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MICROWAVE SYNTHESIS OF MATERIALS:
A bird view with some chosen examples

EL NANO thematic school GDR NAME

Aussois,13th June 2023

ELaboration of NANOmaterials for the recovery, conversion, transport and storage of energy

THIS LECTURE - OUTLOOK

MICROWAVE SYNTHESIS OF MATERIALS:
A bird view with some chosen examples

BASIC of MW PROCESSING of MATERIALS

A QUICK VIEW OF MICROWAVE ASSISTED <u>INO</u>RGANIC SYNTHESIS

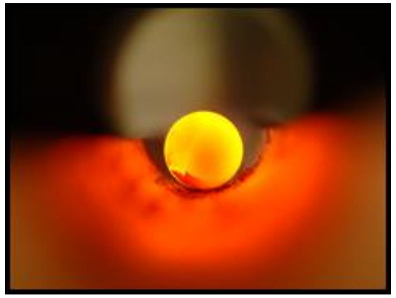
ZnO flower like shape made by sublimation-recrystallisation (CRISMAT)
Microwave Plasma Enhanced CVD (litterature)
Microwave assisted Hydrothermal synthesis (litterature)
Solid-State Synthesis (CRISMAT)
Microwave Sintering: thermal managment to control sintering (CRISMAT)

CONCLUDING REMARKS

I- Basic of MW Processing of Materials

Why is it worth using MW for materials processing?

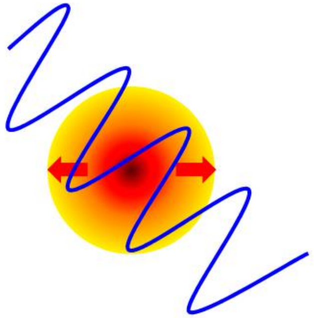
We take advantage of MW-Material *interactions*
300 MHz < f < 300 GHz



Volumetric Heating
And Selective Heating Possible
(depending on the mat. Properties)

Less Energy Consumption
High efficiency
Very High heating rates (>200°C/min)
'Green Technology'

Reliable – Affordable
Our 1987 microwave source Is still in good working conditions



❑ Microwave Heating highly depends on the material electric properties

The absorbed microwave power (case of dielectric materials):

$$Pd = 2\pi f\varepsilon' \tan(\delta) E^2$$

Dielectric Losses

The penetration depth of the Electrical Field:

$$Dp = \frac{\lambda_0}{2\pi\sqrt{\epsilon'}\tan(\delta)}$$

The lower the loss factor
The higher the E field
penetration depth

For conducting material, the penetration depth is very small and tends to zero (skin effect)

Microwaves-Matter Interactions

Dissipated power formula

(usual case for dieletrics) $P_d = 2\pi f \varepsilon' \tan(\delta) E^2$

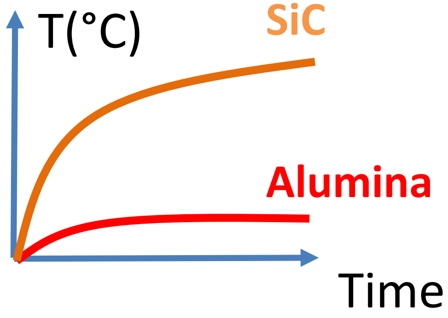
Let us compare Al_2O_3 vs SiC:
A quick comparison to understand
the impact of the loss tangente

Dielectrics properties of alumina versus Silicon carbide @ 2,45 GHz

CERAMIC MATERIALS	FREQUENCY (GHz)	TEMP. (^oC)	ε´	ε´´	tan δ	DEPTH OF PENETRATION D_p (cm)	CRITICAL TEMP. (T_c)
Alumina[1]	2.45	+25	8.9	0.009	0.00010	1.2	800° C (3.89-3.61GHz)[5]
Silicon carbide[2]	2.45	+200	105	110	1.048	0.28	

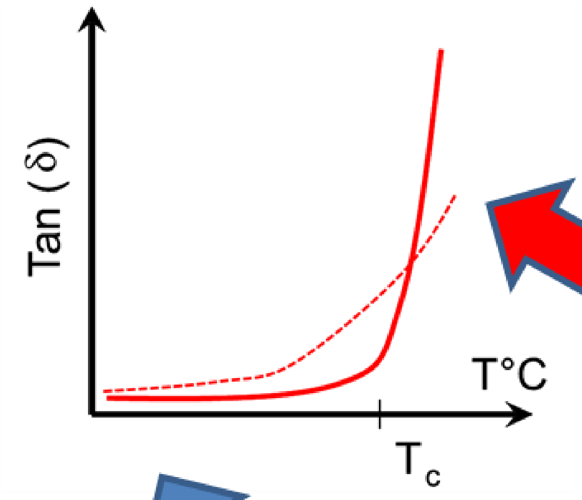
1: Data provided by J. Gerling, Gerling Applied Engineering, Inc., Modesto, CA
2: Leiser, K., "Microwave Behavior of Silicon Carbide/High Alumina Cement Composites," Doctoral dissertation, University of Florida (2001)

Al_2O_3 is a poor coupling material
SiC highly absorbs MW



what is true at low temperature, is not necessarily true at high temperature

Tan(δ) increases with temperature (general trend)

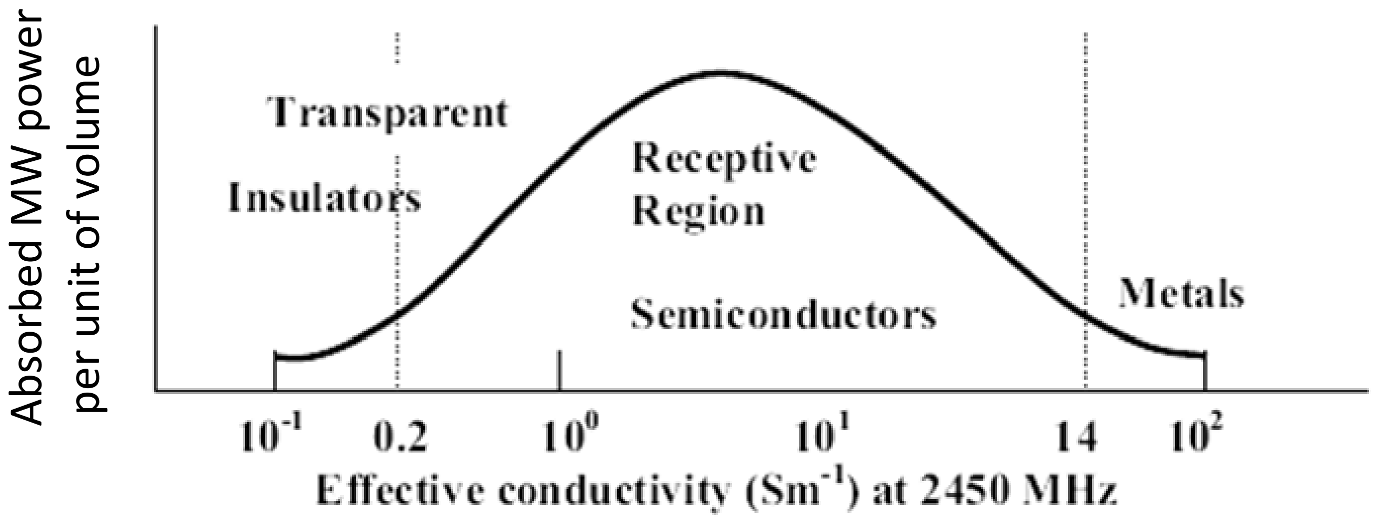


Strong coupling at
high temperature (>800°C)

Transparent (or not coupling)
materials at RT

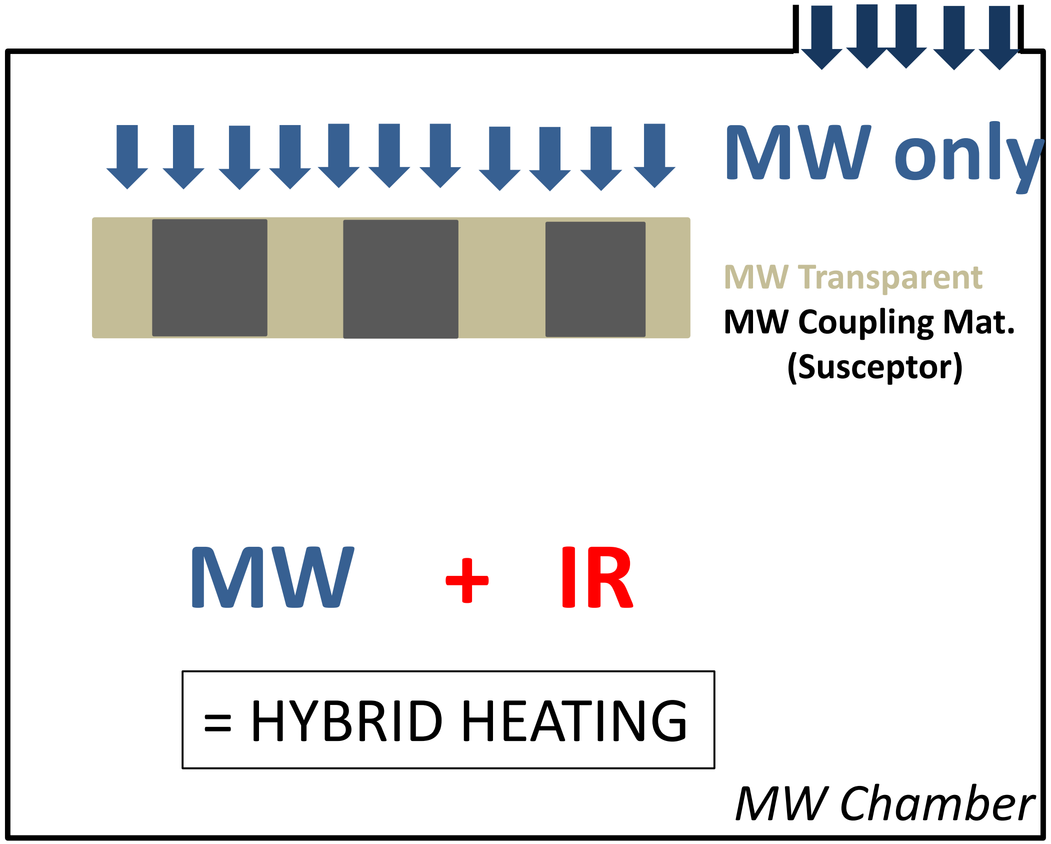
$$(Pd = 2\pi f \varepsilon' \tan(\delta) E^2)$$

Most transparent materials at RT end up coupling (heating) at high temperature



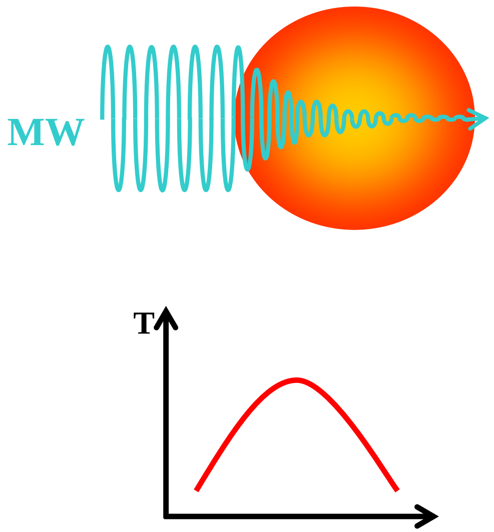
*It is not because only semi-conducting material easily couples with microwave that we cannot use MW for any kind of materials. The adopted solution is to use a **SUSCEPTOR** which provides **hybrid heating.***

SUSCEPTOR: a material which strongly couples with MW converting the MW energy into thermal IR radiation (it becomes like a resistor in a CV furnace)

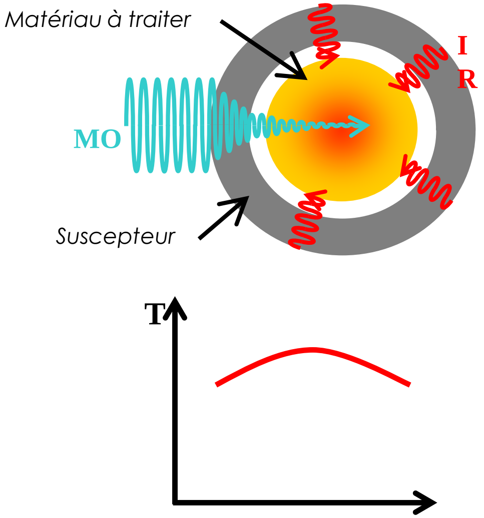


MW mater interactions: Different ways of using MW Energy

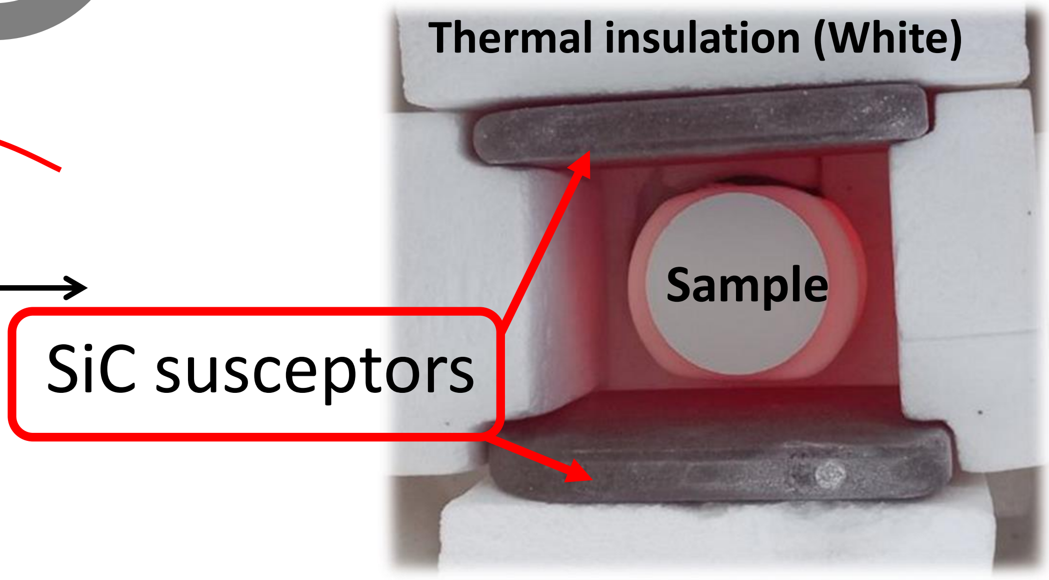
Direct Heating



Hybrid Heating



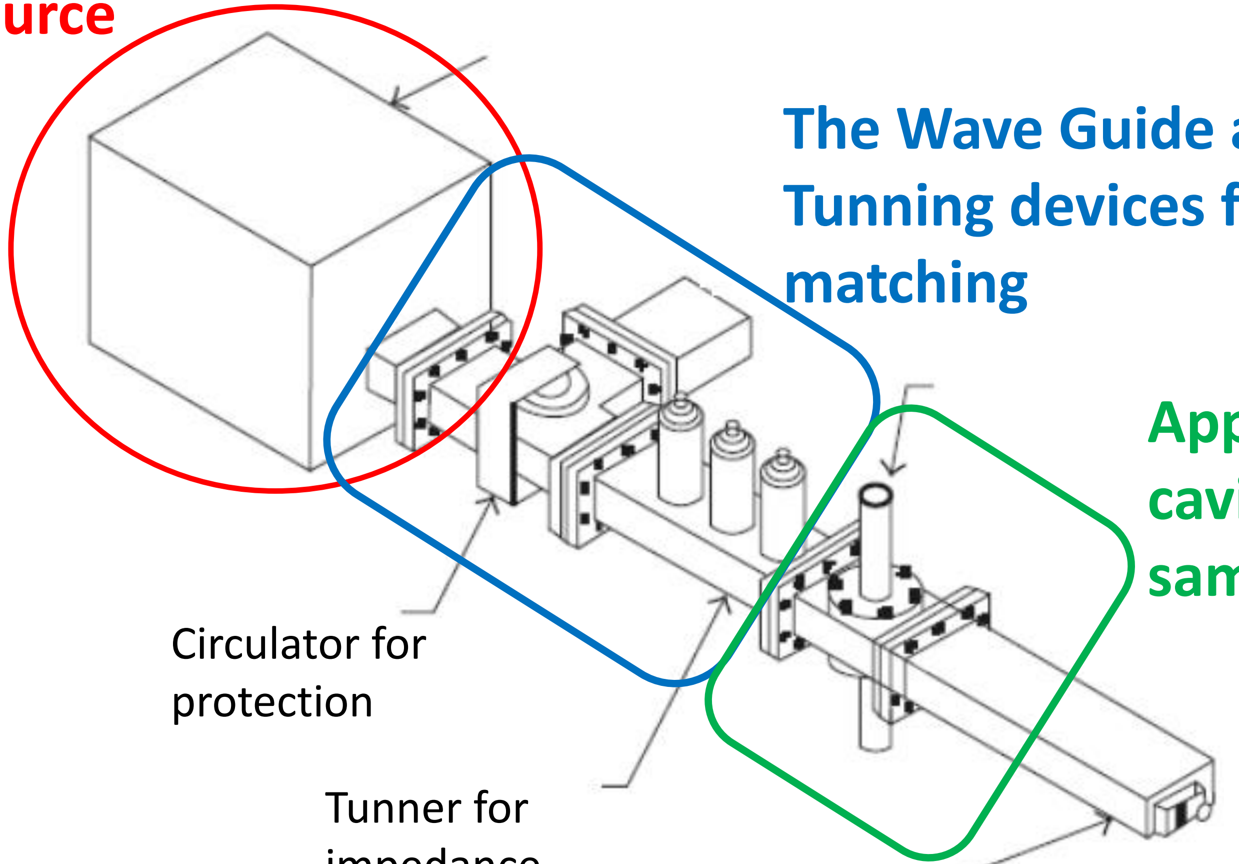
Hybrid Heating Is most often Priviledged when an ***<u>« homogeneous »</u>*** *Temperature Distribution is needed*



Direct heating can be advantageously used when ***<u>selective heating process</u>*** *is researched (Welding, Fast synthesis,etc.)*

MW Sintering:
Some Technical
Details describing
How Energy is transfered from the
source to the sample

TYPICAL SINGLE-MODE MICROWAVE SYSTEM FOR MATERIALS PROCESSING



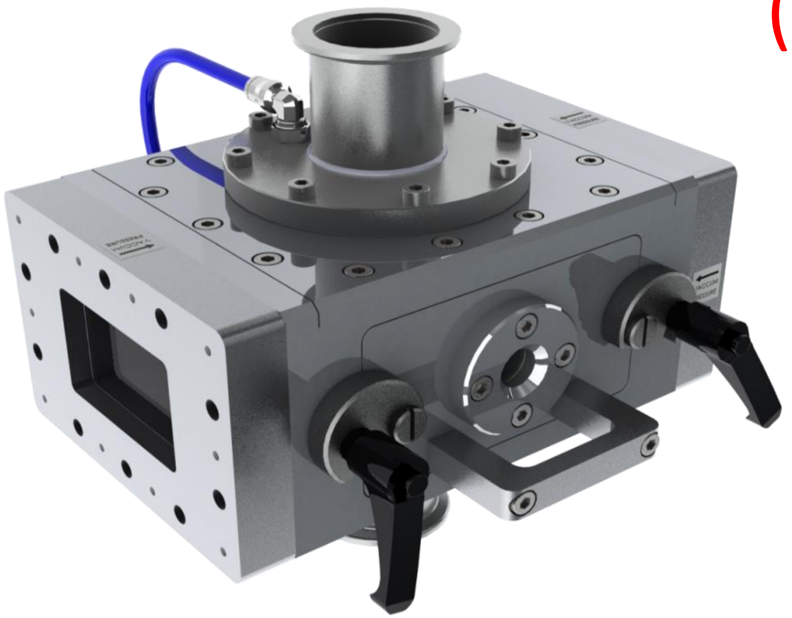
S. Marinel et al.
Advances in Materials Science and Engineering
Volume 2018, Article ID 4158969, 8 pages
https://doi.org/10.1155/2018/4158969

At the end of the line, an applicator or a microwave Cavity is used to convert the MW energy to thermal energy into the material …

There are two types of cavity :

(i) resonant cavity

(ii) multimode cavity (chamber)



Single-mode/resonant cavity:

Small Volume ∝ f^{-1}

E, H fields are amplified
(Standing Waves are formed)

Low Power needed – high efficiency

(E, H) (x,y,z,t) distribution known

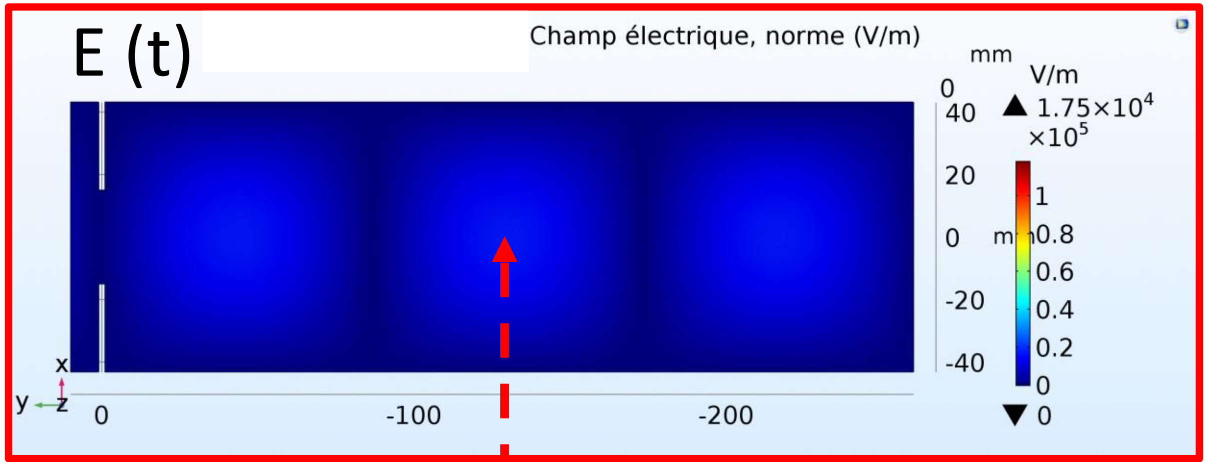
Multi-mode Cavity

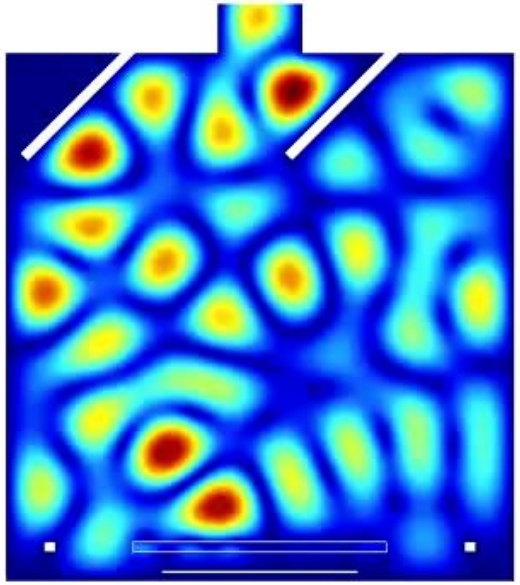
Larger Volume

Stirrer is often used (mixing waves)

Higher MW Power is needed

(E, H) (x,y,z,t) distribution is averaged.

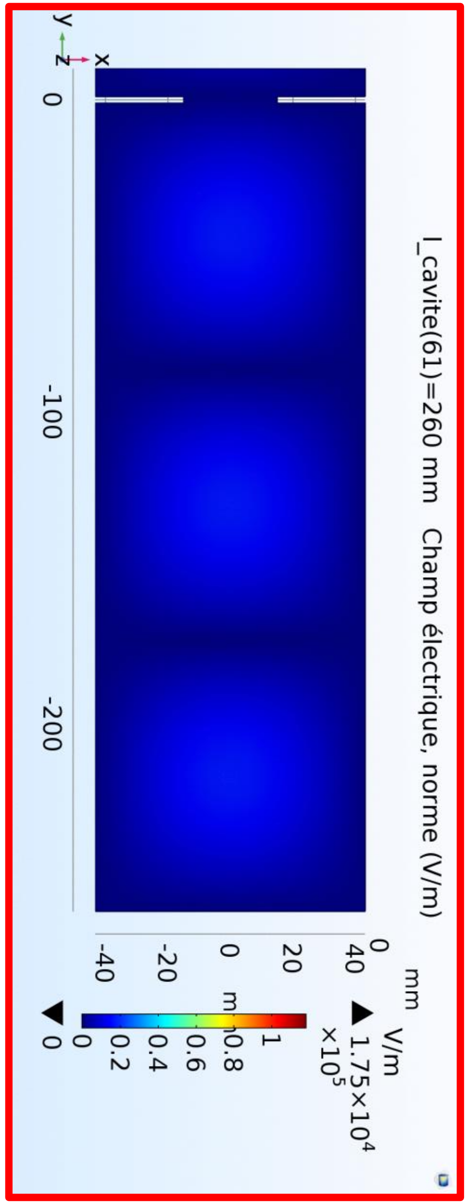


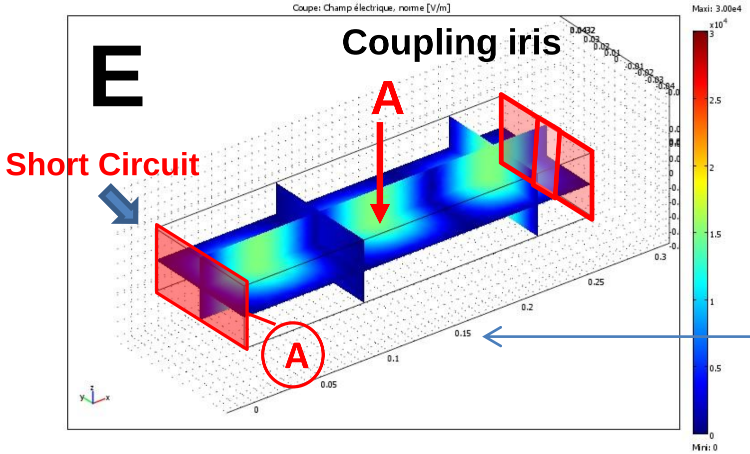


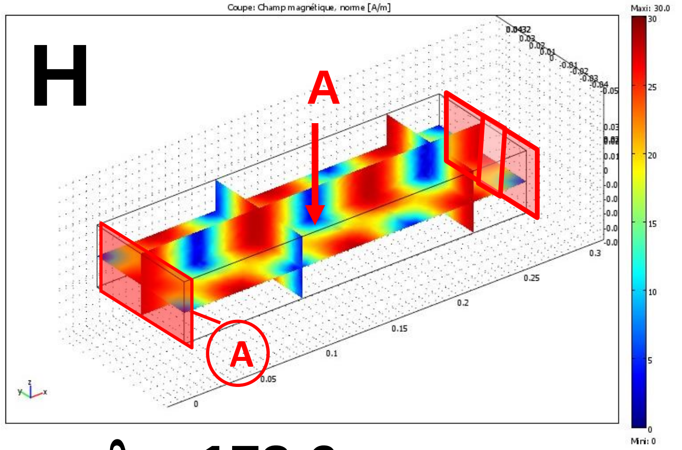
Maximums of the E (V/m) at fixed position

Maximums of the E (V/m) are moving

In a Single- mode Cavity : 'We have stationnary wave'







λ_g=173,6 mm

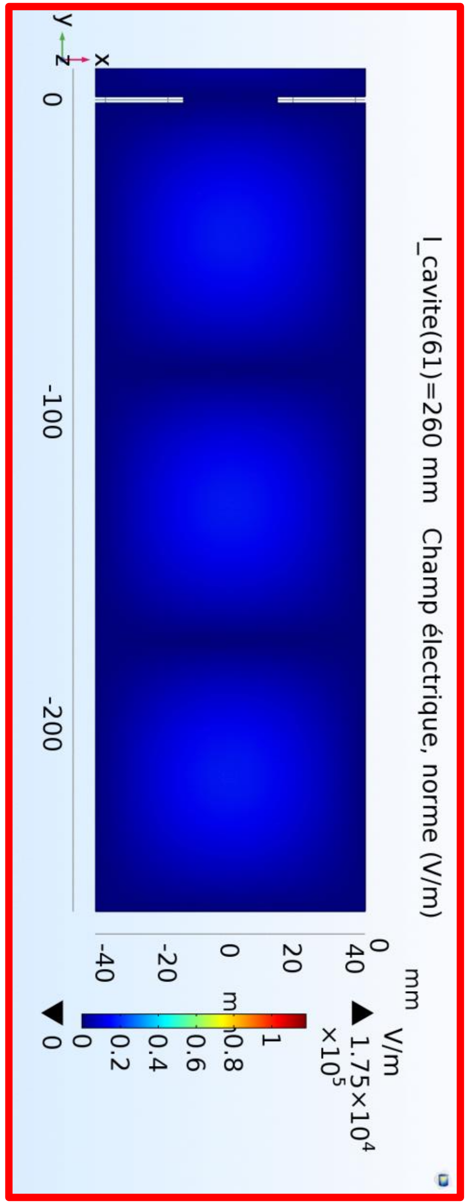
Short Circuit in A position

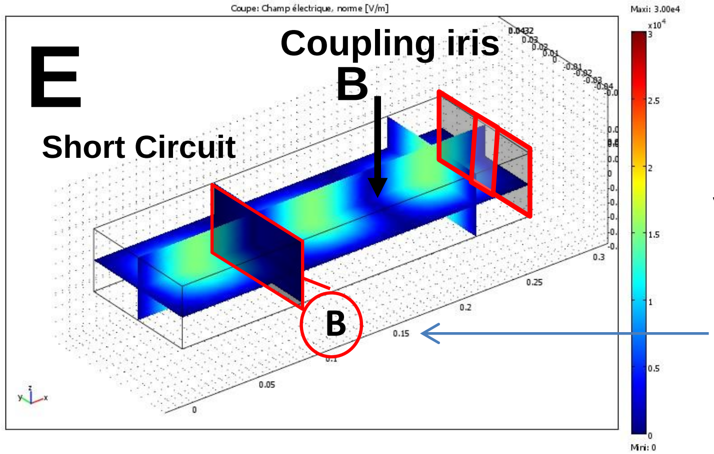
TE103: E field, L=1,5 λ_g

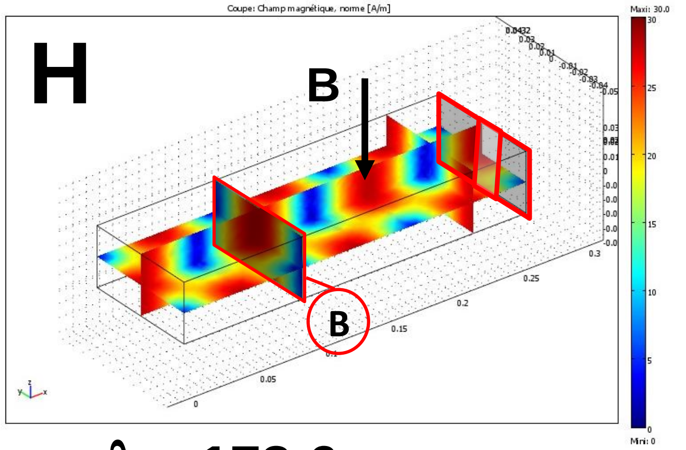
Sample located in a max. of E

<u>E field mode</u>

In a Single- mode Cavity : we have stationnary waves







λ_g=173,6 mm

Short Circuit in B position

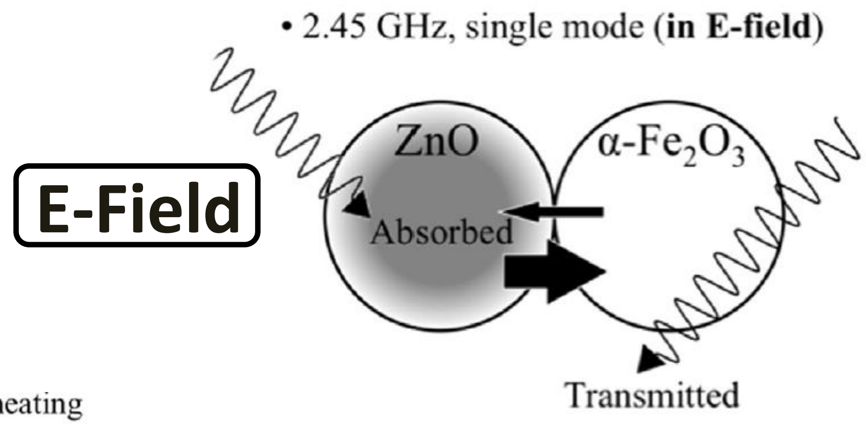
TE102: H field, L=1 λ_g

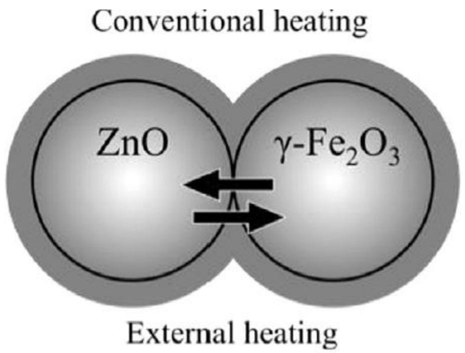
Sample located in a max. of H
<u>H field mode</u>

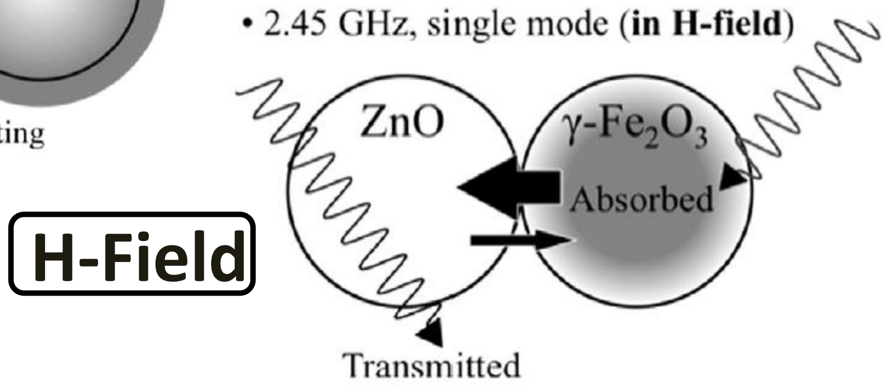
That is why some people say we are working in **« pure » E field (for dielectrics)**

Or **in « pure » H field** (for semi-conductors for instance)

Microwave heating depends on the electrical properties of the materials, but it may also be different if you use the E-field or the H-field



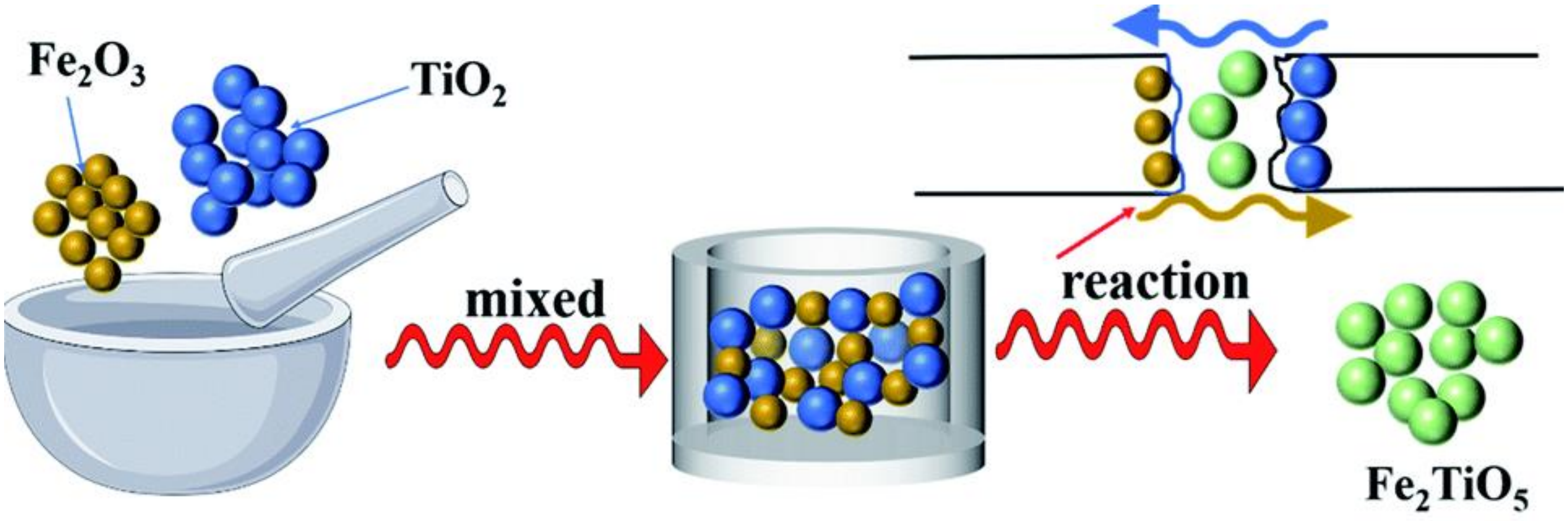




it gives you many possibilities to heat materials - This way of heating can be very versatile

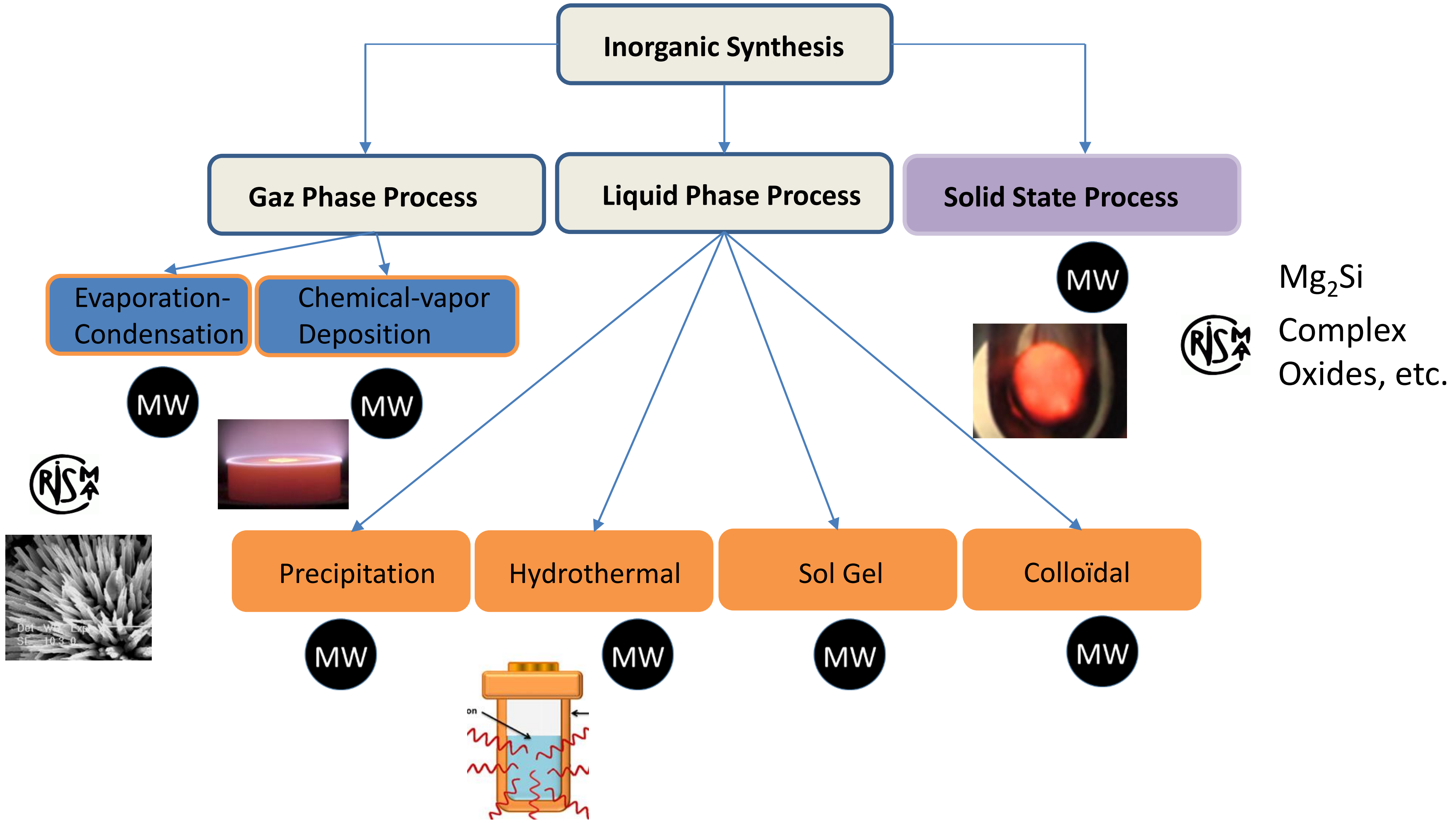
Nagao et al.

Ceramics International 41 (2015) 14021–14028



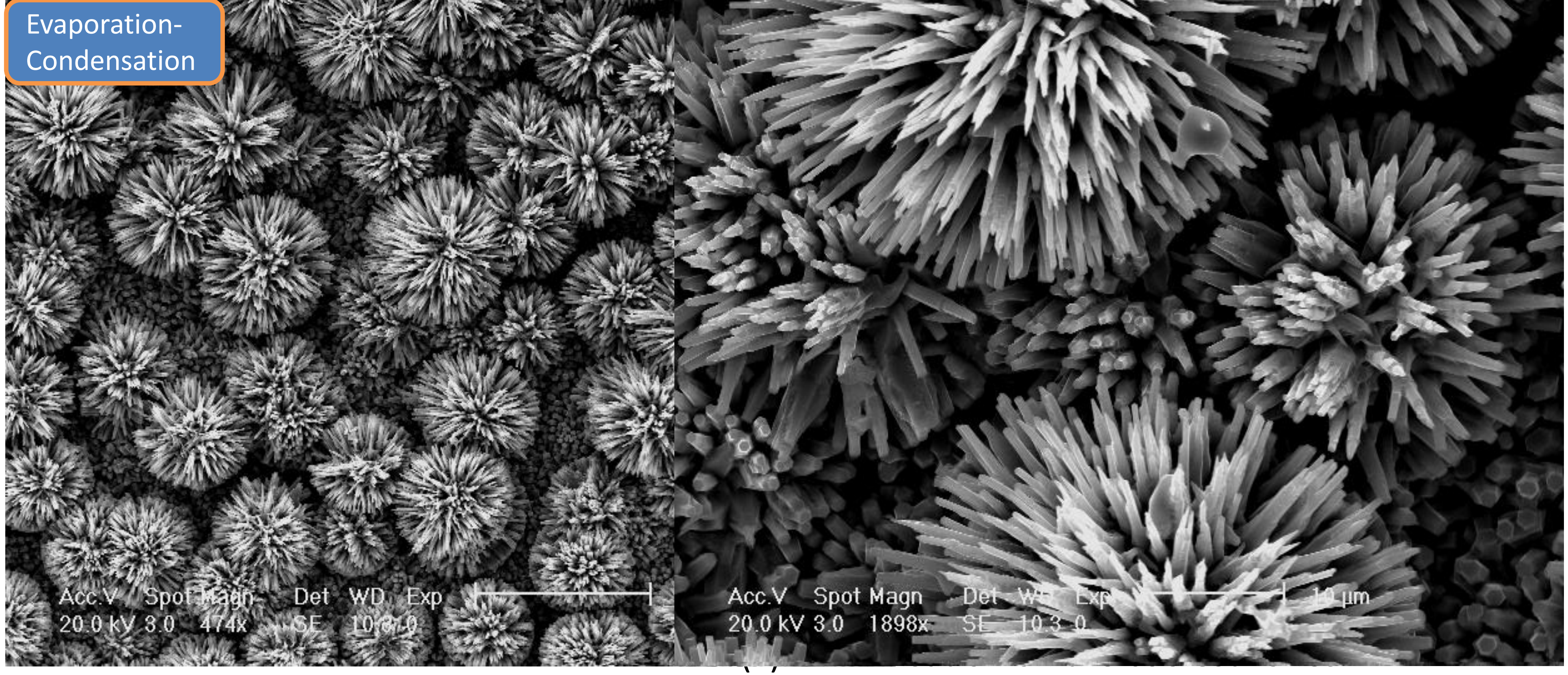
II- A QUICK VIEW OF MICROWAVE ASSISTED <u>**INO**</u>RGANIC SYNTHESIS and SINTERING

Different methods to get inorganic compounds: actually MW technology can be found in most of them :



EXAMPLE 1

Flower-like shape ZnO made by sublimation-recrystallisation

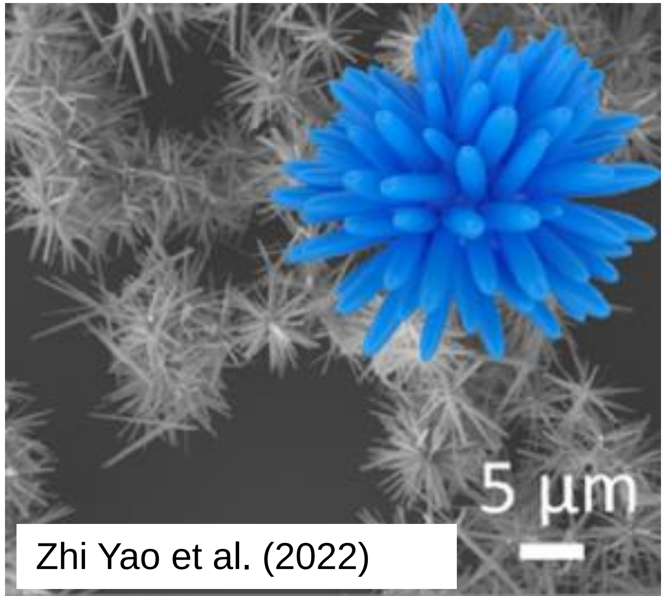


- Similar Nanostructures were obtained by precipitation techniques (C. *Wu et al. MRB (2007), etc.)*

- MW fast heating in vaccum: Simple and fast process !

Some interesting applications:

* flower-like ZnO superstructures embedded in the PVA-co-PE nanofiber membrane, to prepare air-filter.
* flower-like ZnO for luminescence properties
F. Koao et al. [Mat. Sci. Semicond. Proc. 27](), 2014. etc.



Zhi Yao et al. (2022)

EXAMPLE 2

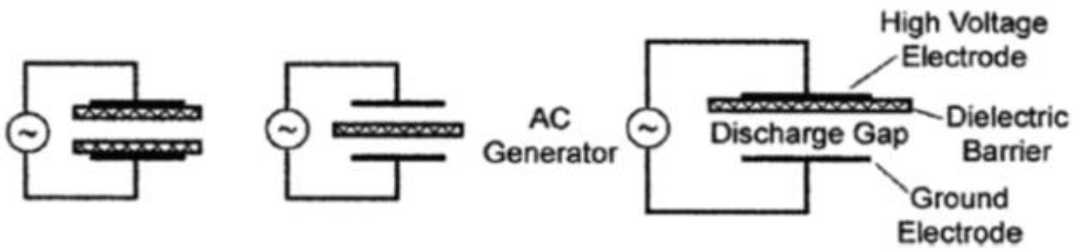
MICROWAVE PLASMA *ENHANCED CVD: Growth of synthetic diamonds*

Chemical-vapor Deposition

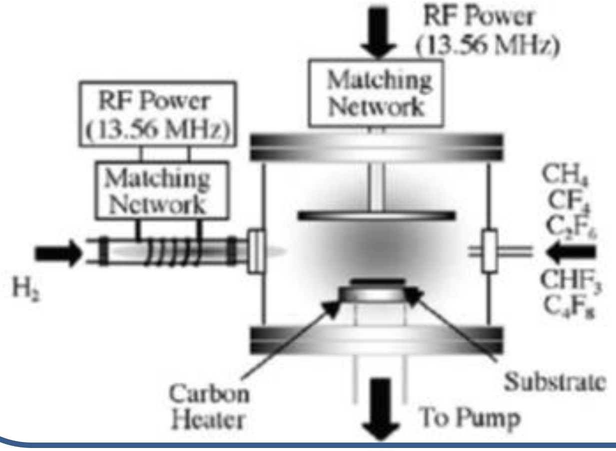
CVD technique is widely used in the field of Thin films technology

Different ways of producing PLASMA

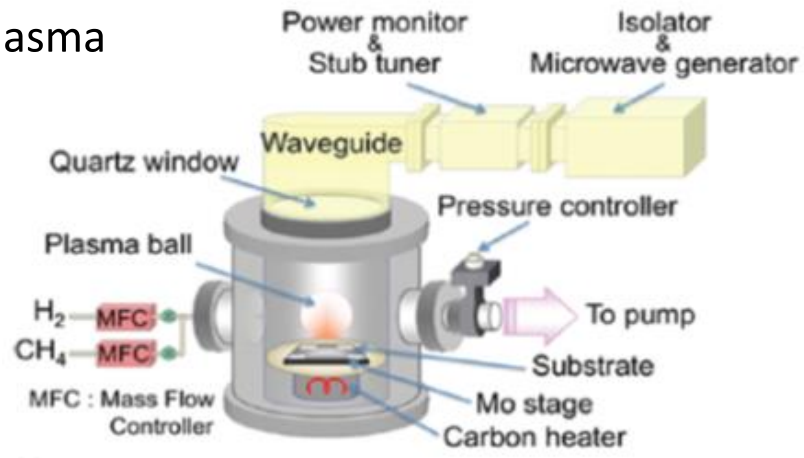
Dielectric discharge barrier



RF plasma



MW plasma



Advantages of MW PLASMA:

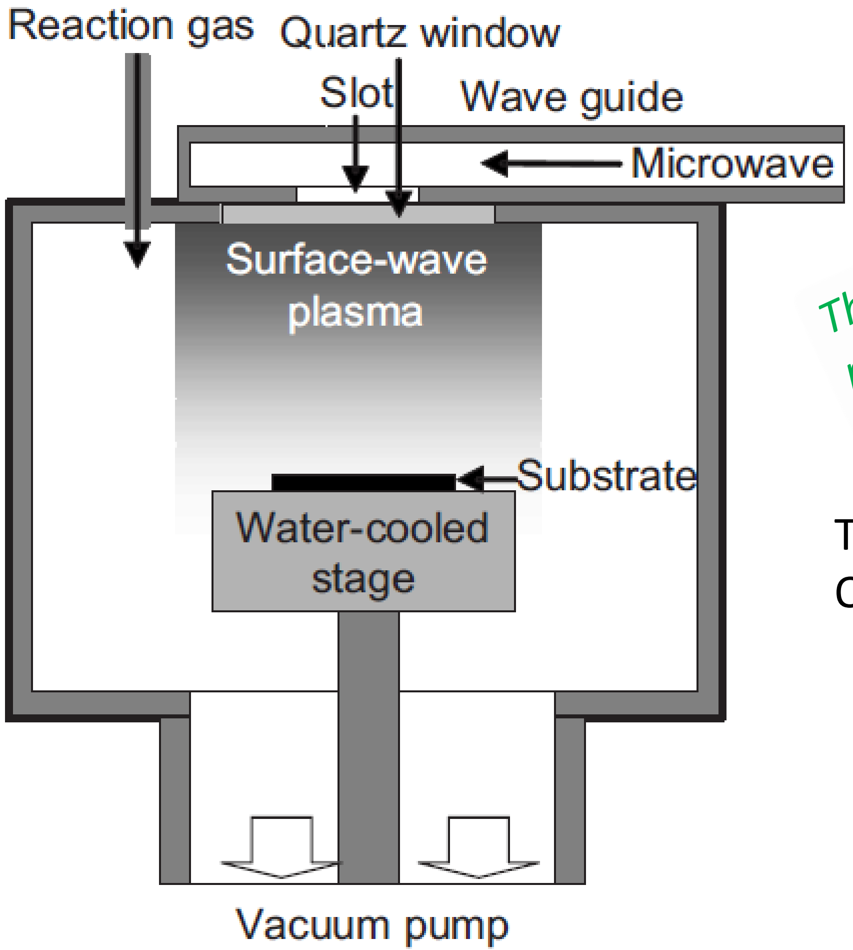
- Usually called « non thermal plasma » thanks to the low plasma temp. (contrarywise to arc plasma)
- No electrodes

Plasma-Enhanced Chemical Vapor Deposition: Where we are and the Outlook for the Future

Yasaman Hamedani Et al.
http://dx.doi.org/10.5772/64654

Chemical Vapor Condensation

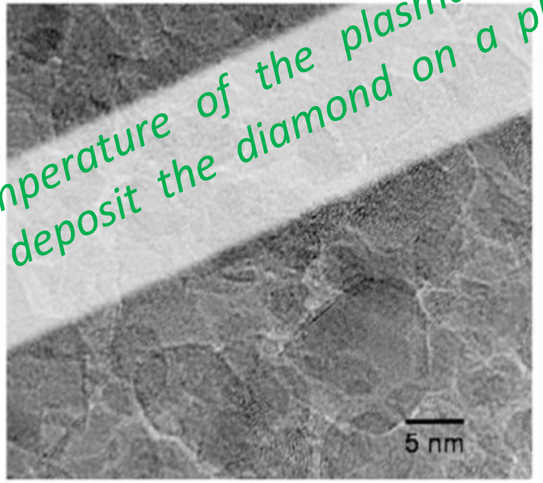
Example of Nanocrystalline diamond film growth on plastic substrates at temperatures below 100 °C from low-temperature plasma



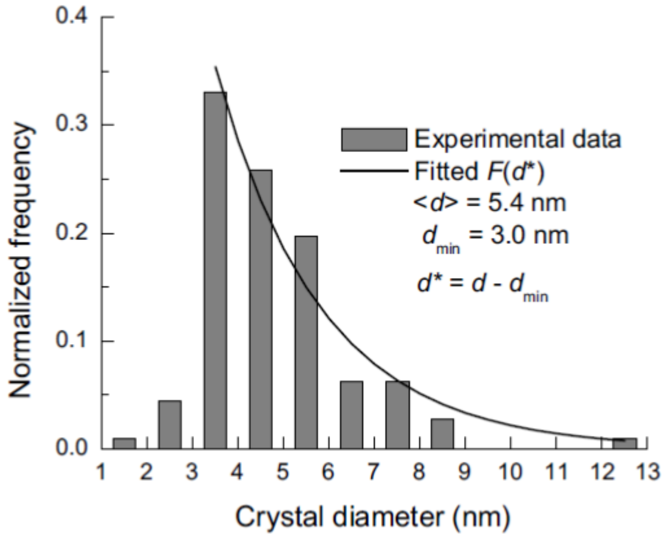
Kazuo Tsugawa et al.

PHYSICAL REVIEW B **82**, 125460 (2010)

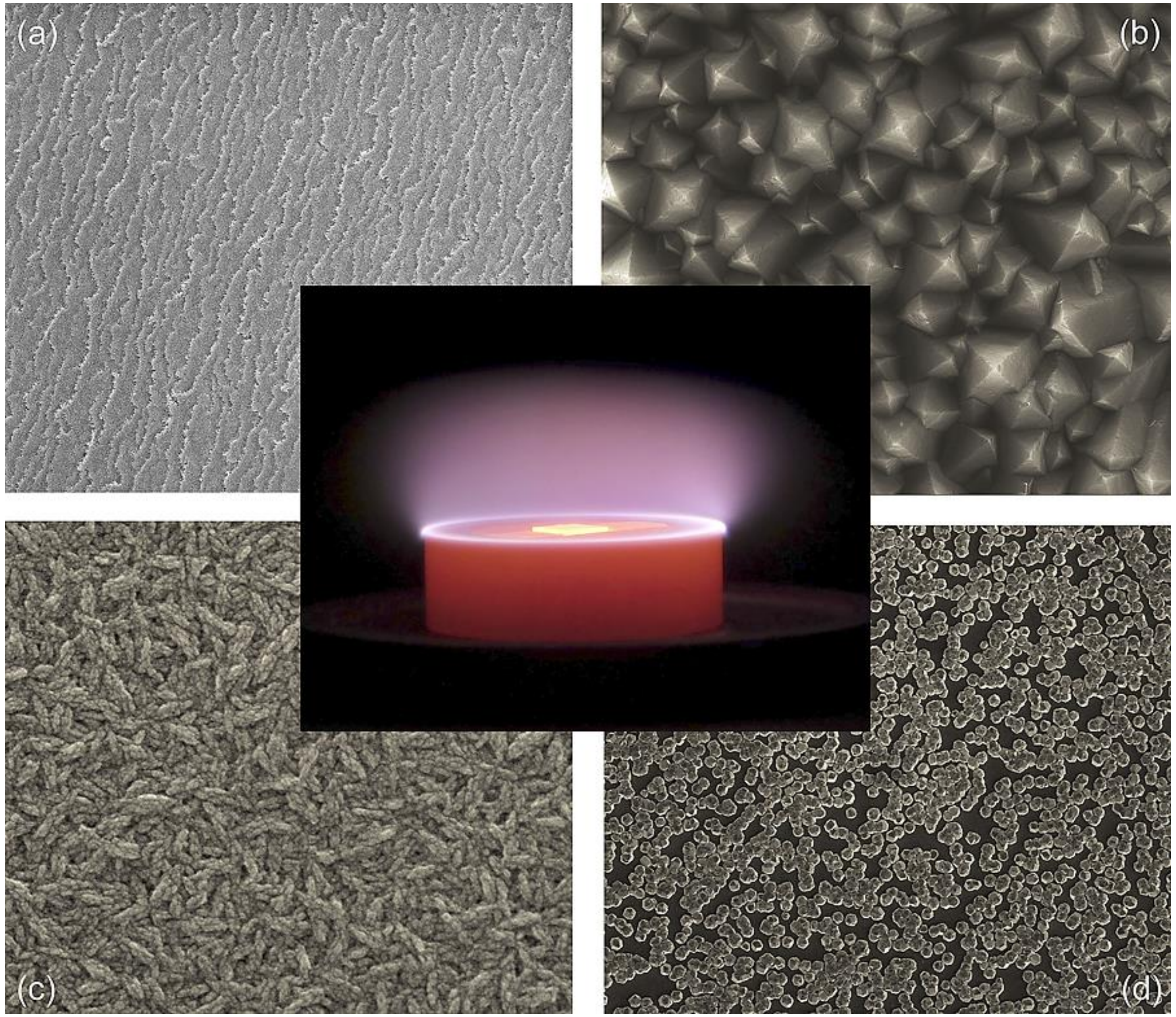
The low temperature of the plasma makes possible to deposit the diamond on a plastic substrate



TEM observation of the NCD deposited
On plastic and the granulometric distribution



http://sekidiamond.com/microwave-plasma-cvd-systems/

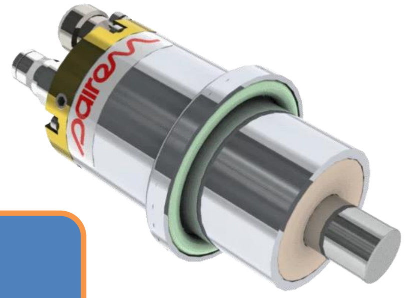


Different type of nano cristalline diamond (depending on the experimental conditions)
Courtesy MRS Bulletin, Vol 39, June 2014

Huge Market in which
MW plasma CVD process
grows over the high temp.
Hig pressure process

Synthetic diamond!

Jewelry
Electronics,
Mining
Machining
Construction etc .



Chemical
Vapor Condensation

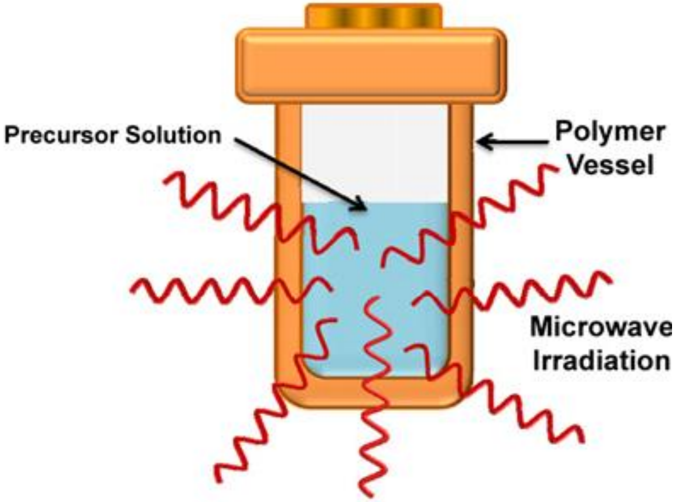
EXAMPLE 3

MICROWAVE HYDROTHERMAL PROCESS

Liquid Phase Method
Ex. hydrothermal

The hydrothermal synthesis process is a method for growing single crystals from an aqueous solution in an autoclave (a thick-walled steel vessel) at high temperature (400 deg. C) and pressure.

Royal society definition



Microwave hydrothermal process was first developped by Komarneni et al In the mid 90s. @ PennState University. USA

Thanks to the volumetric microwave heating, the solvant is rapidly and uniformly heated by microwaves.
This efficient heating way is beneficial for getting
- Finner grain size with a narrow granulometric distribution
- Fast process
- Many types of materials can be produced at 200 °C

There are many commercial MW autoclaves available in the market
From Milestone, CEM, Biotage, MARS, Anton Paar , etc…

Liquid Phase Method
Ex. hydrothermal

Conventional and Microwave Hydrothermal Synthesis and Application of Functional Materials: A Review

Guijun Yang and Soo-Jin Park *Materials* 2019, *12*, 1177; doi:10.3390/ma12071177

Comparison: MW versus CV Hydrothermal process

CV: Zirconia - 24 hours – 150 °C – 20-50 nm

MW: Zirconia - 30 min. 150 °C – 10-20 nm

CV: TiO_2 - >8 hours – 195°C –50 /100 nm

MW: TiO_2 - 10 min. – 195°C –10 nm

Table 2. Comparison of the morphology, particle size, and reaction conditions using the hydrothermal and microwave hydrothermal methods.

	Hydrothermal Method					Microwave Hydrothermal Method				
	Morphology	Raw Materials	Conditions	Size	Ref.	Morphology	Raw Materials	Conditions	Size	Ref.
ZrO_2	Spherical	$ZrOCl_2 \cdot 8H_2O$, NH_4OH, NaOH	150 °C, 24 h	20–30 nm	[87]	Monoclinic	$ZrOCl_2 \cdot 8H_2O$, NaOH	200 °C, 2 h, 2.45 GHz	10–20 nm	[88]
	Rod	$ZrOCl_2 \cdot 8H_2O$, NH_4OH, NaOH	200 °C, 24 h 250 °C, 24 h	50 nm × (200–400) nm 80 nm × (200–500) nm	[87]	Tetragonal-monoclinic	$ZrOCl_4$, NaOH	150–220 °C, 30 min	~20 nm	[89]
Al_2O_3	Hollow	$Al(NO_3)_3 \cdot 9H_2O$, glucose	160 °C, 3–8 h	5.4–6.9 µm	[64]	Hollow	$KAl(SO_4)_2 \cdot 12H_2O$, $CO(NH_2)_2$	180 °C, 40 min, 300 W	0.8–1.2 µm	[90]
	Rod	$Al(NO_3)_3 \cdot 9H_2O$, $N_2H_4 \cdot H_2O$	200 °C, 12 h	8 nm × (220–532) nm	[65]	Fiber	Surfactant Brij 56, H_2SO_4, Aluminum sec-butoxide	80 °C, 30 min, 500 W	~50 nm	[91]
MnO_2	Belt	Mn_2O_3, NaOH	170 °C, 12 h	5–15 nm	[92]	Flower Nanosheet Fiber	$KMnO_4$, HCl	100 °C, 25 min 140 °C, 25 min 180 °C, 25 min	200–400 nm 10 nm 2–6 µm	[94]
	Urchin Urchin Nanowire	$MnSO_4$, $(NH_4)_2S_2O_8$	80 °C, 4 h 110 °C, 4 h 140 °C, 4 h	2–3 µm 30–40 µm ultrathin	[83]	Nanosphere	$KMnO_4$, $MnSO_4 \cdot H_2O$	75 °C, 30 min	70–90 nm	[95]
TiO_2	Nanotube	TiO_2, NaOH	150 °C, 48 h	8.1–27.3 nm	[96]	Nanowire	TiO_2, NaOH	210 °C, 2 h, 350 W	80–150 nm	[96]
	Acicular	$TiOCl_2$	195 °C, >8 h	100 nm × 50 nm	[97]	Spherical	$TiOCl_2$	195 °C, >30 min, 2.45 GHz	10 nm	[97]

Liquid Phase Method
Ex. hydrothermal

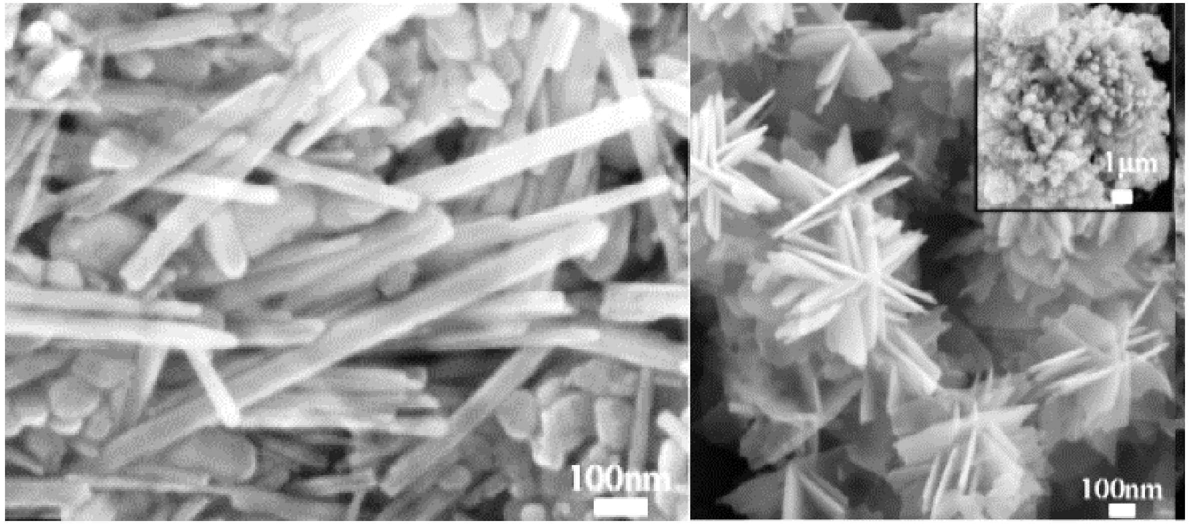
Microwave Hydrothermal and Solvothermal Processing of Materials and Compounds

http://dx.doi.org/10.5772/45626 (2012) Boris I. Kharisov Et al.

Exemple of ZnO nanorods/flowers

$$Zn(NO_3)_2 + 2NaOH \rightarrow Zn(OH)_2 \downarrow + 2NaNO_3$$

$$Zn(OH)_2 + 2H_2O \rightarrow Zn(OH)_4^{2-} + 2H^+ \rightarrow ZnO + 3H_2O$$



Nano-rods and flower-like
Shape ZnO crystal obatined
by
MW hydrothermal process
From Zinc nitrate in basic
medium

Binary metallic oxides: ZnO, CuO, PdO, CoO, MnO, TiO_2, CeO_2, SnO2, HfO_2, ZrO_2, Nd_2O_3, In_2O_3, Tl_2O_3, Fe_2O_3, Fe_3O_4, and Mn_3O_4.[3,15,25–29]

Oxyhydroxides

Nanoporous materials such as zeolites

Complex oxides such as perovskites and spinels: $KNbO_3$, $ATiO_3$ (A = Ba, Pb), $NaTaO_3$, $BiFeO_3$,[15,35–38] and ZnM_2O_4 (M = Al, Ga) and AFe_2O_4 (A = Zn, Ni, Mn, Co).[39,40]

SOLID STATE ROUTE

(Microwave Synthesis and Sintering)

Solid state synthesis

… Now we are going to start from solid compounds !

Energy / high Temperature / solid state diffusion

$$A_{solid} + B_{solid} \longrightarrow C_{solid}$$

- High temperature is required (>800°C)
- Usually it takes time as reaction is controlled by atomic diffusion, which is a thermally activated process

MW often provides an efficient and rapid way to get compounds from solid-state reaction

Three examples:
- Synthesis of the intermetallic Mg_2Si
- Synthesis of the complexe oxide $CaCu_3Ti_4O_{12}$
- Microwave sintering of oxides (process controle and results)

✓ Self-Propagation synthesis of intermetallic compounds by microwave ignition

Fast synthesis of nanocrystalline Mg2Si by microwave heating: a new route to nano-structured thermoelectric materials, E. Savary et al. Dalton Transactions (2010)

MICROWAVE SYNTHESIS of NANO-STRUCTURED Mg_2Si

Expected reaction:

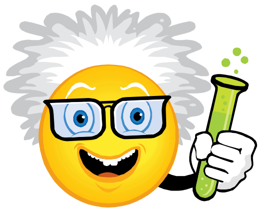
$$2Mg + Si \Rightarrow Mg_2Si$$

Energy

Solid state synthesis

FLASH MW Synthesis of Mg_2Si

Solid state synthesis



➢ The strategy

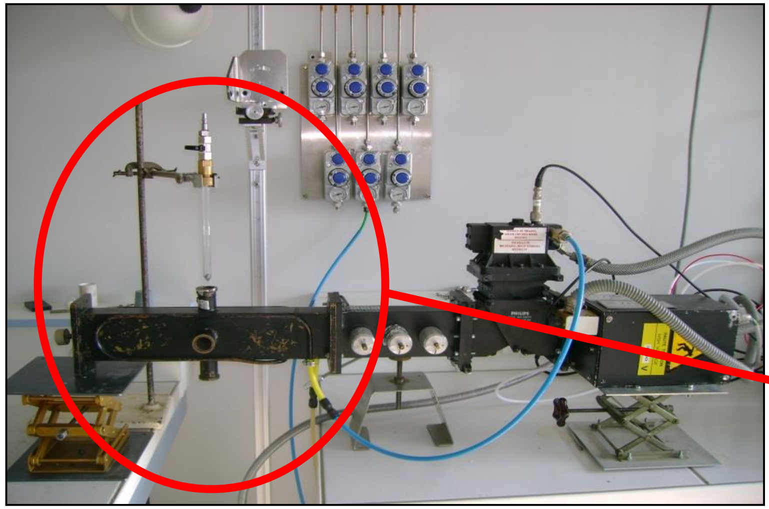
1. High energy ball-milling (mechanical alloying) between 2Mg+Si

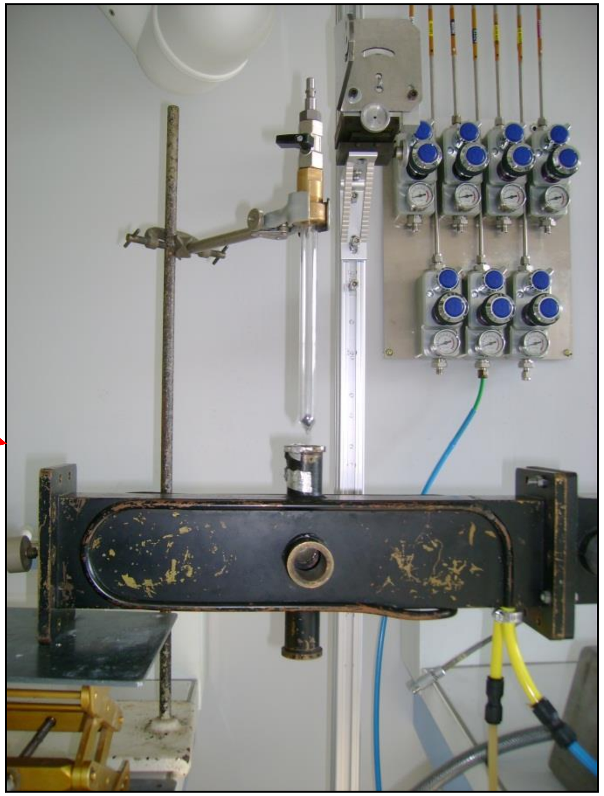
2. Fast MW heating of the mixture

Question : How do Mg and Si react to MW ?

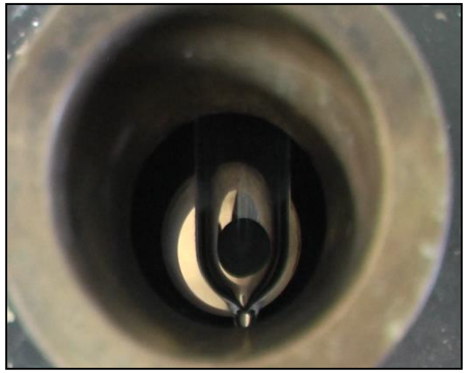
Solid state synthesis

- Behavior of the precursors ‘Silicon and Magnesium’ under MW:

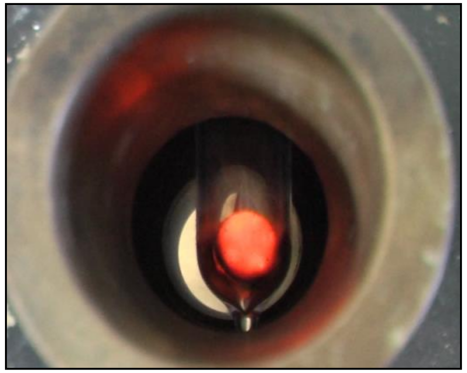




In H field



Mg sample into a sealed tube



Si heats by itself

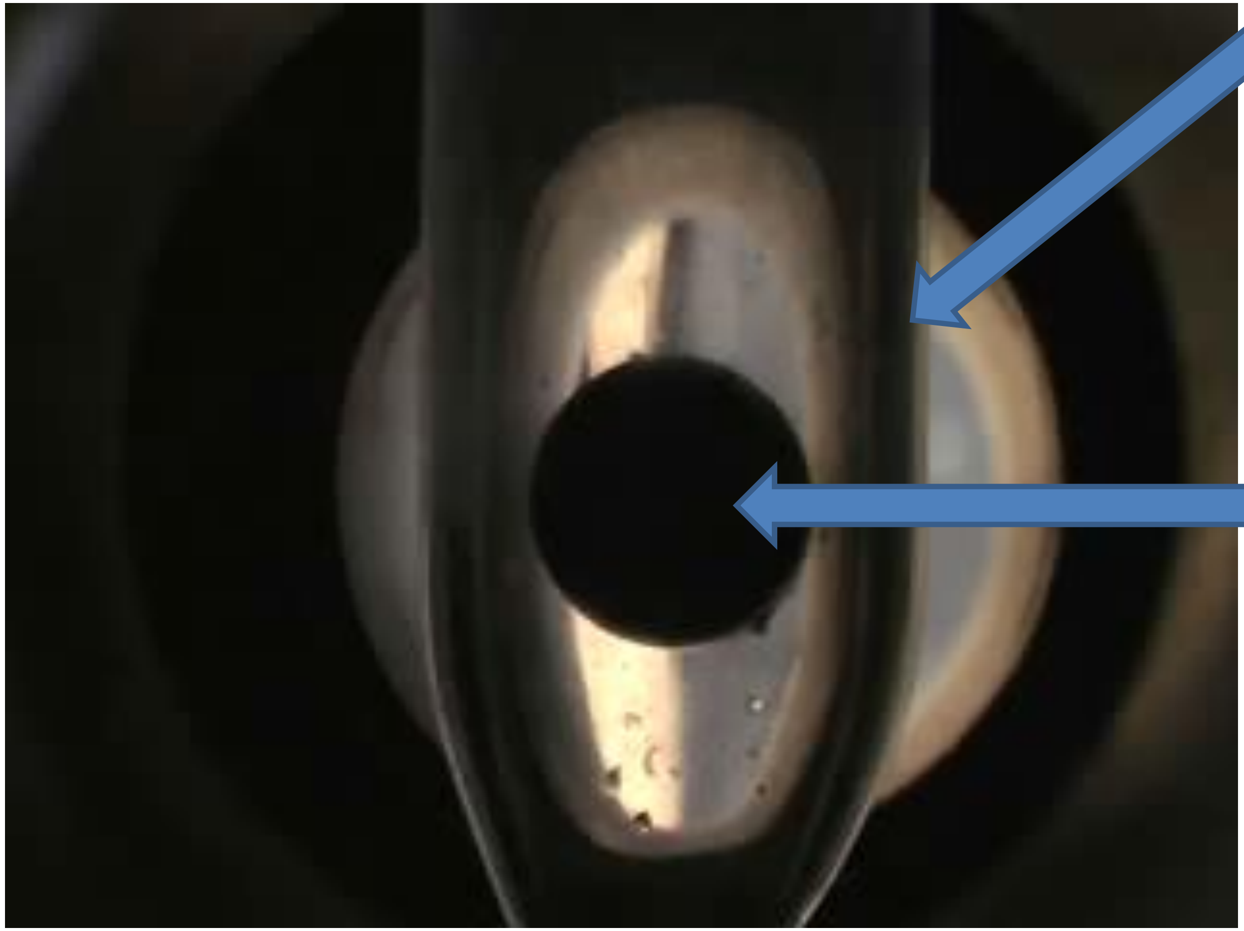
$\sigma(Mg) \approx 10^6$ S.cm^{-1}

$\sigma(Si) \approx 0{,}5\text{-}10$ S.cm^{-1}

Silicon couples well
Magnesium (too conductive) does not

MW Heating of the Si+2Mg mixture **IN REAL TIME**

Solid state synthesis



Sealed quartz Tube (in vacuum)

Pressed 2Mg+Si Powder

MW Power ~ 100 Watts

This Power is switched off when sample starts glowing.

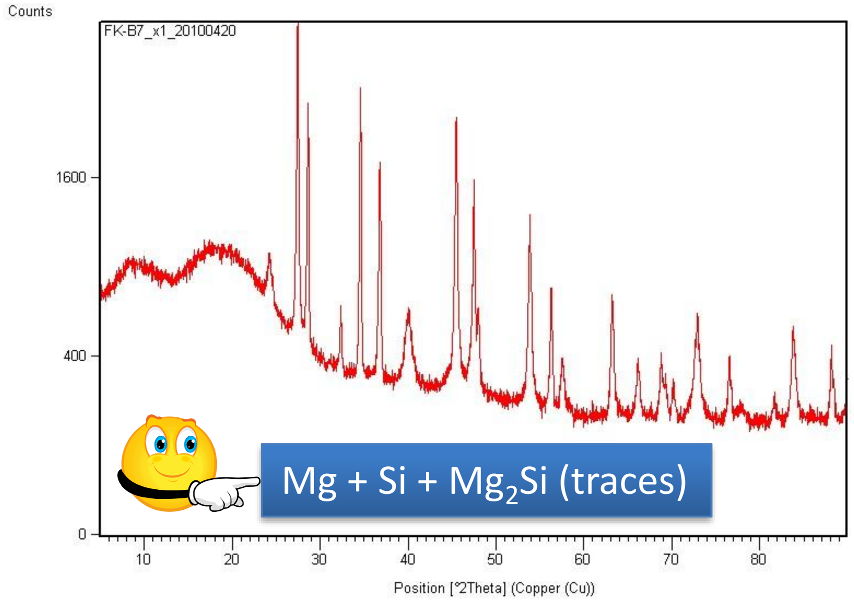
Synthesis of Mg_2Si Nano-grains by MW heating

Solid state synthesis

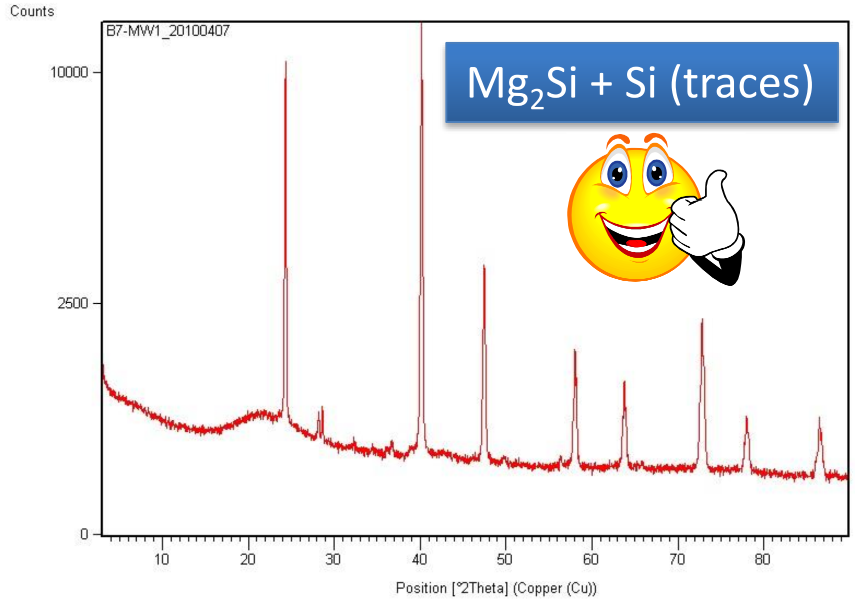
- XRD Diffraction patterns before and after the MW irradiation

(≈ 20 seconds)

XRD of the mixture (prepared by ball milling)



After MW irradiation



The 'Fast Synthesis' Strategy works well !

Savary et al., Fast Synthesis of Nanocrystalline Mg2Si by Microwave Heating: A new Route to Nano-Structured Thermoelectric Materials
2010, Dalton Transactions 39(45):11074-80

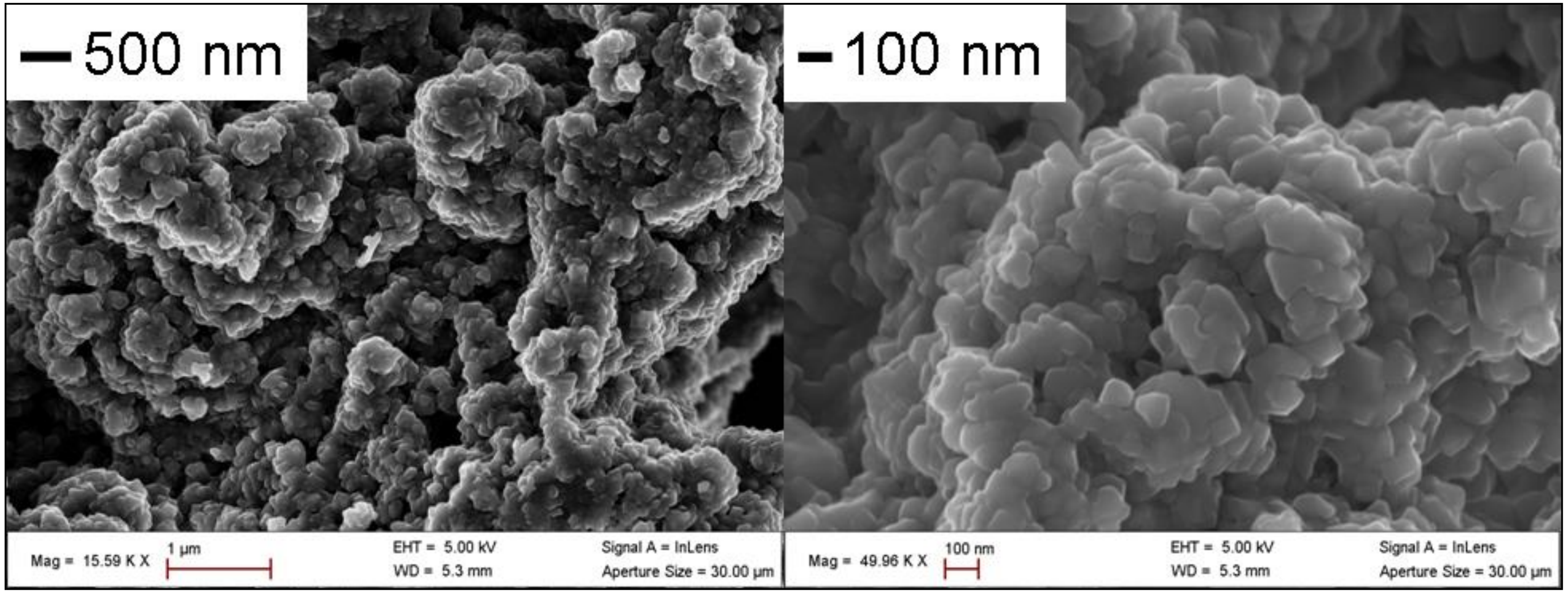
Synthesis of Mg_2Si Nano-grains by MW heating

- TOWARDS Nano-structuration

Grinding duration : 4x30 min ;

Microwave Incident power : 175W ;

Irradiation time after coupling = 2 seconds



Nano-structured Mg_2Si grains

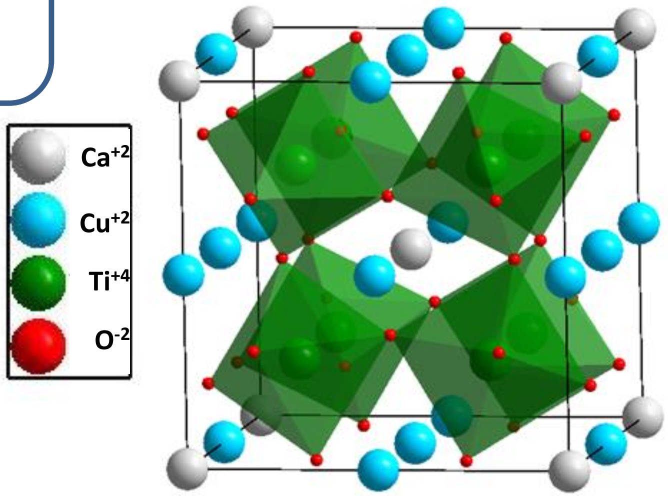
Savary et al., Fast Synthesis of Nanocrystalline Mg2Si by Microwave Heating: A new Route to Nano-Structured Thermoelectric Materials

2010, Dalton Transactions 39(45):11074-80

MICROWAVE SYNTHESIS (SS route) of $CaCu_3Ti_4O_{12}$

$CaCu_3Ti_4O_{12}$

a = 7.393 Å - SG : $Im\bar{3}$



$CaCu_3Ti_4O_{12}$:

- ✓ First synthesis in 1967 in CAEN at CRISMAT Laboratory By Deschanvres *et al. (1)*

- ✓ Subramanian et al. first reported original properties in 2000 (2)

* High dielectric constant $\varepsilon_r > 10^5$ (at room T°, 1 kHz)

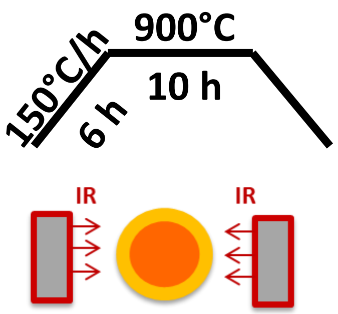
* High dielectric loss tan $\delta \simeq 0{,}1$

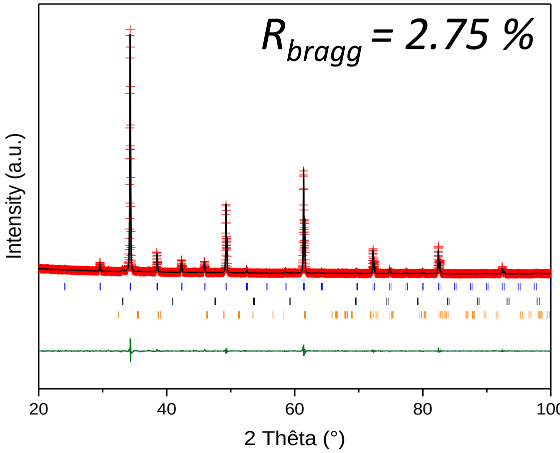
(1) Deschanvres A, Raveau B, Tollemer F (1967) Substitution of copper for a bivalent metal in titanates of perovskite type. Bulletin de la Societe Chimique de France 11:4077
(2) M.A. Subramanian et al. Journal of Solid State Chemistry 151 (2000)

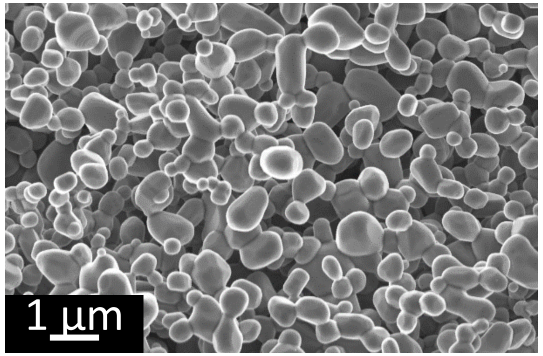
Solid state synthesis

$$CaCO_3 + 3\ CuO + 4\ TiO_2 \longrightarrow CaCu_3Ti_4O_{12} + CO_2$$

Conventionnal heating



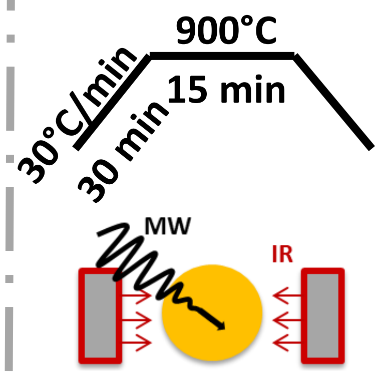


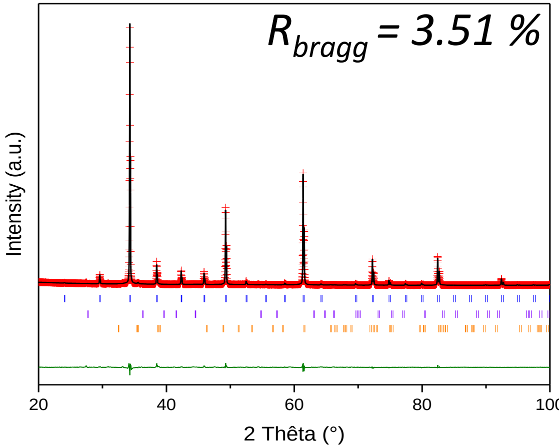


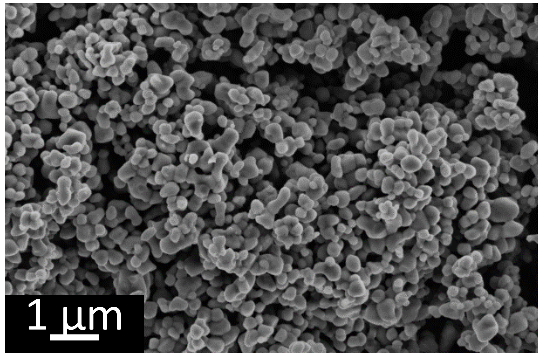
Pure CCTO after 3 calcinations (≈72h)

Grain size (700nm-1µm)

Hybrid microwave heating



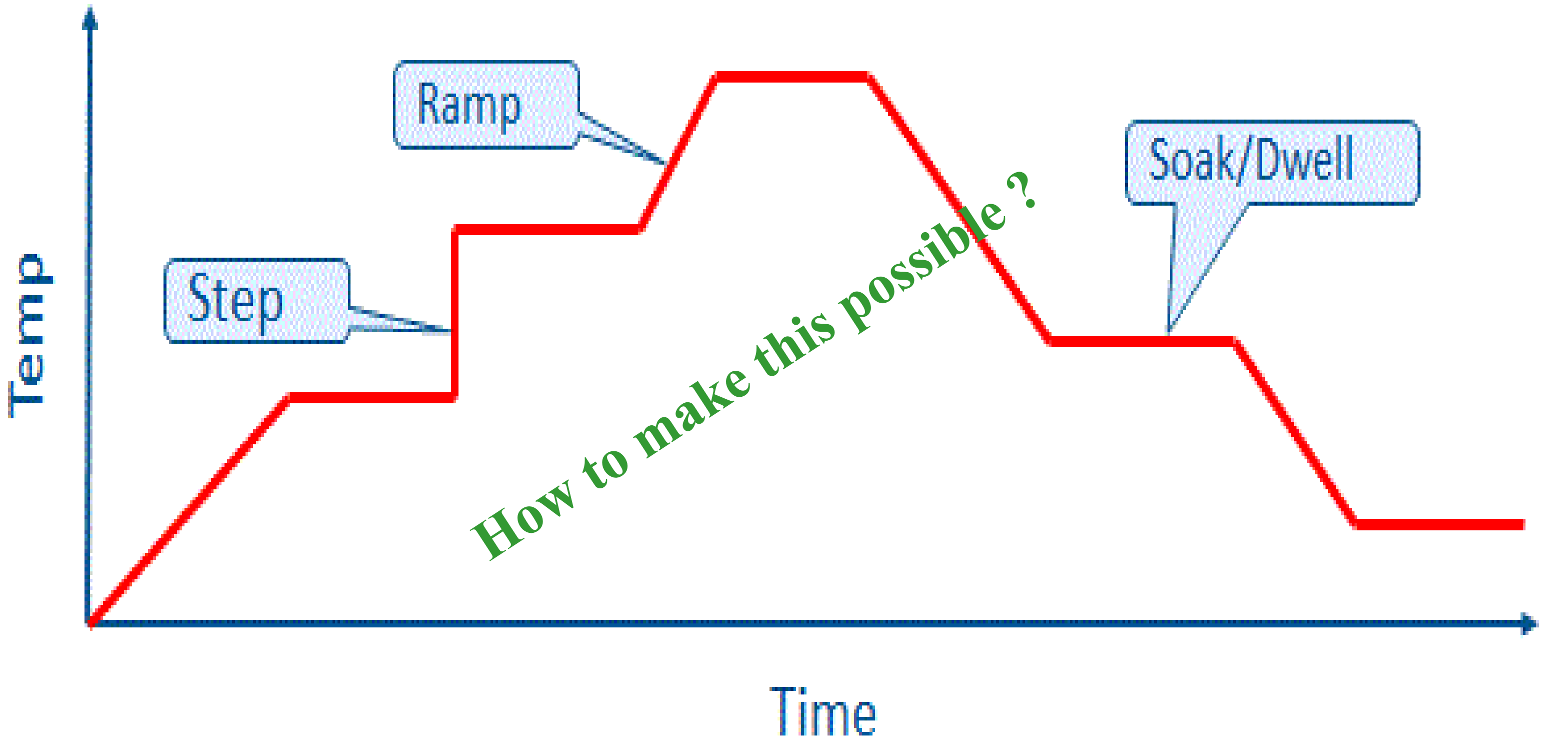




✓ Pure CCTO after 4 calcinations (≈4h)

✓ Grain size (300-500nm)

(1) Riquet et al., *Direct and hybrid microwave solid state synthesis of $CaCu_3Ti_4O_{12}$ ceramic : Microstructures and dielectric properties*, Ceram. Int. (2018)



Process control during high temperature microwave sintering of materials

Development of a fully automatized 915 MHz single-mode applicator for High Temperature Processing

Why 915 MHz ? ***(1) Larger penetration depth than in 2.45 GHz***

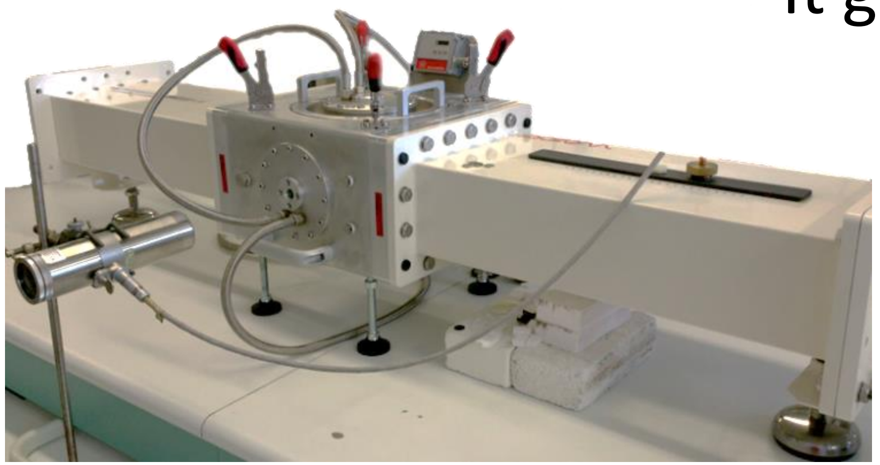
For example: ε^*=5-0,5j
(porous alumina at HT)

Dp [2.45Hz] ~ 8 cm
Dp [915 MHz] ~ 23 cm

More homogeneous temp. distribution expected

(2) Larger useful volume than in 2.45 GHz

It goes from 0,35 liter [2.45 GHz] to 9 liter [915 MHz]

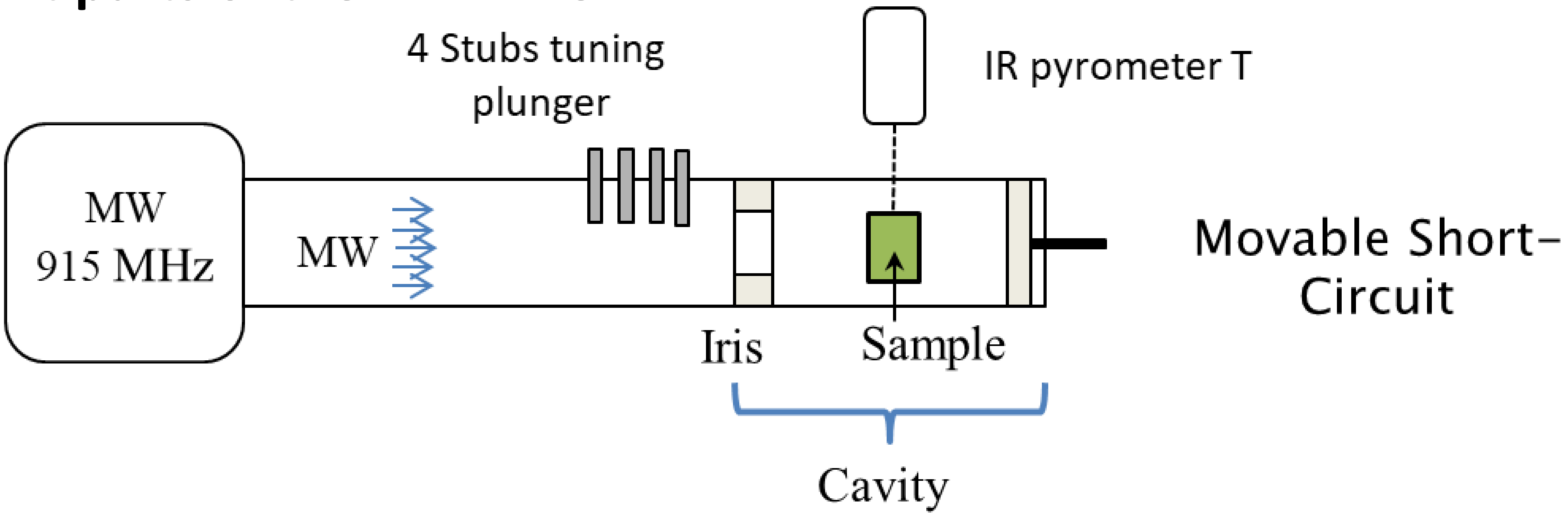


915 MHz

Why Using a single-mode applicator?

- **E,H Field distribution well known**
- **Higher energy efficiency than In multi-mode chamber !**

The different parts of the MW Line



The sample temperature results from the **balance power between absorbed power and dissipated power:**

$$\rho C_p \frac{\partial T}{\partial t} = \nabla.(k\nabla T) + p(x,T)$$

Absorbed Power *(If dielectric Losses)*

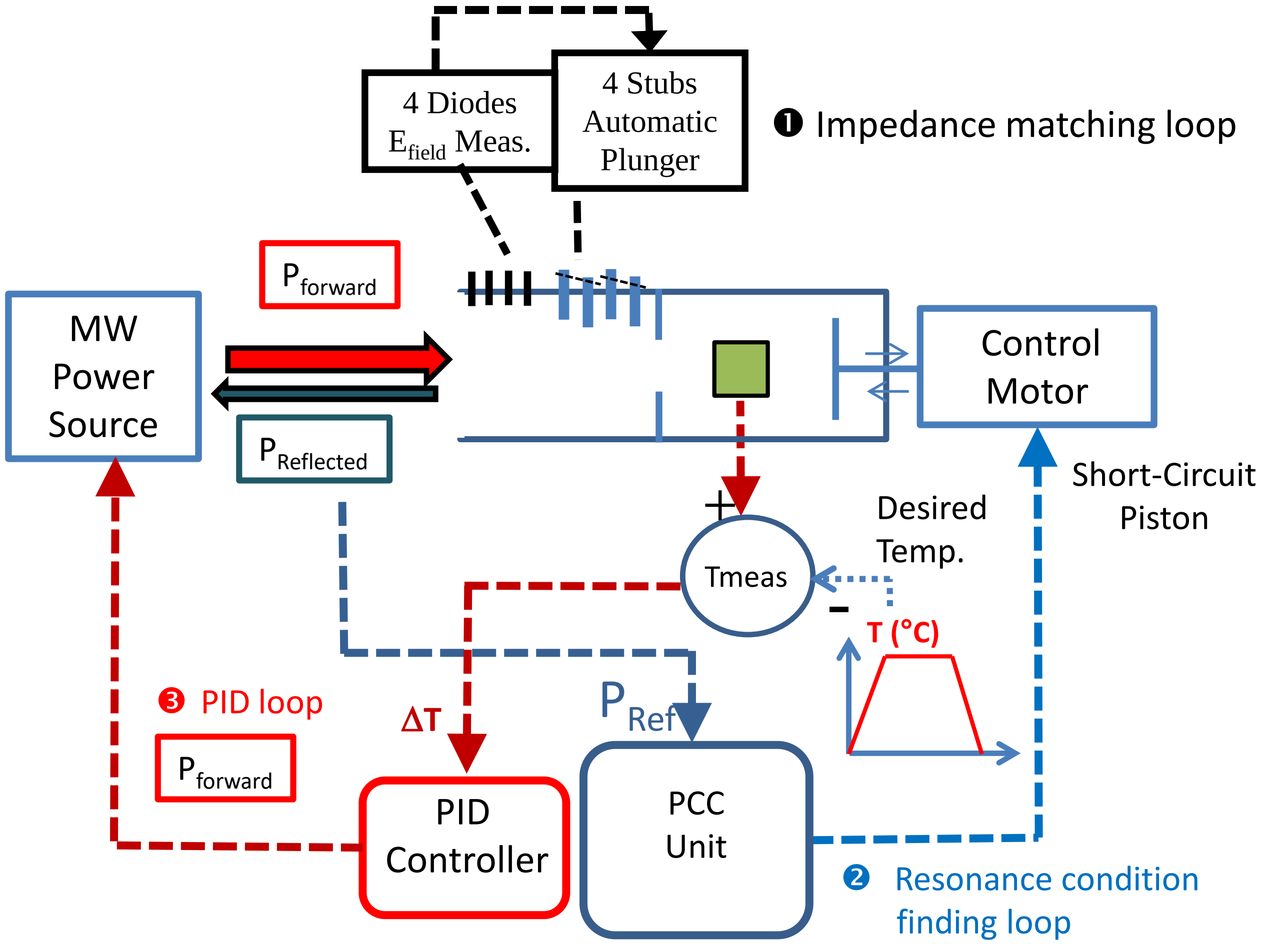
$$p(x,T) \propto E^2$$

Dissipated Power:

Thermal losses come from thermal radiation & thermal conduction mechanisms

MW heating highly depends on the material properties (T)

Block Function Diagram – Implementation: the system quite complicated but it works !



Maximum amount of the Microwave energy needs to be directed to the cavity (impedance matching)

❶ **Impedance Matching**:
First Loop: **Automatic Impedance Tuning is required**

Maximum E
(resonance conditions–amplification)

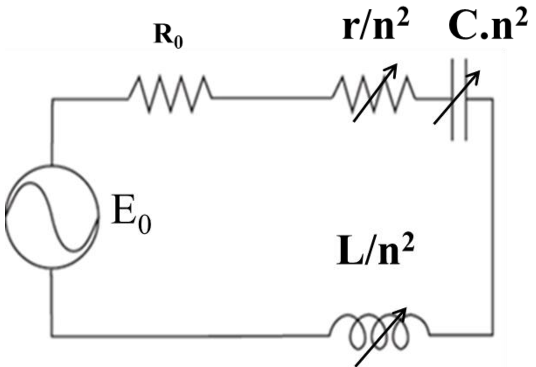
❷ **Resonance Mode:**
Second Internal Loop: **Resonance Conditions must be tuned automatically**

A PID module on the incident power must be implemented (to follow-up the Thermal cycle)

❸ **Incident Power:**
Microwave Power must be tuned to get the wished Thermal cycle (auto adaptive PID)

***More details:** Marinel et al. Adv. Mat. Sci. Eng. 2018*

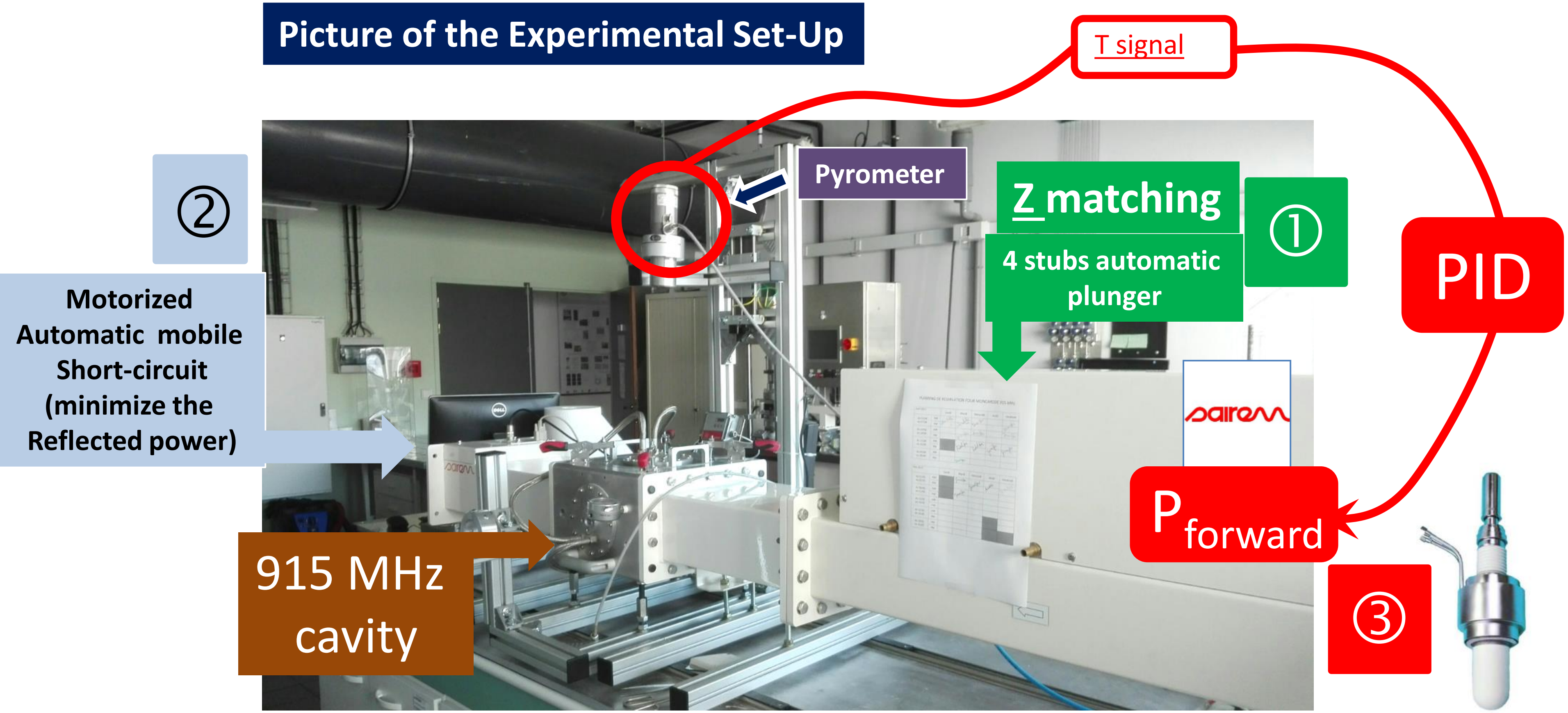
Advances in Materials Science and Engineering
Volume 2018, Article ID 4158969, 8 pages
https://doi.org/10.1155/2018/4158969



Analoguous Electrical Circuit
More details in *Marinel et al. Adv. Mat. Sci. Eng. 2018*

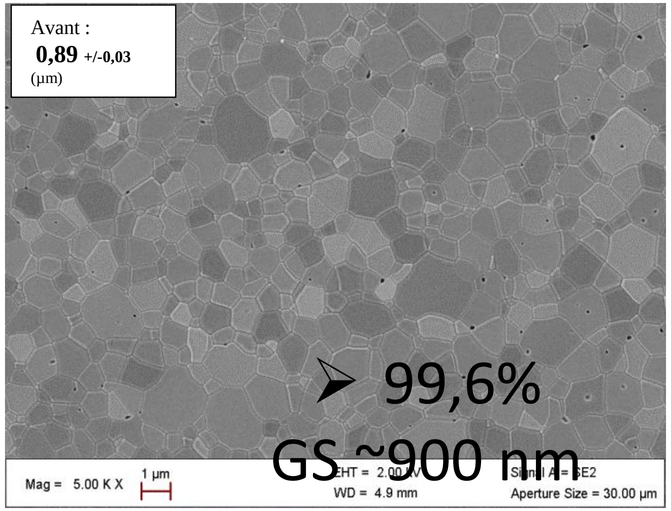
A fully automatic 915 MHz MW process for sintering:

How does it look like?



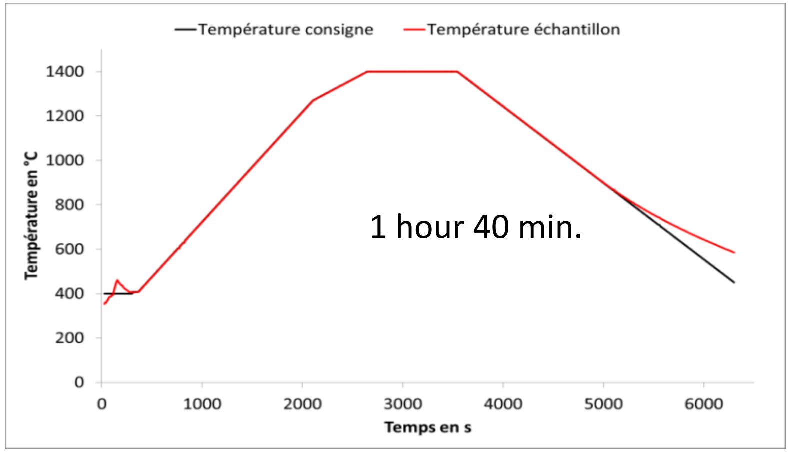
Microwave Sintering of large hexagonal pieces of Alumina: Microstructure Distribution & Reproducibilty

- Highly Pure Materials, Alumina from Baikowski (BMA15)
- Green Hexagonal shaped samples were slip-casted by our industrial Partner SOLCERA,
- Green density was ~60 % of the theoritical density

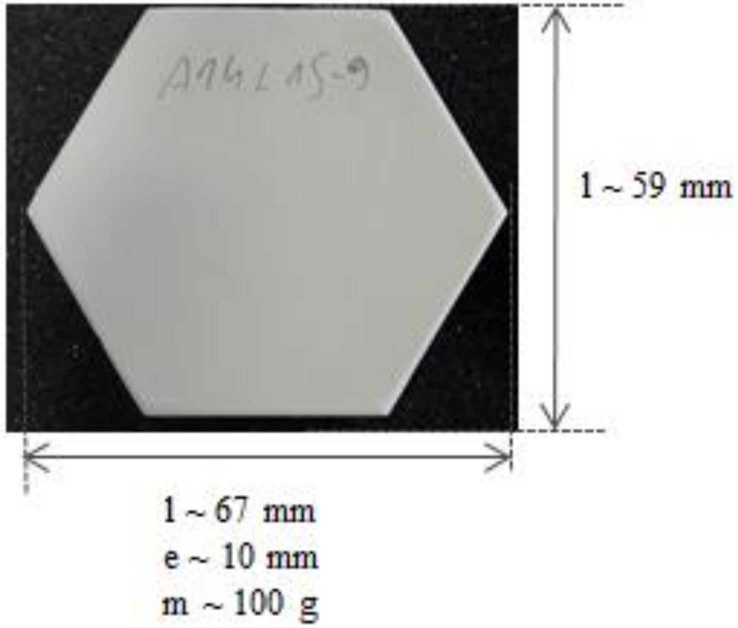


- 99,6%

GS ~900 nm



TYPICAL TEMP/TIME CYCLE



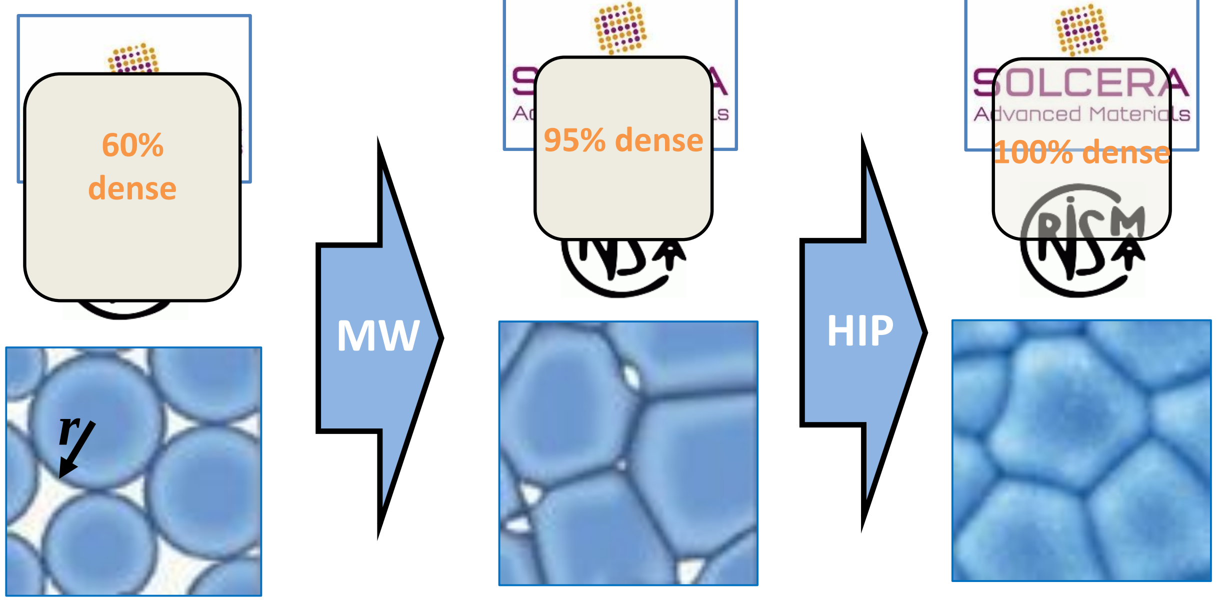
DGA project

MINES Saint-Étienne

SOLCERA Advanced Materials

INVESTIGATION OF THE MW SINTERING FOLLOWED BY HIP PROCESS TO GET TRANSPARENT SPINEL CERAMIC

Driving force $2\times\frac{\gamma}{r}$ $2\times\frac{\gamma}{r}+PHIP$



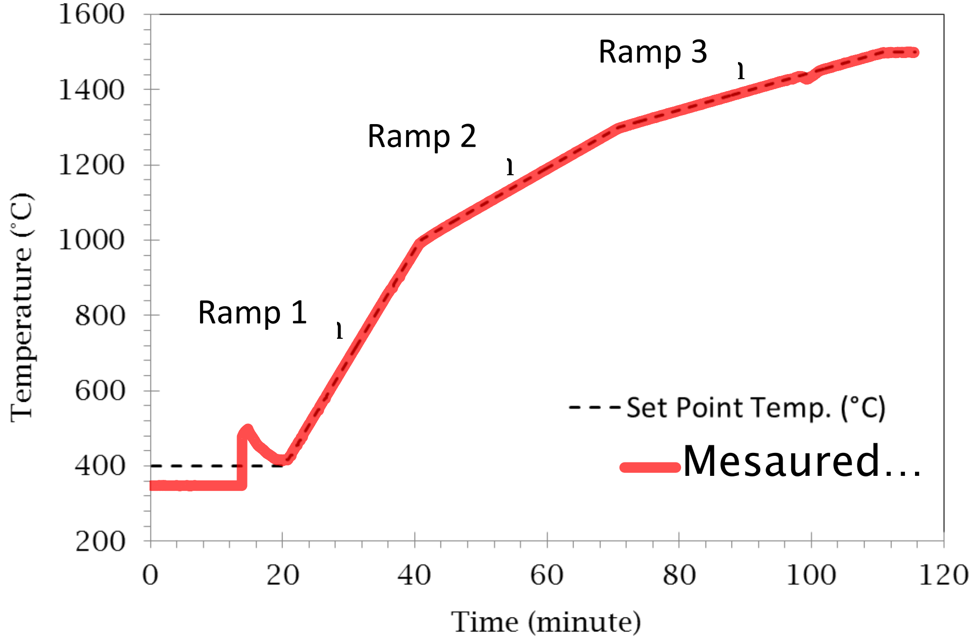
Green Sample
Slip casting
High-Quality
Green compact

MW Sintering
Open porosity ~ 0 %
Closed porosity remains

HIP Process
100 % dense
Transparent Ceramic
(no remaining porosity)

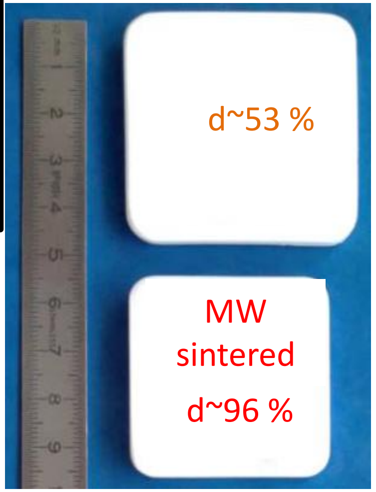
$MgAl_2O_4$

Multi-Step MW sintering of $MgAl_2O_4$ phase @ ~ 1500°C



65 mm × 65 mm Spinel Green Part Slip-Casted

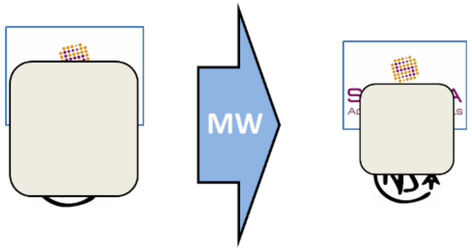
~ 50 mm × 50 mm MW sintered



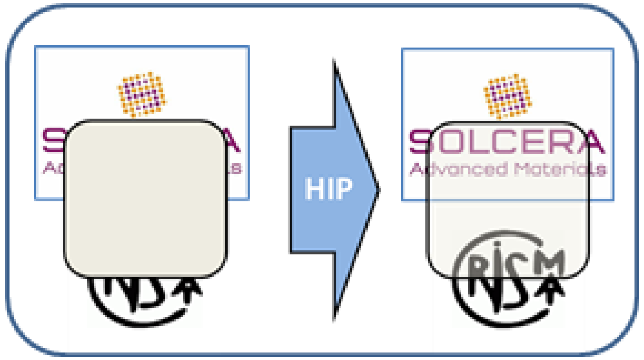
~ 4 % closed porosity

HIP process

(T°C – Pressure Confidential)



After HIP treatment to remove the remaining porosity

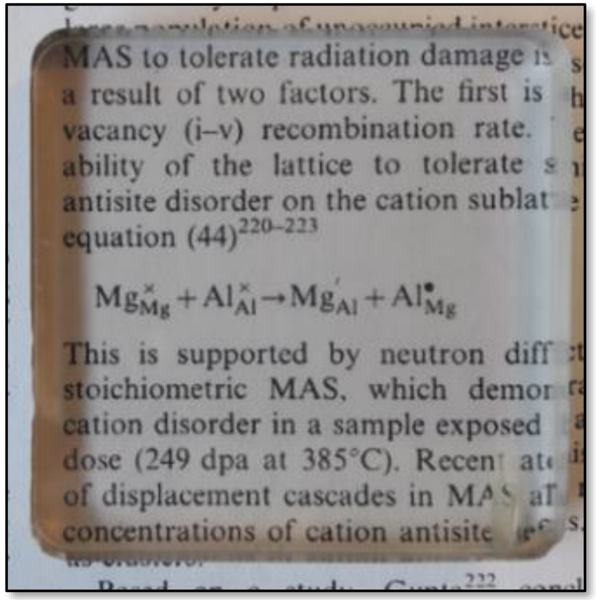


Spectrophotometre Perkin Elmer 1050 Lambda
(From CIMAP, CAEN)

At λ = 640 nm RIT ≈ 70 % and RIT ≈ 80 % In the Infra Red Region

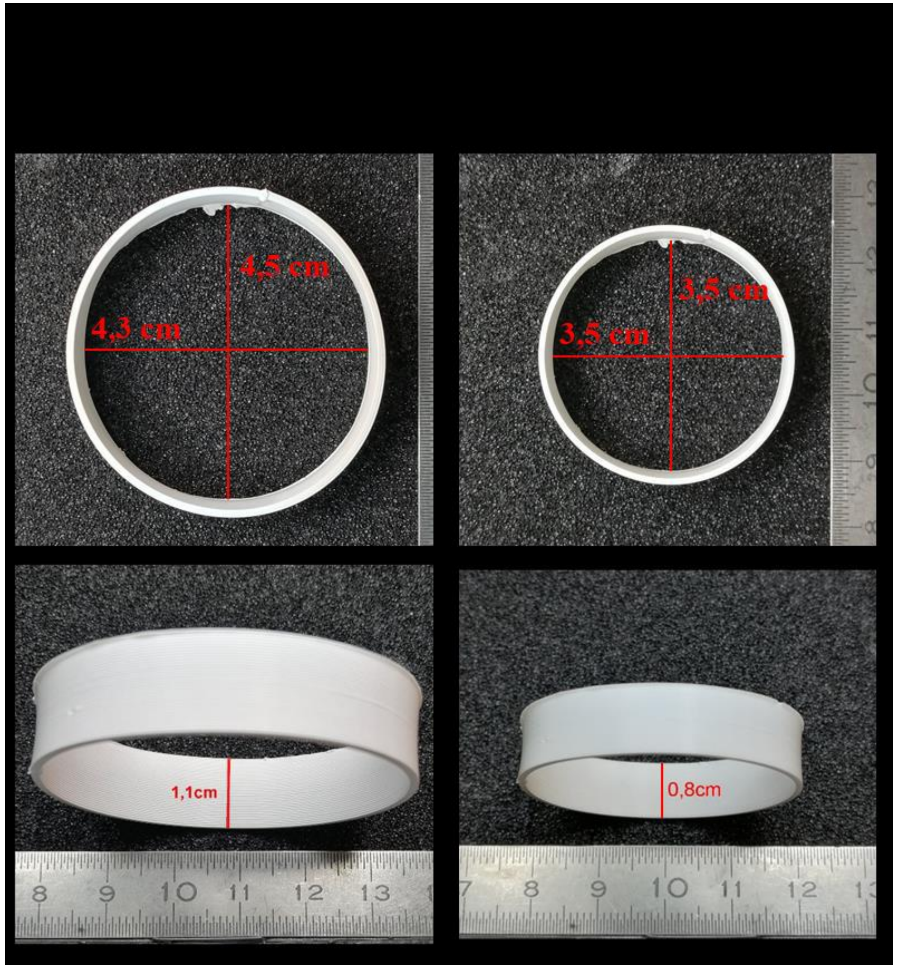
CiMap

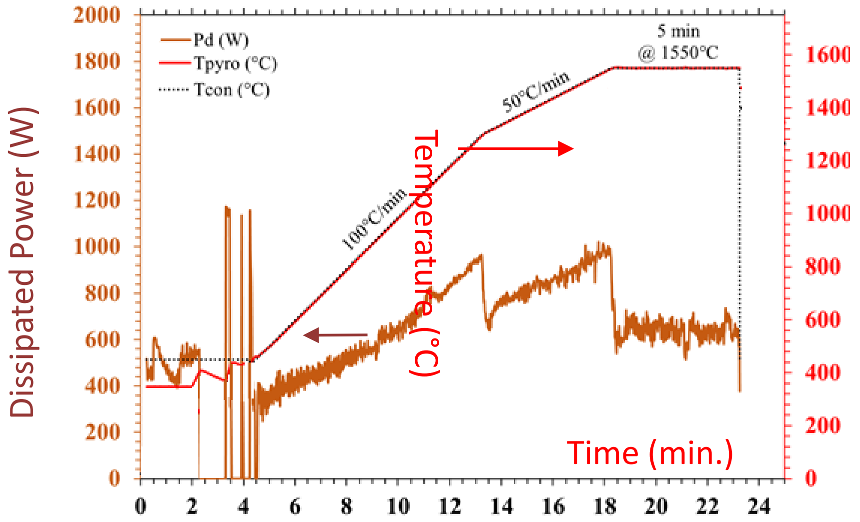
SOLCERA
Advanced Materials



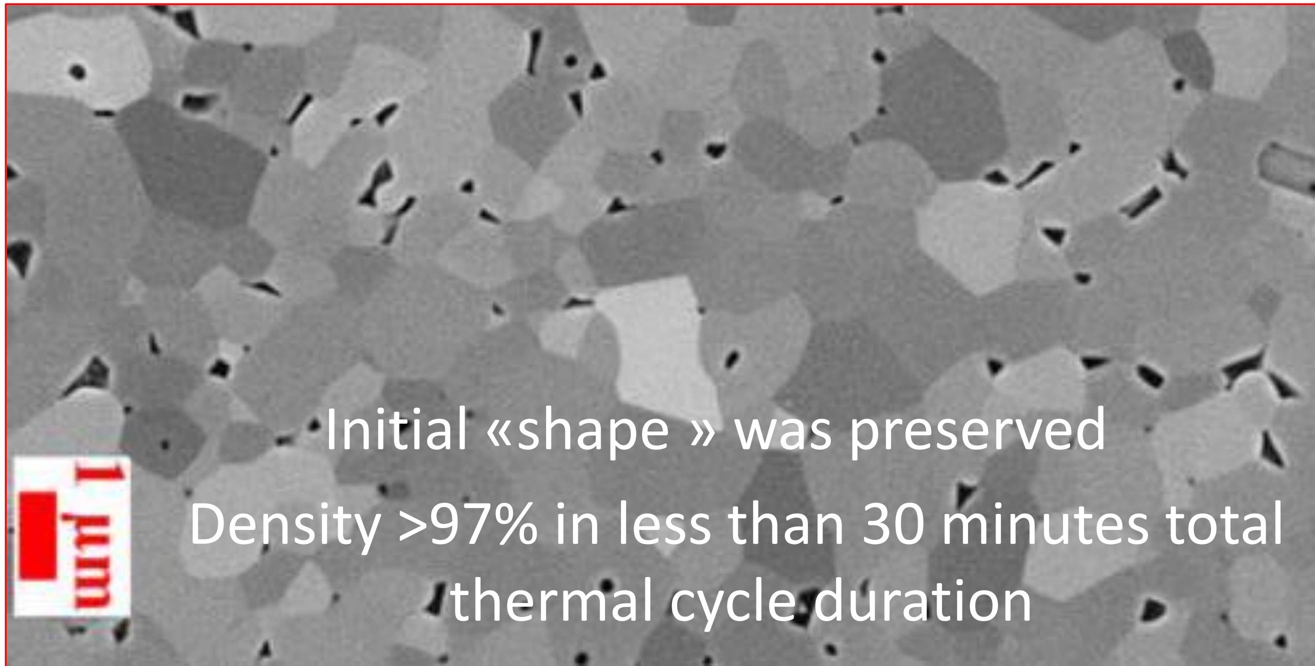
>1 cm thickness transparent spinel ceramics made by MW sintering followed by HIP

Microwave Sintering of 3D printed alumina complexe shape @ 915 MHz (MW assisted with susceptors)





Green alumina part made by INSA LYON



Initial «shape » was preserved

Density >97% in less than 30 minutes total thermal cycle duration

SUMMARY-CONCLUDING REMARKS

Basic aspects of the microwave processing of materials were presented showing different Applications:

- The fast synthesis of magnesium silicide compounds (with nano-sized grains)
- The synthesis of oxides (SSR)
- The sublimation and recrystallisation of ZnO to get flower-like ZnO nano-structure.
- Microwave is also widely spread in thin films technology and liquid phase processing

(Hydrothermal)

Microwave processes are fast …

« Conventional thermal heated runner »

« Microwaved runner »

Thermal cycle control works well (magnetron) but it requires a relatively complicated instrumentation.

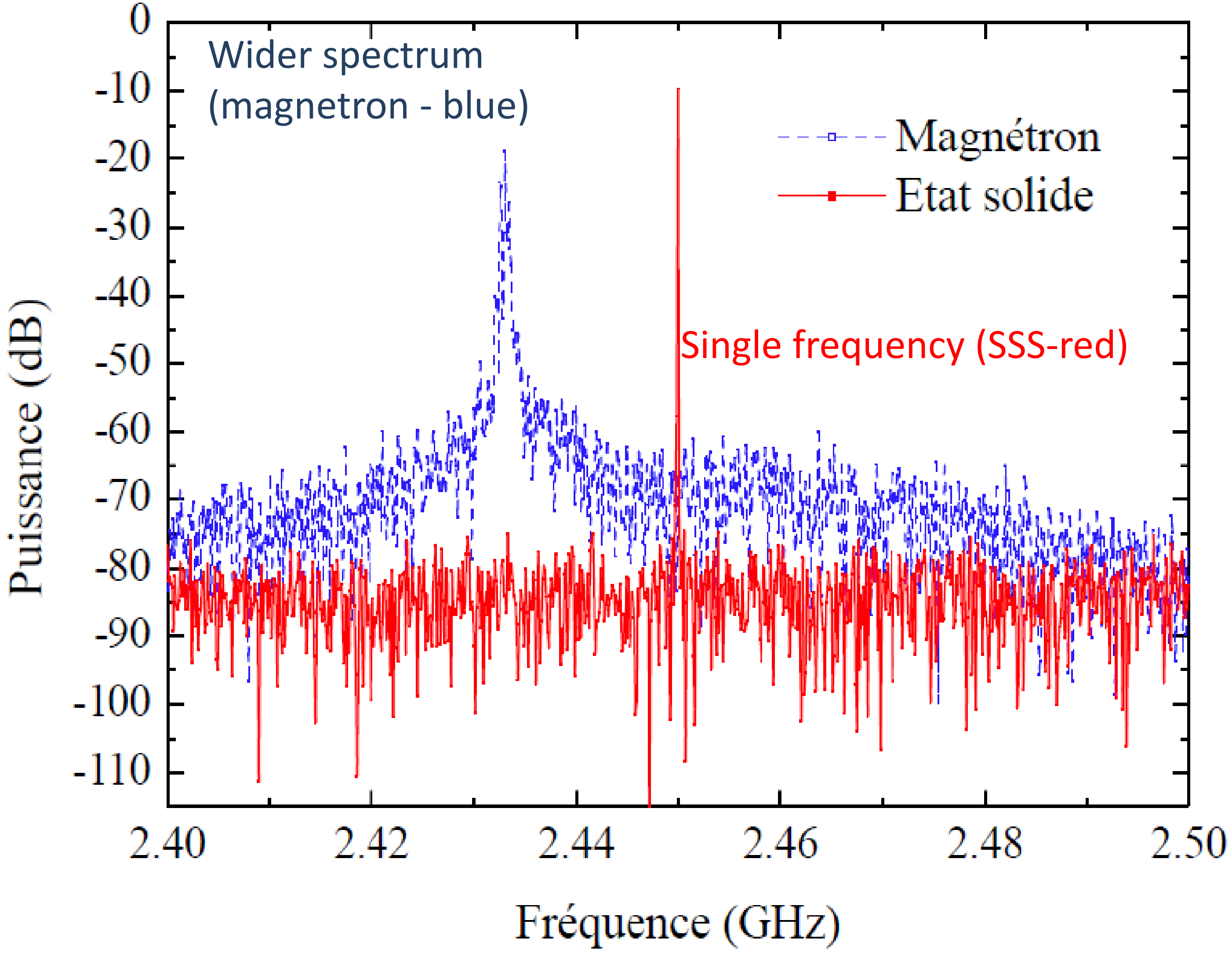
Things are about to change with the new generation of microwave generator…

Magnetron

Magnétron 2.45 GHz, 1.2 kW
(4.5 kV, 0.4 A, η = 60%)

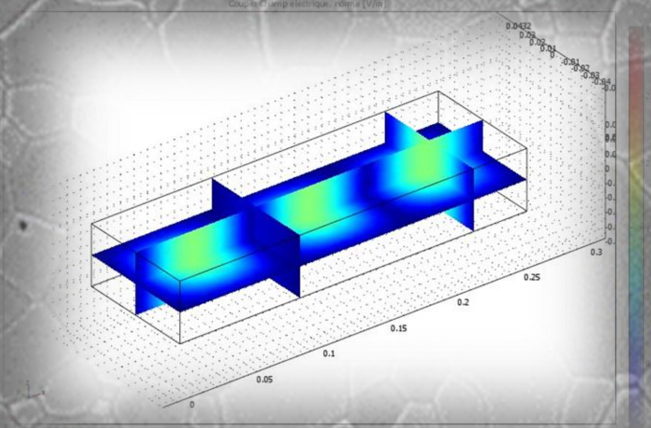
VS

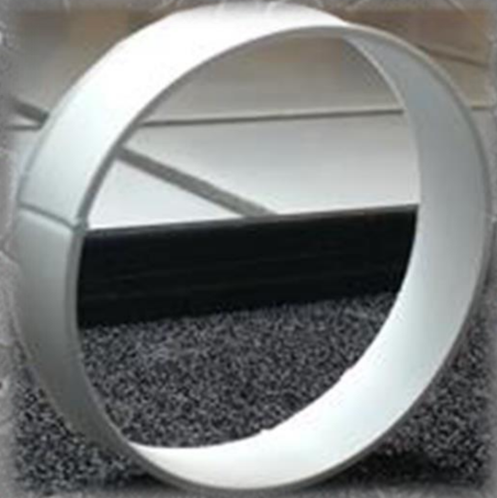
Solid-State RF Amplifier

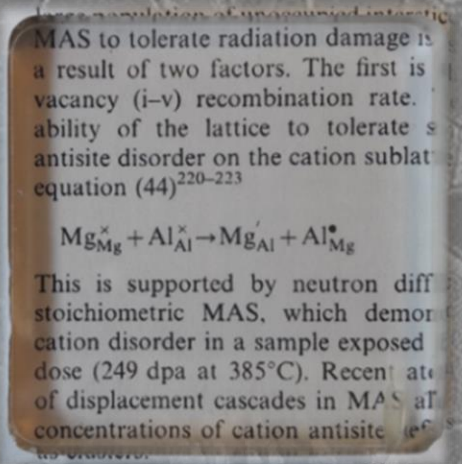


Vaccum tube operating à high voltage (>
High power available
Reliable and it has a long lifespan
Frequency not stable (not adjustable)
Wide Spectrum (vs power)

UNIVERSITÉ
CAEN
NORMANDIE

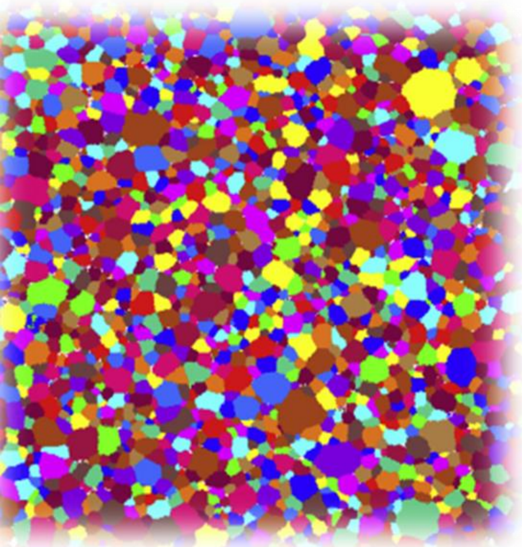




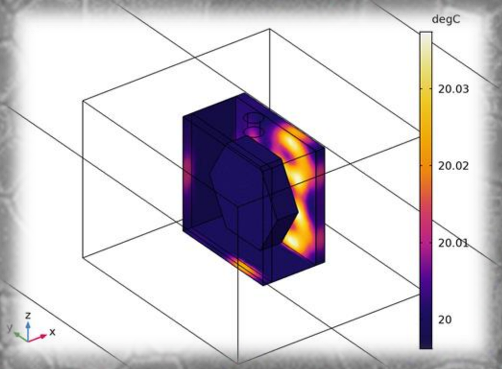

MAS to tolerate radiation damage is a result of two factors. The first is vacancy (i–v) recombination rate. ability of the lattice to tolerate s antisite disorder on the cation sublat equation (44)[220–223]

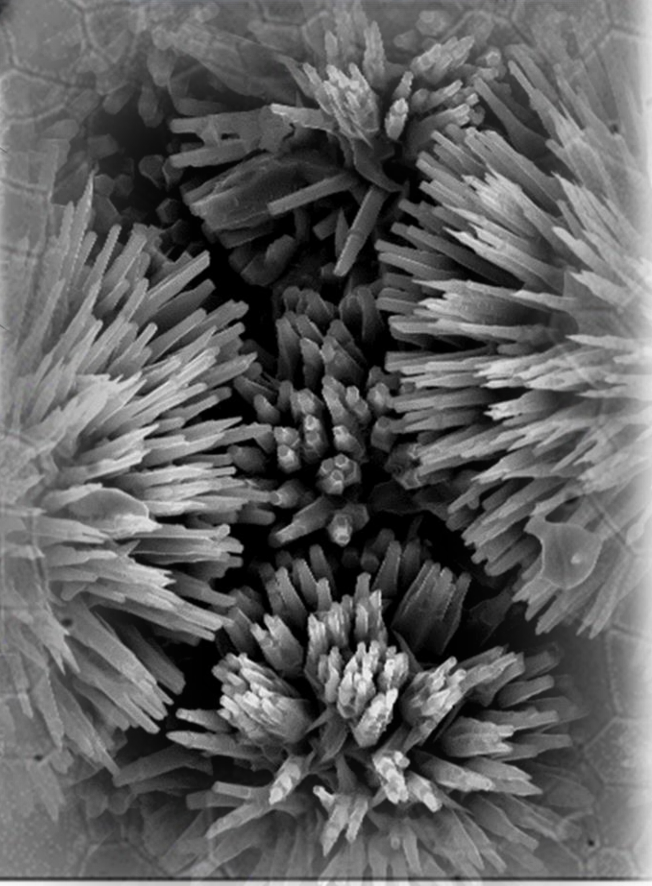
$$Mg_{Mg}^{\times} + Al_{Al}^{\times} \rightarrow Mg_{Al}^{\prime} + Al_{Mg}^{\bullet}$$

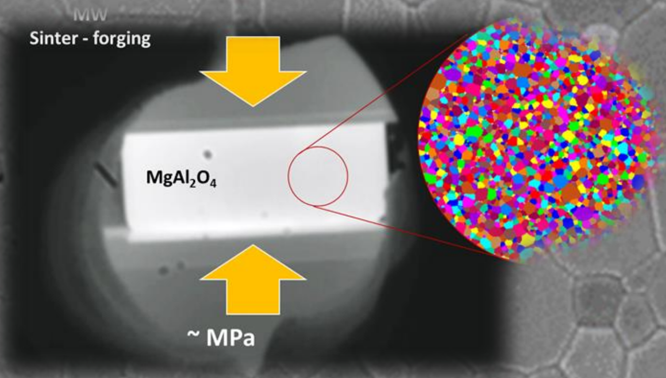
This is supported by neutron diff stoichiometric MAS, which demon cation disorder in a sample exposed dose (249 dpa at 385°C). Recent at of displacement cascades in MAS al concentrations of cation antisite



Thank you for your attention !







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Jonathan Kenny (B_4C-SPS)
Guillaume Riquet (Synthèse $CaCu_3Ti_4O_{12}$)
Gabriel Kerbart (M-0 sous charge)
Nicolas Renaut (projet Andra)
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