

## NEW TECHNIQUES DEDICATED TO THE CHARACTERIZATION OF INNOVATIVE FUELS

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#### Introduction

- Irradiation performances of HTR fuels impose requirements on HTR coating particles :
  - Low standard deviation of kernel diameter and sphericity
  - Close control of coating thickness
- FEM calculations require the exact knowledge of properties of each specific coating layer :



- Thermal property
- Mechanical property



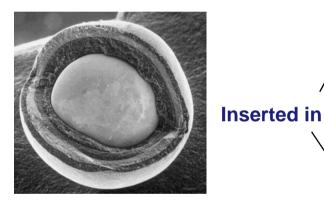
We are implementing new characterization techniques to fulfill these requirements.



#### HTR fuel description



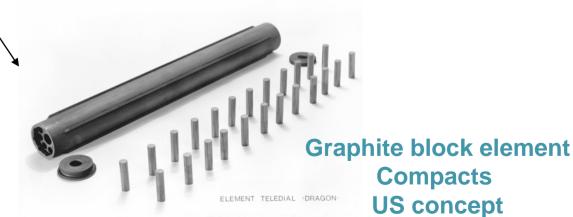
based on spherical coated particles inserted in graphite blocks element



**Particle fuels** 



Graphite block element
Pebbles
German concept

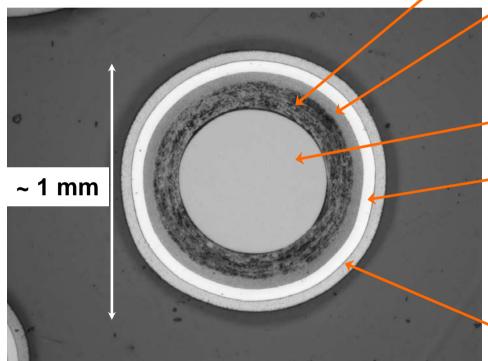




### HTR fuel particle description



Each layer in the TRISO particle design plays a role in fuel performance and fission product (FP) retention.



Metallographic section of a TRISO particle

Buffer ~ 95 µm

Provides a void volume for gaseous FP and accommodates kernel swelling

Dense Inner PyC ~ 40 µm
Reduces tensile stress on SiC
and acts as diffusion barrier to metallic FP

 $-UO_2$  kernel  $\sim 500 \mu m$ 

-SiC ∼ 35 µm

Ensures leak tightness to metallic FP during normal and accidental situations ⇔ cladding in PWR fuels

Dense Outer PyC ~ 40 µm Reduces tensile stress on SiC as IPyC and provides bonding surface for matrix material

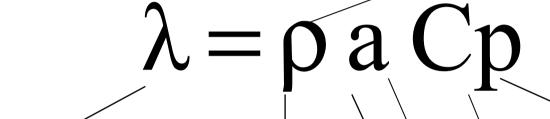
#### Thermal characterization

**Principle** 

 To predict the temperature of the fuels, thermal conductivity has to be determined:

It's deduced from this equation :

sink-float method



[Wm<sup>-1</sup>K<sup>-1</sup>]

• With:

- ρ

: Density

– a

: Thermal diffusivity

 $[kgm^{-3}]$   $[m^2s^{-1}]$ 

Ср

: Heat capacity

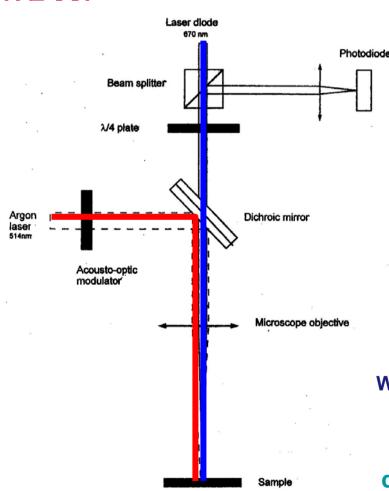
microcalorimetry

Thermo reflectance microscopy

 $[Jkg^{-1}K^{-1}]$ 



## The method consists in photothermal effect detection which allows a no-contact thermal diffusivity measurement



This technique is based on the measurement of the temperature increase induced by the absorption of an intensity modulated laser beam (pump beam)

The temperature increase is determined at the sample surface with the help of a secondary continuous laser beam (probe beam),

Argon laser and Laser diode are focused through optical microscope to perform very local measurements.

We use this technique to measure each layer's thermal diffusivity on polished particles

This technique allows to extract thermal diffusivity with a precision often better than 5 %

Diffusivity (mm<sup>2</sup>.s<sup>-1</sup>)

Conductivity  $(W.m^{-1}K^{-1})$ 

5.8

 $7.4 < \lambda < 8.2$ 

Preliminary results on dense pyrolytic carbon layers at room temperature: Simulant HTR particles Literature data Inner dense PyC Outer dense PyC  $Cp\left(J.Kg^{-1}K^{-1}\right)$ 710 710 Density (g.cm<sup>-3</sup>) →  $1.8 < \rho < 2$  $1.8 < \rho < 2$ **Specification** 99Porosity (%)

• In accordance with literature data (strong dispersion because of various structures of PyC)

13

 $16.6 < \lambda < 18.5$ 

- Difference is observed between I-PyC and O-PyC, whereas both PyC are apparently processed in the same conditions
- May be correlated with the fact that I-PyC is annealed between 1500-1600 °C during SiC deposition Process

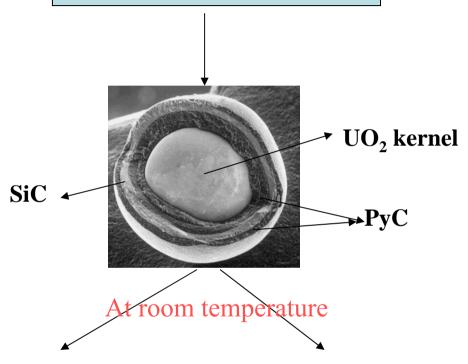
Measurement



- These first experimental values need to be confirmed by more statistical measurements
- Determination of the thermal conductivity of the buffer layer
- SiC layer thermal characterization
- In-temperature tests up to 1500 °C







**Acoustic microscopy** 

Nano indentation

UO<sub>2</sub> and SiC

PyC



 Ultrasonic measurements provide information on sound velocities and attenuation coefficients. In an isotropic material,
 volumic acoustic modes exist :

Longitudinal velocity (V<sub>L</sub>)



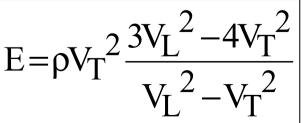
Rayleigh wave velocity (V<sub>R</sub>)

Calculation of the  $transverse(V_T)$ 

To perform both velocities measurements, the use of a special device is necessary



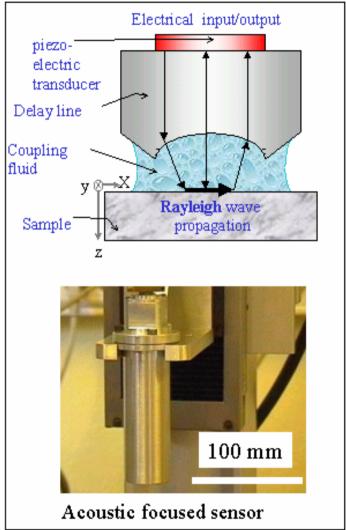
Elastic moduli calculation E, G, K, v...



$$v = 2\left(\frac{Vt}{VI}\right)^2 - 1/2\left(\left(\frac{Vt}{VI}\right)^2 - 1\right)$$

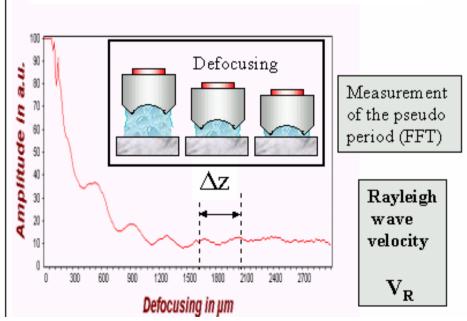






The sensor focuses the ultrasonic wave on the sample

#### Principle of the measurement

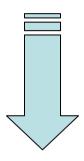






On  $UO_2$  and SiC we have shown that E could be calculated with only  $V_R$ 

$$E = 3\rho V_R^2$$



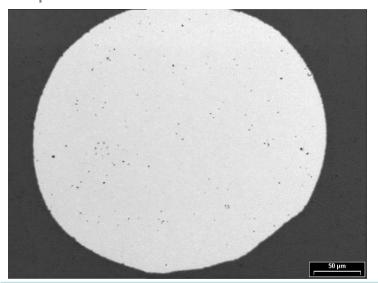
### This technique is used to:

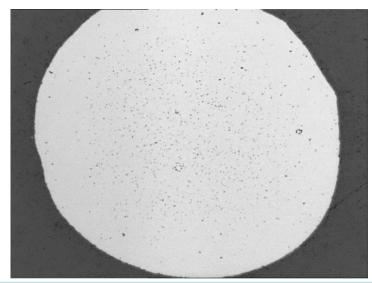
- Local measurements
  - High accuracy
- Fast (a few seconds)

For  $UO_2$ :  $p = [1 - (V_R/2593)]/0.91$ 



 UO<sub>2</sub> Kernels : ≠ batches produced in CEA with different condition processes





No influence of the fabrication process on the elastic

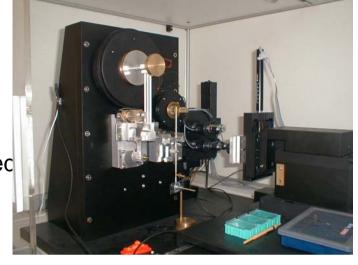
The difference of the amount of porosity is correlated by the metallographic section

Porosity measurements by SEM image progressing are in progress to confirm the amount of porosity deduced from acoustic values



#### Features:

- diamond Berkovich indenter
- indentation rate: 2,5 mN.s<sup>-1</sup>
- Load max: 200 mN
- 10 indentations in each layer
- Measurement on equatorial polished section



	250 -	
Load (mN)	200 -	
	150 -	O-PyC <b>←</b>
	130	I-PyC
	100 -	
	50 -	
	0 -	
	(	500 1000 1500 2000 2500
		Indentation depth (nm)

_	IPyC	ОРуС
E(GPa)	18	29

Difference may be correlated with the annealing of the IPyC during the SiC deposition process

Load-displacement curves generated using the nanoindenter

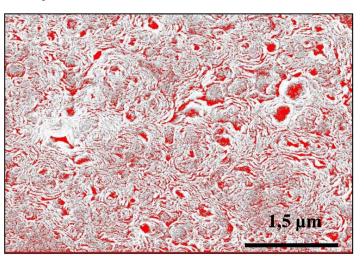


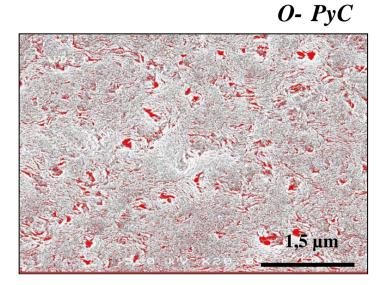


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 Assumption confirmed by SEM micrographies on I-PyC and O-PyC

AREVA I- PyC





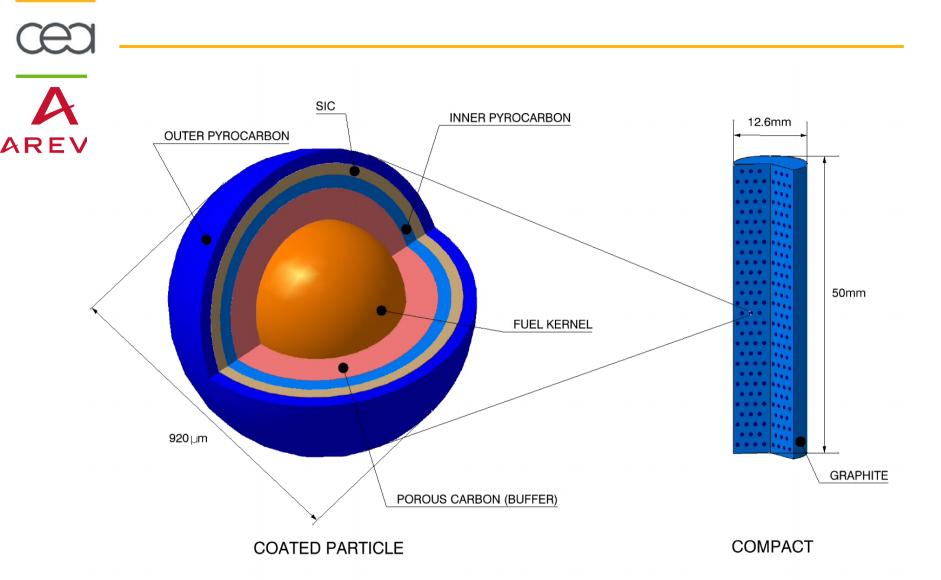
- These preliminary results show the importance of layer particle's characterization
- For the comprehension, correlation between the structure and the properties has to be done



### Summary and outlook

- Thermal characterization
  - The first results we have obtained allow us to demonstrate the feasibility of the thermo reflectance microscope to characterize each layer of the particle
  - Characterization will be continued
  - Thermal conductivity of each layers will be evaluated this year until 1500 °C
- Mechanical characterization
  - Two different devices are used to determine elastic properties of UO<sub>2</sub> kernel and dense layers of particle at room temperature
  - Other techniques will have to be defined to obtain in-temperature elastic properties
  - Fracture stress will have to be also determined, probably thanks to internal pressure tests on hemispherical sample obtained directly from particles
- In each case, the correlation between properties measurement and structure of material will have to be done





Thank you for you attention