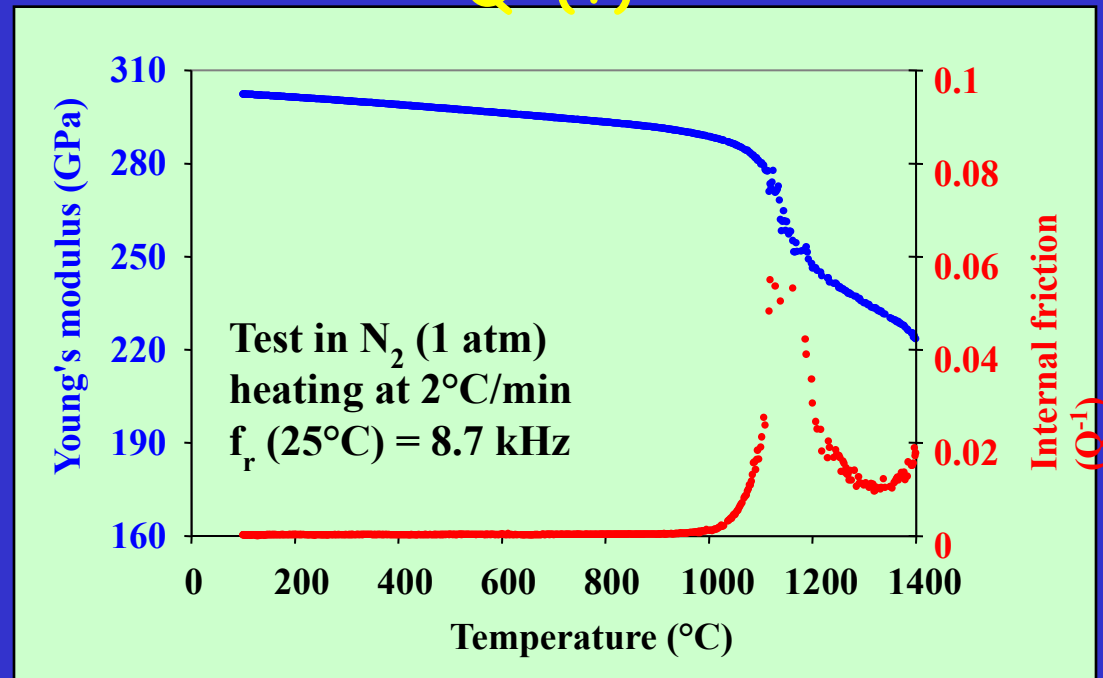
⇒ Measurement of  $E$ ,  $G$ ,  $\nu$ , and  $Q^{-1}$  at RT  
⇒ Measurement of  $E$  and  $Q^{-1}$  at elevated  $T_{app}$   
Applicable to monoliths, coatings and laminates

# Elastic and damping properties of materials

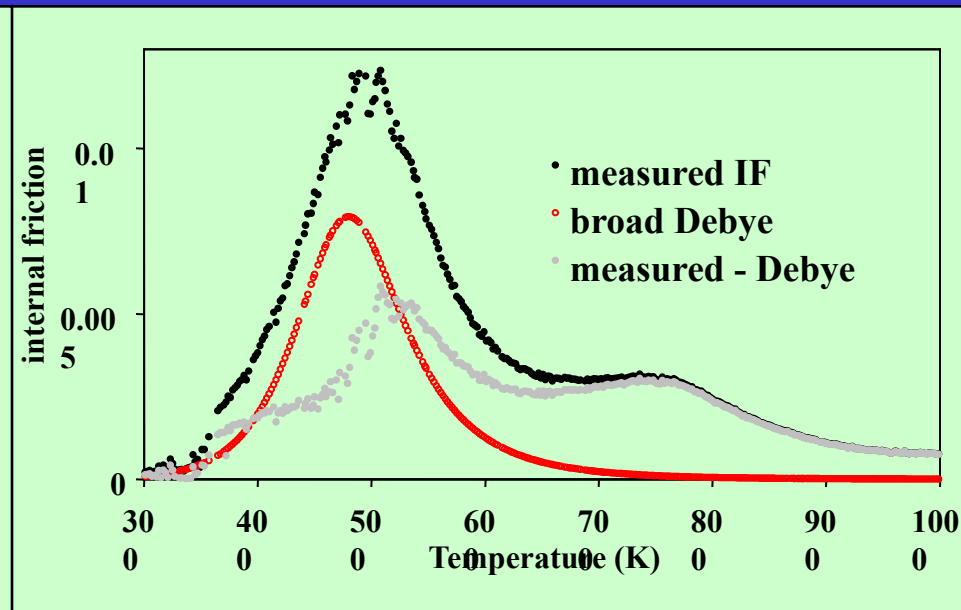
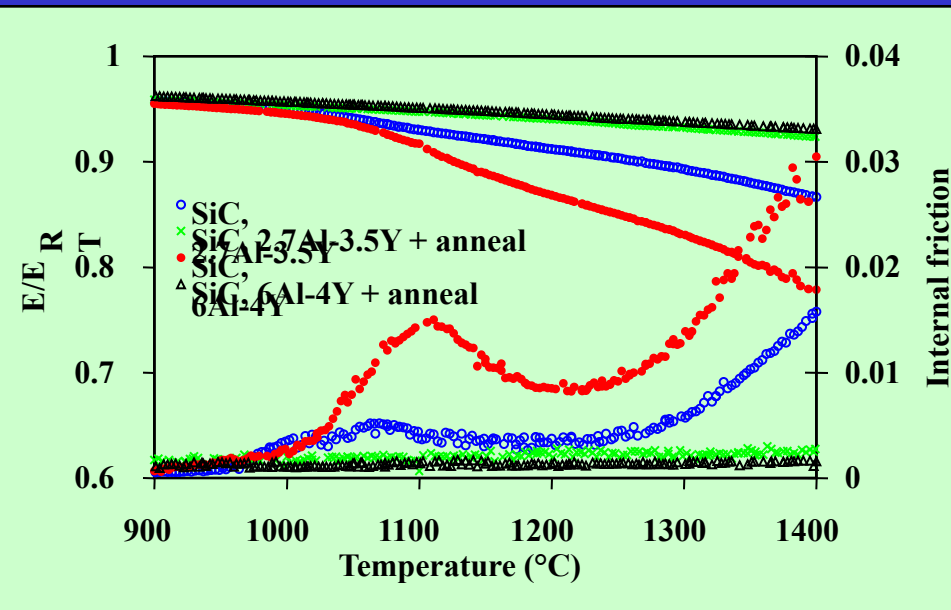
## Schematic of an IET-furnace



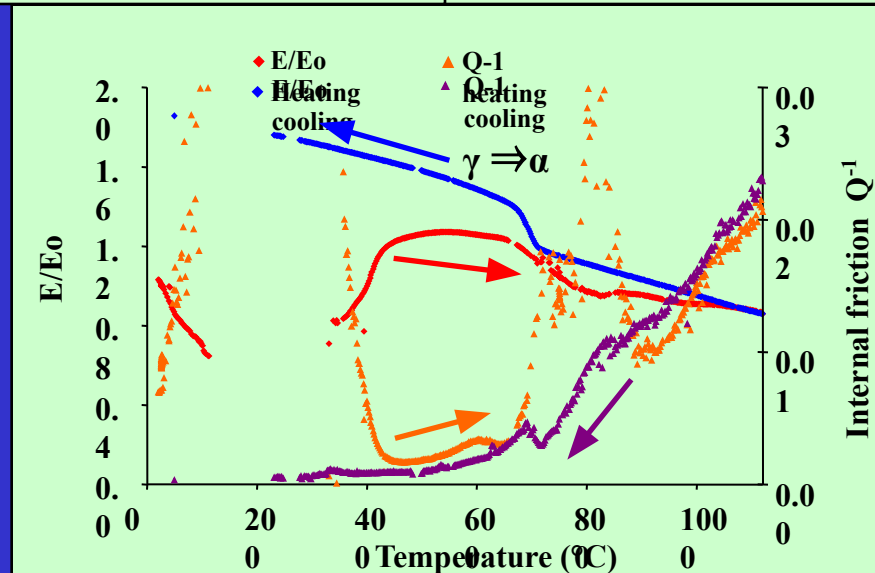
$\text{Si}_3\text{N}_4$  result :  $f_r$  or  $E(T)$  and  $Q^{-1}(T)$



# Elastic and damping properties of materials



Hot pressed SiC

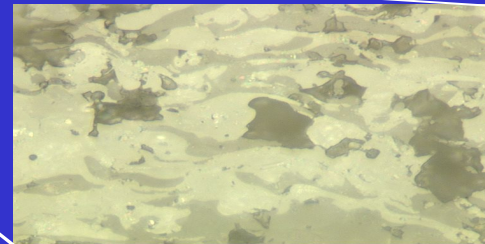
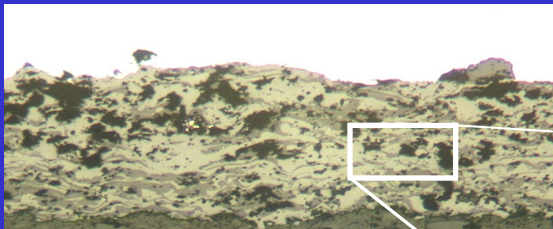
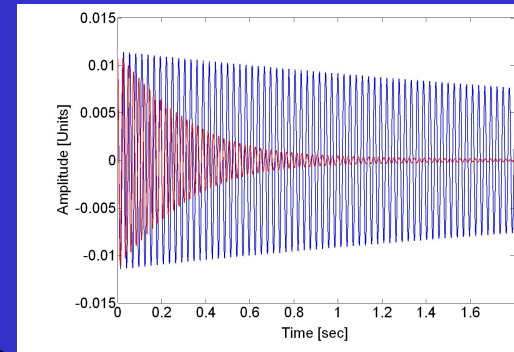
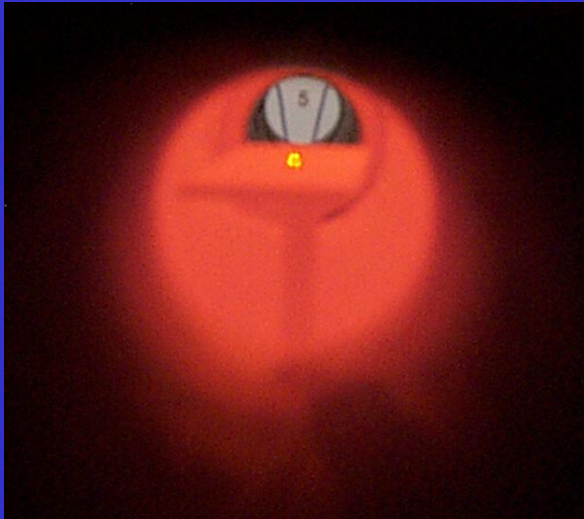


Sintered 2Y-TZP

Sintering PM steel

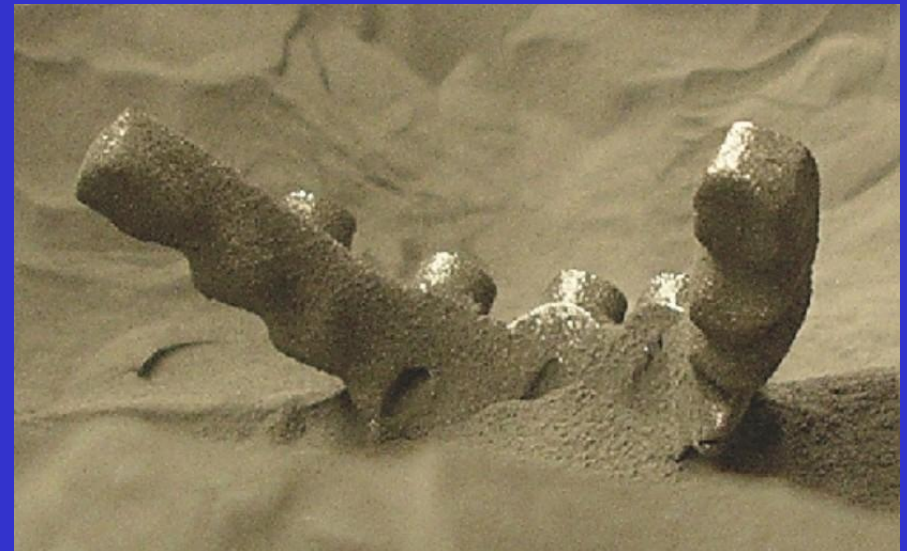
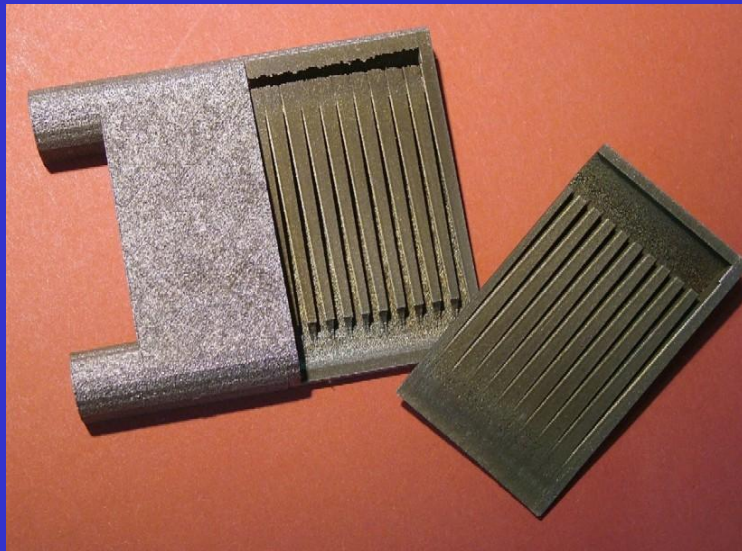
# Elastic and damping properties of materials

## Structural Integrity of Ceramic Multilayers and Coatings”



# Selective laser sintering and melting

Direct rapid manufacturing of metallic and ceramic parts

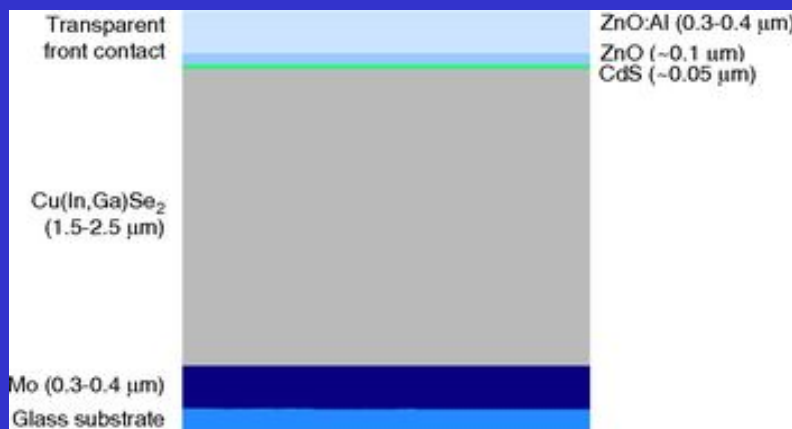


SBO project: **DiRaMaP** (2008-2012)

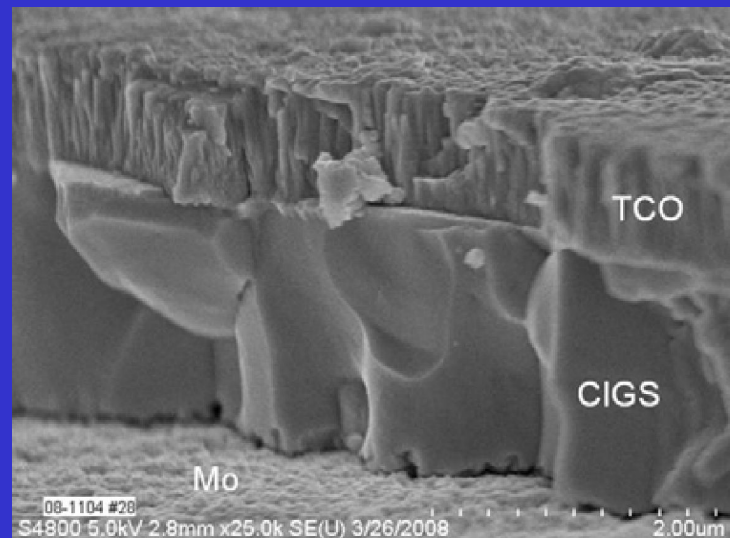
Project Coordinator:  
PMA, K.U.Leuven

# Solution Deposition Technologies for CIGS and TCO

- Powder-based opposed to vacuum sputtered photovoltaics
- Selenisation studies of Cu(In,Ga)-Selenides
- Assessment of fast selenisation processes
- Rapid annealing processes of transparent conductive oxides (TCO)



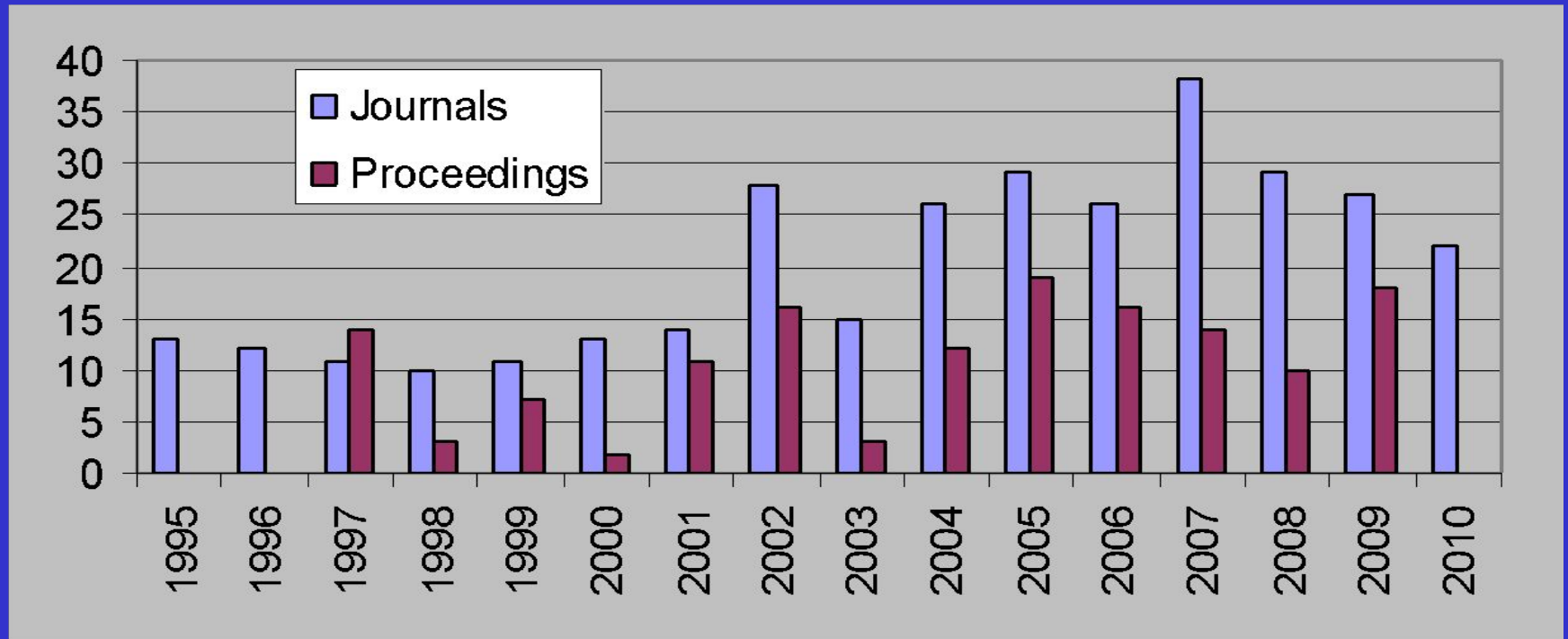
Cross-section of Cu(In,Ga)Se<sub>2</sub> solar cell



SIM project: **SoPPoM** (2010-2014)



# Publications ceramics research group



# Ceramics research group

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