ActiLab Closing Meeting

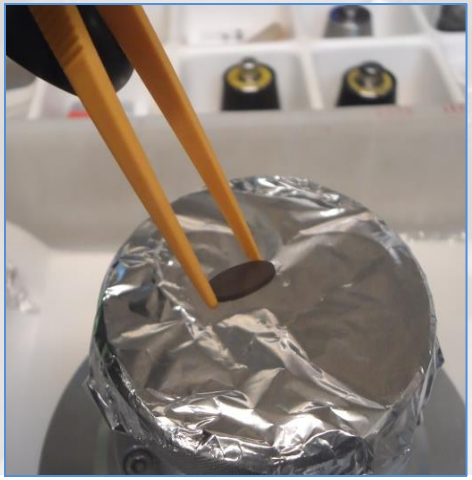
ActiLab Activities at INFN

Stefano Corradetti
and Alberto Andrighetto
SPES Target Group

Outline

- UC_x synthesis (Task #1): 2013 activity

- UC_x thermal characterization (Task #2): 2014 activity




UC_x synthesis

New equipment (purchased in Jan ‘13)

- Reduce and homogenize the grain size of oxide and carbon source ⟶ selection and control of the precursors (as defined in ActiLab SC May 2012, strategy for UC_X production at INFN)

↓

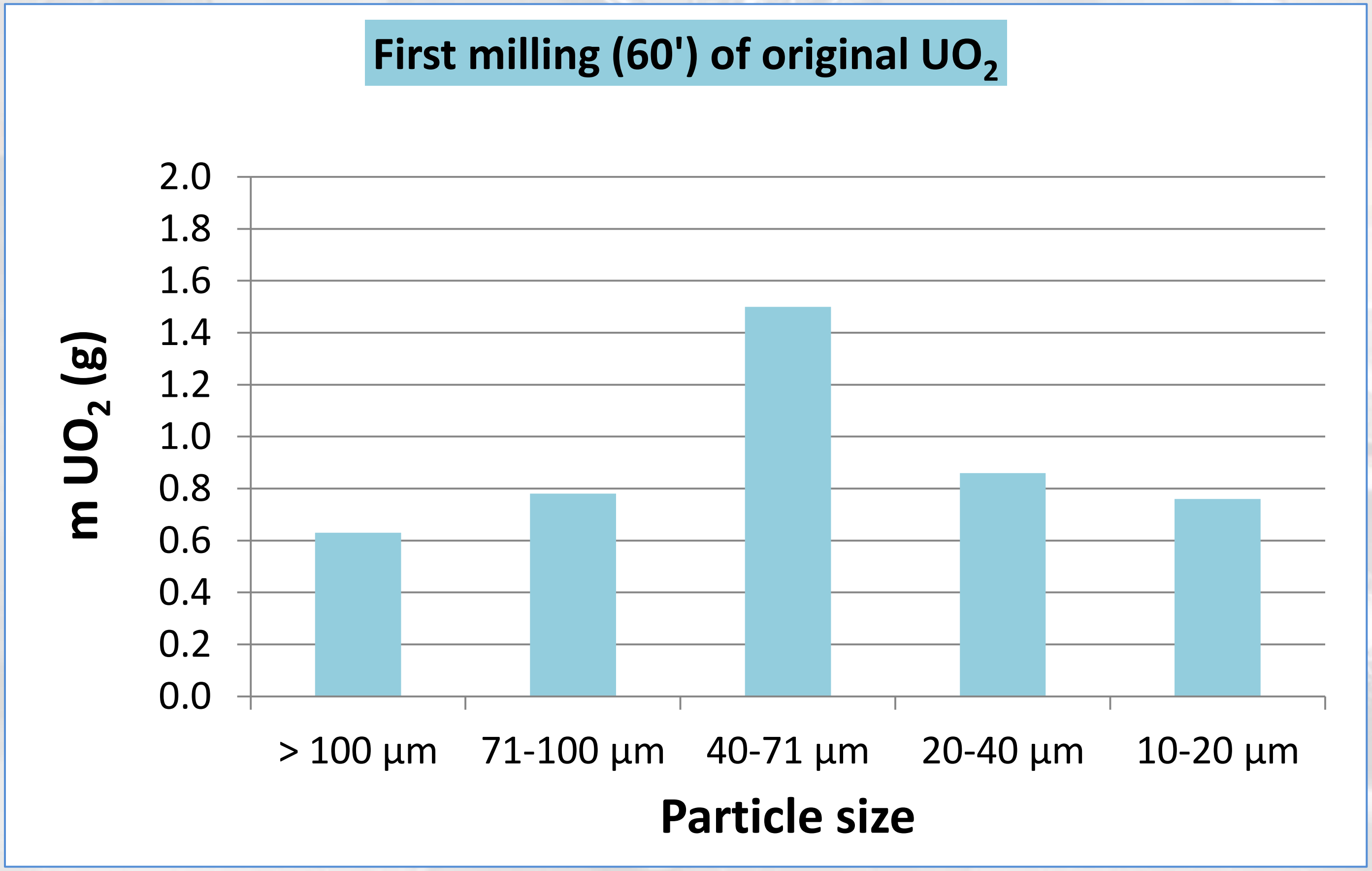
- Vibratory micromill and sieves purchased at UNIPD lab (grain size down to few μm) to treat small quantities of precursors

UC_x synthesis

Milling and sieving → UO_2 particle size control

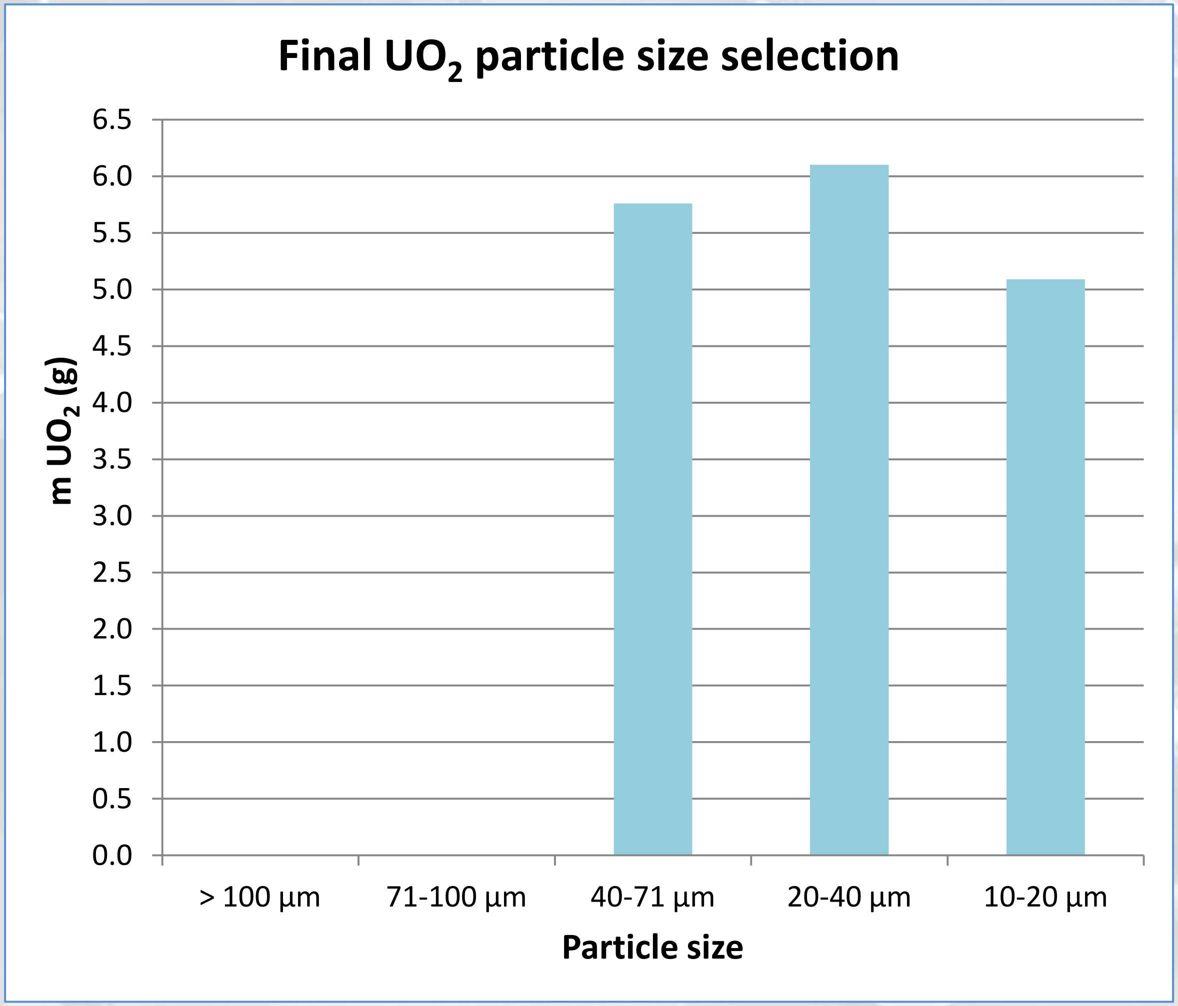
'Original' UO_2 batch: ~ 20 g

First milling → ~ 5 g of UO_2



Subsequent millings to produce enough UO_2 for 10 UC_x pellets

Final particle size population



- Sieves below 10 µm mesh also available (Nylon mesh) and tested
- Small amount of powder ground to < 10 µm size in reasonable time
- < 20 µm ("10-20 µm") was selected as a precursor size to produce 10 pellets, sent to IPNO for off-line characterization and irradiation tests

UC_x synthesis

Pellets production → 10 UC_x discs

"Green" UO_2 + C pellets

% wt. UO_2	% wt. C	% wt. binder	Weight (g)	Diameter (mm)	Thickness (mm)	Density (g/cm³)
77.60	20.30	2.10	0.57	13.01	0.95	4.50

Thermal treatment:

- 2°C/min up to 1250°C (binder decomposition occurs)
- 8 hours at 1250 °C, then 2 °C/min up to 1350 °C (carburization)
- 8 hours at 1350 °C, then 1.5 °C/min up to 1730°C (carburization)
- 6 hours at 1730 °C

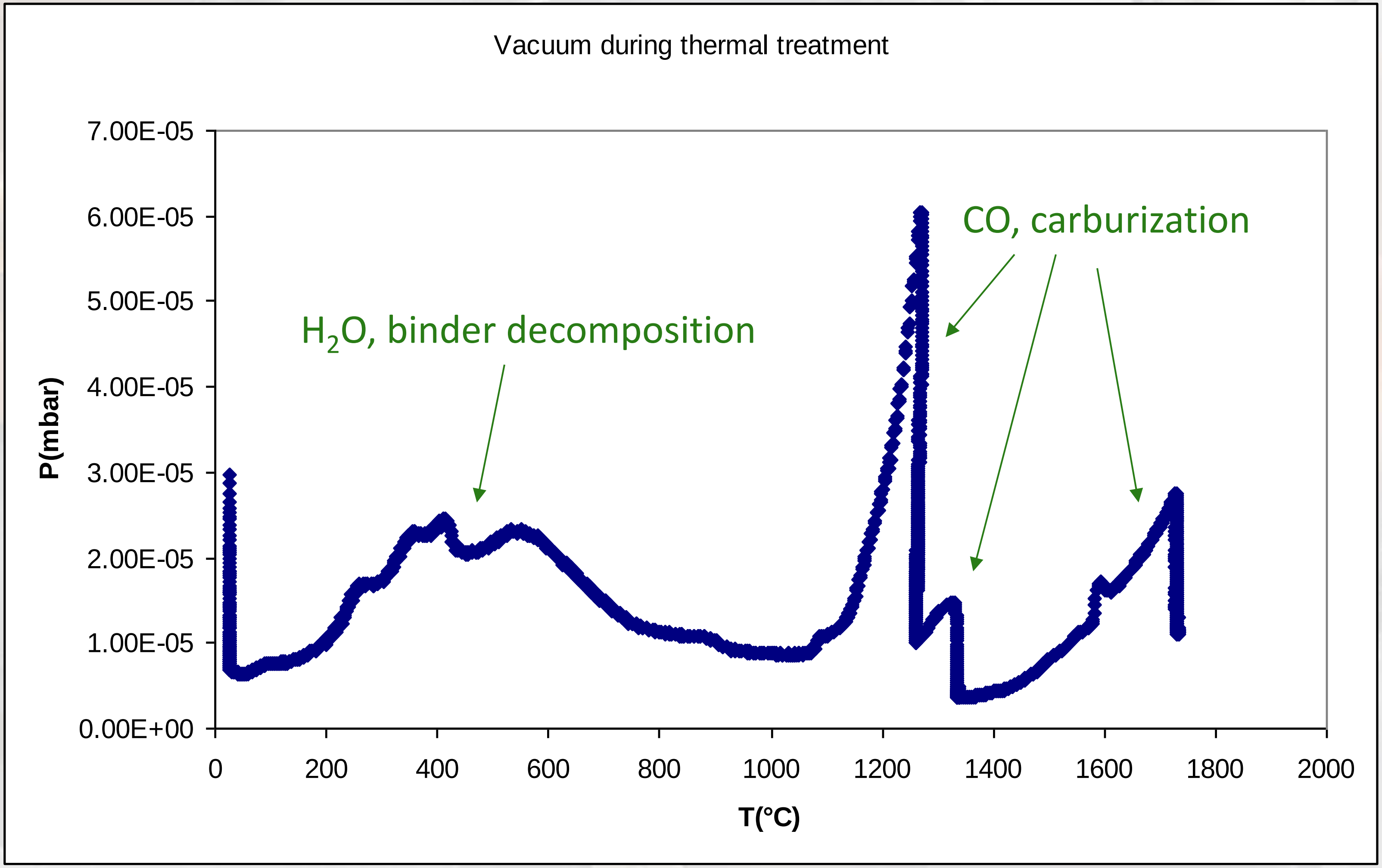
UC_2 + 2C pellets after carburization ($UO_2+6C \rightarrow UC_2+2C+2CO$)

Weight (g)	Diameter (mm)	Thickness (mm)	Density (g/cm³)	Total porosity (%)	Weight loss (%)	Theoretical weight loss (%)
0.47	12.66	0.86	4.33 **Similar to SPES standard**	58.90	17.57	16.90

Expected composition of the samples: UC_2, graphite and a minor amount of UC

UC_x synthesis

Pellets production ⟶ 10 UC_x discs



UC_x synthesis

Pellets production ⟶ 10 UC_x discs

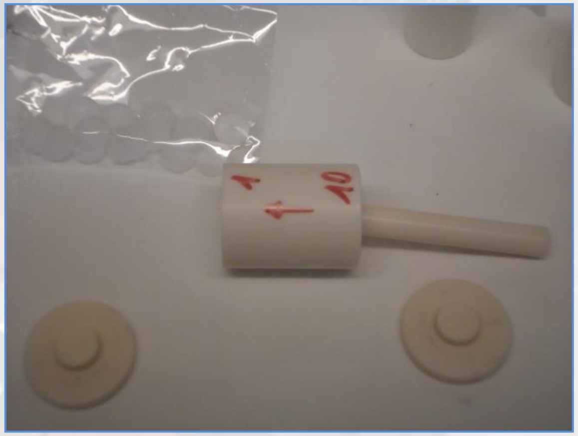
Sample before treatment

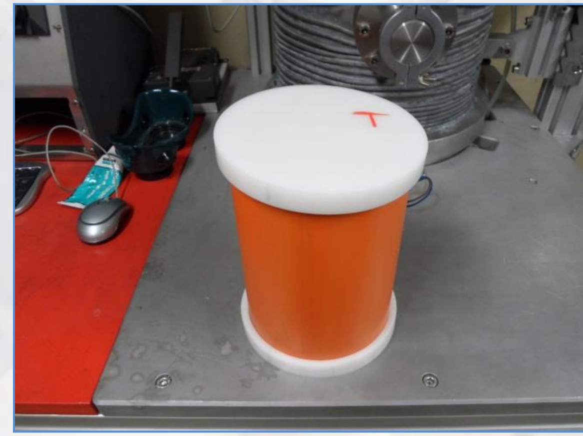
Samples after treatment

Disc	Weight (mg)	ρ (g/cm³)	Thickness (mm)
1	466	4.31	0.86
2	476	4.36	0.87
3	467	4.37	0.86
4	466	4.31	0.86
5	475	4.31	0.87
6	473	4.38	0.86
7	476	4.29	0.88
8	473	4.42	0.85
9	457	4.37	0.83
10	474	4.22	0.89

UC_x synthesis

Shipping of the pellets to IPNO





1) **Authorization from IPNO to send the samples took 3 – 4 months**

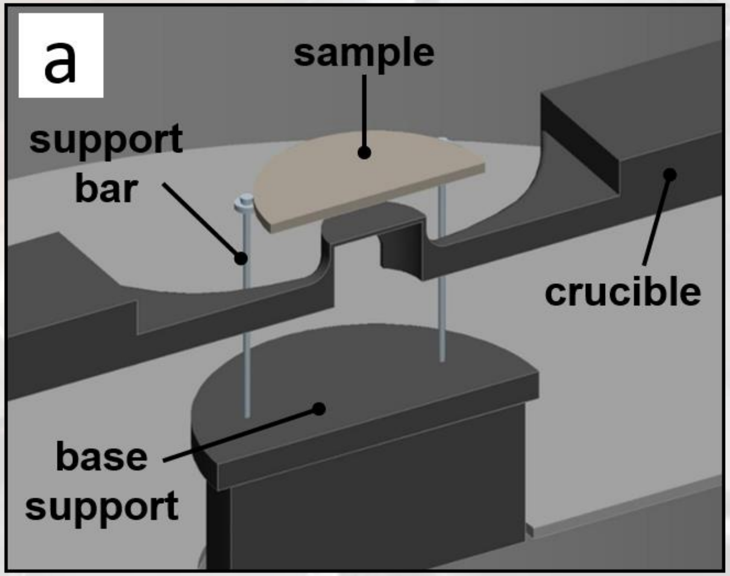
2) **Shipping to Orsay (September 2013) took less than two weeks (Cost: ~ 1k€)**

INFN (UNIPD) →→→→ IPNO OK

UC_x characterization

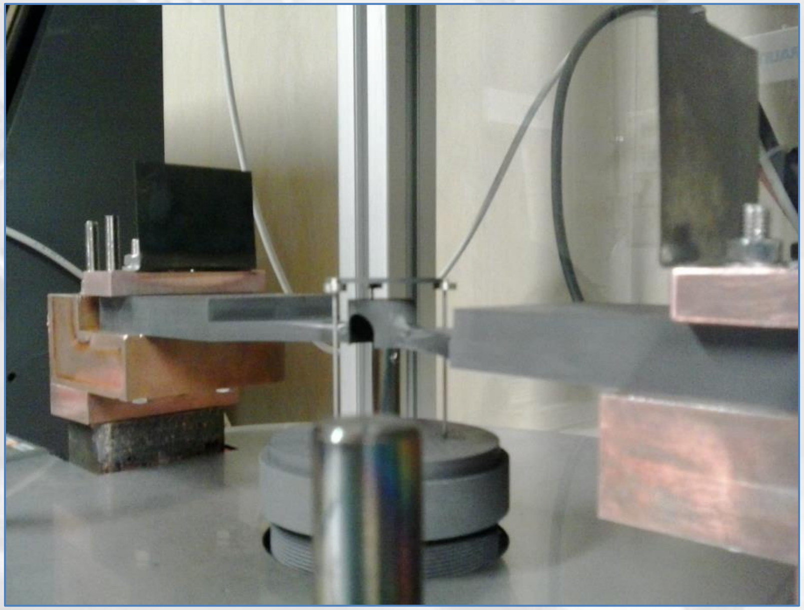
Thermal conductivity

Original setup – INFN/LNL



M. Manzolaro et *al.*, Rev. Sci. Instr. 84 (2013) 054902.

Newly developed setup (Padova)



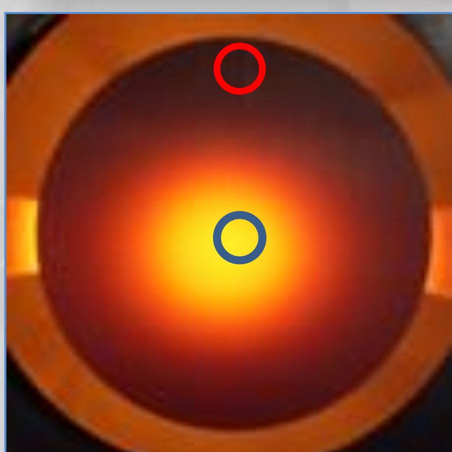
Allowing measurements for samples of smaller sizes (down to ∅ ~30 mm)

<u>Creation of a thermal gradient on the tested disc by means of irradiation from a heated graphite crucible</u>

UC_x characterization

Thermal conductivity

M. Manzolaro, S. Corradetti, A. Andrighetto, L. Ferrari, Rev. Sci. Instrum. 84, 054902 (2013)

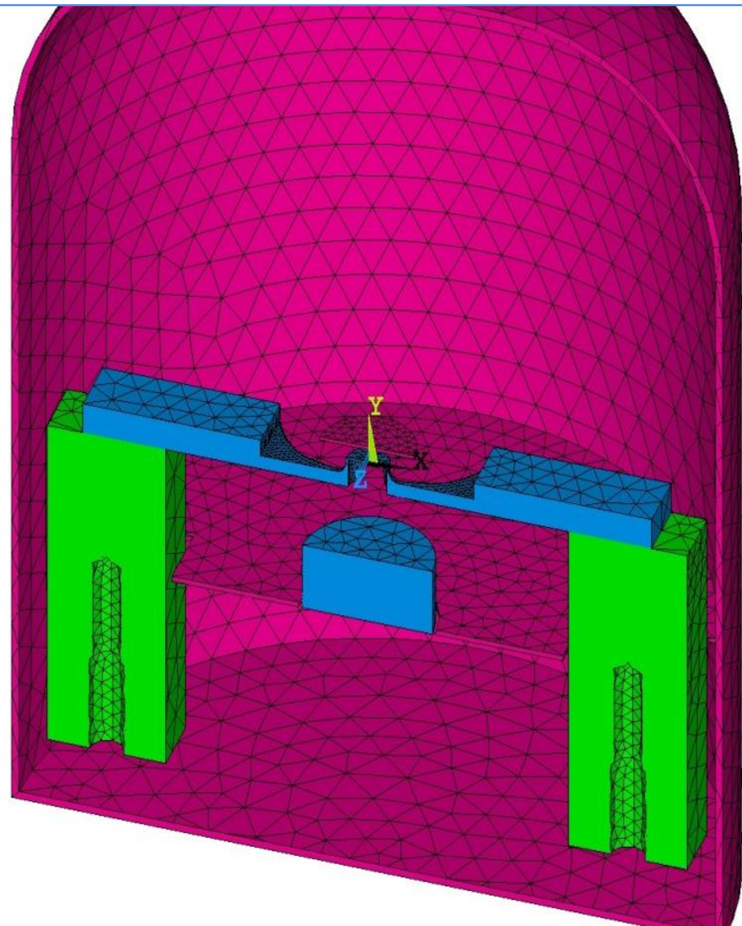


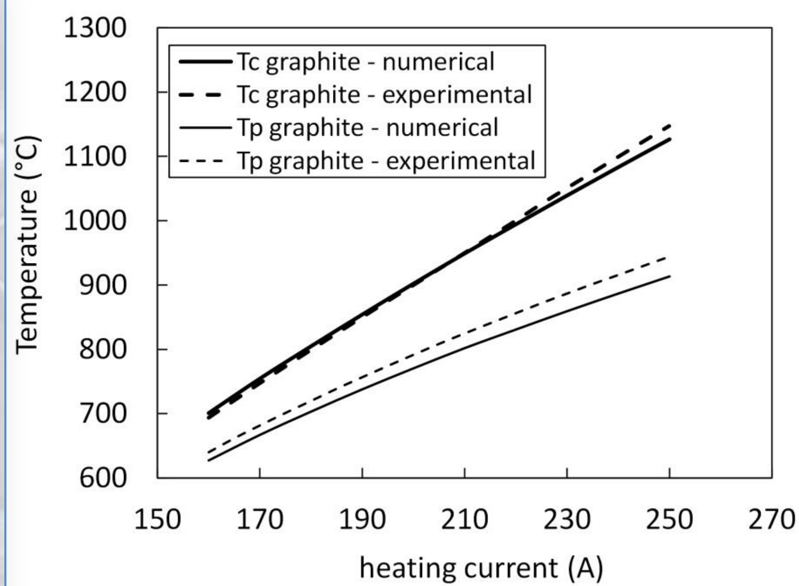
- Creation of thermal gradients in the disc caused by irradiation from the crucible
- A low temperature pyrometer (range 600 ÷ 1400 °C) is used to monitor temperature in the disc **center** and **periphery** (repeated cycles of heating)
- Inverse analysis → k (T) (tipically in 600 ÷ 1200 °C range)

UC_x characterization

Thermal conductivity

FEM model of the chamber – crucible – disc system





- ΔT between center and peryphery: comparison between numerical and experimental
- k (T) is obtained minimizing the differences between the ΔTs
- k (T) = C_0+C_1*T (average T)

UC_x characterization

Thermal conductivity

ϕ 30 mm samples produced

Treatment →

1) UO_2 + C + binder, UO_2 < 20 μm → same material sent to IPNO (only this was tested)

2) UO_2 + C + binder, 20 μm < UO_2 < 40 μm

3) UO_2 + C + binder, 40 μm < UO_2 < 71 μm

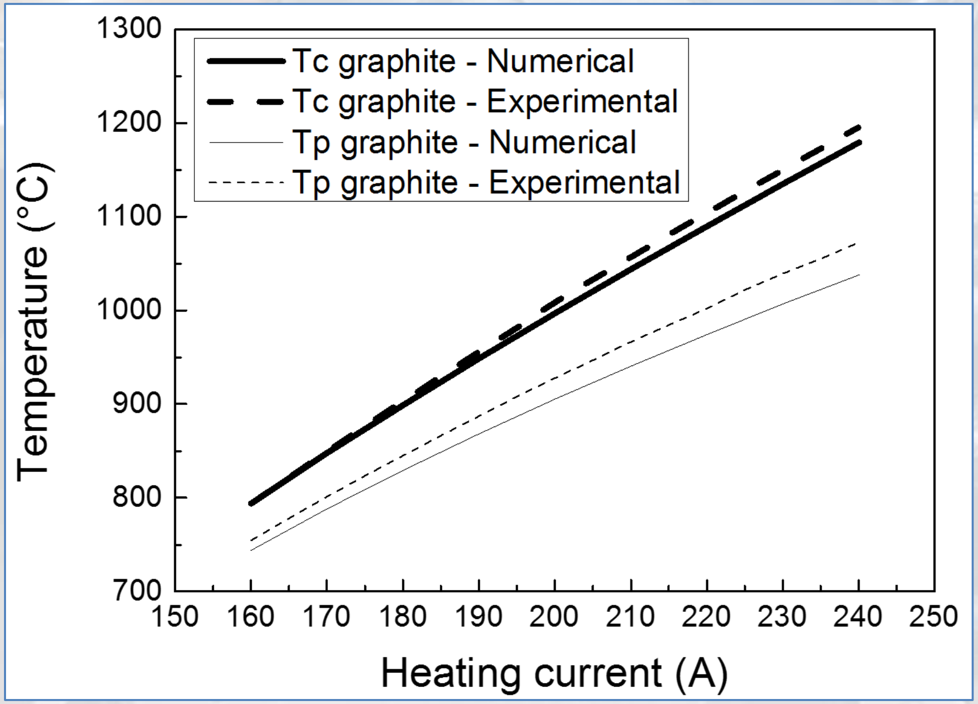
Mat.	Weight (g)	ρ (g/cm^3)	Thickness (mm)
1	3.51	3.87	1.38
2	3.57	3.85	1.40
3	3.58	3.48	1.54

UC_x characterization

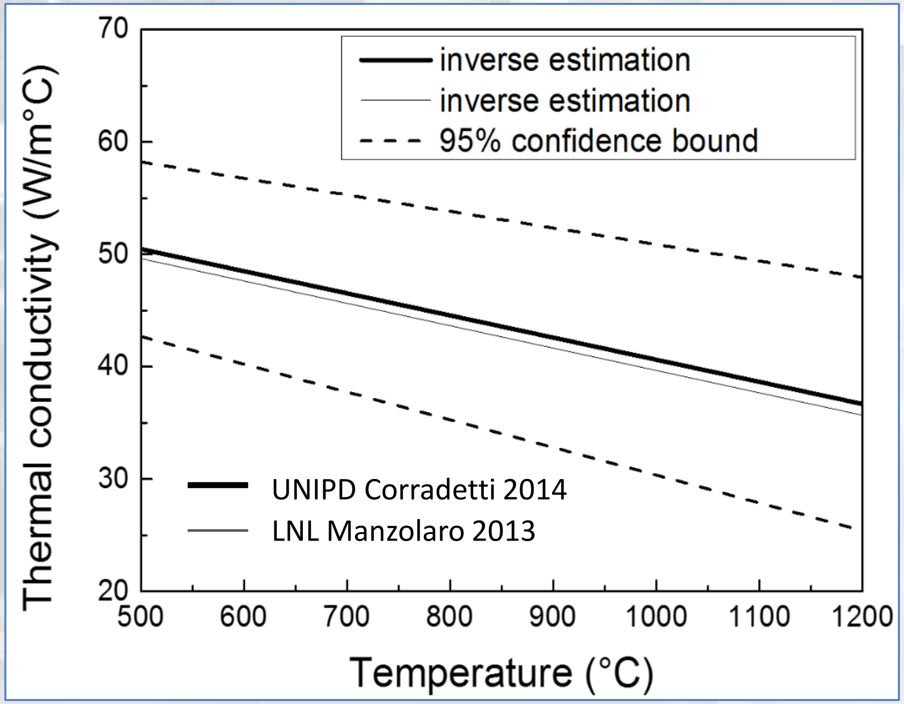
Thermal conductivity

Calibration of the system with a graphite disc

Temperature [°C] measurements vs. numerical data



Inverse analysis gives Thermal conductivity [W/m°C]



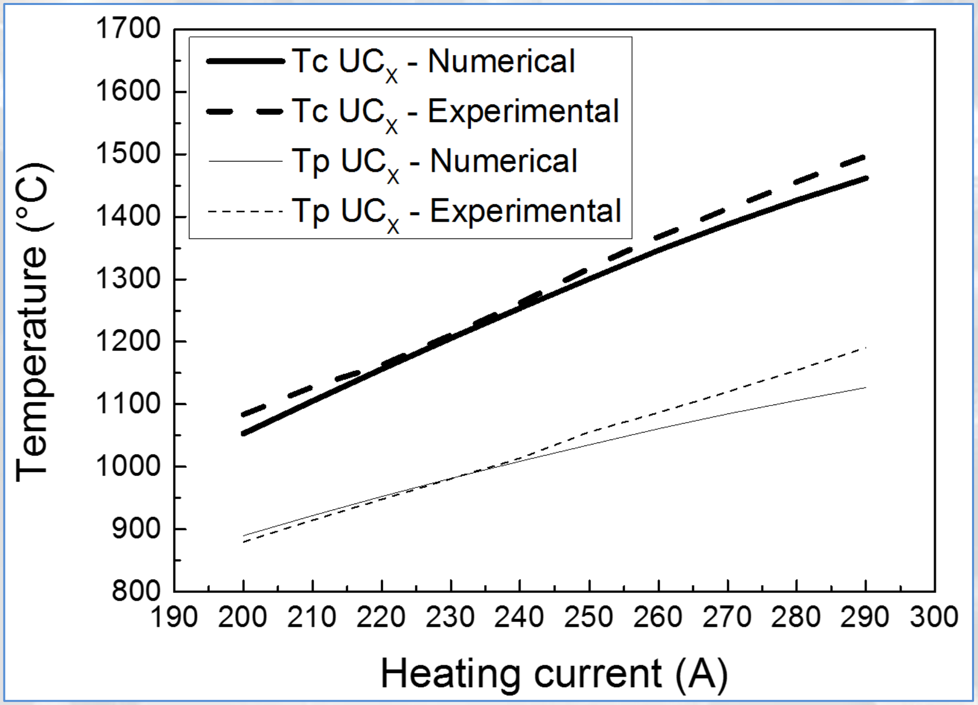
UC_x characterization

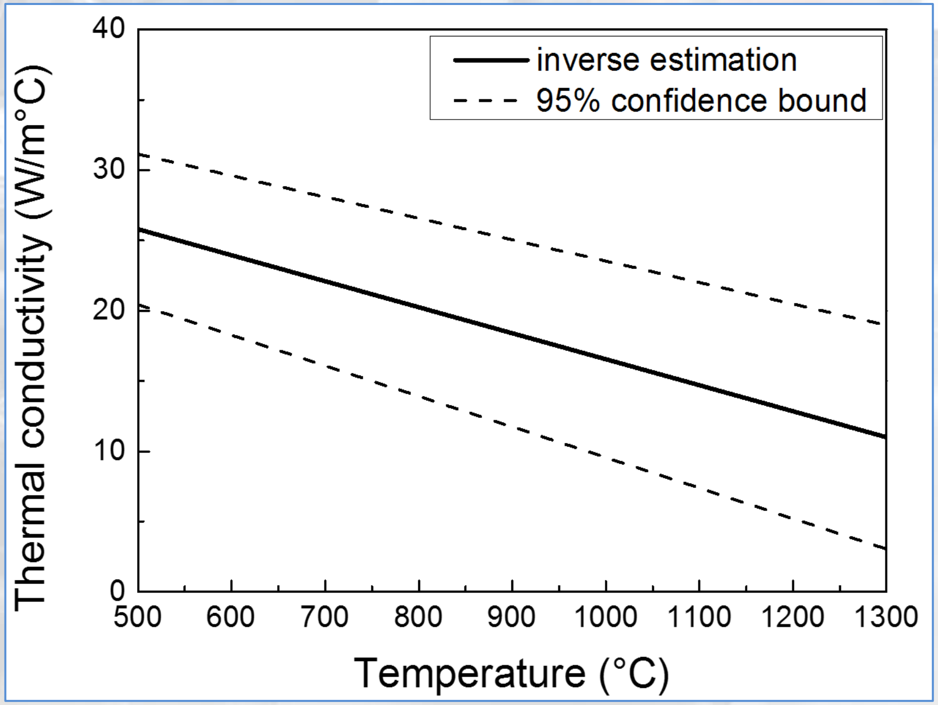
Thermal conductivity

Measurements on UC_x discs

Temperature [°C] measurements vs. numerical data

Inverse analysis gives Thermal conductivity [W/m°C]

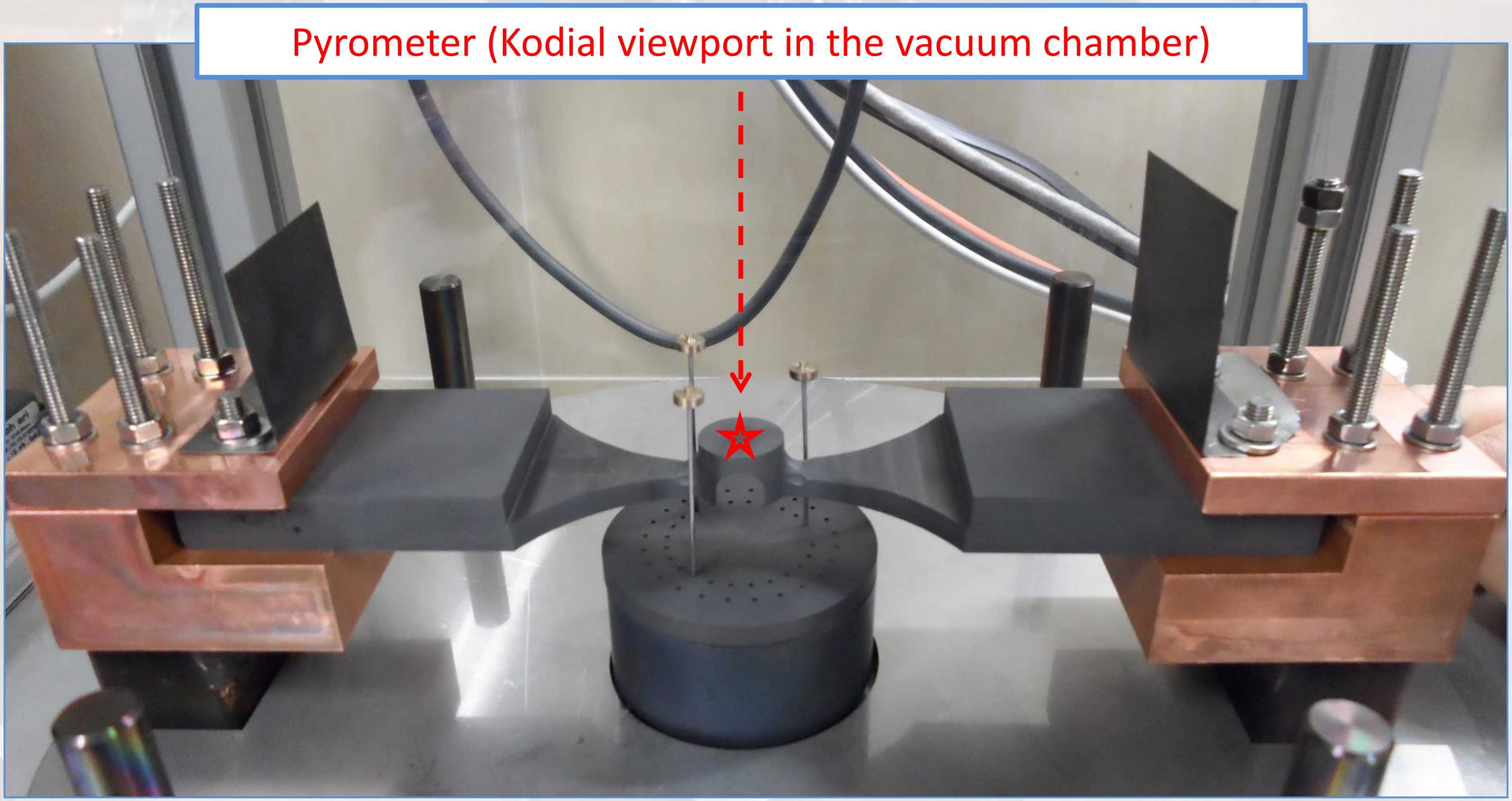




UC_x characterization

Emissivity

Measurement of the thermal emissivity of a sample placed on a heated graphite crucible by means of a dual-frequency pyrometer



UC_x characterization

Emissivity

Pellets shipment from IPNO

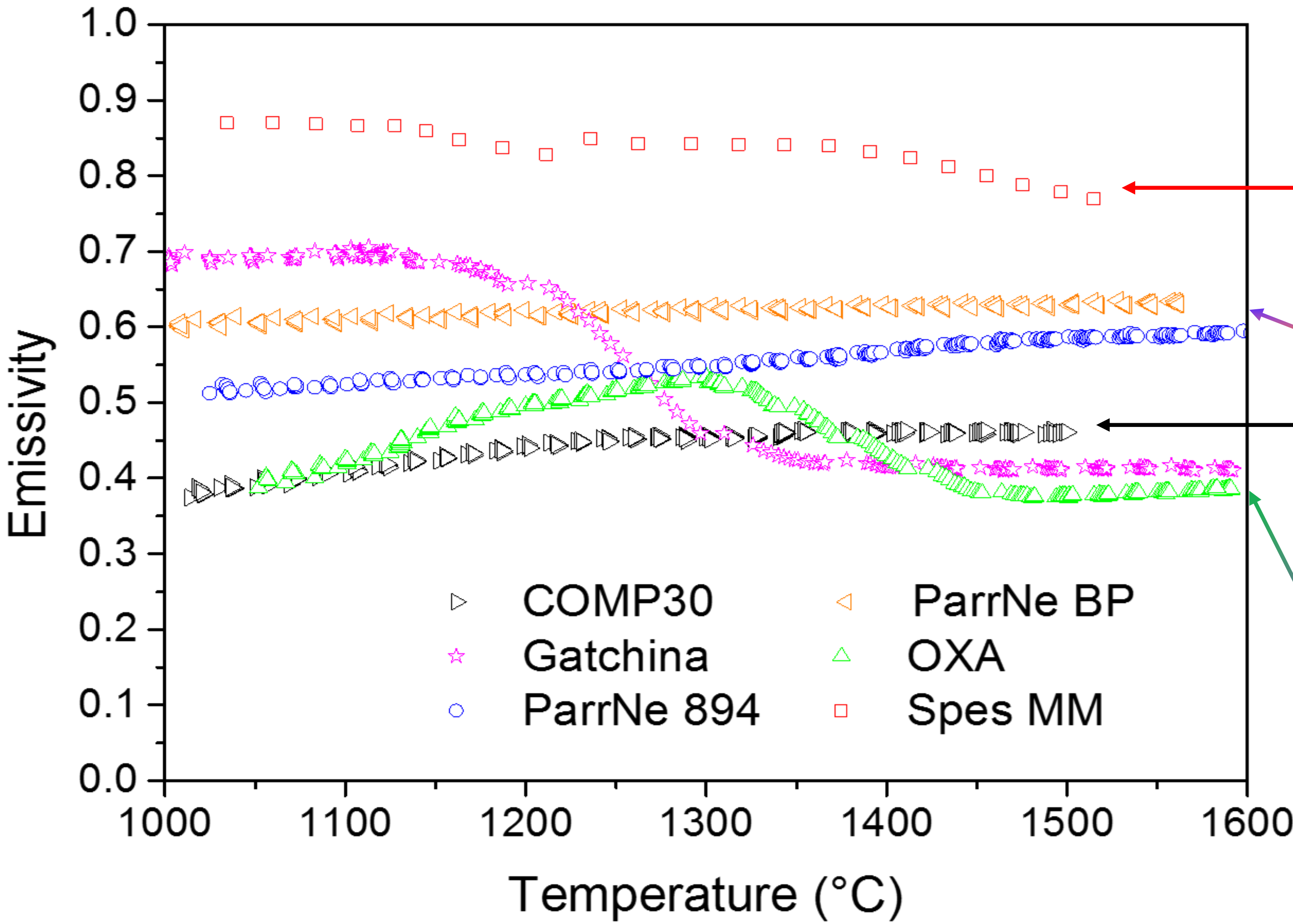
pellet	n°	mass (g)	main phase	mass of U (g)	diameter (mm)	thickness (mm)
GATCHINA	299	1.80	UC	1.71	13.17	1.03
PARRNe894	3	0.82	UC_2	0.74	12.95	1.92
OXA	1 of the 7	0.61	UC	0.70	7.40	1.87
PARRNeBP	7	0.87	UC_2	0.79	12.63	1.53
COMP30	4	0.68	UC_2	0.62	8.28	2.51
TOTAL		**4.78**		**4.57**		

- Selected samples from the batch tested in March 2013 at IPNO (unirradiated)
- Authorization to send requested in summer 2013
- Samples received at UNIPD in July 2014

IPNO →→→→ INFN (UNIPD) **~OK**

UC_x characterization

Emissivity



SPES UC_X (ϕ 30 mm) with <20 μm UO_2

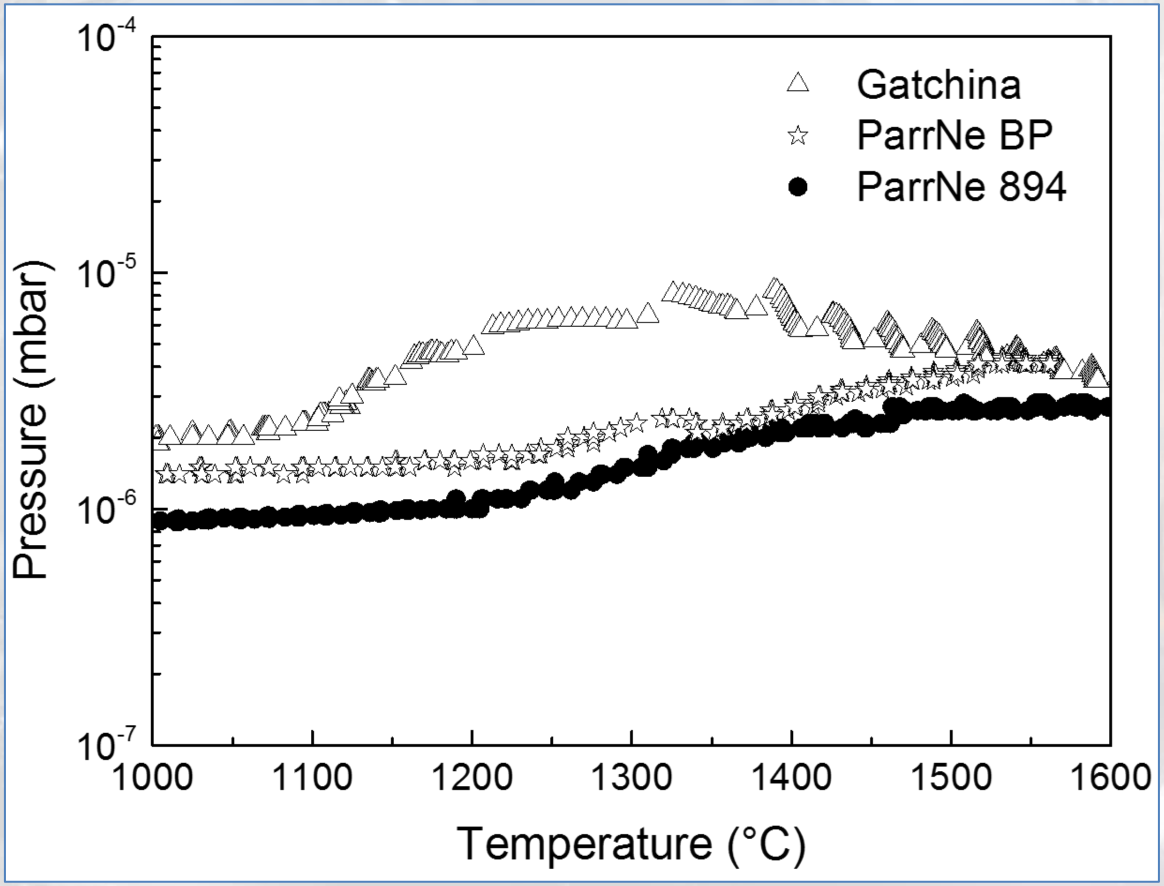
IPNO UC_X by U_3O_8 + C

Designed to obtain UC, but result is UC_2 + minor am. C

HD UC

UC_x characterization

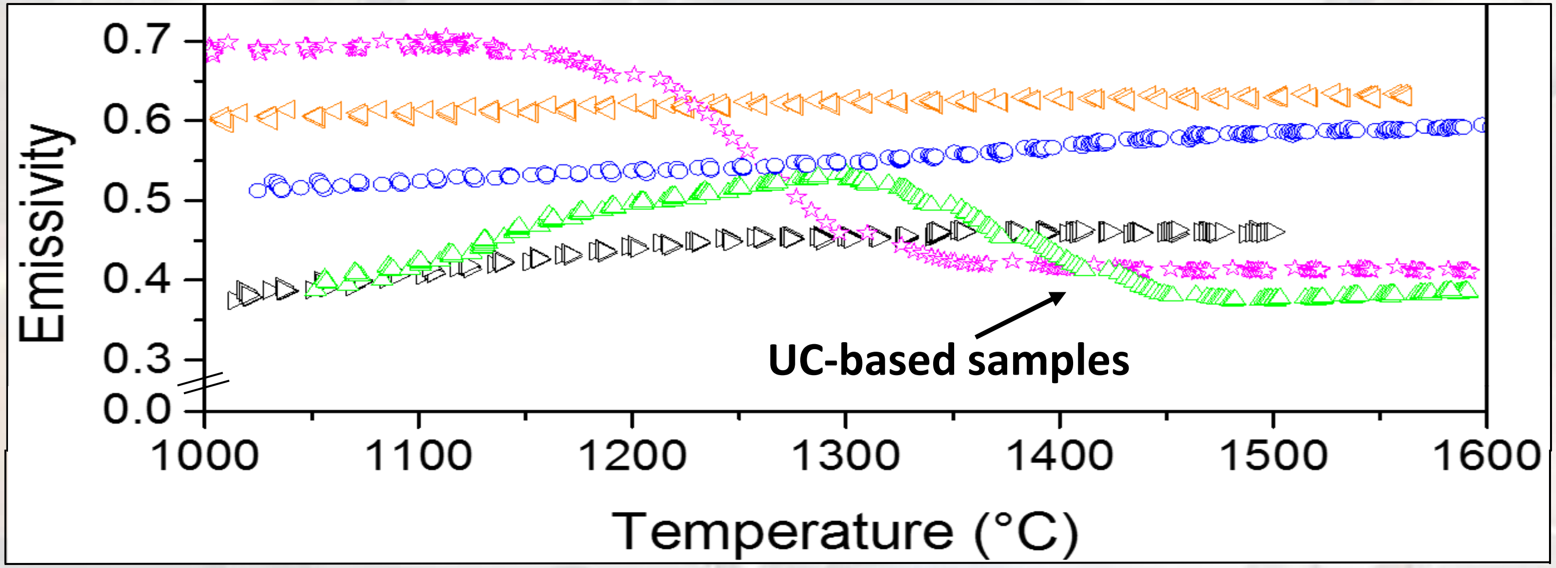
Emissivity

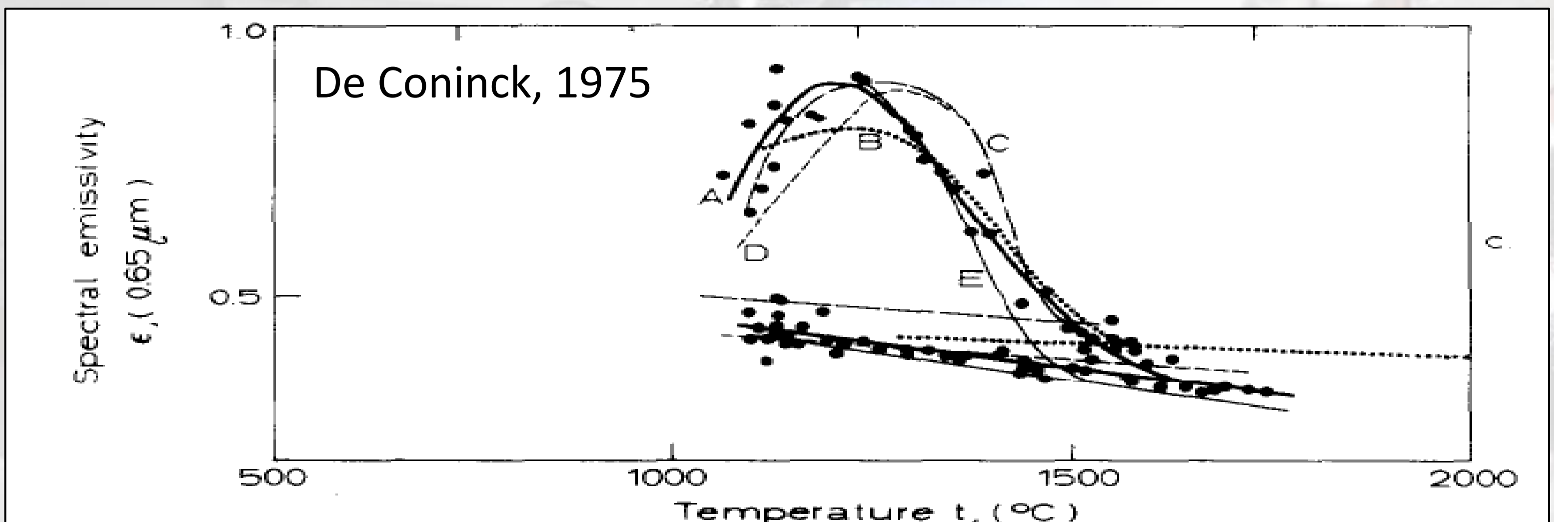


Instability in emissivity can be related to reaction with the graphite crucible (both UC-based samples, Gatchina and OXA, were found sticked on the crucible)?

UC$_x$ characterization

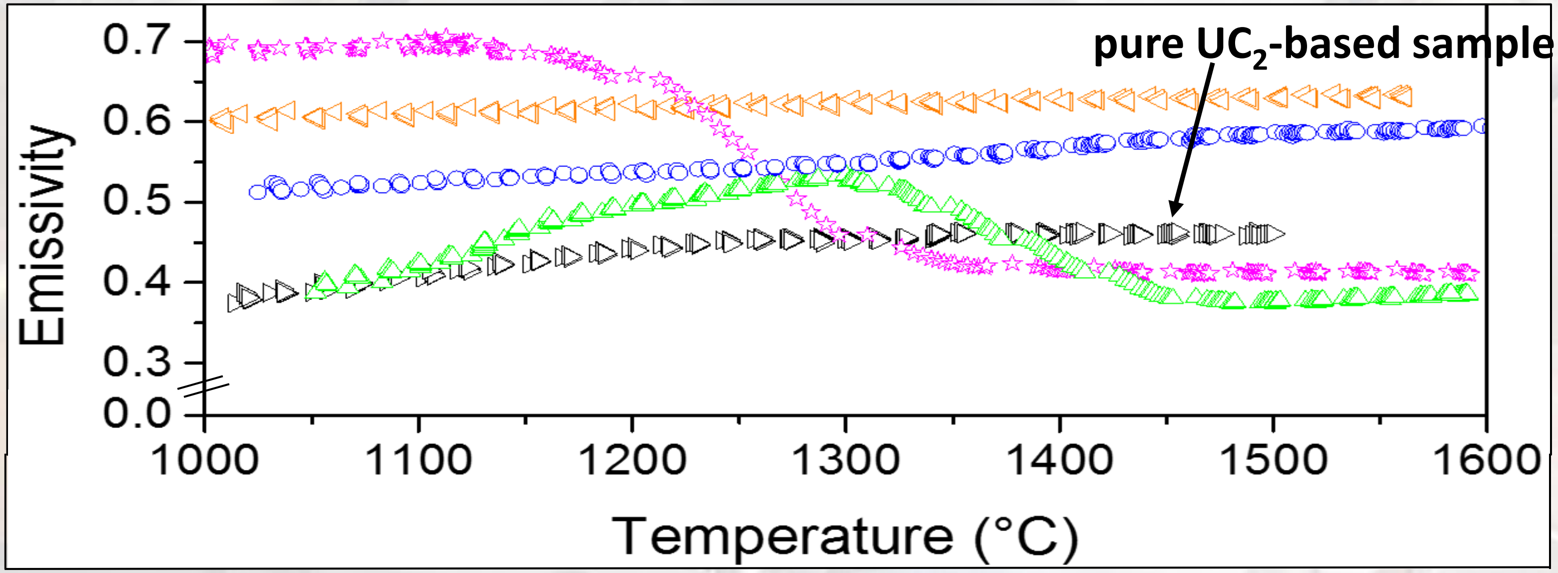
Emissivity

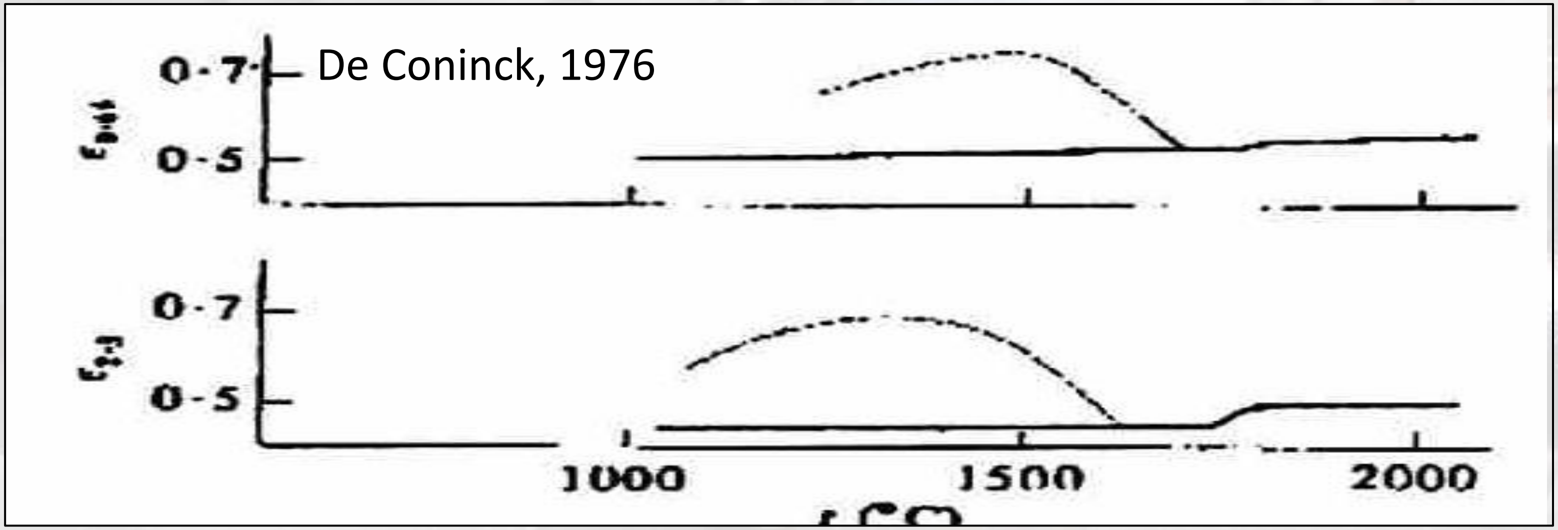




UC_x characterization

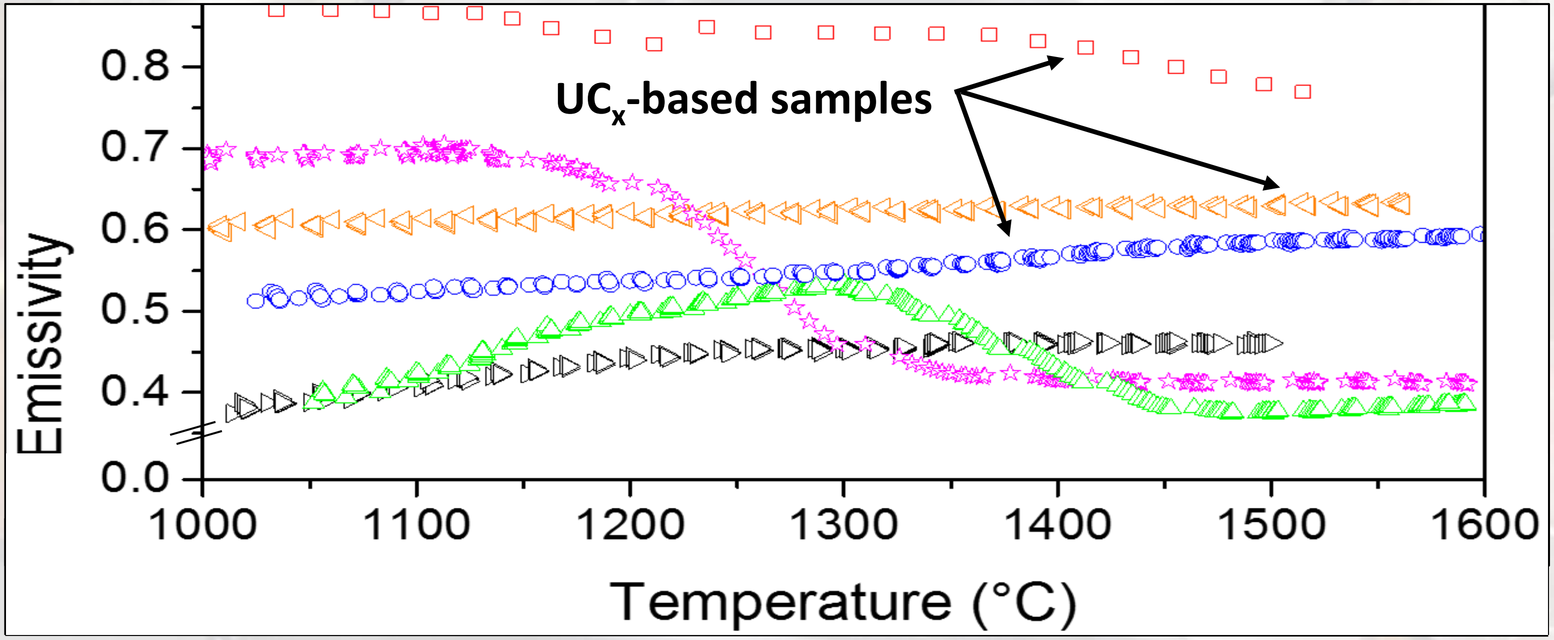
Emissivity

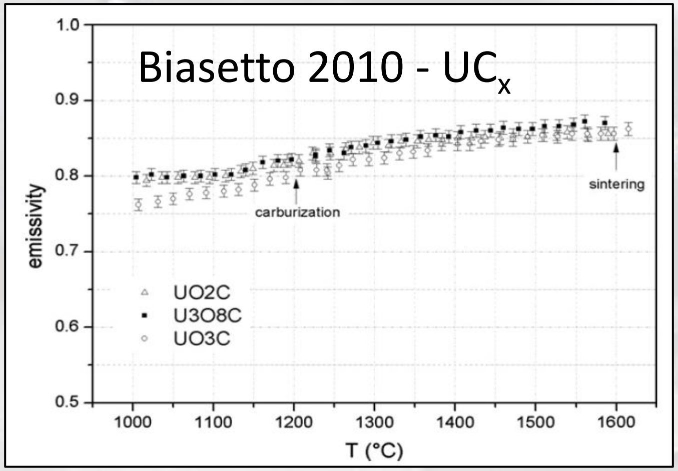


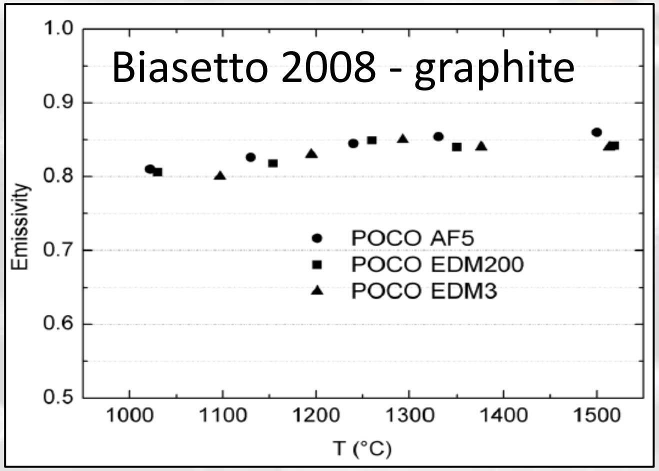


UC_x characterization

Emissivity







UC_x characterization

Conclusions

- Consistency of the thermal conductivity setup with the previous one verified (graphite test samples)
- Among the first results of ISOL-like UC_x materials thermal conductivity obtained
- Numerical model still to be improved (non-linear behavior for high temperatures, reduction of differences between experimental and numerical leading to narrower confidence bands)
- Temperature spectrum to be widened to explore more realistic operating conditions
- Emissivity correlation to structure and phases → useful in defining parameters for thermo-electrical simulations

Thank you for your attention