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TECH2FAB

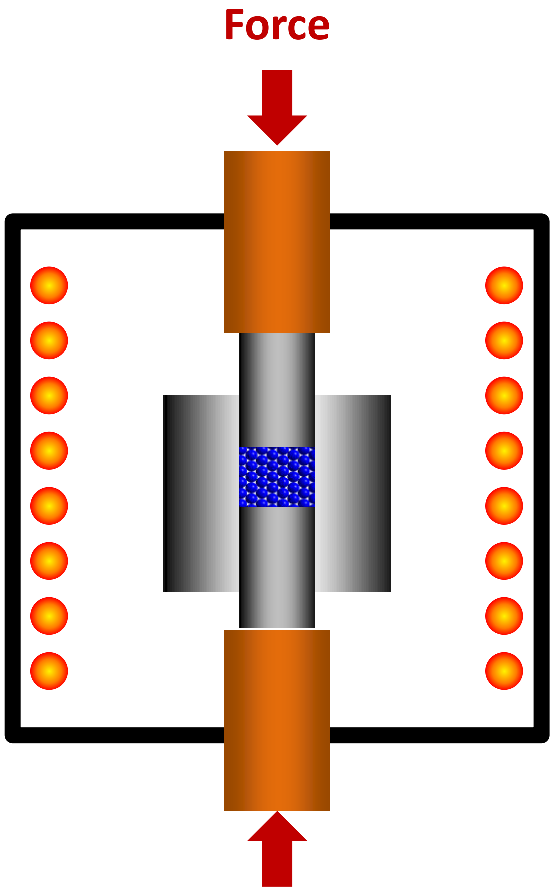
Field assisted sintering techniques
SPS and Flash Sintering

Jean-Pierre Erauw

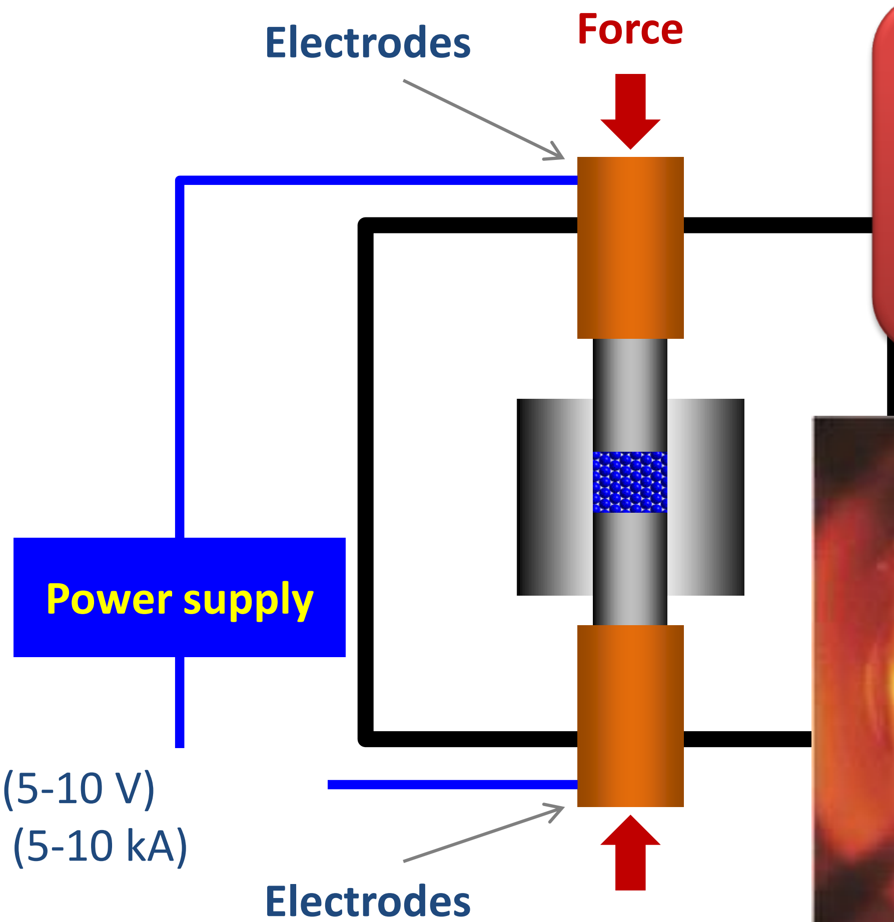
Belgian Ceramic Research Centre

SPARK PLASMA SINTERING - SPS

Principle

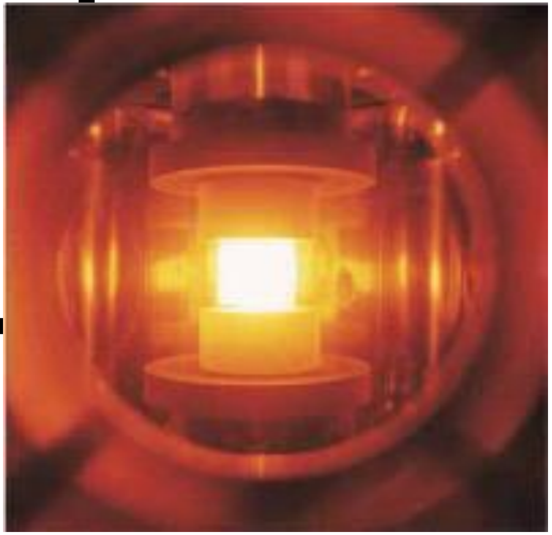


Principle



Low voltage (5-10 V)
High current (5-10 kA)

Direct Joule heating
of the die
and **of the compact**
(if conductive)



Source FCT Systeme

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Recognised merits of SPS

Advantages of the technique

- Applicable to a large range of materials, conductive as non-conductive
- Applicable to materials difficult to sinter
- Very short thermal cycles
- Lower sintering temperatures
  - leads to reduction of power consumption
  - phases integrity can be maintained
  - offers potential microstructural control (nanomaterials ...)

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These merits stem from

Thermal effects

- very high heating rates ➔ surface diffusion mechanisms inhibited
- Important local temperature gradients ➔ thermal diffusion (Ludwig-Soret effect)
- Macroscopic thermal grandients ➔ thermomechanical stresses amplifying the creep mechanisms
- Heterogeneous local distributions of temperature ➔ local melting phenomena at contact points

Athermal effects

- electromigration / enhancement of diffusion in ionic conductors
- electroplasticity phenomena
- dielectric breakdown of oxide layer at the surface of the grains and enhanced formation of defects at GB
- …

These merits stem from

Thermal effects

- very high heating rates ➔ surface diffusion mechanisms inhibited
- Important local temperature gradients ➔ thermal diffusion (Ludwig-Soret effect)
- Macroscopic thermal grandients ➔ thermomechanical stresses amplifying the creep mechanisms
- Heterogeneous local distributions of temperature ➔ local melting phenomena at contact points

Athermal effects = Field effect

Field Assisted Sintering technology (FAST)

Some limitations remain

Advantages of the technique

- Applicable to a large range of materials, conductive as non-conductive
- Applicable to materials difficult to sinter
- Very short thermal cycles
- Lower sintering temperatures
  - leads to reduction of power consumption
  - phases integrity can be maintained
  - offers potential microstructural control (nanomaterials …)

Limitations of the technique

- Limited geometrical complexity of the parts
- Batch process – further improvement of the productivity is needed
- Homogeneity often remains an issue (especially for large parts)

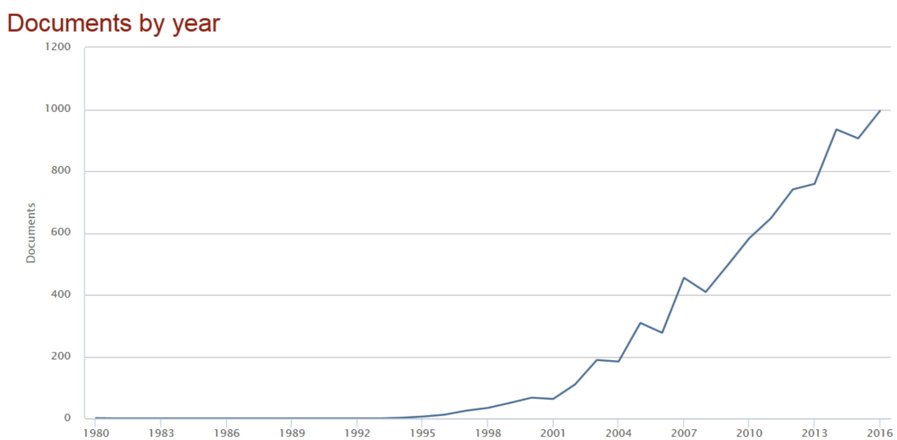
Nevertheless SPS attracts a large interest

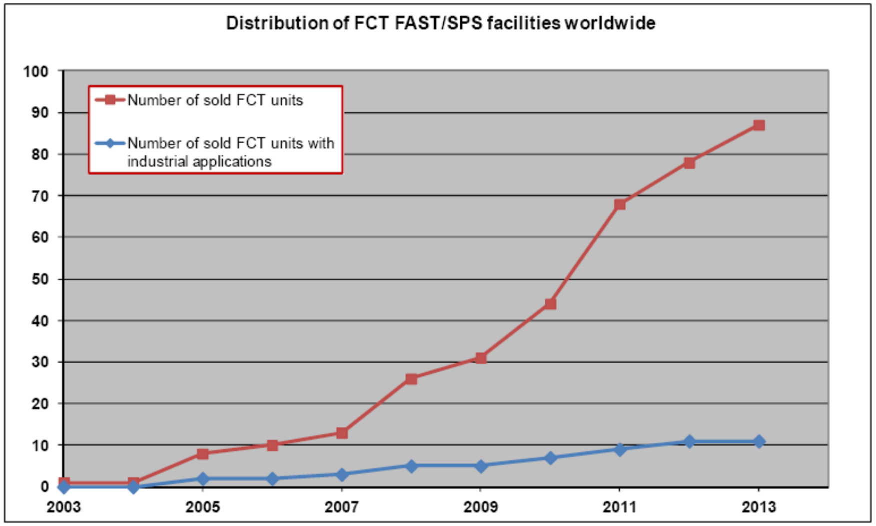
from the academic world

as well as from industry

Source : Scopus

Keyword(s) : Spark Plasma Sintering





SPARK PLASMA SINTERING @ BCRC

Spark Plasma Sintering at BCRC

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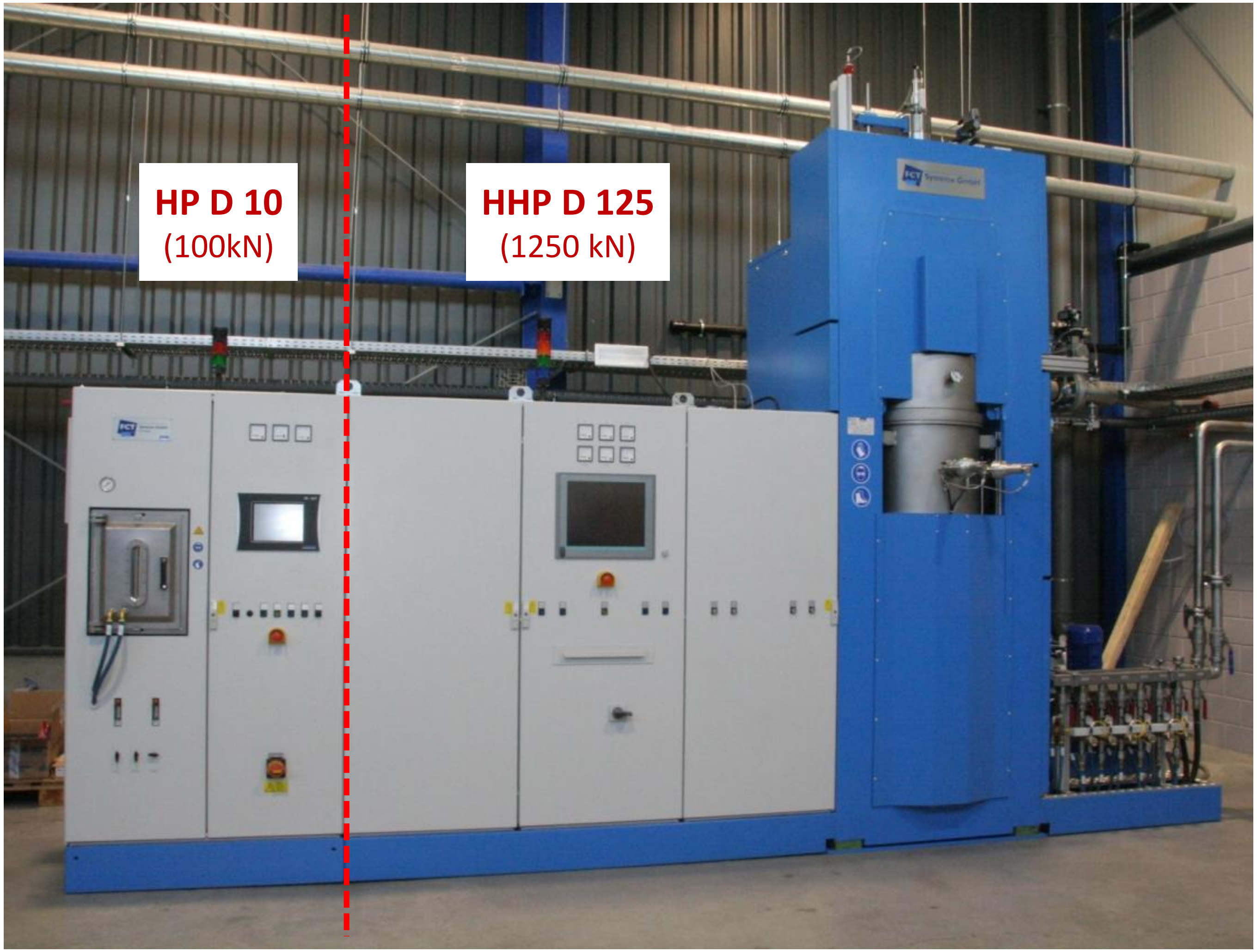
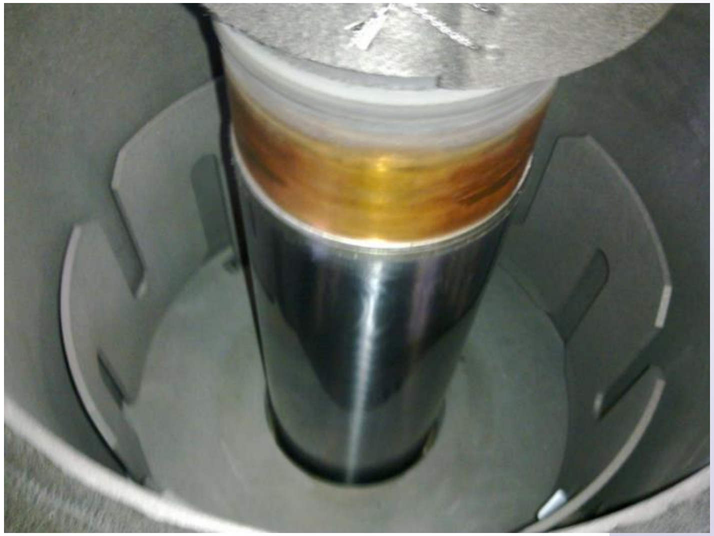
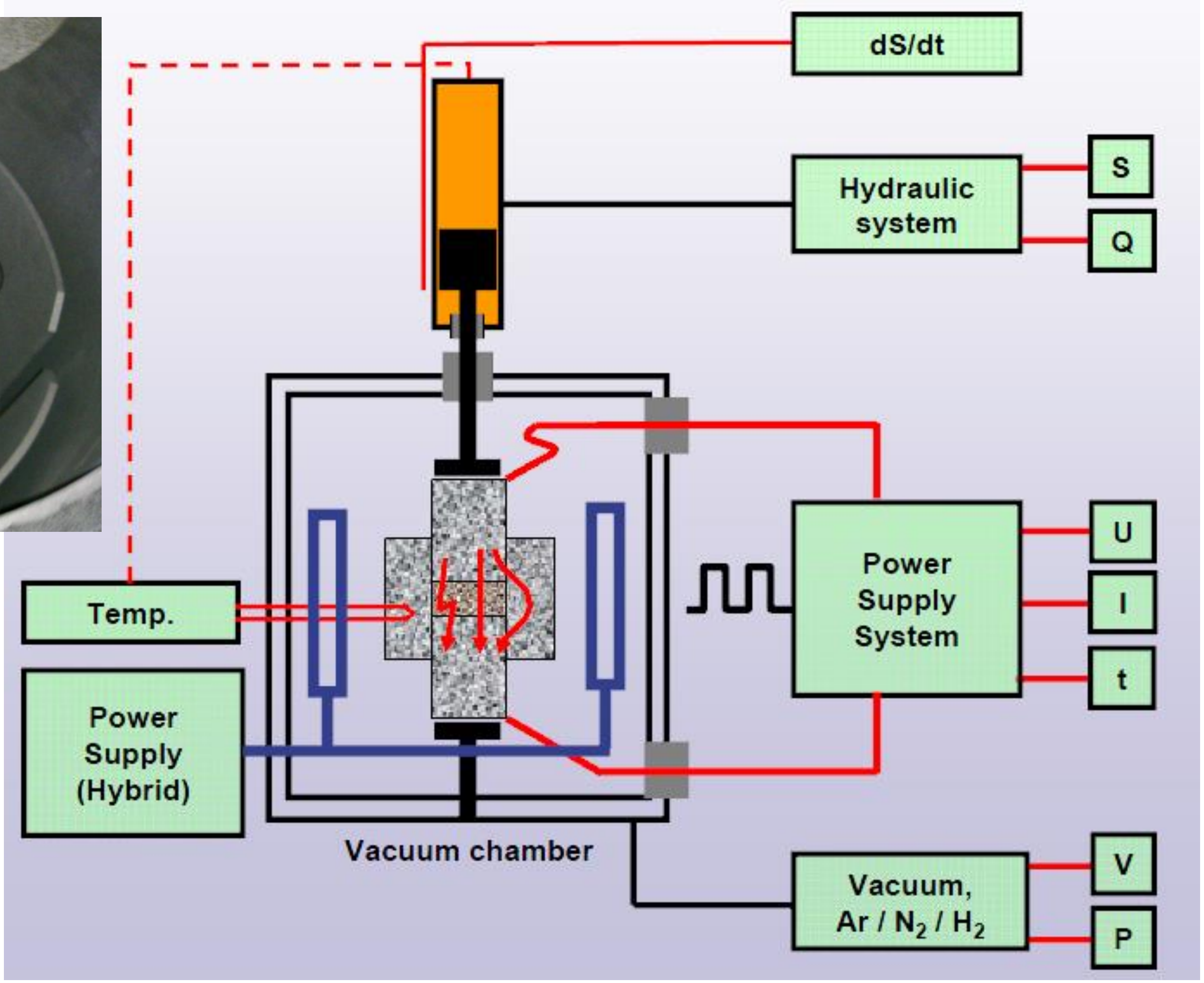
Twin-Hybrid system - HHP D 125-SP (FCT Systeme GmbH)



Spark Plasma Sintering at BCRC

Twin-Hybrid system - HHP D 125-SP (FCT Systeme GmbH)





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Our research topics

Basic research	• Insight in the fundamental mechanisms • Process modelling
Innovative Materials	• Wear resistant — Cermets and composites • (Electro) optics — YAG, Ba(Sr)TiO_3 • Thermoelectrics — (Semi) Heussler compounds • « Polyvalent » — MAX phases
Technical Improvements	• Upscaling • Shape complexity

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- Process modelling

Innovative Materials

• Wear resistant	Cermets and composites
• (Electro) optics	YAG, Ba(Sr)TiO_3
• Thermoelectrics	(Semi) Heussler compounds
• « Polyvalent »	MAX phases

Technical Improvements

- Upscaling
- Shape complexity

Project(s)	PhD thesis (First DOCA DGO6), Cerapide (ERDF) Vulcanus traineeship
Contributors	M. Demuynck, V. Dupont, J.P. Erauw, Y. Hasagawa T. Van Herck
Collaboration(s)	KULeuven, UMONS-FPMs

INSIGHT IN THE FUNDAMENTAL MECHANISMS

First DOCA - SPS

SPS vs conventional HP

Real advantages and limitations?

Influence of thermal and electrical properties?

Available online at www.sciencedirect.com
SciVerse ScienceDirect
Journal of the European Ceramic Society 32 (2012) 1957–1964
www.elsevier.com/locate/jeurceramsoc

Densification of alumina by SPS and HP: A comparative study

Maryse Demuynck [a,*], Jean-Pierre Erauw [a], Omer Van der Biest [b], Francis Delannay [c], Francis Cambier [a]

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Influence of conductive secondary phase on thermal gradients development during Spark Plasma Sintering (SPS) of ceramic composites

Maryse Demuynck [a,*], Jean-Pierre Erauw [a], Omer Van Der Biest [b], Francis Delannay [c], Francis Cambier [a]

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[c] EMPA UCLouvain, Place Sainte Barbe 2, Louvain-la-Neuve, B-1348 Belgium

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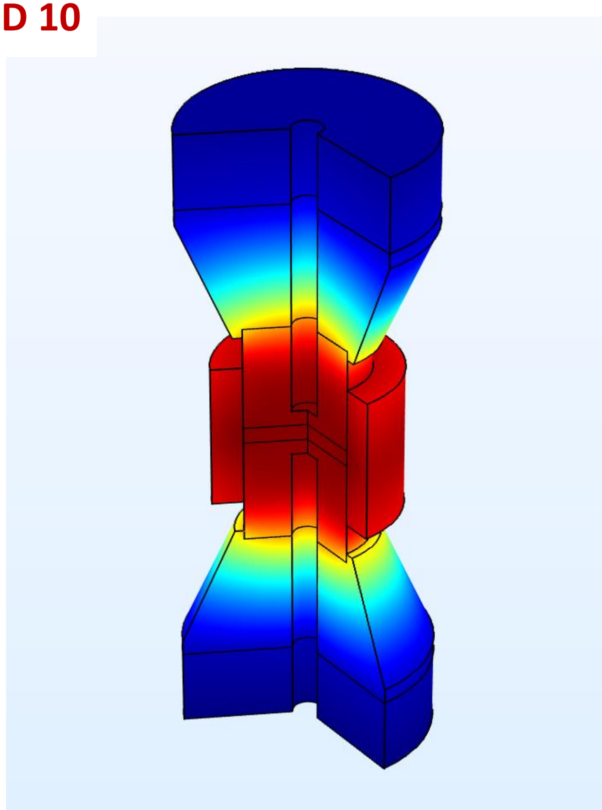
Project(s)	Cerapide (ERDF)
Contributors	M. Cambier, V. Dupont, J.P. Erauw, T. Van Herck
Collaboration(s)	UMONS-FPMs

MODELLING THE PROCESS

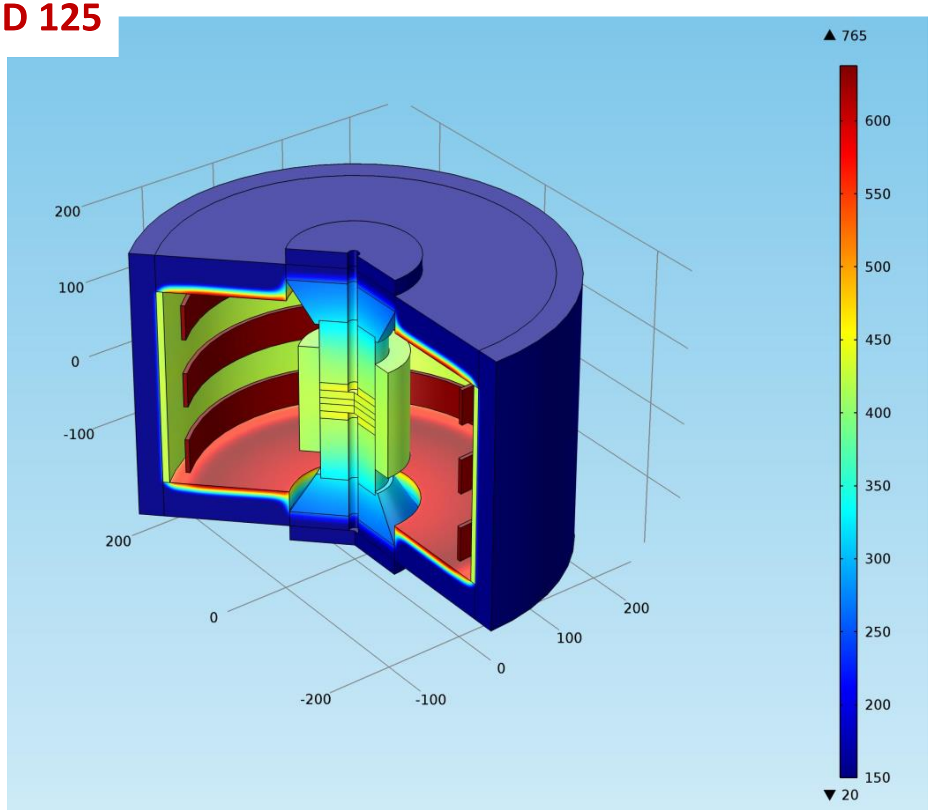
Finite element modelling

- helps clarifying the experimental observation
- supports the optimization of the tools (dies, plunger…)
- Supports the optimization of the sintering cycle (e.g. in hybrid mode)

HP D 10



HHP D 125

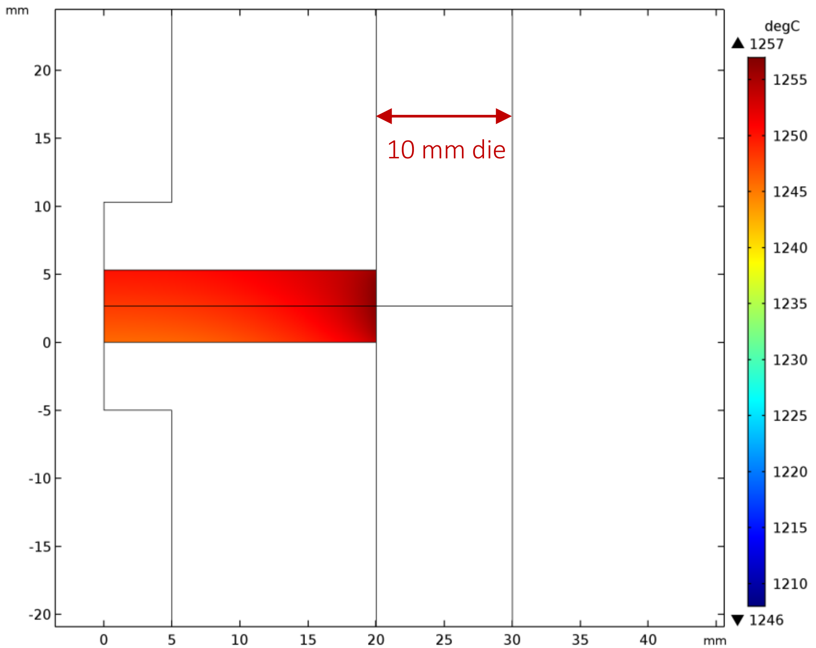


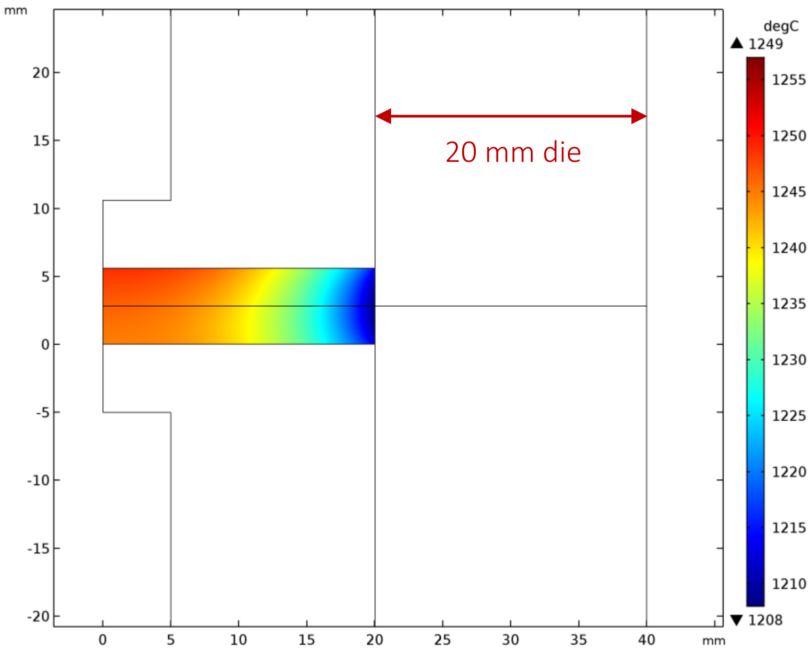
Die thickness optimization

Zirconia – Ø 40mm – Temperature distribution after 5’@1250°C

T_{min} : 1246 °C T_{mean} : 1251 °C
T_{max} : 1257 °C DT_{max} : 11 °C

T_{min} : 1208 °C T_{mean} : 1231 °C
T_{max} : 1249 °C DT_{max} : 41 °C





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Our research topics

Basic research	• Insight in the fundamental mechanisms • Process modelling	
Innovative Materials	• Wear resistant • (Electro) optics • Thermoelectrics • « Polyvalent »	Cermets and composites YAG, $Ba(Sr)TiO_3$ (Semi) Heussler compounds MAX phases
Technical Improvements	• Upscaling • Shape complexity	

Project(s) STEELFSW & FSW-PME (DGO6), contractual projects

Contributors L. Boilet, M. Demuynck, V. Dupont, J.P. Erauw

Collaboration(s) Belgian Welding Institute, CEWAC

WEAR RESISTANT MATERIALS

WC-Co-cBN composites

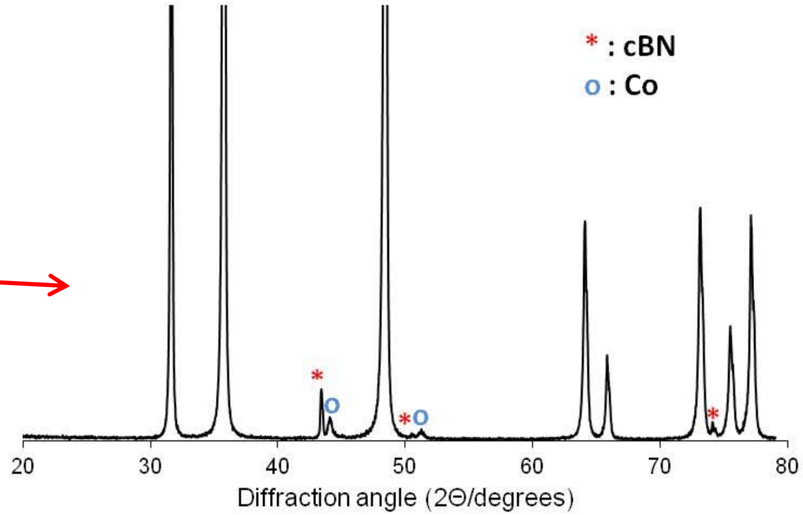
- Addition of cobalt to WC → increases sinterability and fracture toughness
  → decreases hardness and wear resistance
- cBN is the second hardest material after diamond
- Addition of cBN to cemented carbides → increases hardness
  → decreases sinterability
- Thereupon, the phase transformation of cBN to its hexagonal phase limits the sintering temperature .

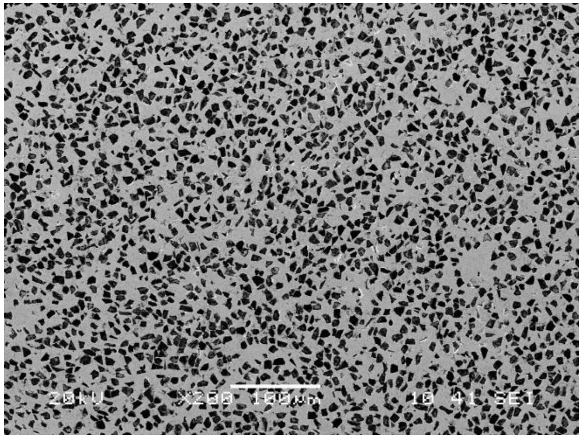
Combination of cBN addition and SPS technique
(with lower sintering temperature)
↓
Materials with
enhanced mechanical properties

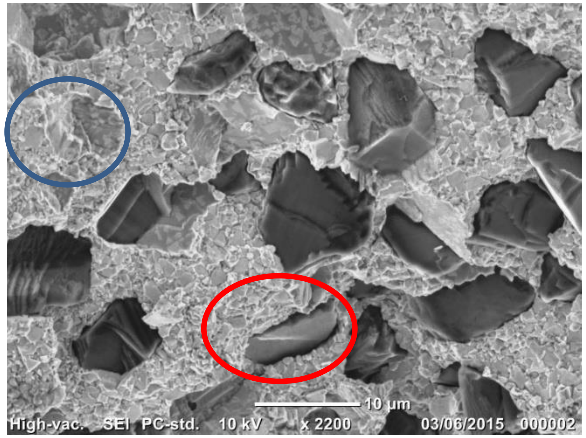
WC-Co-cBN composites

Optimization of the density by adjusting sintering parameters (T, P, dwell time)

	Temperature (°C)	Dwell time (min)	Pressure (MPa)	Relative density (%)
WC 6 - 0	1200	10	60	99,4
WC 6 - 15	1200	15	60	99,4
WC 6 - 25	1220	15	60	99,7
WC 6 - 35	1220	15	70	99,3
WC 12 - 0	1150	15	60	100
WC 12 - 15	1160	15	60	99,9
WC 12 - 25	1160	15	60	100
WC 12 - 35	1160	15	70	99,9



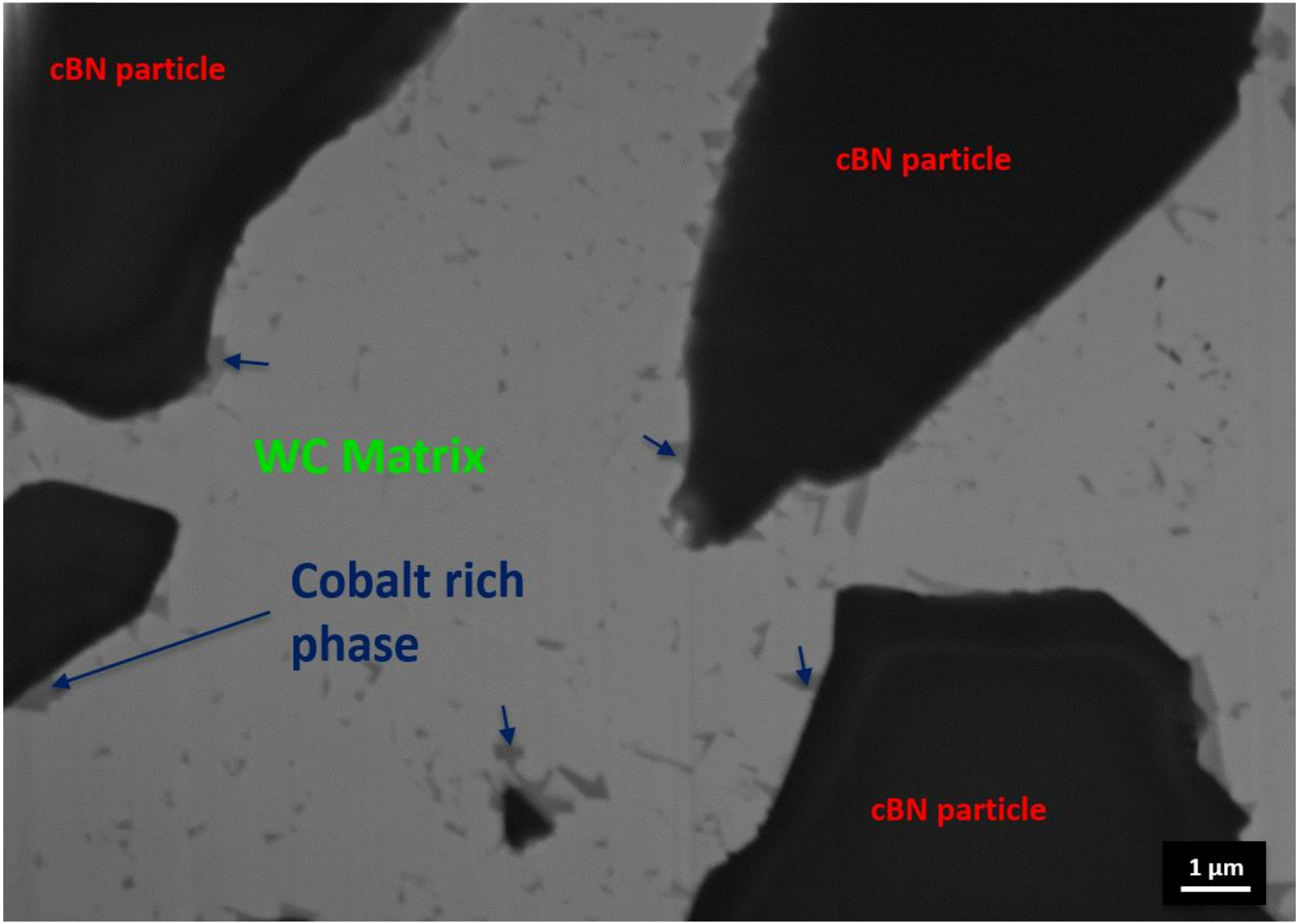




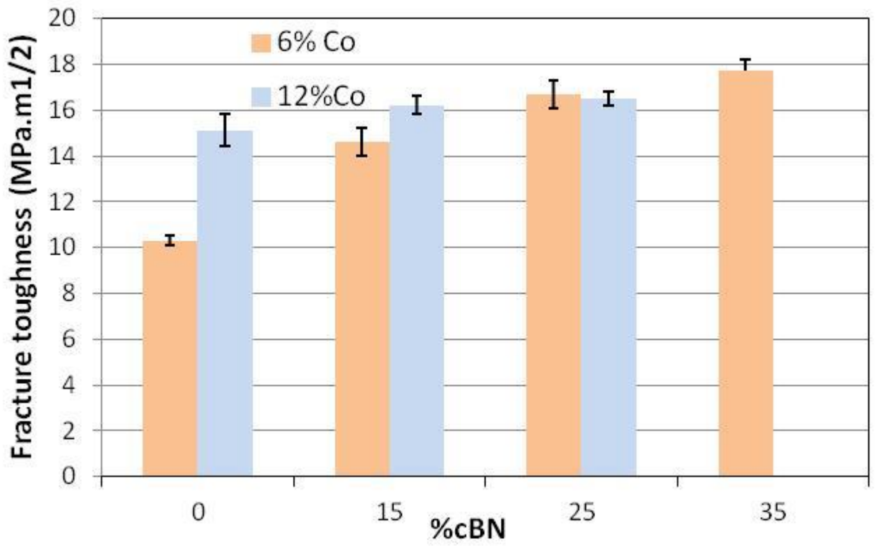
WC 12 - 35

- higher sinterability with 12% Co (densities ≅ 100%)
- hBN phase not detected
- homogeneous distribution of cBN in the WC-Co matrix
- Mix of intergranular and transgranular fractures

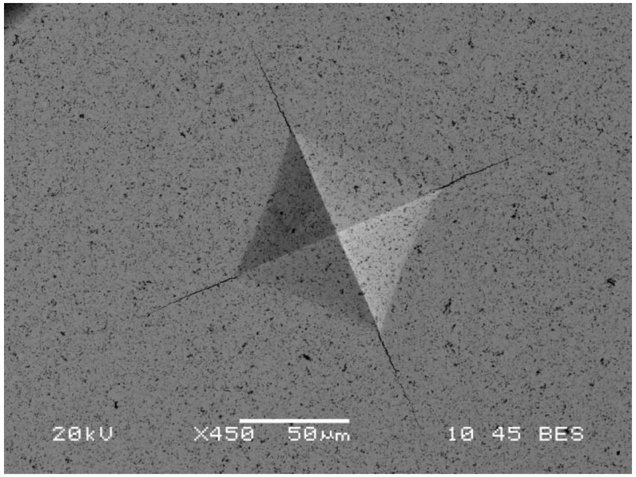
WC-Co-cBN composites



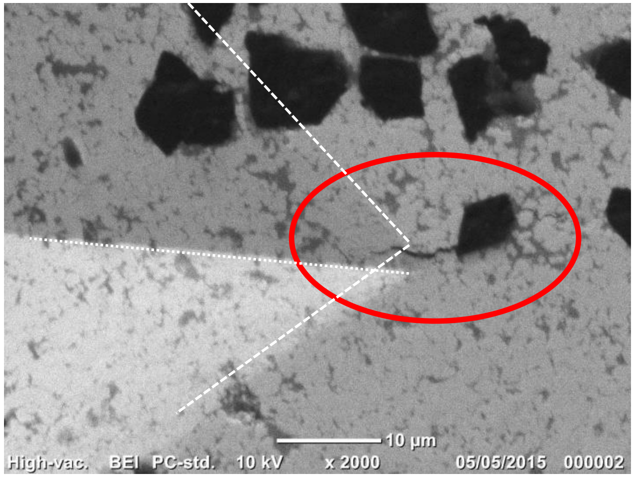
Mechanical properties



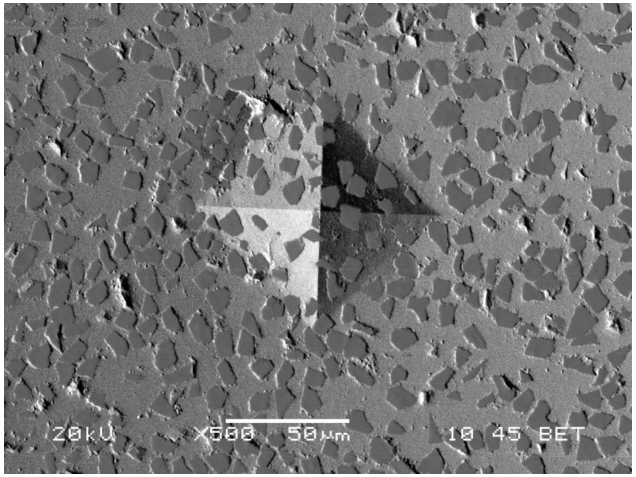
Increase of indentation fracture toughness due to crack deflection and/or arrest by cBN particles



WC 6-0

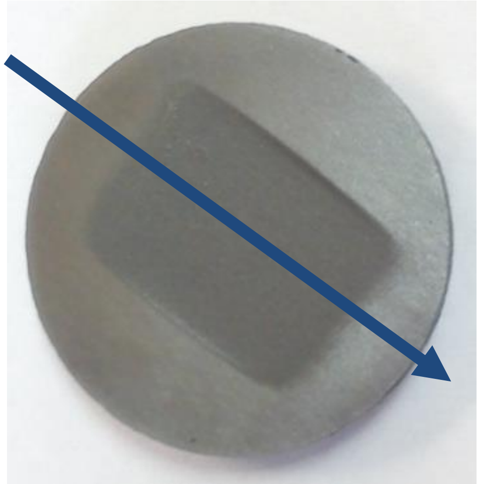


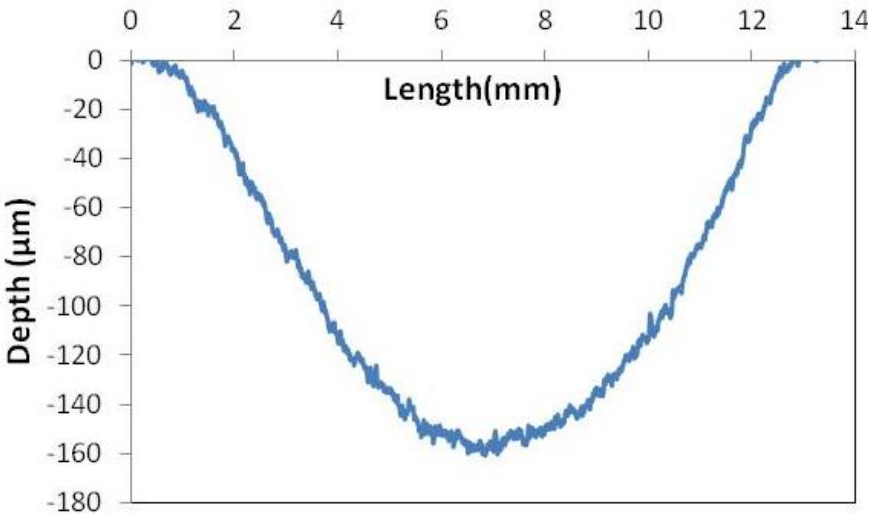
WC 12-15

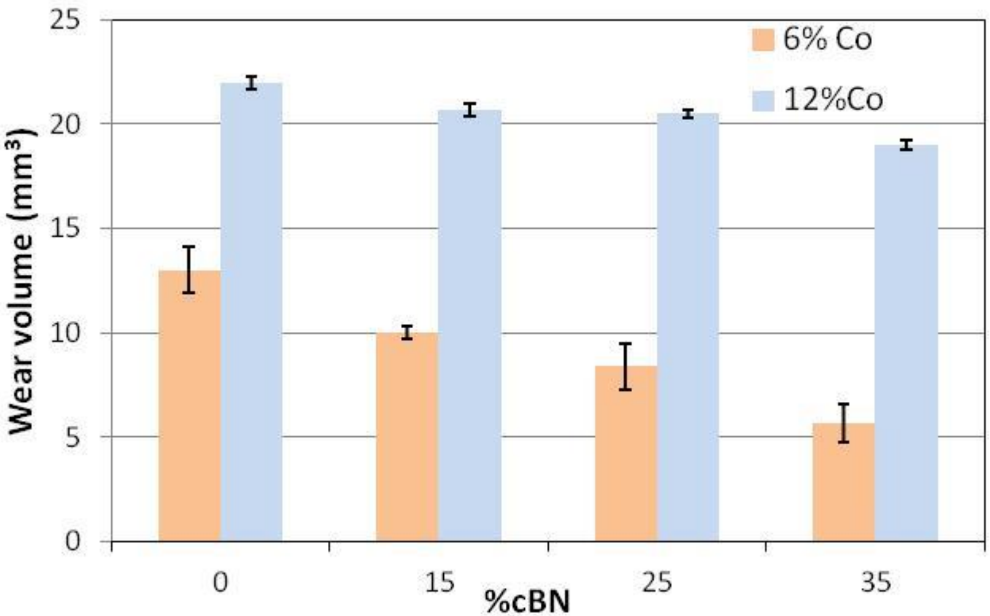


WC 12-35

Wear tests results



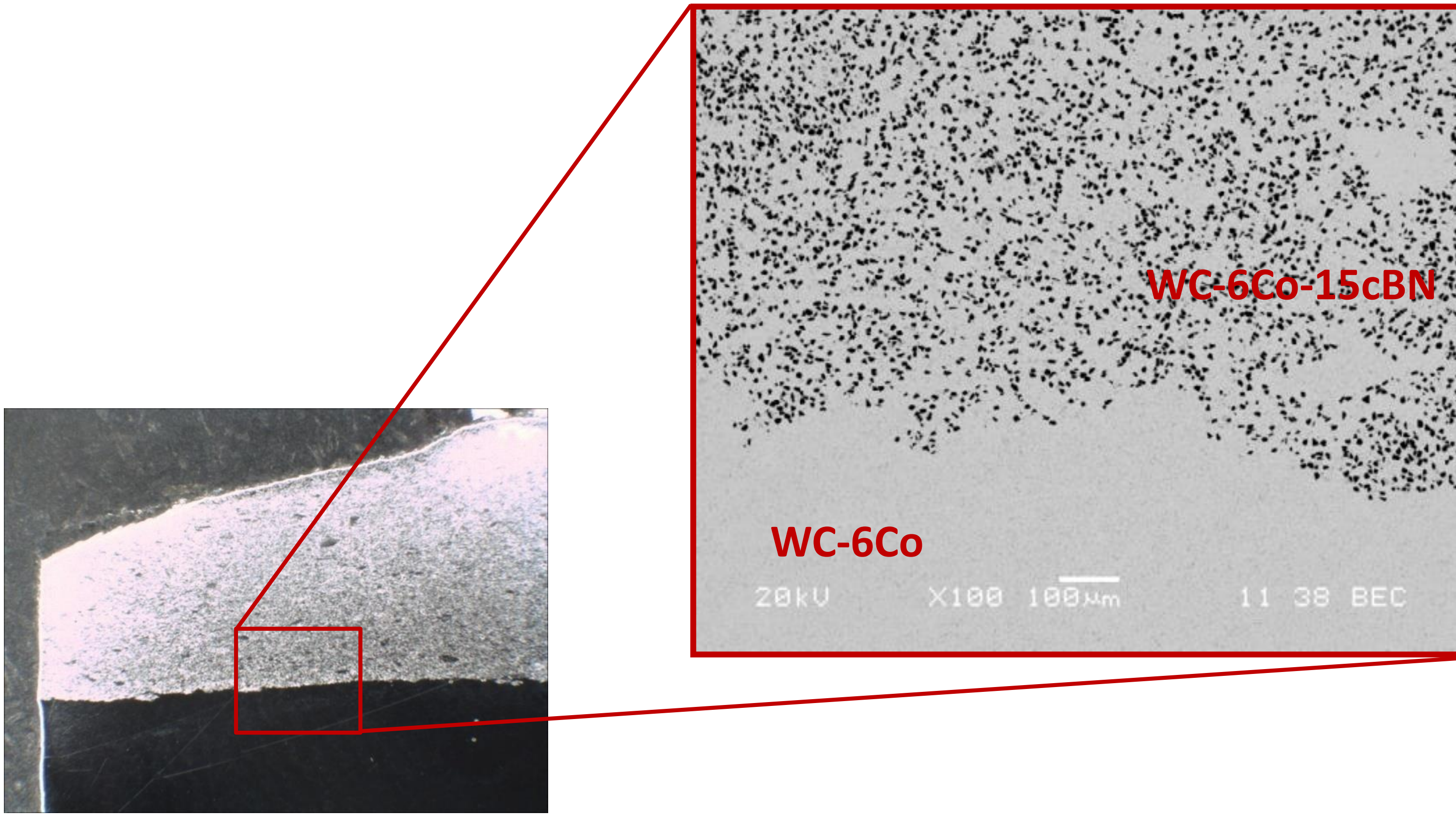




- ↓ Co volume → ↓ wear
- ↑ cBN volume → ↓ wear

WC-Co-cBN based FGM material

Development of a graded composition has been demonstrated



Project(s)	PhD thesis (First-DOCA DGO6)
Contributors	R. Moronta Perez, P. Aubry, L. Boilet
Collaboration(s)	University of Namur, Politecnico di Torino

(ELECTRO)OPTICS

Objectives

- Achieve high transparency on both YAG and $Ba(Sr)TiO_3$
- Achieve tunable optical properties in the specific case of $Ba(Sr)TiO_3$
- Identify SPS sintering conditions leading to full density while keeping the grain size small
- Identify, where needed, optimum pre-treatment of the raw materials and/or post-treatment of the compacts enabling an improvement of the optical/electrical performances

Commercial YAG powder

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Temperature influence

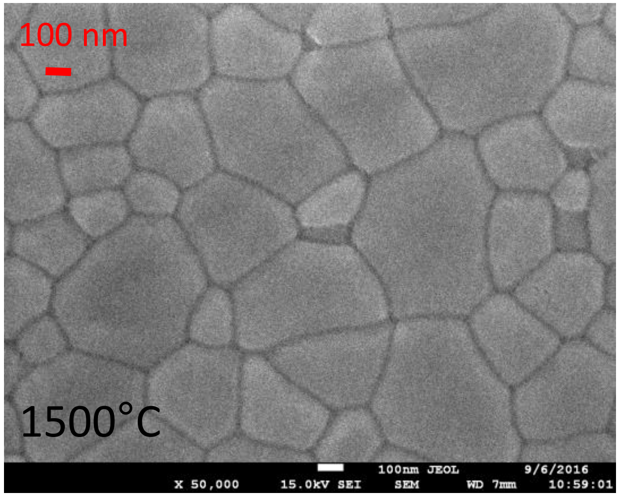
50MPa
100°C/min 1050°C
10 °C/min 1450 or 1500°C
15min

1450°C
ρ ~100%

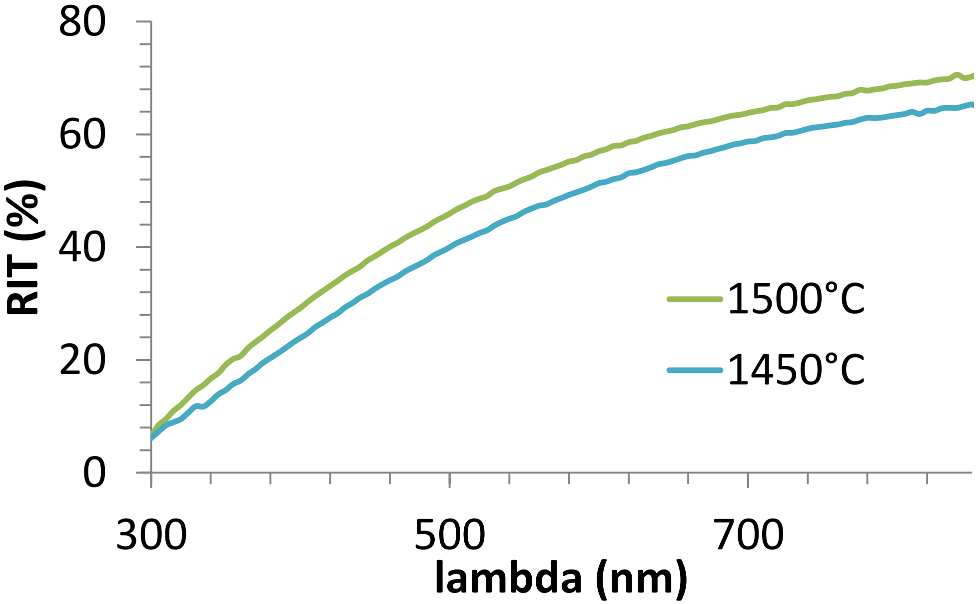
1500°C
ρ~ 100%

DRX- pure YAG

Slight coloration



d = (200-800nm)
No porosity



Commercial YAG powder

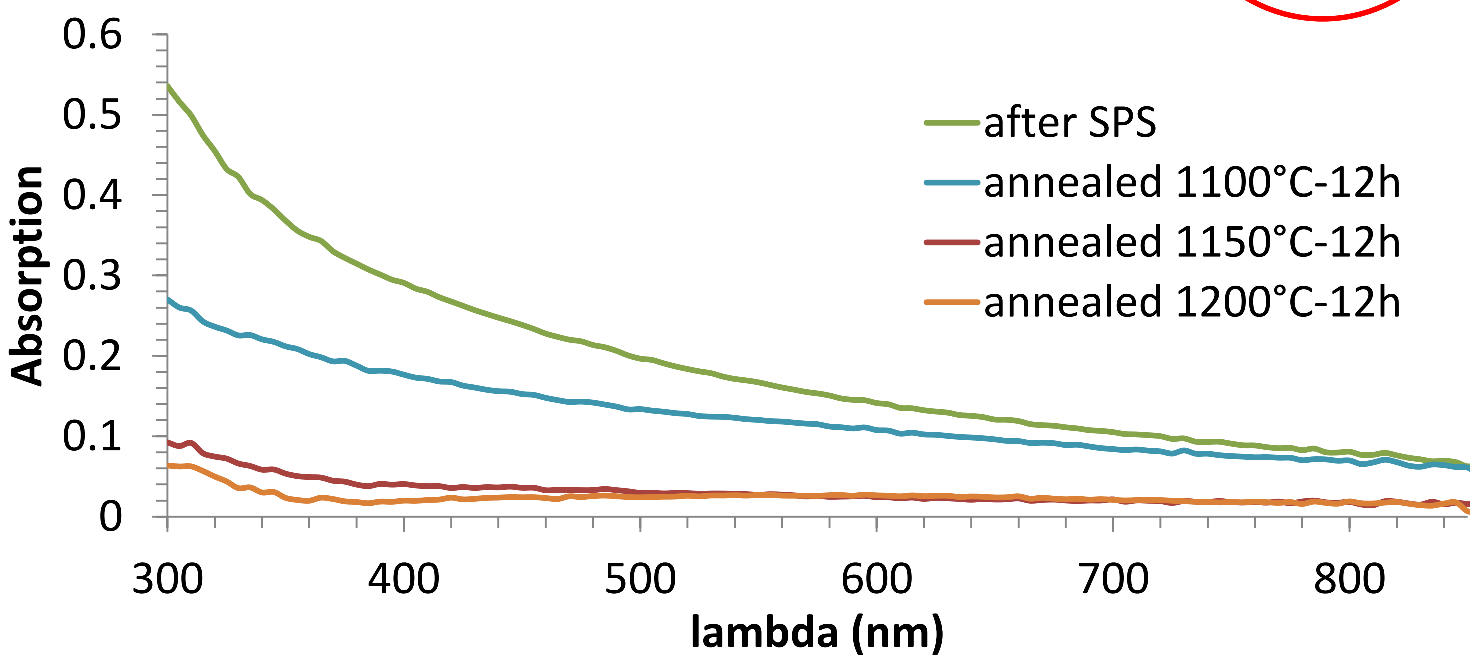
Post treatment

Cycle:
5°C/min
Dwell at T_{max} 12 h

Colouration ↓

After annealing

As produced | 1100°C-12h | 1150°C-12h | 1200°C-12h



Best results
TFT: 75%
RIT: 66%

Commercial $BaTiO_3$ powder

Optimization by dispersion

Dispersion and drying conditions

Isopropanol +Turbula BM 24h

Drying: rotavapor

After SPS

Better homogeneity

Grain size < 1µm

➡ Less agglomerates lead to a more homogenous compaction and exaggerated grain growth removal

Air annealing

Different annealing conditions tested (700-950°C for 1-12h)

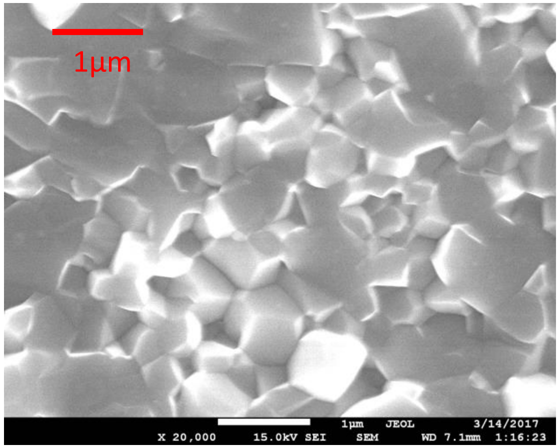
Chosen conditions: 800°C 6h

➡ Darkening decreases → Translucent

Grain size constant

1050°C

99.9%



t = 1mm

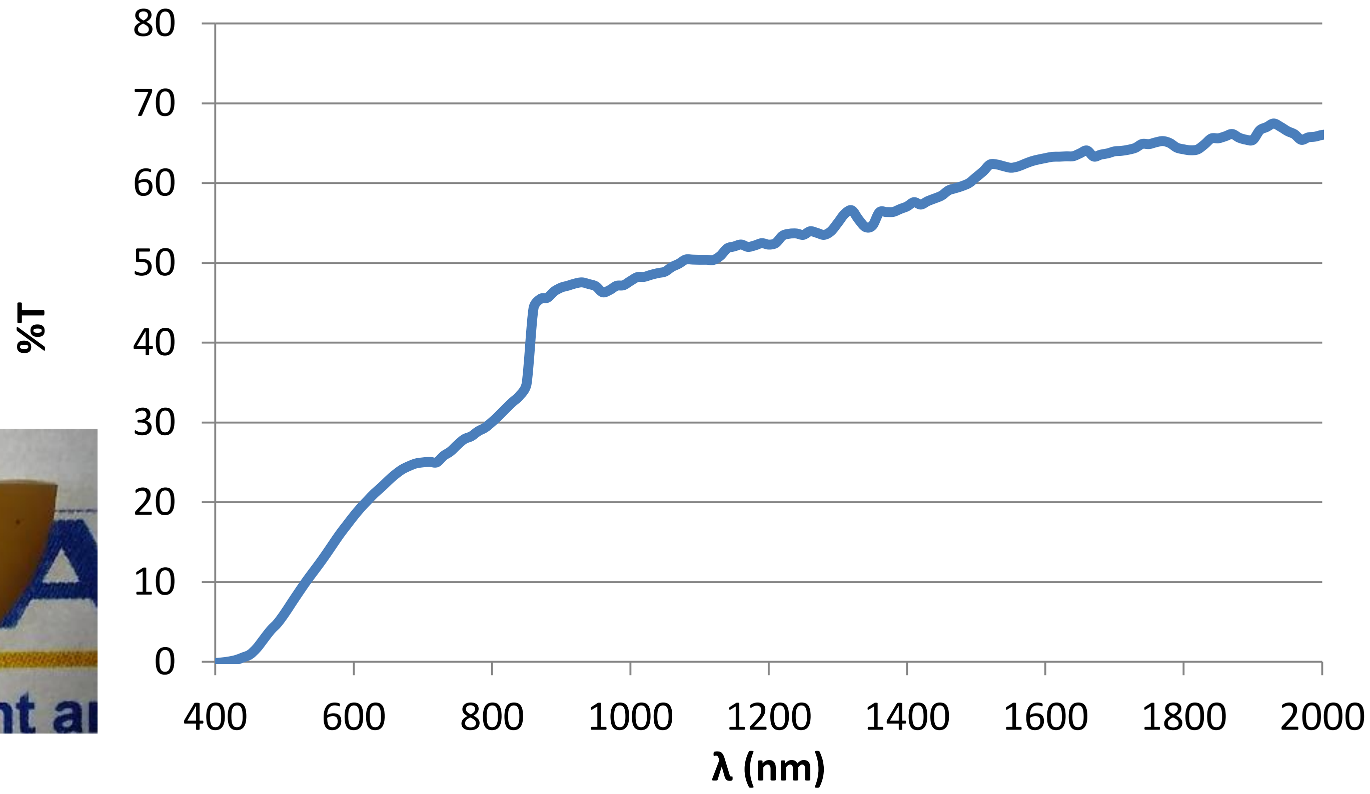
Commercial $BaTiO_3$ powder

Optical transmission

After annealing

Slight tranmission in the VIS (17% TFT at 600nm)

Good tranmission in the IR



Project(s)	LoCoTED (ERDF), master and PhD thesis UCLouvain
Contributors	P. Aubry, V. Dupont, J.P. Erauw, C. Ott, D.H. Makuanga, F. Timmermans, C. van der Rest
Collaboration(s)	UCLouvain, Ulg, CRM group

THERMOELECTRICS

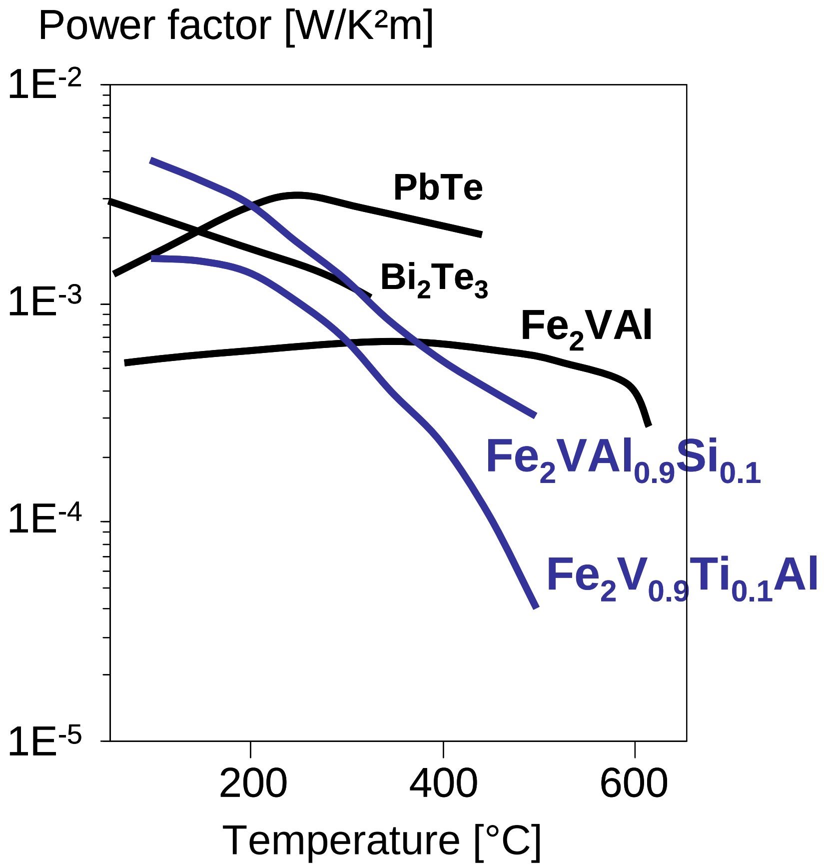
Fe_2VAl - an attractive TE material

The Fe_2VAl compounds are thermoelectric materials that meet most requirements for industrial applications

The constitutive elements are abundant, cheap and non-toxic

The power factor ($PF = \alpha^2\sigma$) is similar to that of Bi_2Te_3 below 250°C

The Seebeck coefficient can be modulated by doping



Based on Y. Nishino published data

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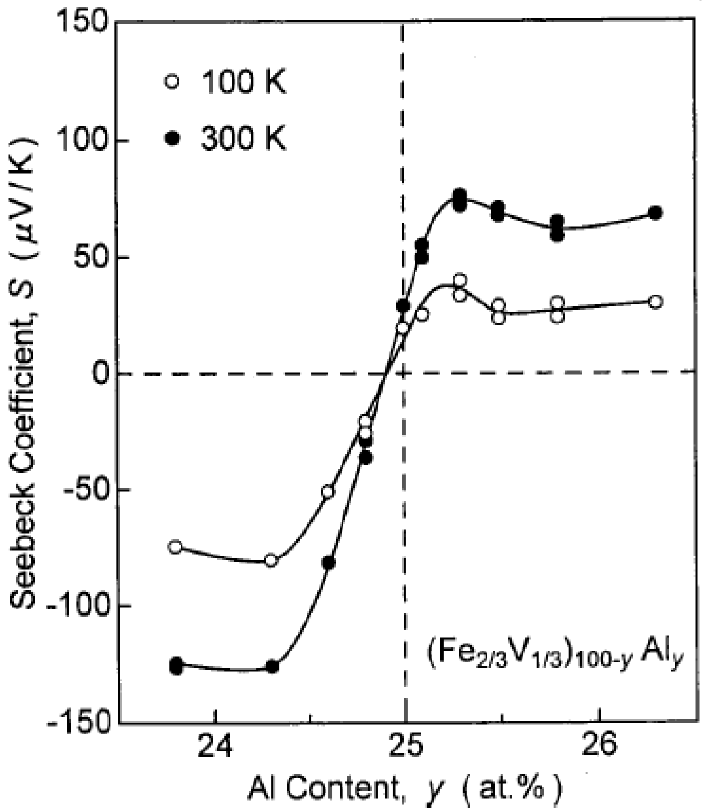
The Seebeck coefficient can be modulated by doping and/or off-stoechiometry



Y. Nishino *et al.*, Physical Review B, [63] 233303

Fe_2VAl - Elaboration strategies

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Classical Metallurgy Route

Fe, Fe-V, Al → Melting → Casting → Post-treatments

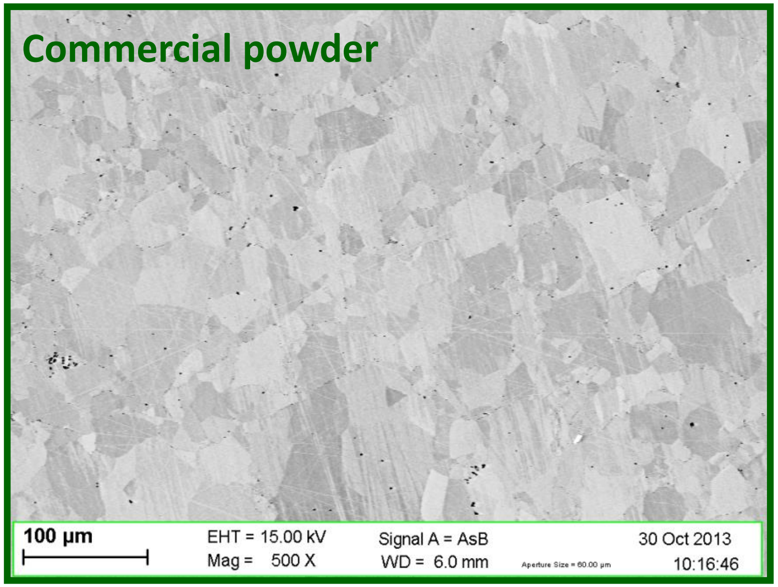
Casting → Crushing / Milling → Fe_2VAl powder

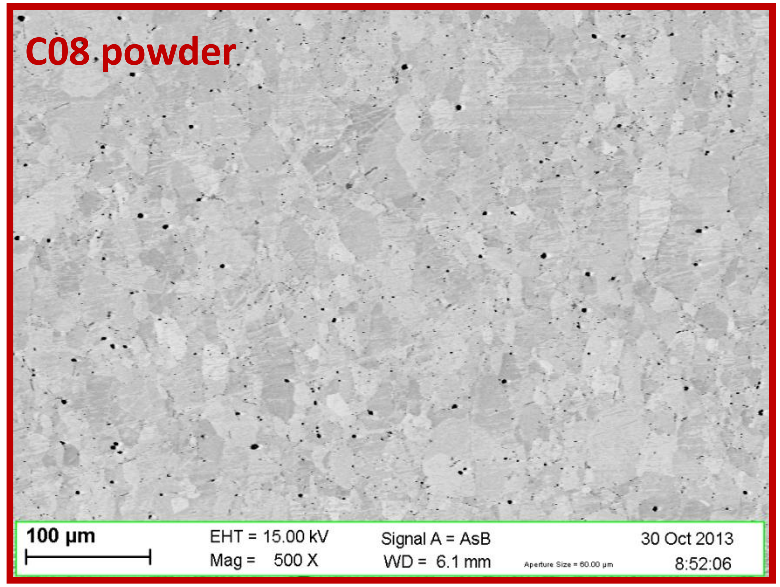
Powder Metallurgy Route

Fe_2VAl powder → Pre Compaction → Field Assisted Sintering

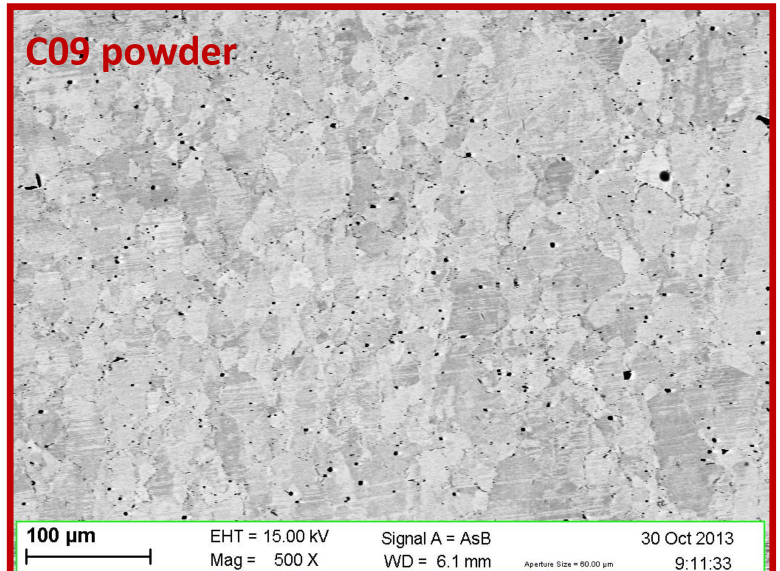
Fe, V, Al → Mixing → Pre Compaction → Field Assisted **Reactive** Sintering

Near full density @ 1200°C

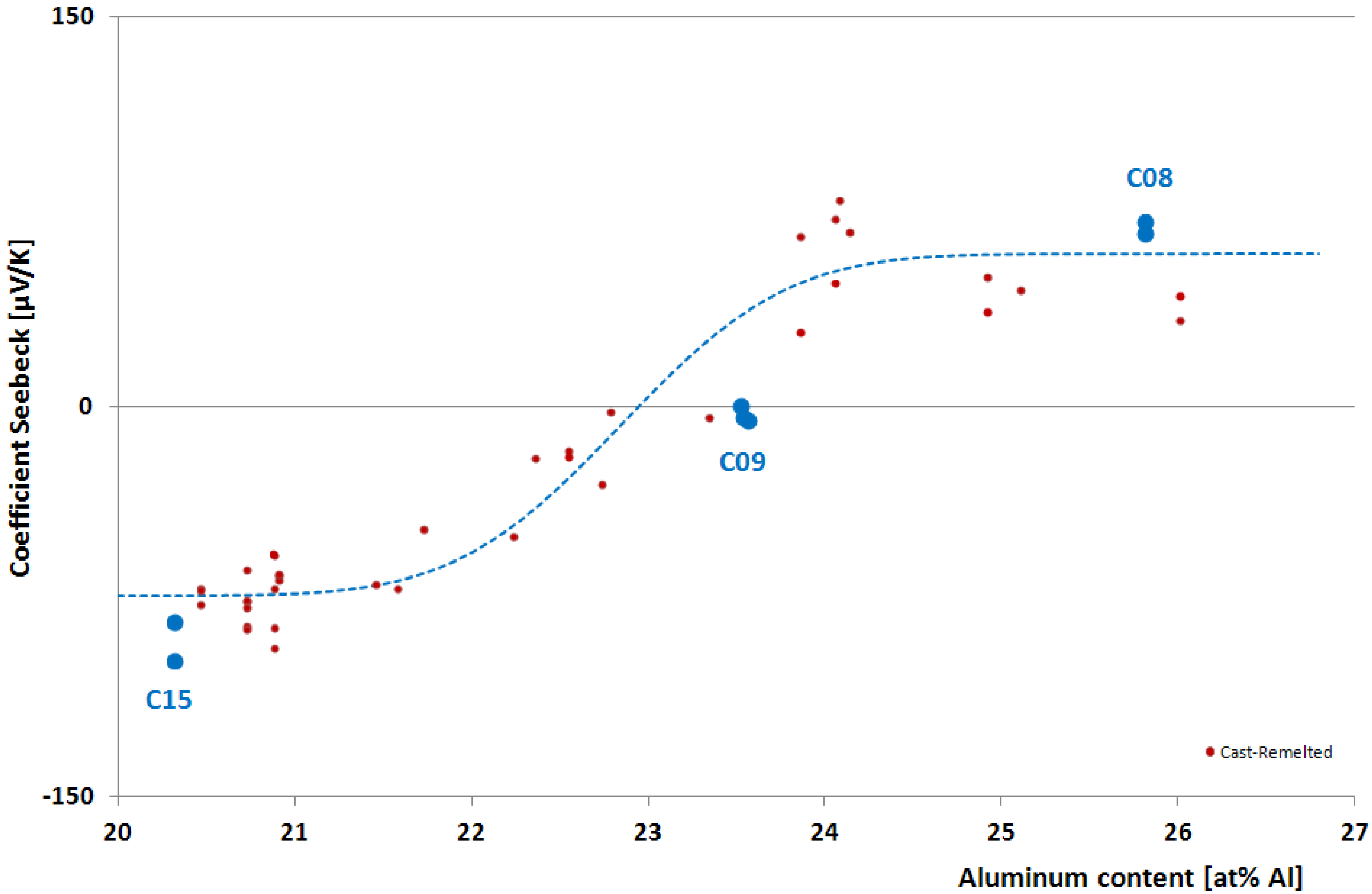




Homogeneous dense microstructure with a few isolated pores and fine globular precipitates along grain boundaries and in isolated clusters

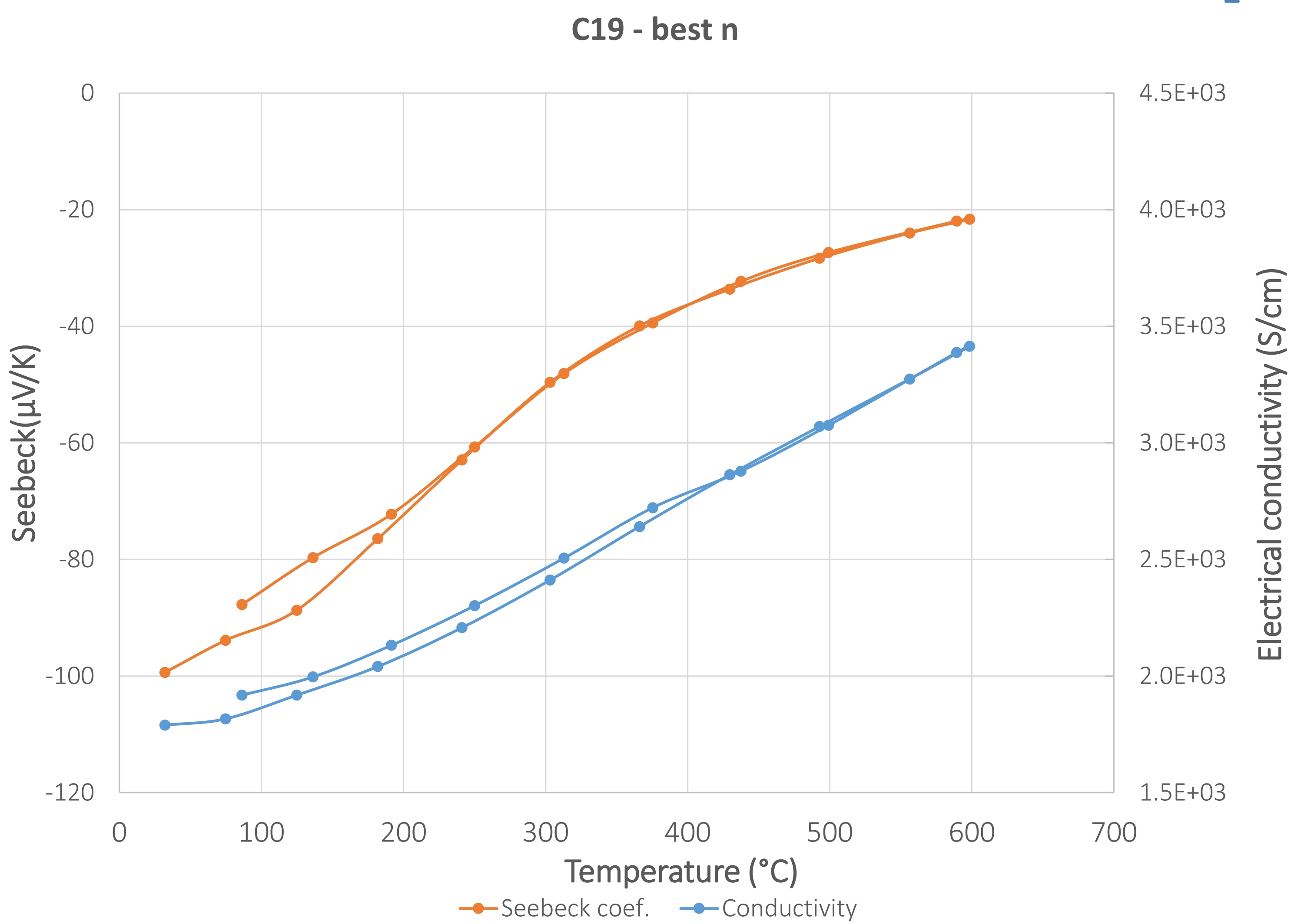


Seebeck coefficient as expected



On-going work - Optimization

Further development led to highest Seebeck coefficient for Fe_2VAl



On-going work – Sputtering targets

Near full density, homogeneous 80 mm diameter sputtering targets have been prepared under hybrid sintering mode on the HHP D125 unit.

They are currently under investigation for the deposition of thin thermoelectric films.

ID	Diamètre mm	ρ_{app} g/cm³	Open poro %	$\rho_{squel.}$ g/cm³
Batch H289	80	6.54 6.56*	0.3 0.2*	6.56 6.57*
Batch H290	80	6.54	0.1	6.55
* after grinding				

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Project(s)	CeraMAX (ERDF), PhD thesis University of Blida
Contributors	P. Aubry, M. Demuynck, V. Dupont, J.P. Erauw, Y. Hadji
Collaboration(s)	UCLouvain, UMONS-FPMs, University of Blida

MAX PHASES

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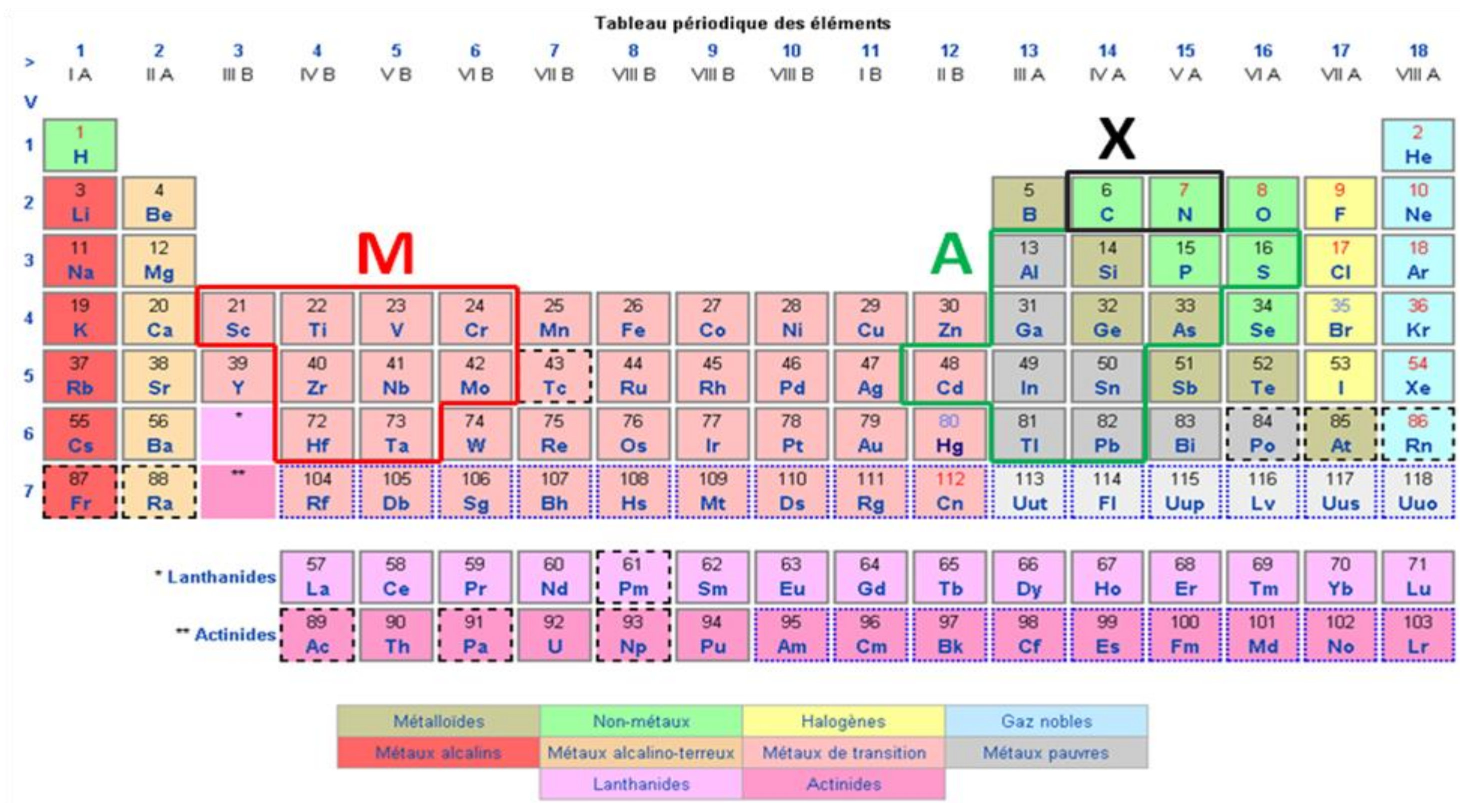
MAX Phases ?

Class of « new » compounds in which :

- **M** is a transition metal (Sc, Ti, V, Cr, Zr, Nb, Mo, Hf ou Ta)
- **A** is an element of group A (Al, Si, P, S, Ga, Ge, As, Cd, in, Sn, Ti, Pb)
- **X** is either carbon or nitrogen.



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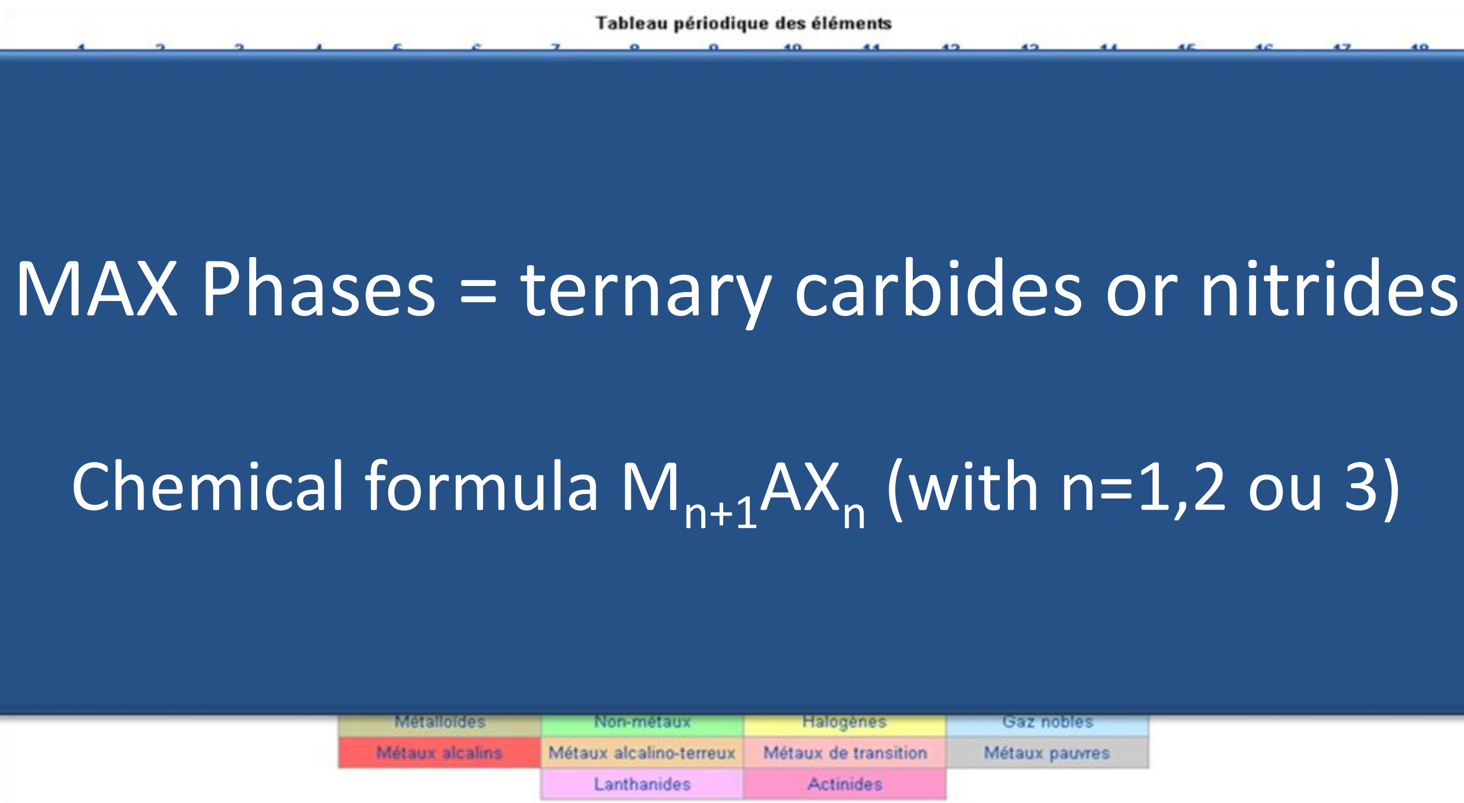
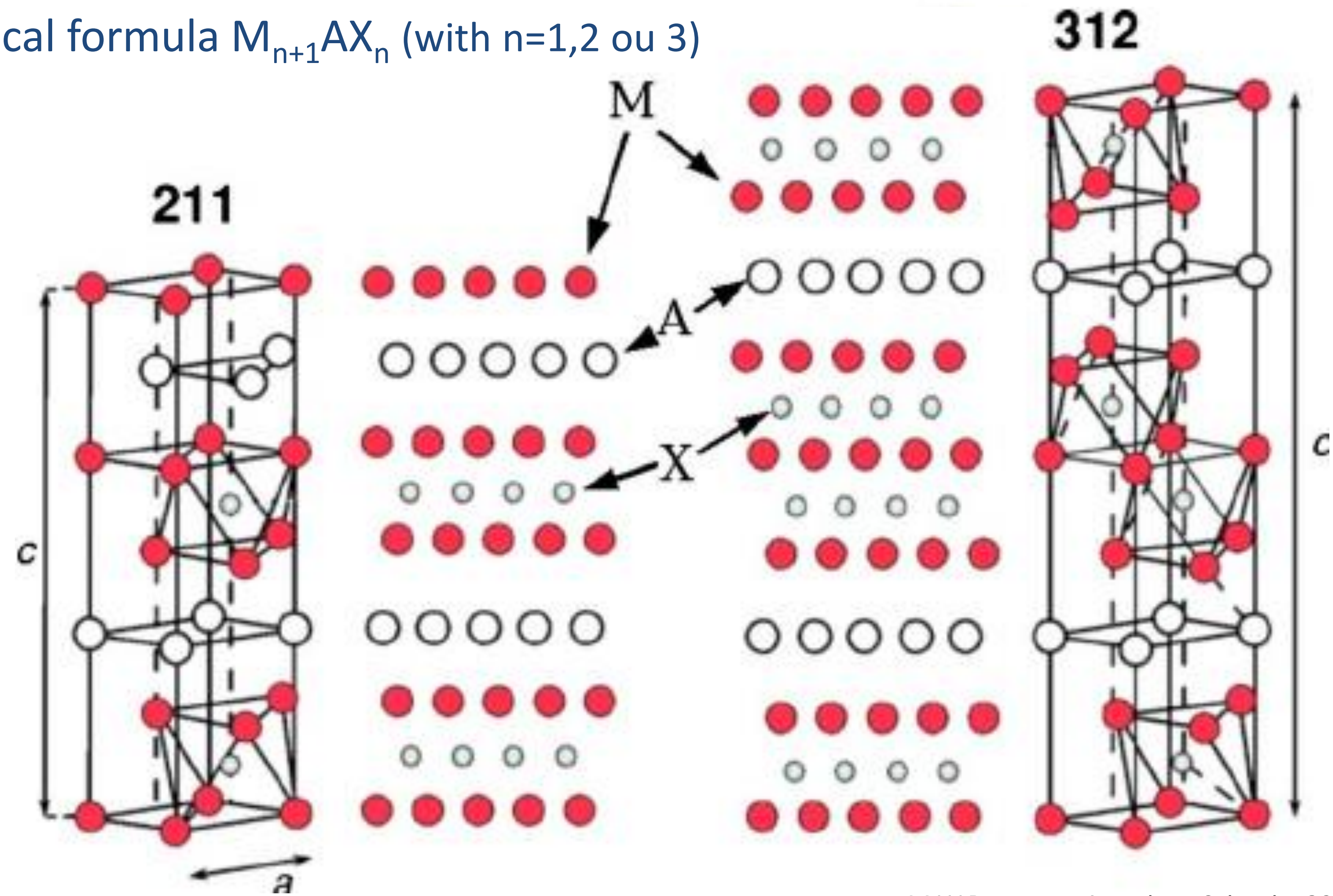


MAX Phases = ternary carbides or nitrides

Chemical formula $M_{n+1}AX_n$ (with n=1,2 ou 3)

Layers of $M_{n+1}X_n$ carbide/nitride intercalated with monolayers of element A

Chemical formula $M_{n+1}AX_n$ (with n=1,2 ou 3)



M.W.Barsoum, American Scientist 89, 34 (2001)

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UNION EUROPÉENNE
EUROPESE UNIE
TECH2FAB

Layers of $M_{n+1}X_n$ carbide/nitride intercalated with monolayers of element A

Chemical formula $M_{n+1}AX_n$ (with n=1,2 ou 3)

312

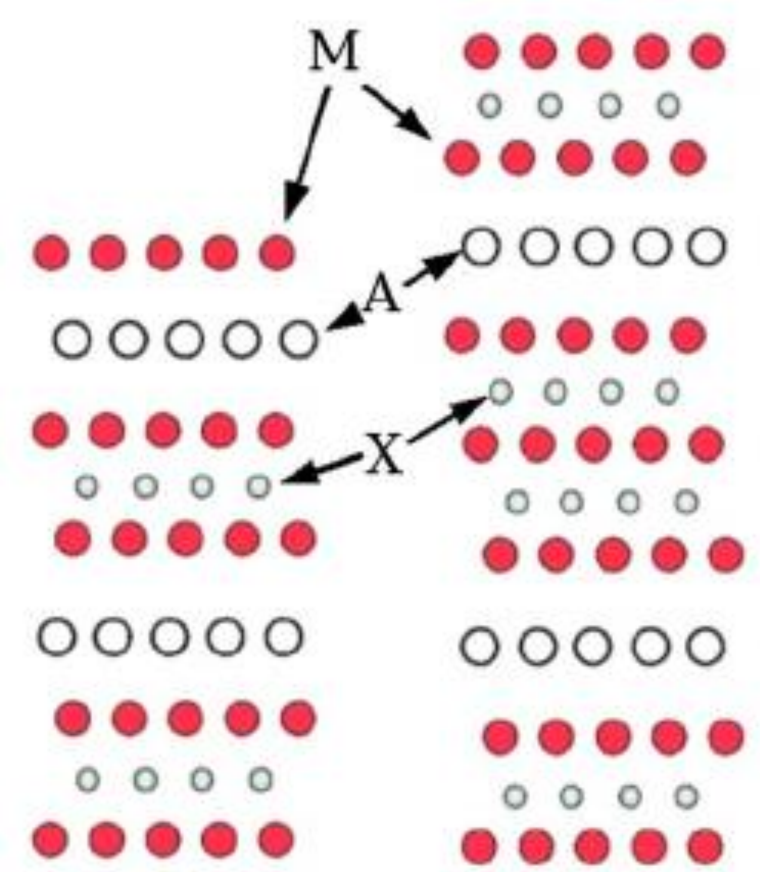
MAX Phases are characterized by their (nano)lamellar structure

M. Naguib et al., Advanced Materials 23, 37 (2011)

a

M.W.Barsoum, American Scientist 89, 34 (2001)

Properties



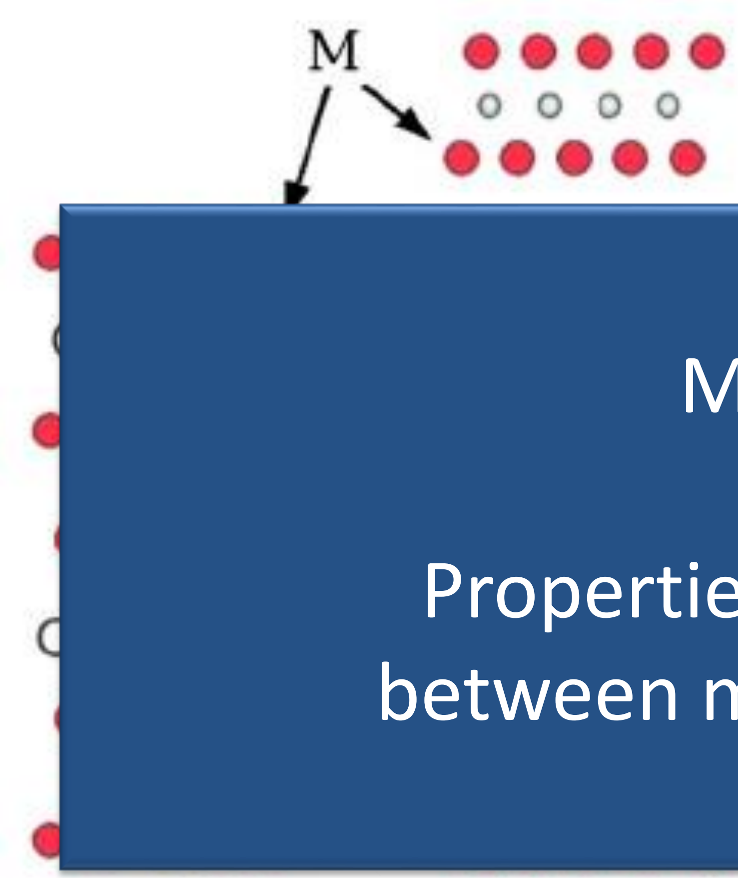
Carbide/nitride > strong covalent bonding M-X

Excellent thermal, chemical, electrical ... properties

Nano lamellar structure

Specific deformation mechanisms

Properties



Carbide/nitride > strong covalent bonding M-X

MAX Phases

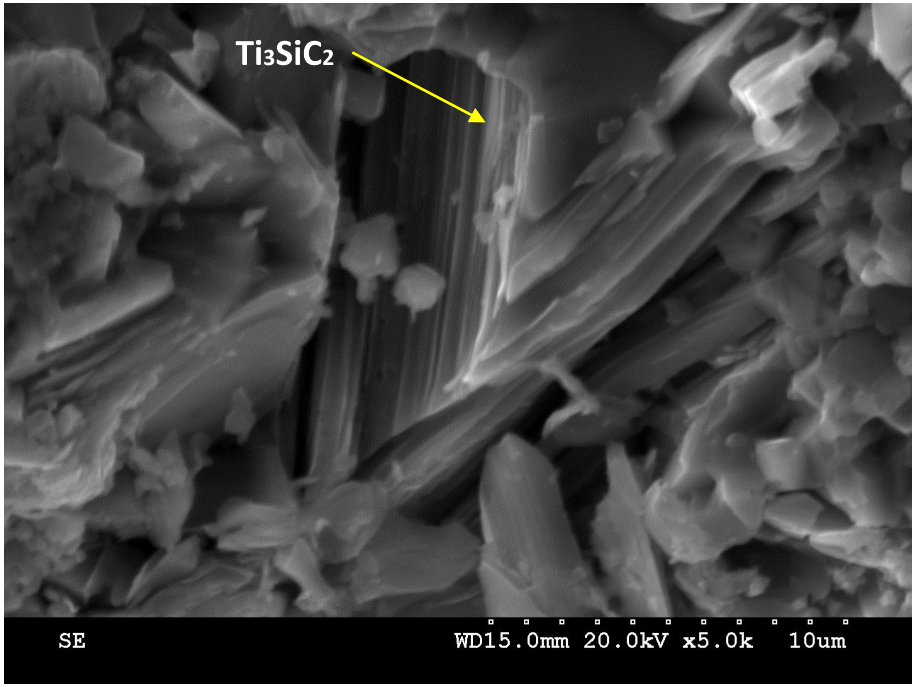
Properties bridging the gap between metals and ceramics

Reference materials

Ti_3SiC_2

Bulk sample reactively SPS sintered

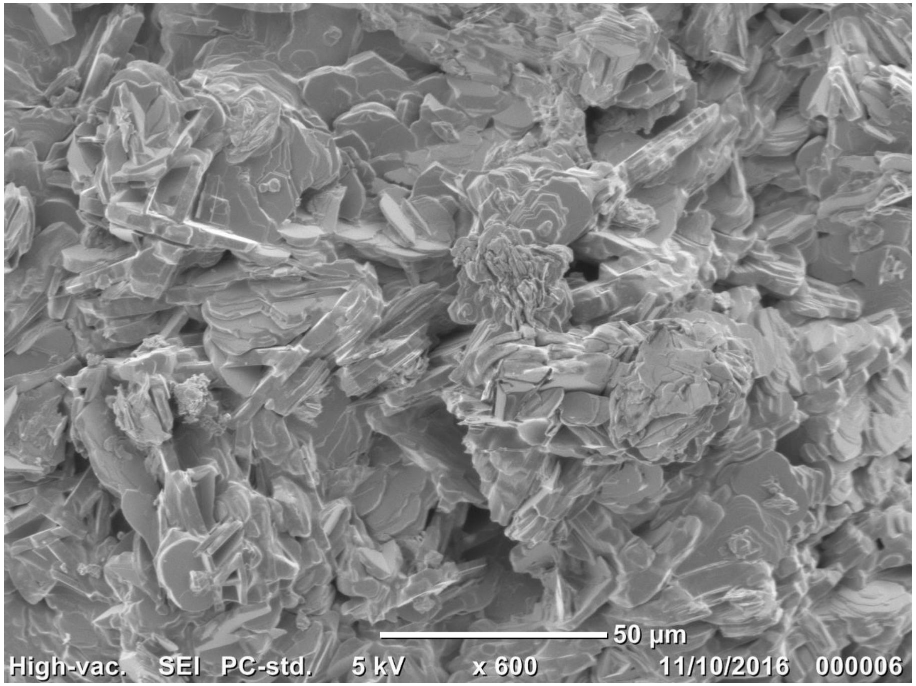
@ 1500°C



Ti_2AlC and Ti_3AlC_2

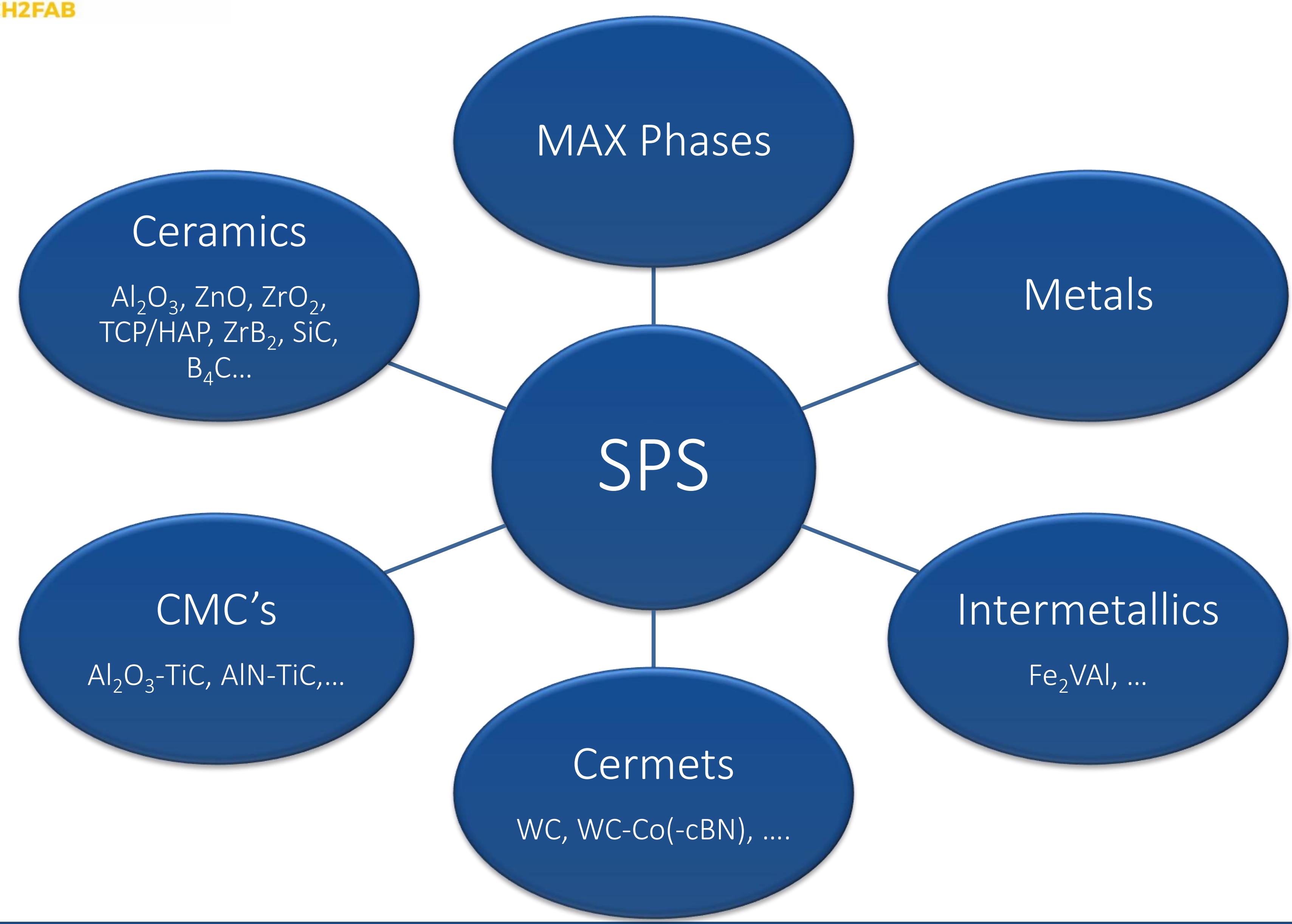
Powder reactive synthesized in SPS

@ 1350°C



Mixture of 211 and 312 phases

Summary


MAX Phases
Ceramics
Al_2O_3, ZnO, ZrO_2, TCP/HAP, ZrB_2, SiC, B_4C…
Metals
SPS
CMC's
Al_2O_3-TiC, AlN-TiC,…
Intermetallics
Fe_2VAl, …
Cermets
WC, WC-Co(-cBN), ….

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Our research topics

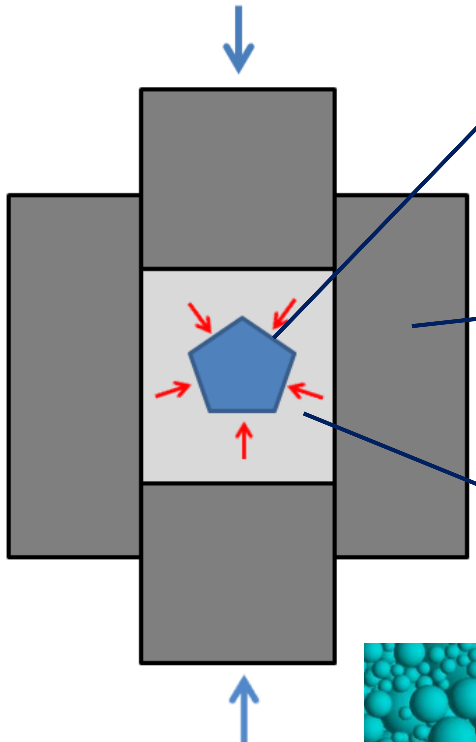
Basic research	• Insight in the fundamental mechanisms • Process modelling
Innovative Materials	• Wear resistant — Cermets and composites • (Electro) optics — YAG, $Ba(Sr)TiO_3$ • Thermoelectrics — (Semi) Heussler compounds • « Polyvalent » — MAX phases
Technical Improvements	• Upscaling • Shape complexity

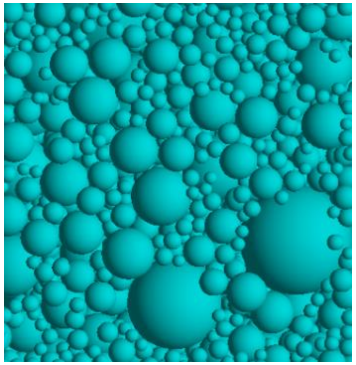
Project(s) SHAPESPS (DGO6)
Contributors M. Demuynck, V. Dupont, S. Hocquet
Collaboration(s) Ulg-GRASP

SHAPE COMPLEXITY

Rationale of the ShapeSPS project

Quasi isostatic SPS





1 - Green part processing

- ❑ Take into account the <u>dimensional variations during sintering</u>
- ❑ Take care of debinding problem: compatible with the powder bed, low volume of gas → debinded?, pre-sintered?

2 - Sintering parameters

- ❑ Reach the wished density
- ❑ Take into account any deformation induced by the pressure

3 - Powder bed properties and numerical modeling

- ❑ Optimal properties of the powder bed
  - *Powder flowability*
  - *Indentation problem*
  - *Forces dispersion*
  - *Recycling of the powder bed (i.e. does not sinter)*
- ❑ Relative dimensions (part←→die)

WC-12%Co

- Uniaxial and isostatic pressing of powder of WC-12%Co
- Laser machining based on CAD model
- SPS cycle under optimal conditions of load and temperature

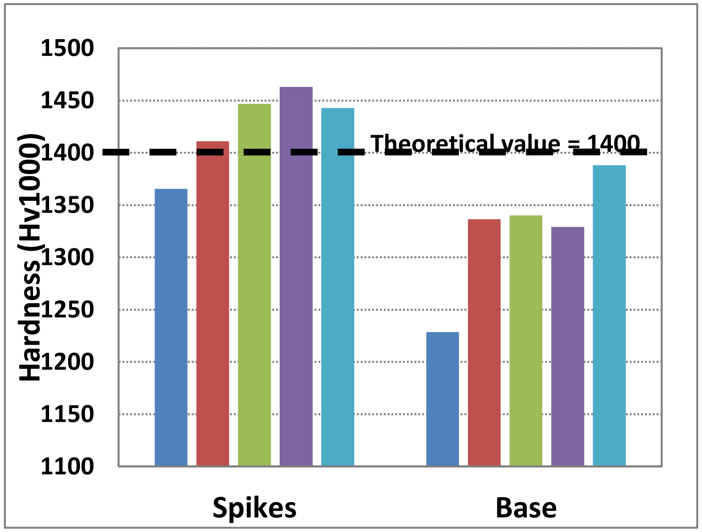
Hardness
- Pure WC ~ 2200
- WC + 6% Co ~ 1550
- WC + 12% Co ~ 1400

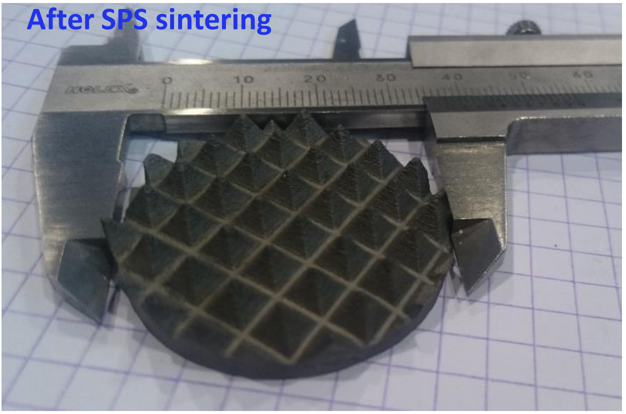
$H(tot)_{ini}$ = 10,65mm

$H(spike)_{ini}$ = 5,90mm

« Boucharde » part – Tool 80mm
1280°C-20min-100kN

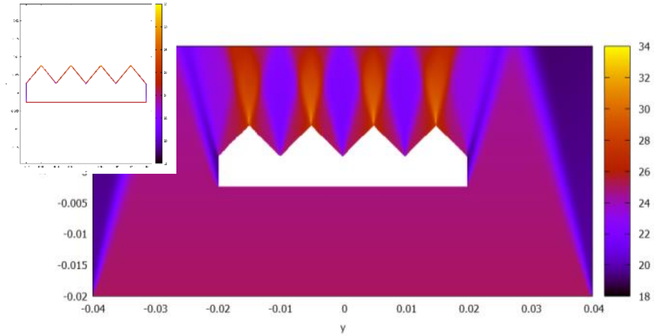
Hardness measurement (HV1000) on base and spikes





Density after sintering : 13.96 or 97.6%

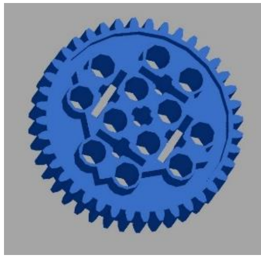
Reinforcement on spikes, explained by 2D model (larger force applied by the granular media on spikes)

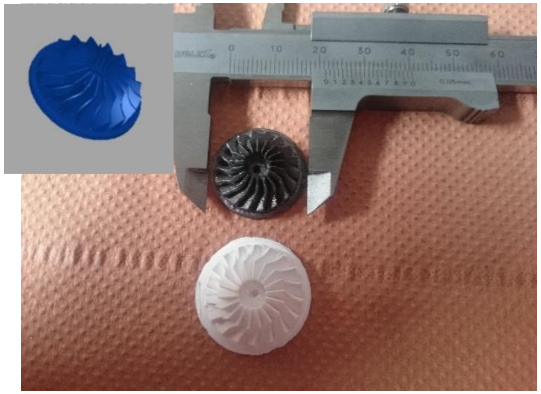


$H(tot)_{fin}$ = 7.85mm $H(spike)_{fin}$ = 4.30mm

Alumina

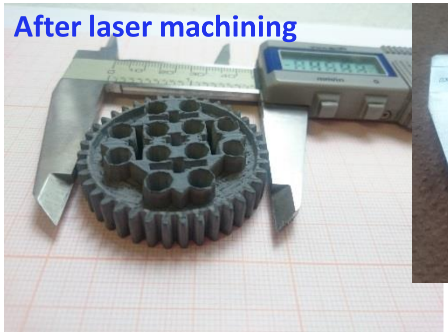
- Uniaxial and isostatic pressing of alumina powder
- Laser machining based on CAD model
- Debinding (700°C) and pre-sintering at 1100°C/3h
- SPS cycle under optimal conditions of temperature and load

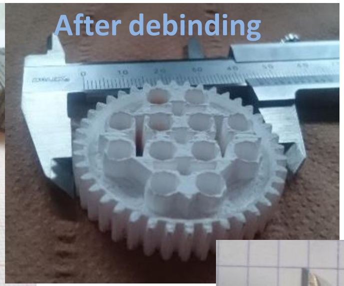




Turbine – Tool 40mm
1600°C-10min-3kN

Density after sintering: 3.78 (95.0%)

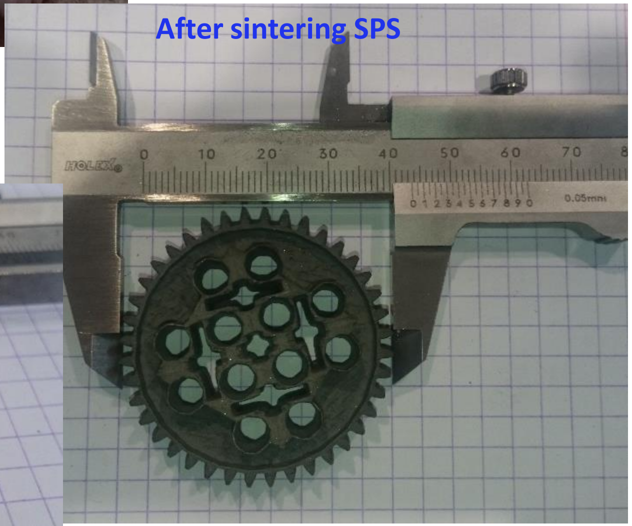




Gear – Tool 80mm
1500°C-10min-40kN

Density after sintering: 3.82 (96,1%)

Shrinkage diameter : 2%
Shrinkage height : 26%



FLASH SINTERING

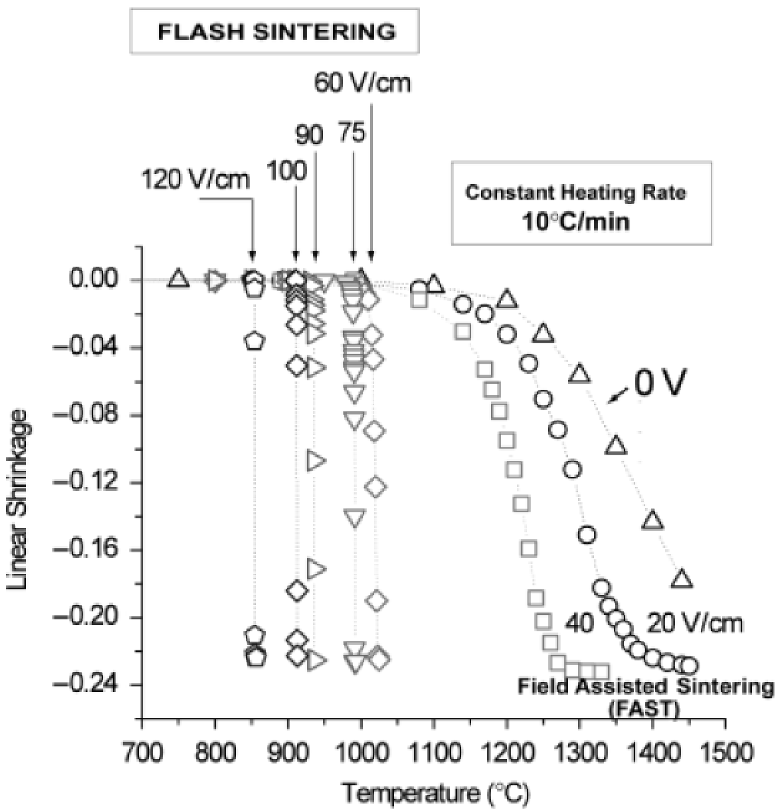
Mentionné une 1ère fois fin 2010

journal

J. Am. Ceram. Soc., 93 [11] 3556–3559 (2010)
DOI: 10.1111/j.1551-2916.2010.04089.x

Flash Sintering of Nanograin Zirconia in <5 s at 850°C

Marco Cologna,[†] Boriana Rashkova,[§] and Rishi Raj[†,‡]



- densification quasi-instantanée
- d'un compact porté progressivement en température
- en présence d'un champ électrique dépassant une valeur seuil

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J. Am. Ceram. Soc., 96 [3] 697–700 (2013)
DOI: 10.1111/jace.12176

journal

Rapid Communication

In Situ Thermometry Measuring Temperature Flashes Exceeding 1,700°C in 8 mol% Y_2O_3-Stablized Zirconia Under Constant-Voltage Heating

Jungdeok Park and I-Wei Chen†

Mécanismes invoqués

- **Forte augmentation locale de température aux joints de grains** (d'autant plus marquée que les cols entre particules sont restreints)
- Nucléation de défauts (paires de Frenkel)
- Interaction avec les charges d'espace

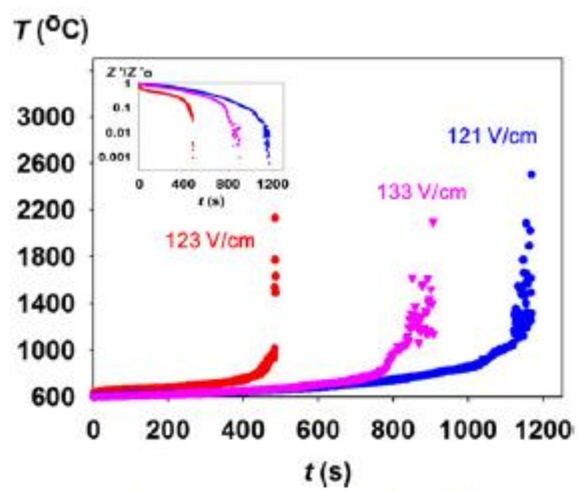

Fig. 6. Temperature of 8YSZ samples (0.6–0.69-mm thick) under constant voltage (8–8.5 V). Inset: decrease in resistance relative to initial value. Furnace heating rate: 10°C/min.

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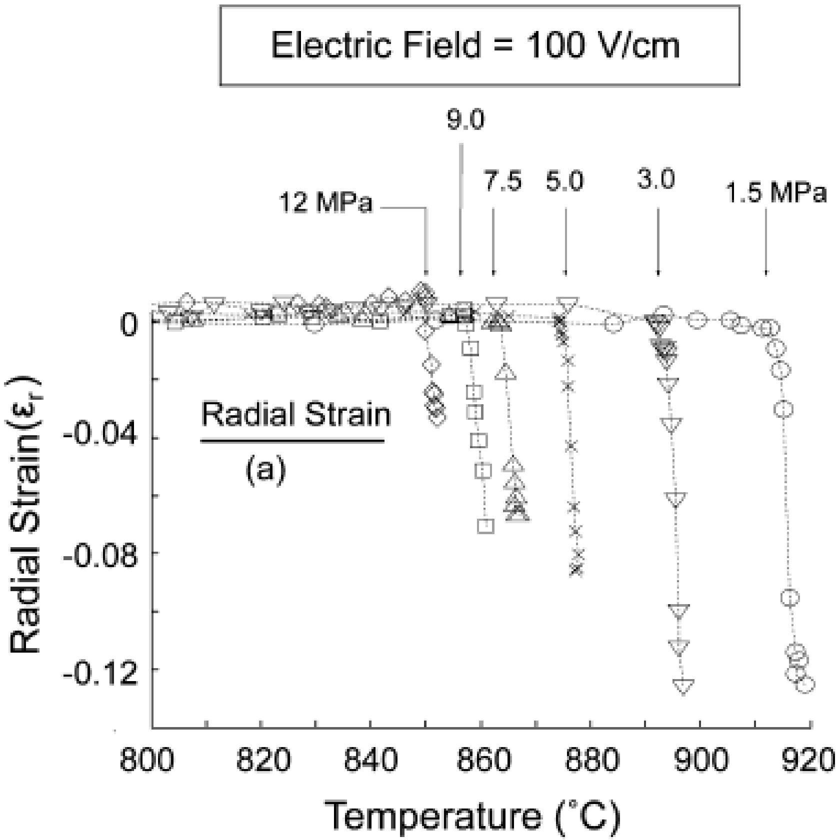
J. Am. Ceram. Soc., 95 [1] 138–146 (2012)
DOI: 10.1111/j.1551-2916.2011.04855.x

journal

Flash-Sinterforging of Nanograin Zirconia: Field Assisted Sintering and Superplasticity

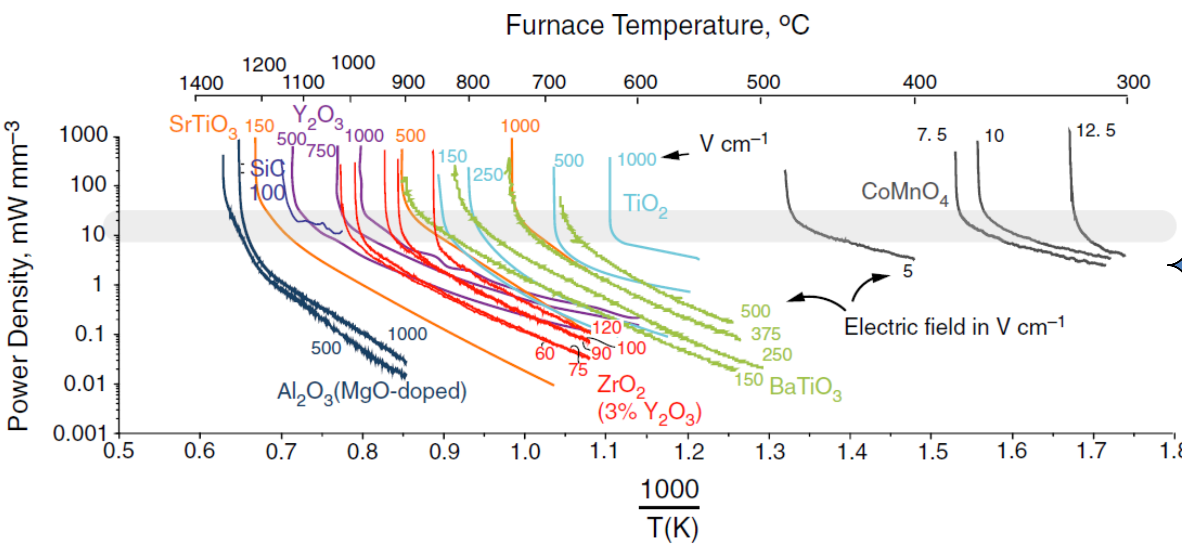
John S.C. Francis† and Rishi Raj



- A champ électrique fixé, on relève une diminution de la température seuil pour des contraintes appliquées croissantes

Applicable à tout matériau ?

- Rapporté essentiellement (mais pas uniquement) pour des oxydes
- Larges variations de la valeur seuil du champ électrique selon le matériau considéré



3%mol tetragonal YSZ
8%mol cubic YSZ
Co_2MnO_4 spinelle (SOFC)
Al_2O_3 dopé MgO
TiO_2 (rutile)
Multilayer NiO - 8YSZ
Y_2O_3
$BaTiO_3$

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ou pour applications ciblées ?

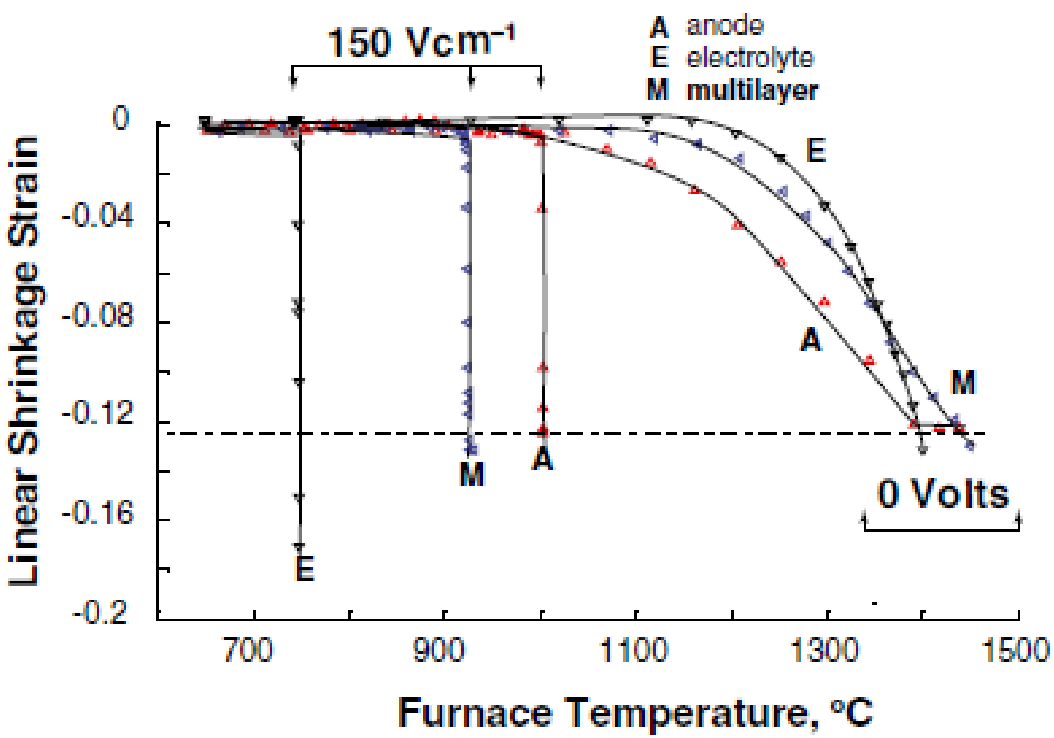
journal

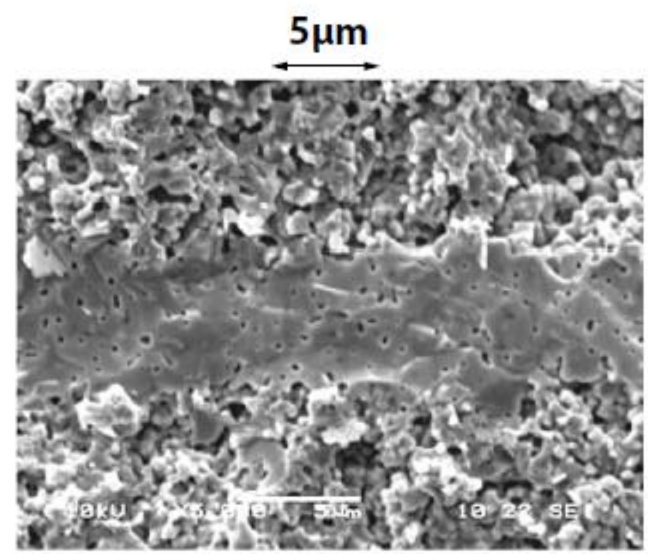
J. Am. Ceram. Soc., **96** [5] 1352–1354 (2013)
DOI: 10.1111/jace.12330

Rapid Communication

Flash Sintering of Anode–Electrolyte Multilayers for SOFC Applications

John S. C. Francis,‡ Marco Cologna,‡ Dario Montinaro,§ and Rishi Raj‡,†





FLASH SINTERING @ BCRC

Project(s)	FlashSint (ERDF)
Contributors	L. Boilet, M. Demuynck, S. Hocquet
Collaboration(s)	Université de Liège (Institut Montefiore)

FRITTAGE FLASH A L'ECHELLE PILOTE

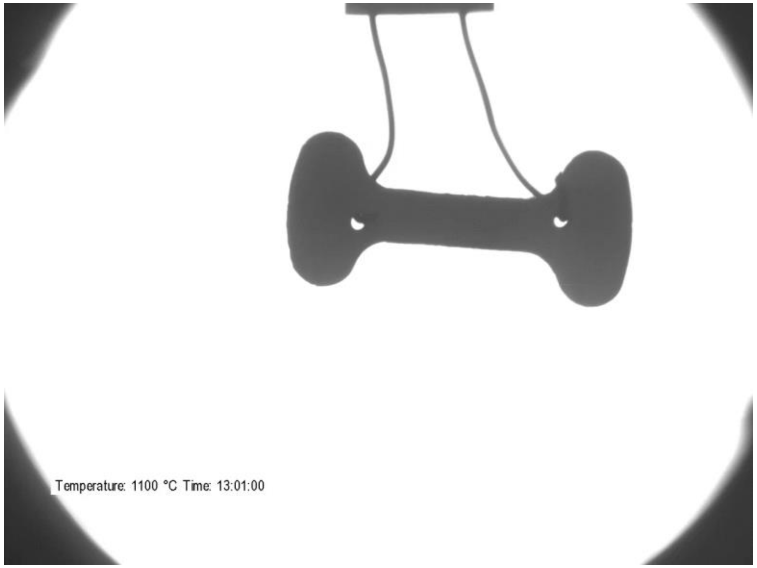
Essais sur Zircone

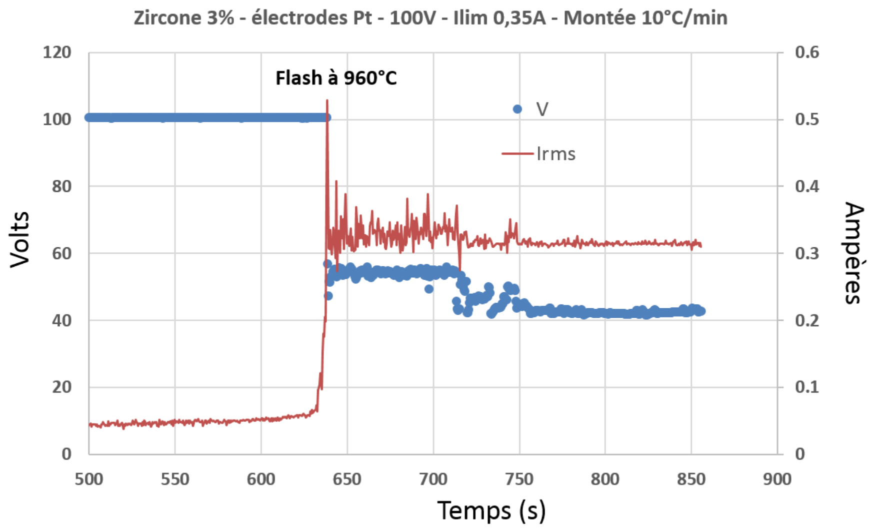
Essais à température constante

Température four (°C)	Tension (V/cm)	Imax (A)
950	115	0,35
1000	106	0,35
1050	100	0,35
1100	92	0,35
1150	62	0,35

Essais à vitesse de chauffe/tension constante

Tension (V/cm)	Température four (°C)	Imax (A)
100	980	0,35
125	950	0,35
150	840	0,35





Pas seulement une curiosité de labo

Low Energy Firing Project

Lucideon (UK)

Objectif :

commercialisation de la technologie de frittage sous champ électrique (frittage flash) pour la production continue de produits céramiques à basse température (750-900°C)

20-metre kiln (source www.ceram.com)

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- The DGO6 (Wallonia, Belgium) is greatly acknowledged for the financial support of the research projects STEELFSW, FSW-PME, SHAPESPS, First Do.C.A. SPS, First Do.C.A. Ceropt
- The European Regional Development Fund (ERDF) and Wallonia, are gratefully acknowledged for their financial support to the research projects T-REX-EquiDeR, T-REX-Cerapide, IMAWA-CeraMAX, IMAWA-FlashSint, FMF-LoCoTED in the frame of the “Convergence programme” and “Transition programme”

ACKNOWLEDGMENTS

Wallonie

The European Regional Development Fund and Wallonia invest in your future

My colleagues

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Michel Cambier
Maryse Demuynck
Vedi Dupont
Stéphane Hocquet
Rosa Moronta Perez

ACKNOWLEDGMENTS

Merci pour votre attention
Questions?

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