

DE LA RECHERCHE À L'INDUSTRIE



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GRAIN GROWTH AND MO HOMOGENIZATION IN U-7MO ATOMIZED PARTICLES

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^D KAIST - Republic of Korea

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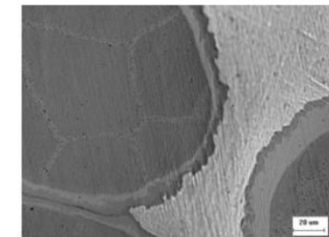
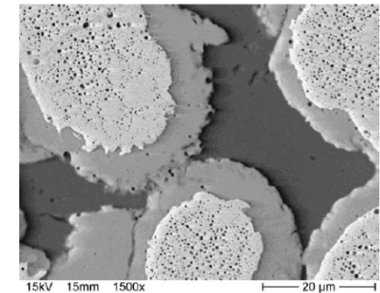
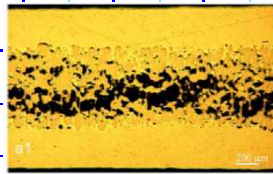
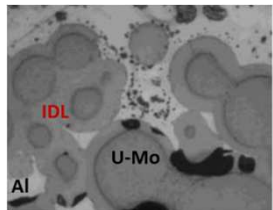
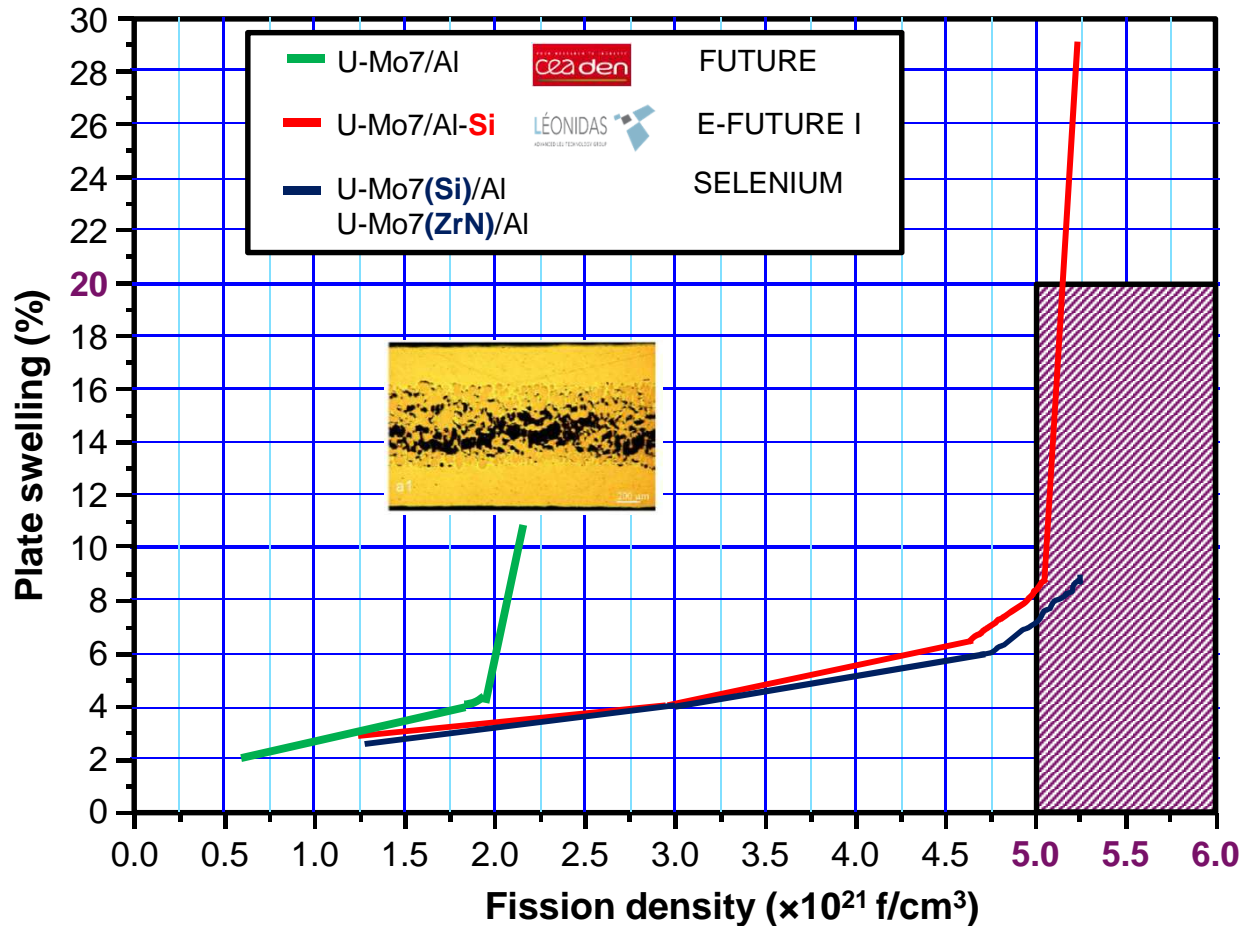
^G SCK-CEN

^H INL, USA

^I AREVA-CERCA



INTRODUCTION: DISPERSE U-MO DEVELOPMENT NEEDS



Two main sources for the swelling:

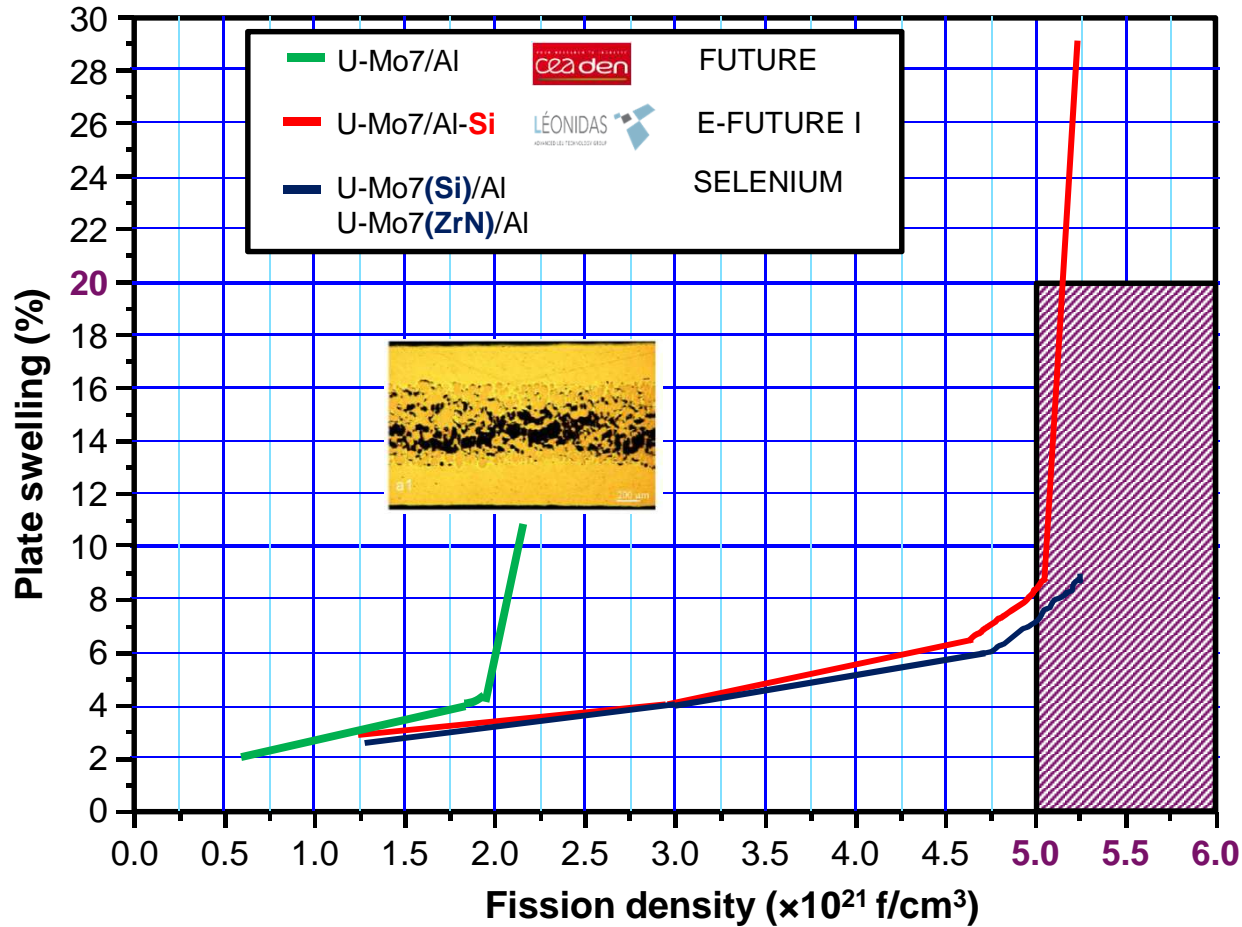
1. Growth of an IDL \Rightarrow **Coating of U-Mo particles with micrometer thickness**

2. Enhanced precipitation of fission products after U-Mo grain recrystallization \Rightarrow **U-Mo microstructure optimization (grain growth, Mo homogenization)**

S. Van den Berghe, *et al.*,
J. Nucl. Mater. 442 (2013) 60–68

Y.S. Kim, *et al.*,
J. Nucl. Mater. 454 (2014) 238-246.

INTRODUCTION: DISPERSE U-MO DEVELOPMENT NEEDS



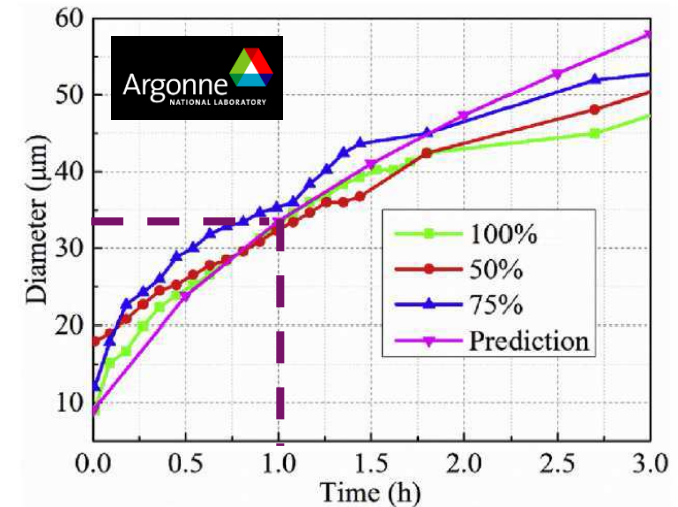
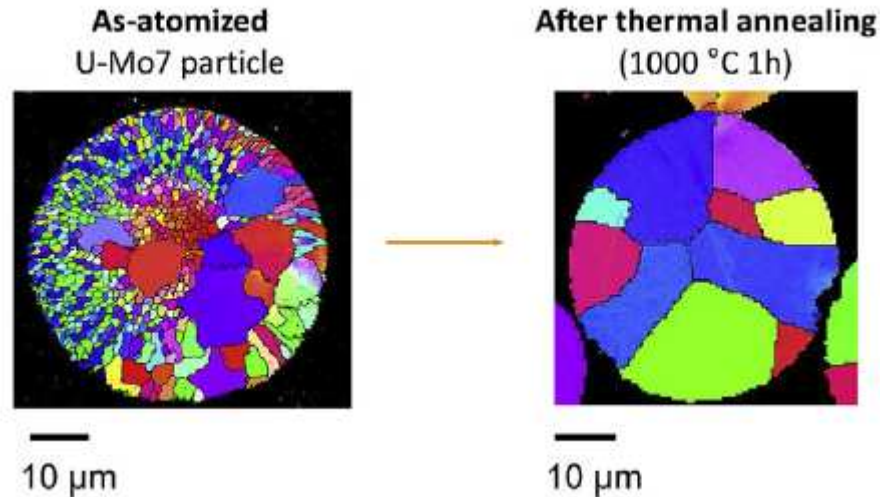
U-Mo microstructure optimization is based on thermal treatments. Reference conditions (1000°C, 1h) were set to provide both:

- Grain growth,
- Mo homogenization.

Can both mechanisms be de-correlated?

Outline

- 1. Grain Growth**
- 2. Mo-homogneization**
 - 1. Fresh powder**
 - 2. Methods: neutron diffraction**
 - 3. Mo-homogenization kinetics**
 - 4. Destabilization kinetics**
- 3. Conclusions**



In depth analysis of atomized microstructures (EMPIrE batches):

The as-atomized case:

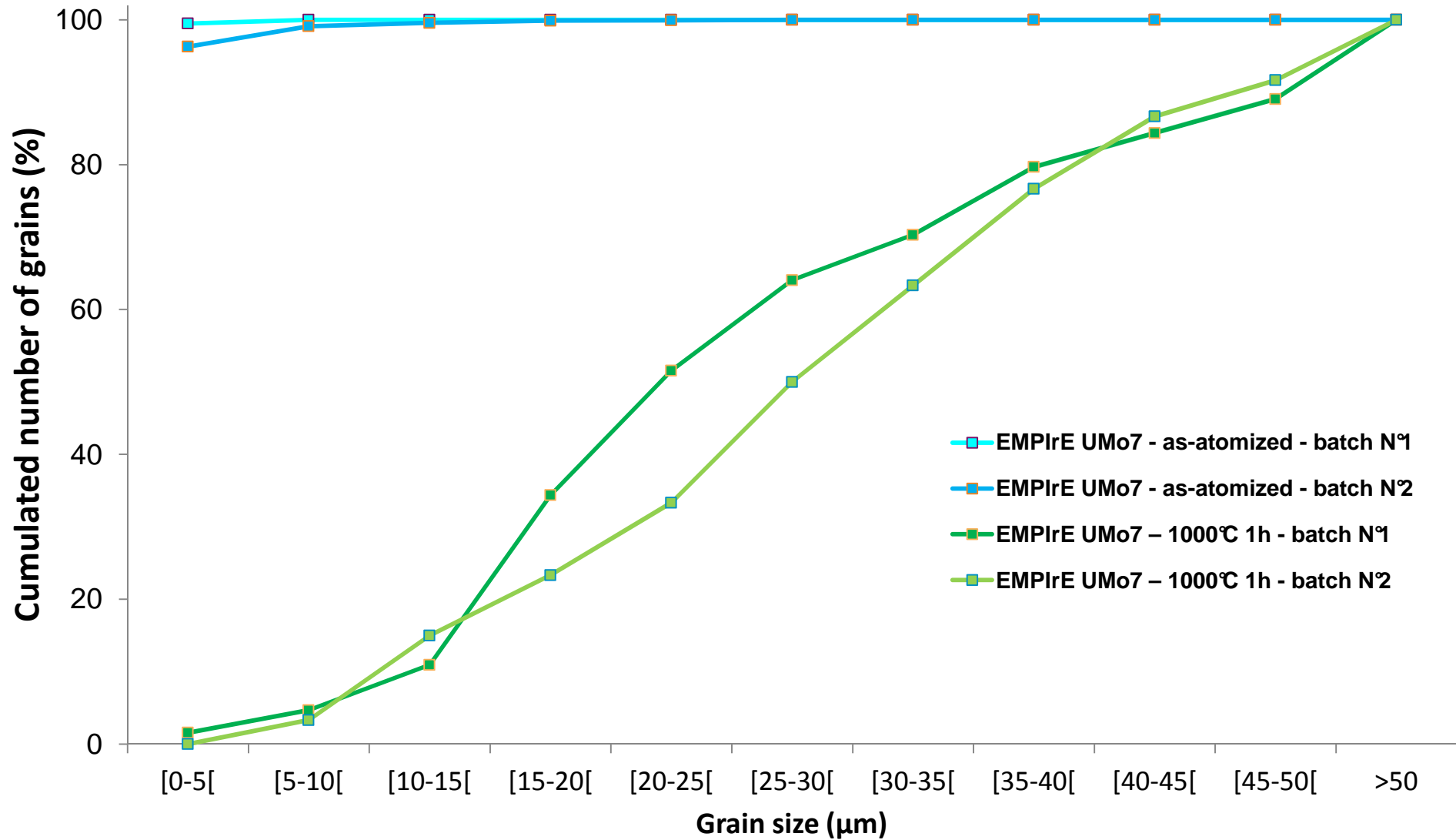
- 2-3 µm average grain size in excellent agreement with nano-XRD CT experiments

After heat treatment at 1000 °C during 1 h:

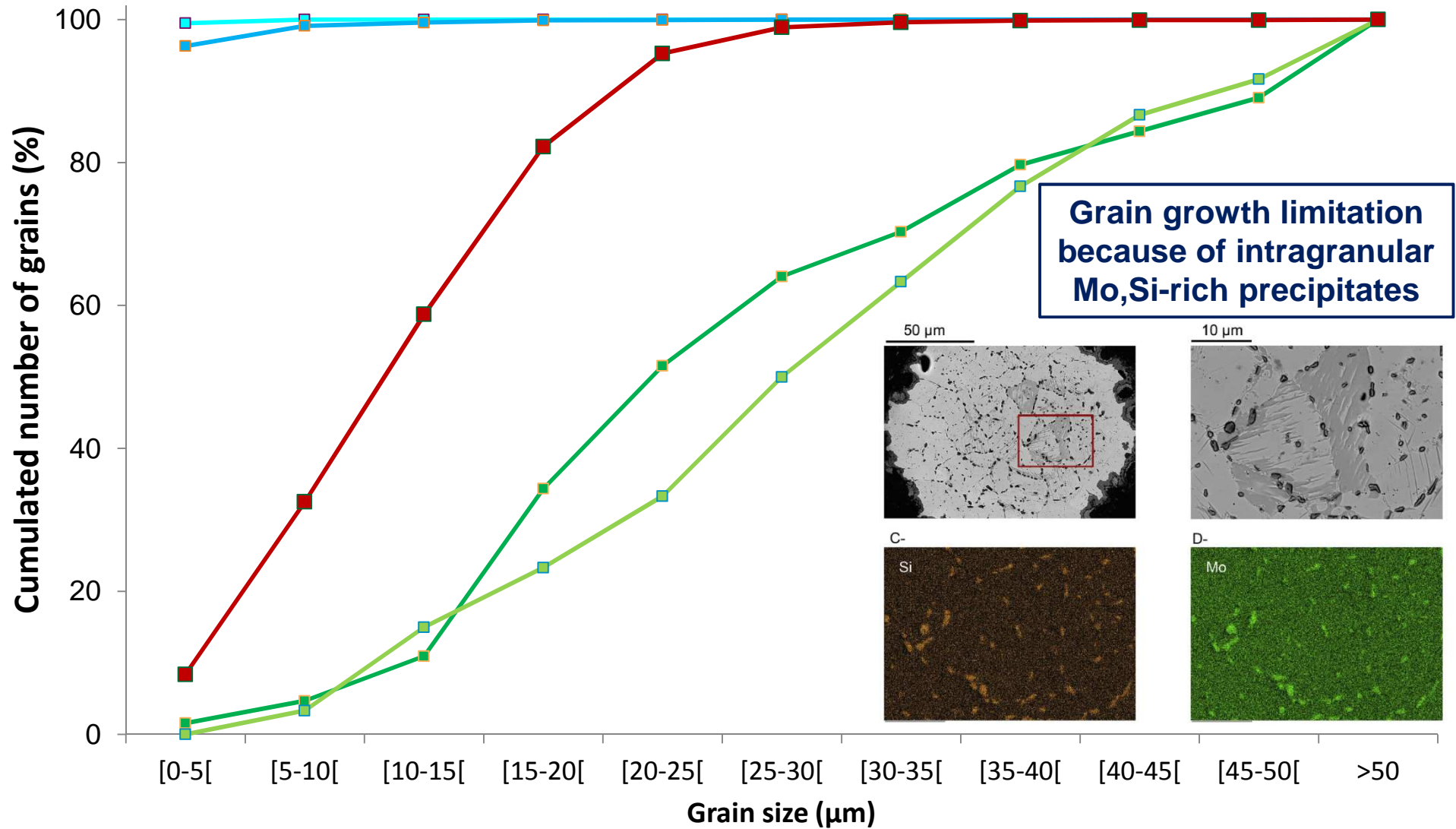
- 32-35 µm average grain size in excellent agreement ANL modelling
- Significant grain growth
- Good reproducibility of the heat treatment, whatever the equipment and the operator

**Annealing conditions validated for “optimized” in-pile performances
(EMPIrE, SEMPER-FIDELIS tests)**

GRAIN GROWTH IN ATOMIZED U-7MO PARTICLES



GRAIN GROWTH IN ATOMIZED U-7MO PARTICLES



Outline

1. Grain Growth

2. Mo-homogenization

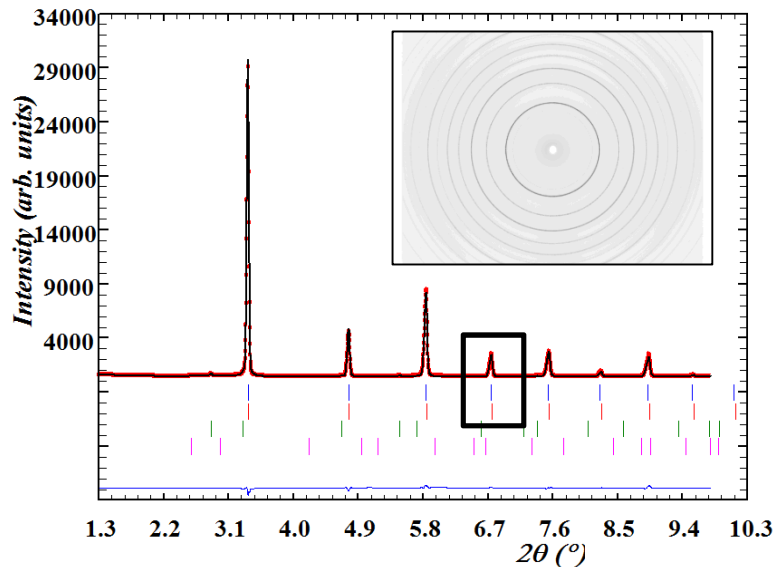
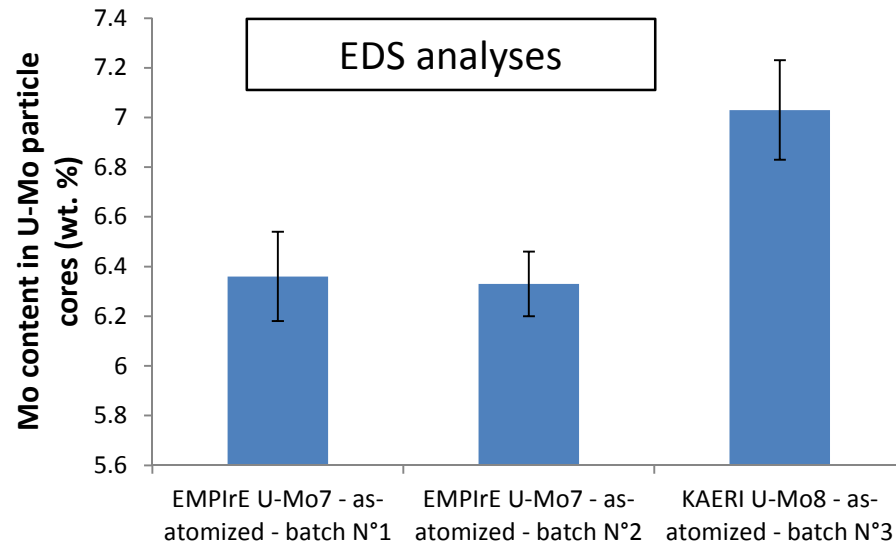
1. Fresh powder

2. Methods: neutron diffraction

3. Mo-homogenization kinetics

4. Destabilization kinetics

3. Conclusions



Mo concentration (wt%):

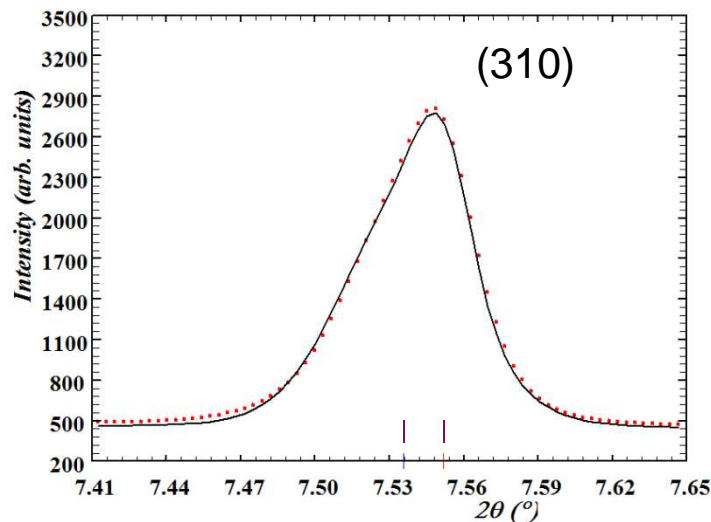
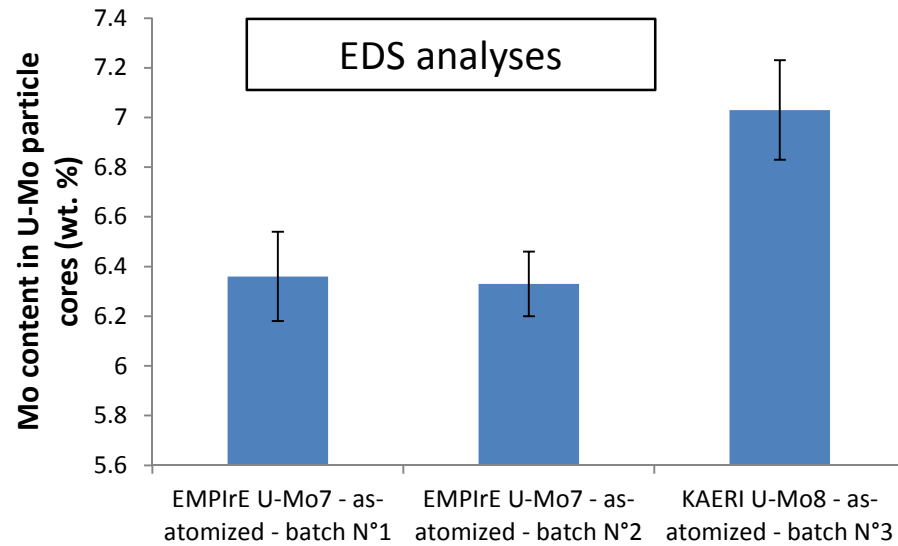
- KAERI : 8 ± 0.3 wt%
- EDS:
 - Semi-quantitative analysis
 - 0.6 wt% higher than for EMPIrE powders
- XRD :
 - Two UMo phases with different lattice constants

3.425113 3.417783

- Dwight's Law
- Result : ~8 wt%

Impurities (wt%):

| | Weight fraction (%) | Comments |
|-----------------|---------------------|------------|
| U(C,O, N) | 2 | High value |
| UO ₂ | ~0.3 | |

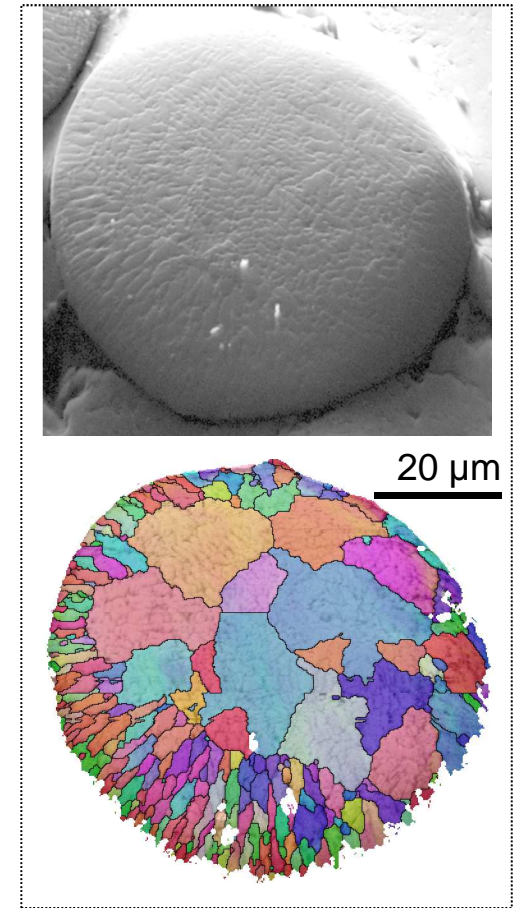
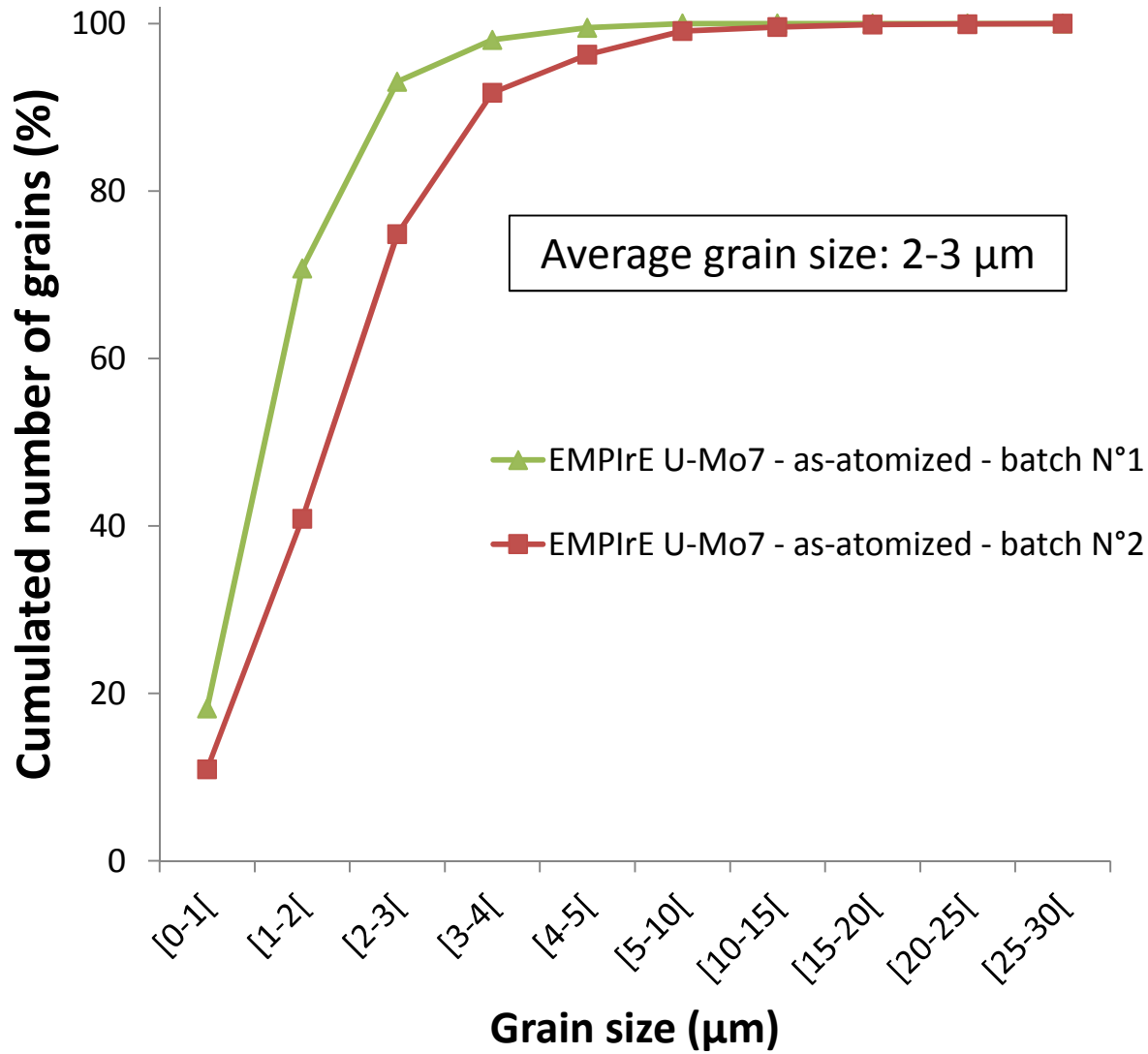


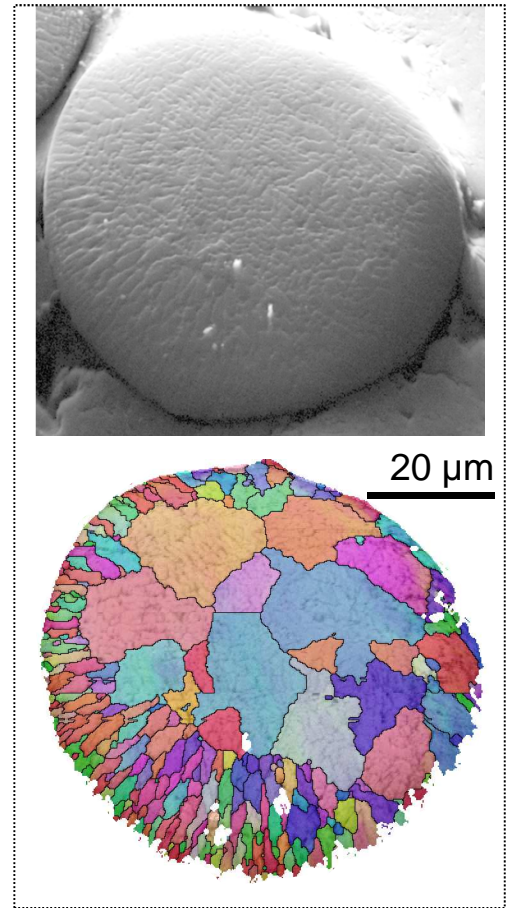
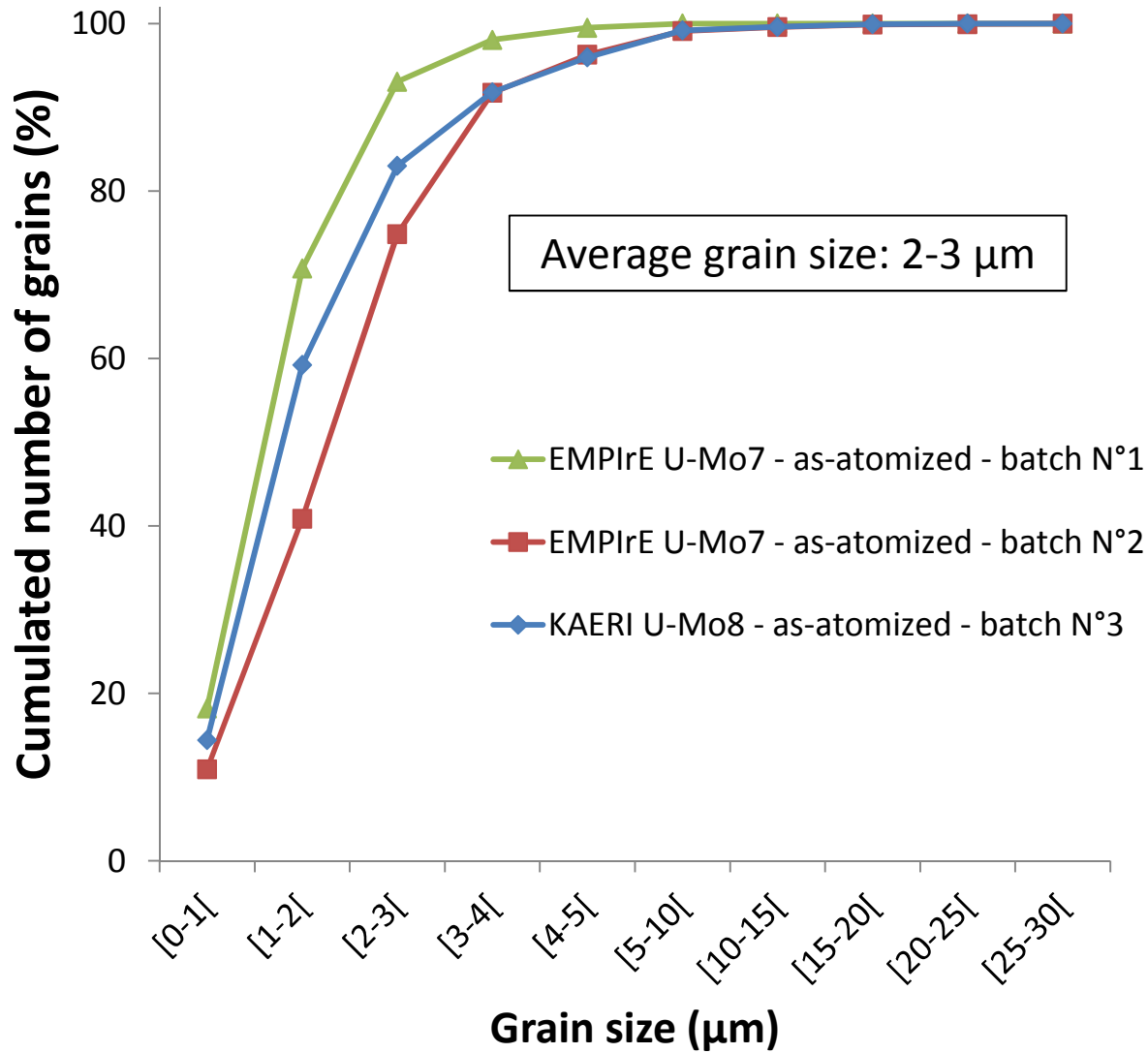
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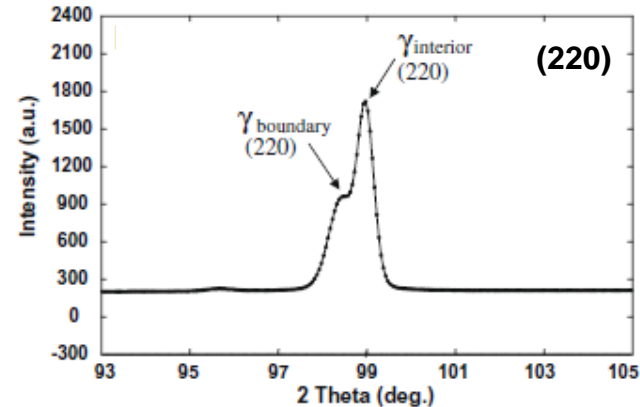
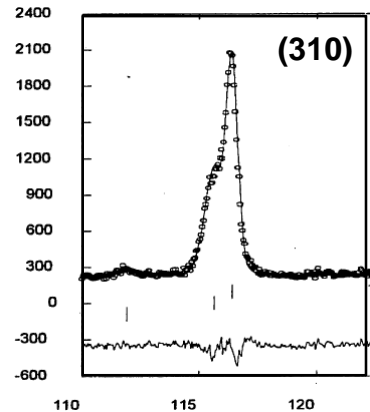


Reliability of EBSD analyses
on atomized powders and
reproducibility of production

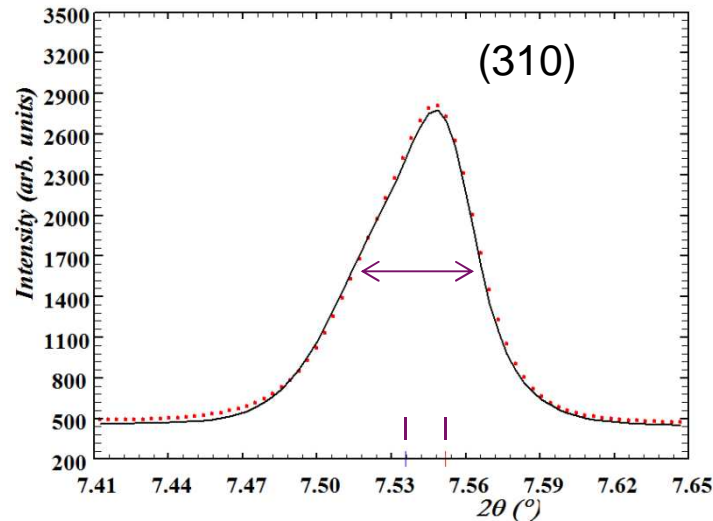
BASIS OF THIS WORK

Litterature

Neutron diffraction on as-atomized U-10 Mo powders



This XRD work



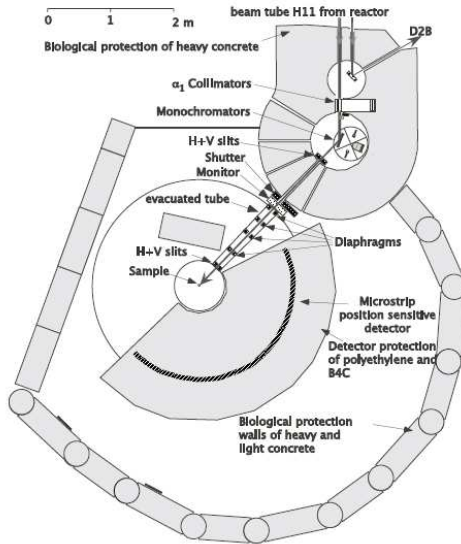
FWHM decrease (« narrowing ») of γ U-Mo Bragg lines is a marker of Mo homogenization in γ U-Mo.

Kinetics can be determined by an in situ diffraction analysis during TT

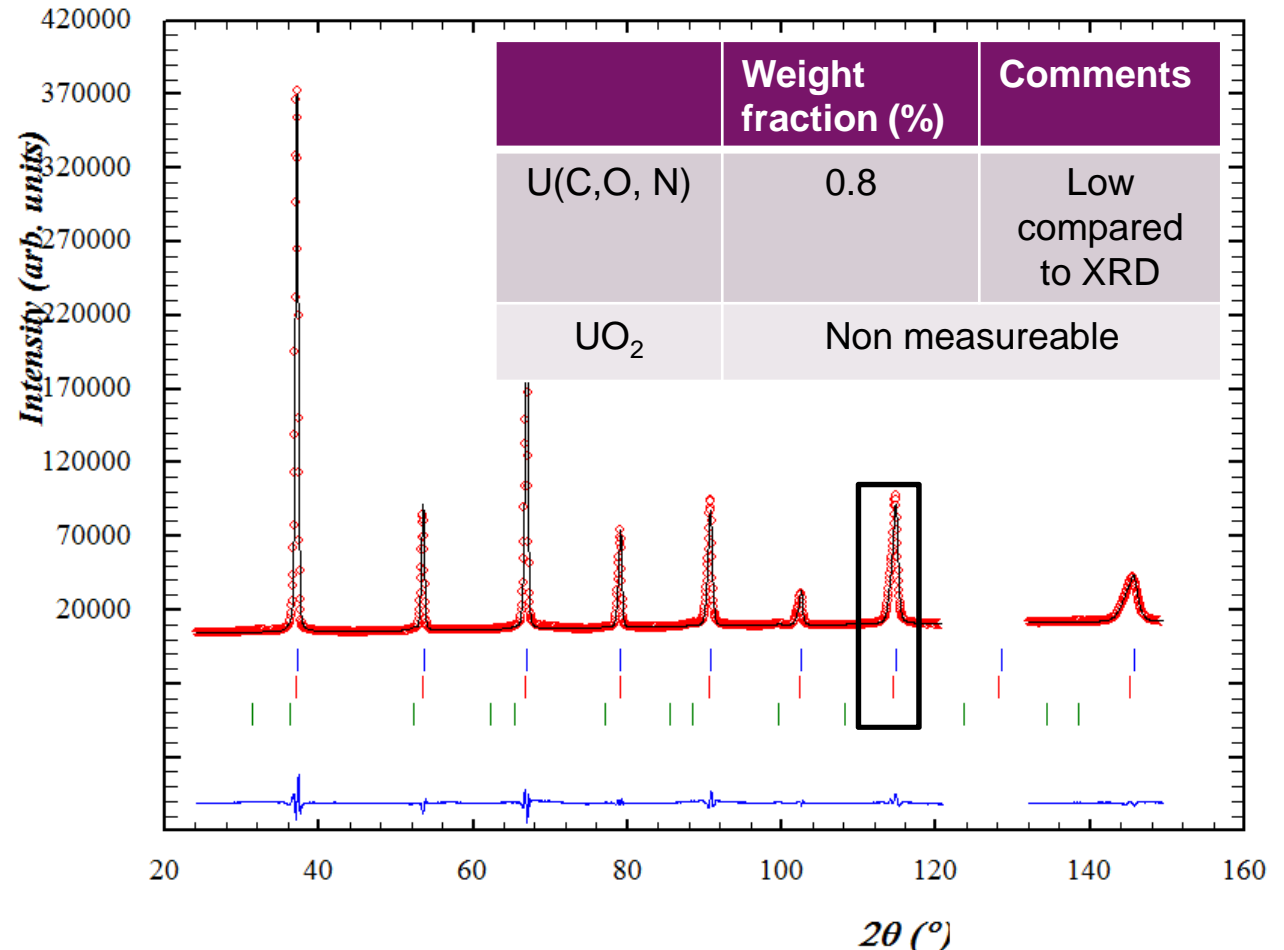
IN SITU NEUTRON DIFFRACTION: MEDIUM-TO-HIGH ANGULAR RESOLUTION



D20 beamline

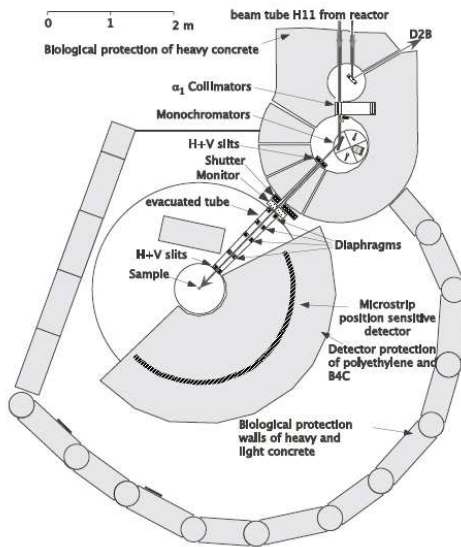


$$\lambda = 1.54 \text{ \AA}$$

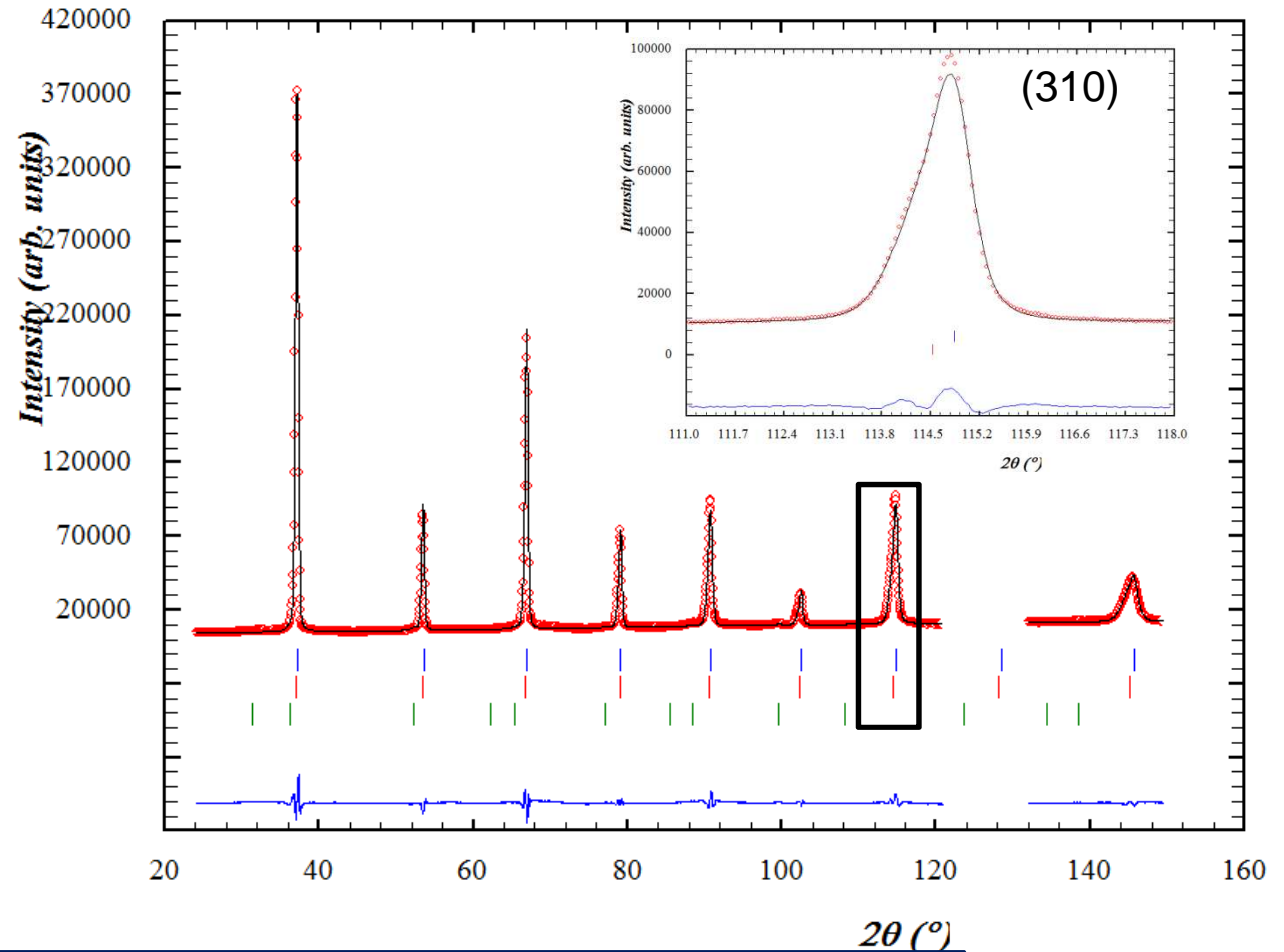


Diffractometer well suited for this experiment

D20 beamline



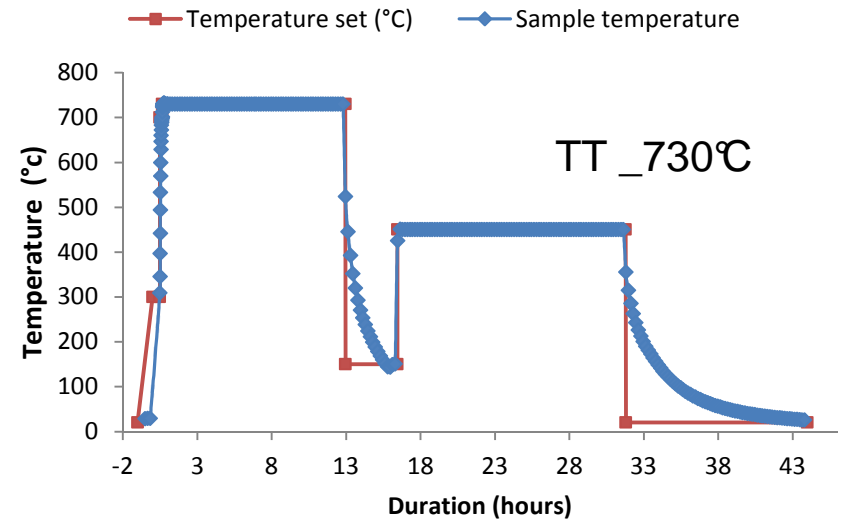
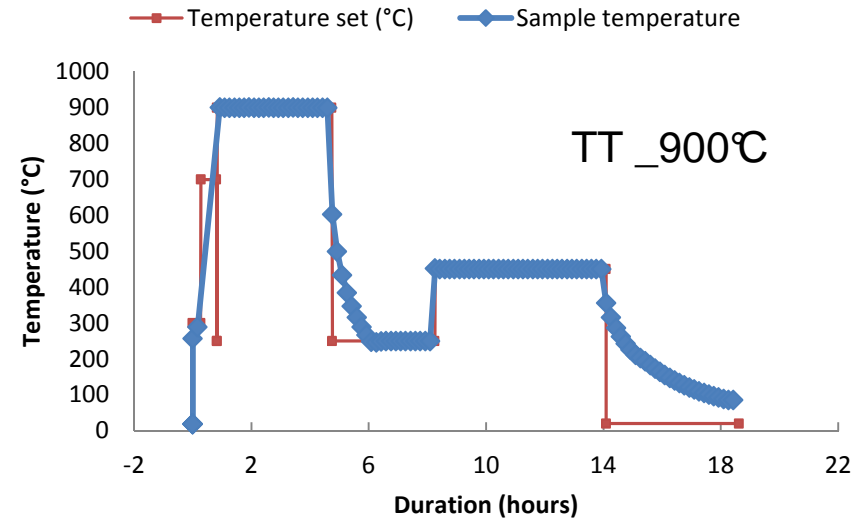
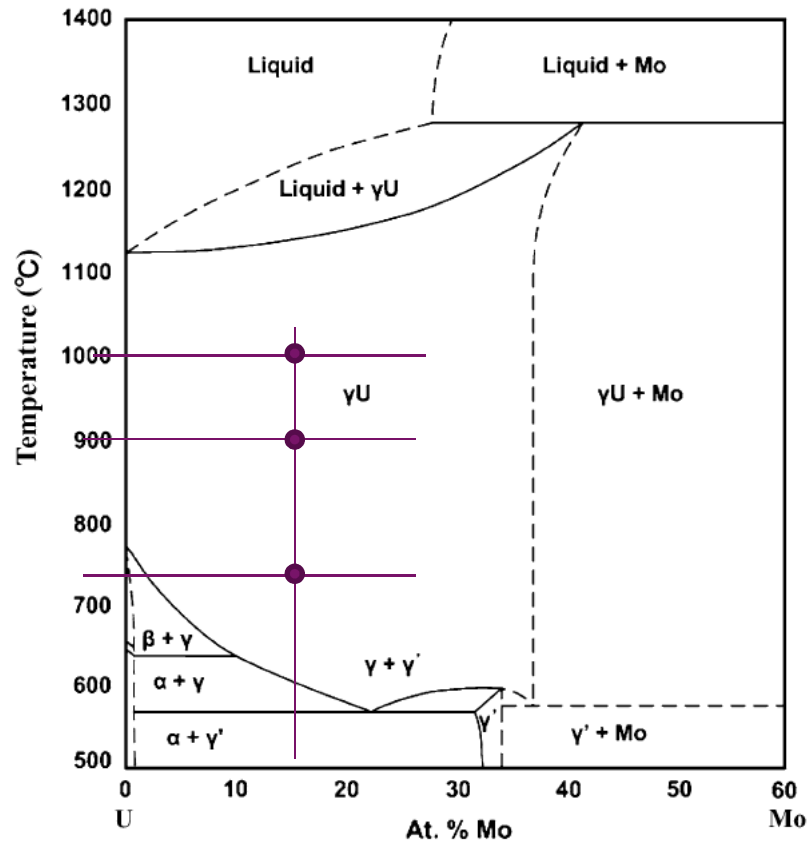
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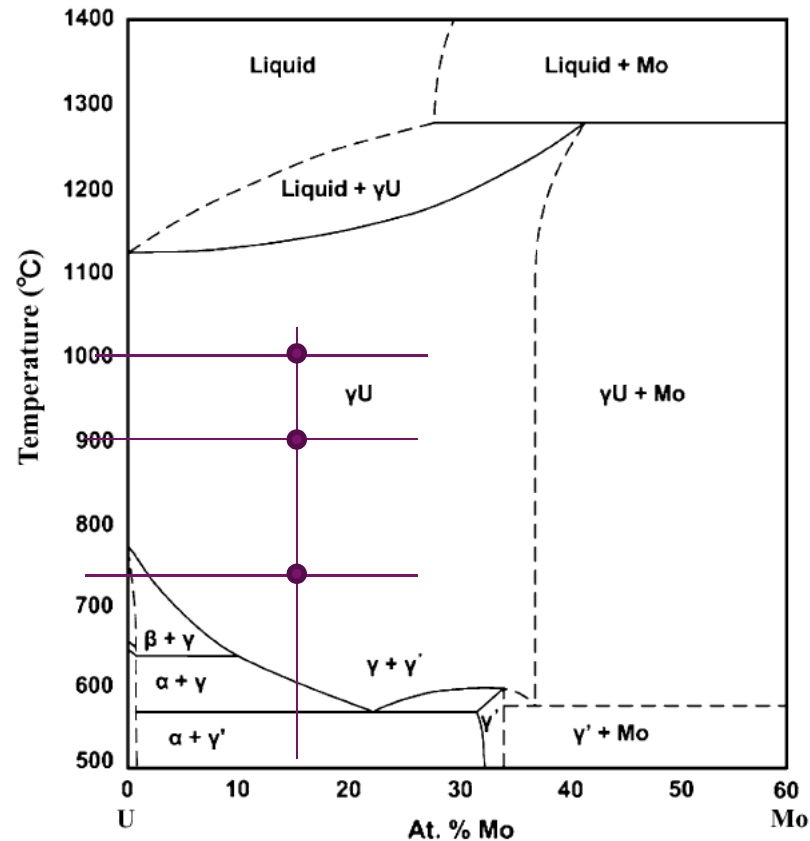
Diffractometer well suited for this experiment

SELECTED THERMAL TREATMENT CONDITIONS

Influence of temperature on homogenization kinetics



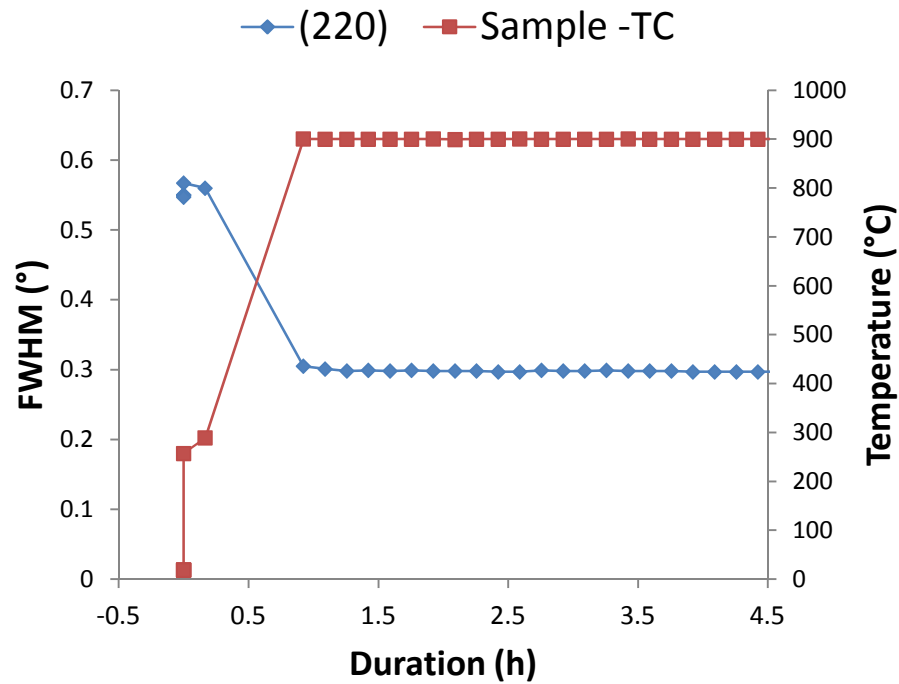
Influence of temperature on homogenization kinetics



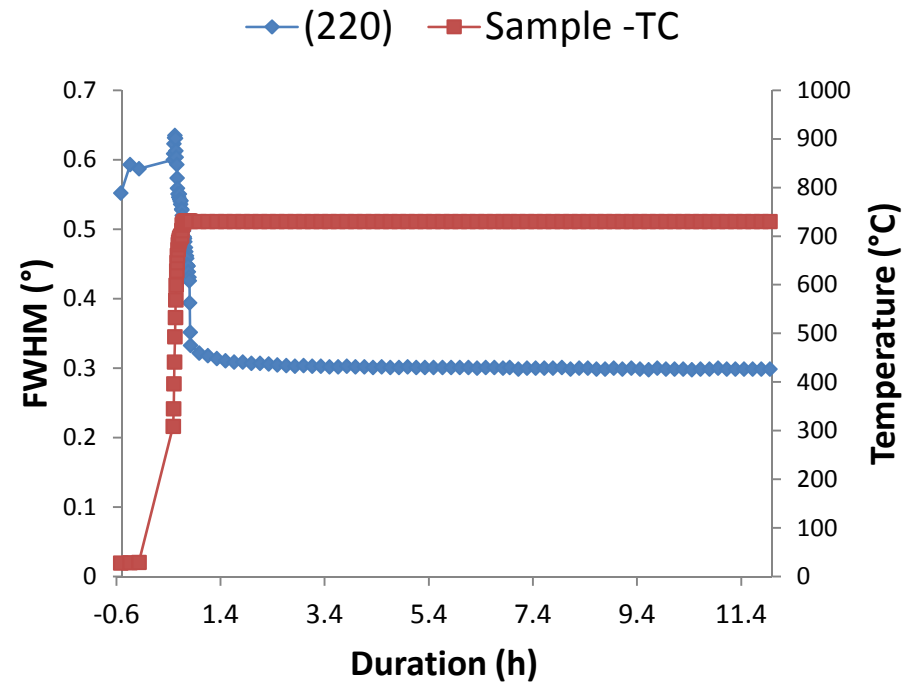
| | Homogenization | | Destabilization at 450 °C |
|---------------|------------------|--------------|---------------------------|
| | Temperature (°C) | Duration (h) | Duration (h) |
| TT_900 | 900 | 4 | 6 |
| TT_730 | 730 | 12 | 15 |
| TT_ref | 1000 | 1 | < 3 h |

HOMOGENIZATION KINETICS

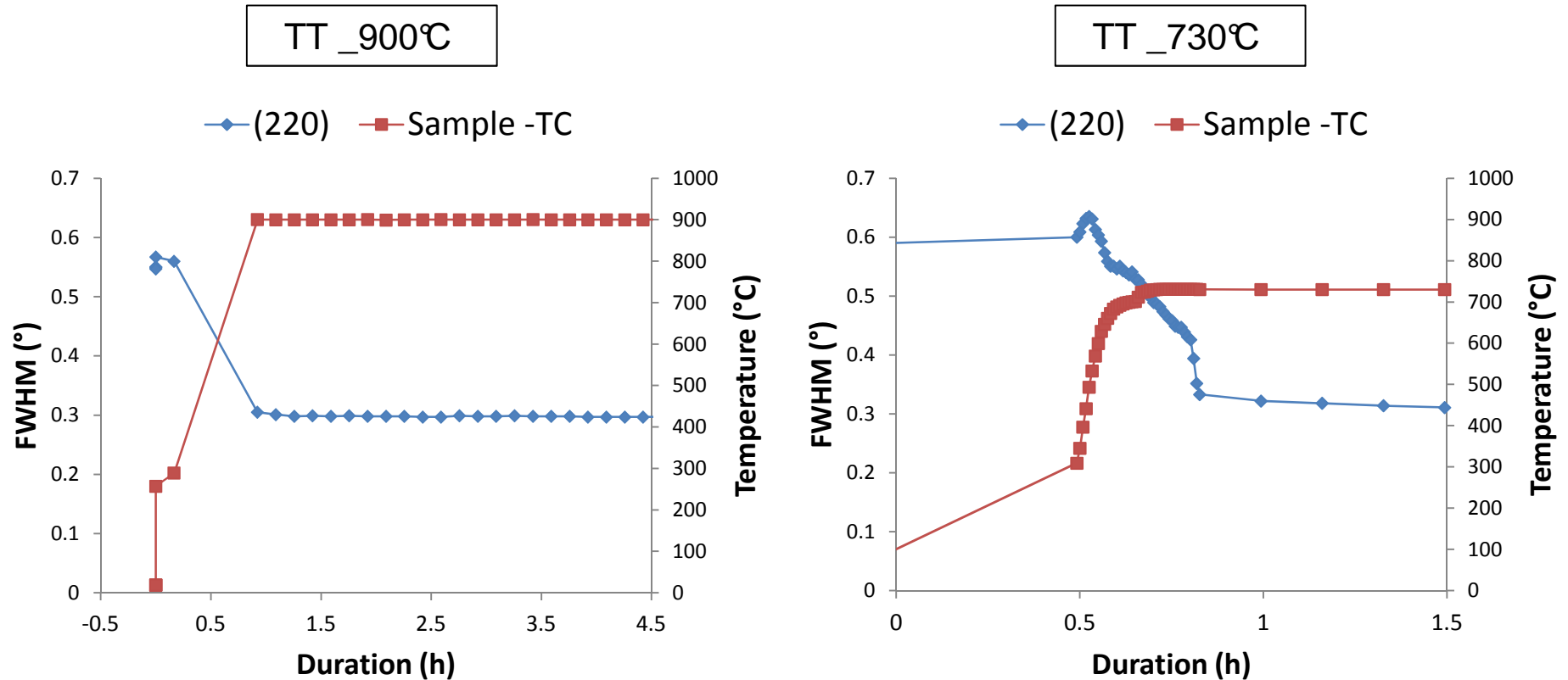
TT_900°C



TT_730°C



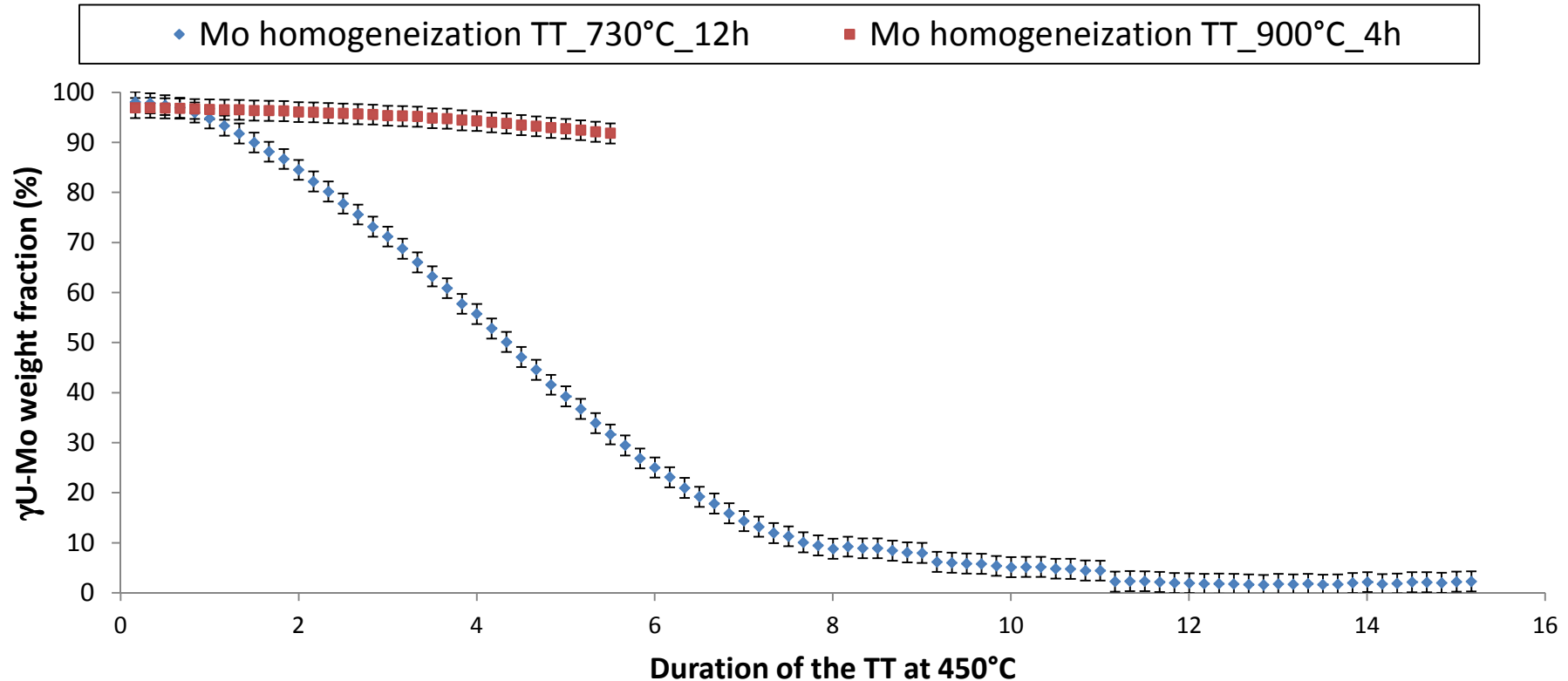
HOMOGENIZATION KINETICS



For both temperatures (730 and 900 °C), Mo homogenization is completed (i.e. the most significant part of this process) before sample temperature is stable.

For the reference case (1000°C, 1h), Mo is therefore homogenized

DESTABILIZATION KINETICS AT 450°C



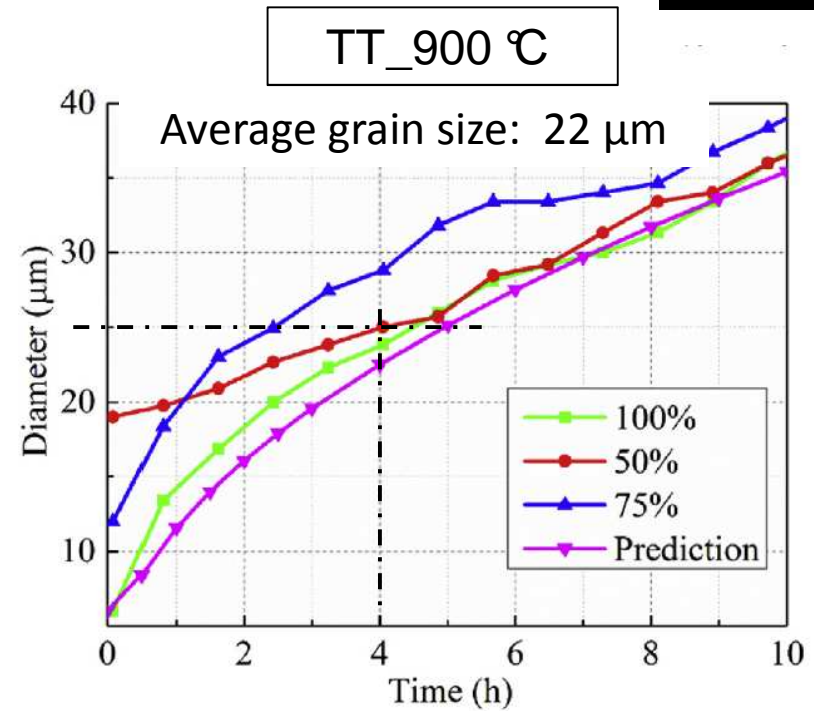
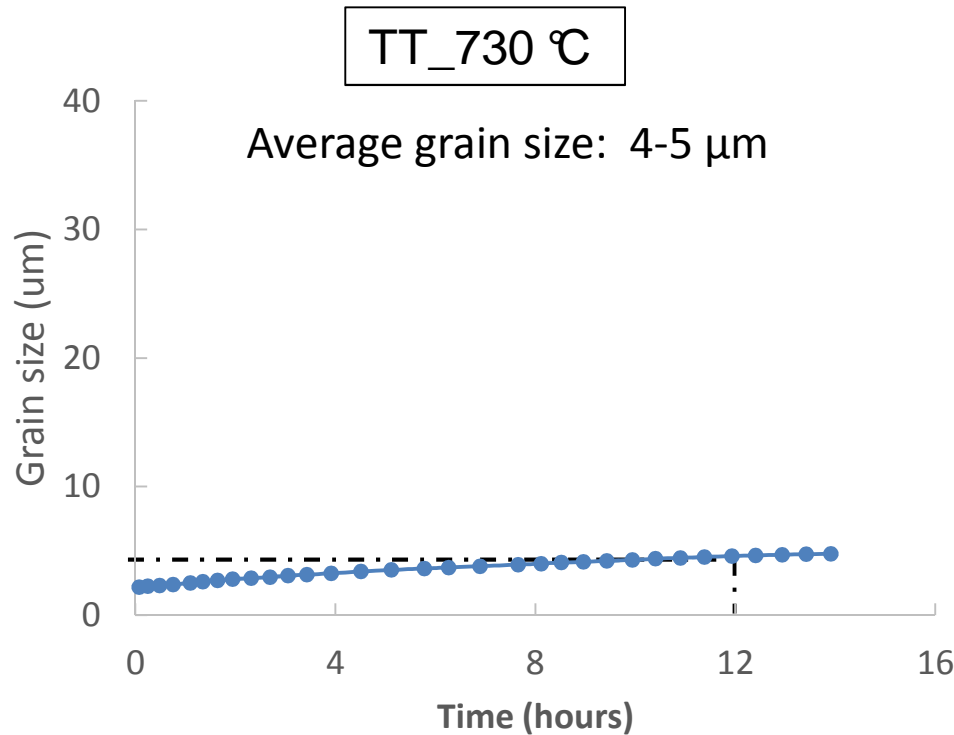
Destabilization kinetics are different for both powders whereas they exhibit the same initial Mo homogenization level - Interpretation ?

In the reference case (homogenization at 1000°C during 1h), a very low destabilization level in the particles is expected after fuel plate fabrication

Evaluation of the U-Mo grain size after homogenization TT:

- Modelling

Collaboration with Z.-G. Mei, L. Liang, A.M. Yacout



After Mo homogenization TT at 730 and 900°C, modelling shows that grain size is very different

Evaluation of the U-Mo grain size after homogenization TT:

- First SEM/EBSD characterizations (after destabilization TT however)

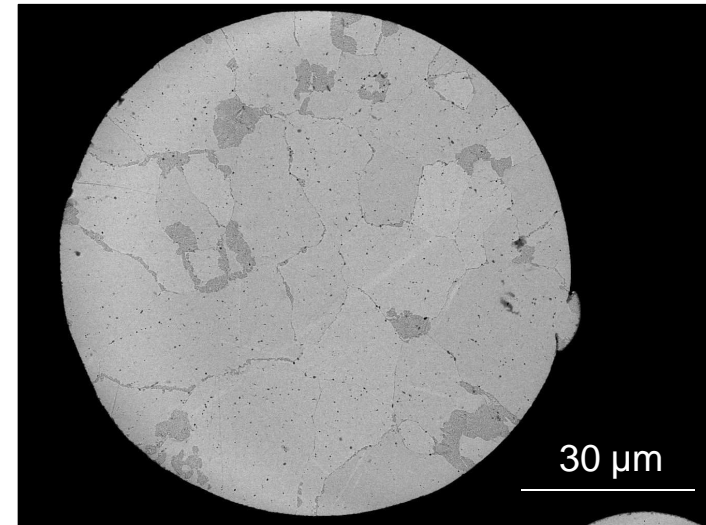
TT_730 °C

Average grain size: very low



TT_900 °C

Average grain size EBSD: ~13 µm

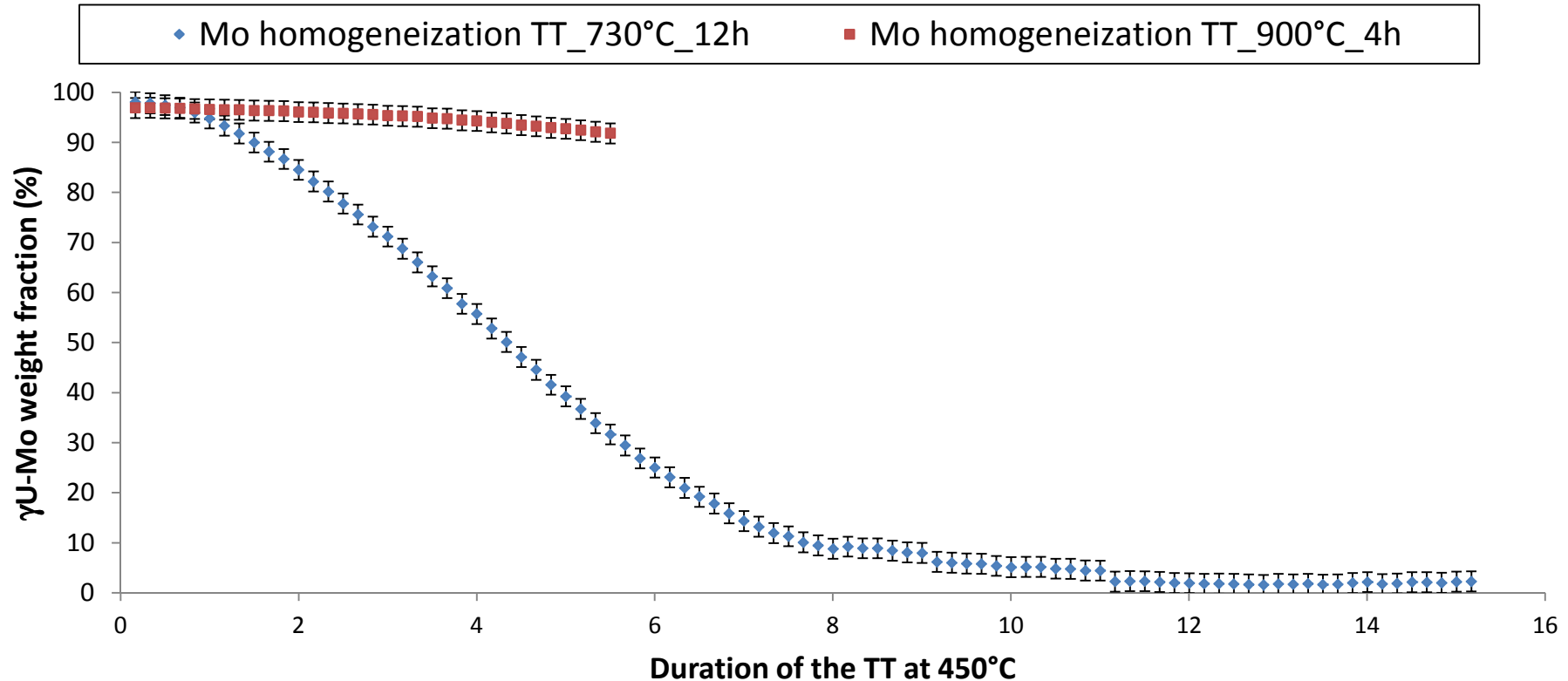


High level of destabilization: observation are difficult

Destabilization ? High UC content ?

After Mo homogenization TT at 730 and 900°C, modelling and experiment show that grain size is very different

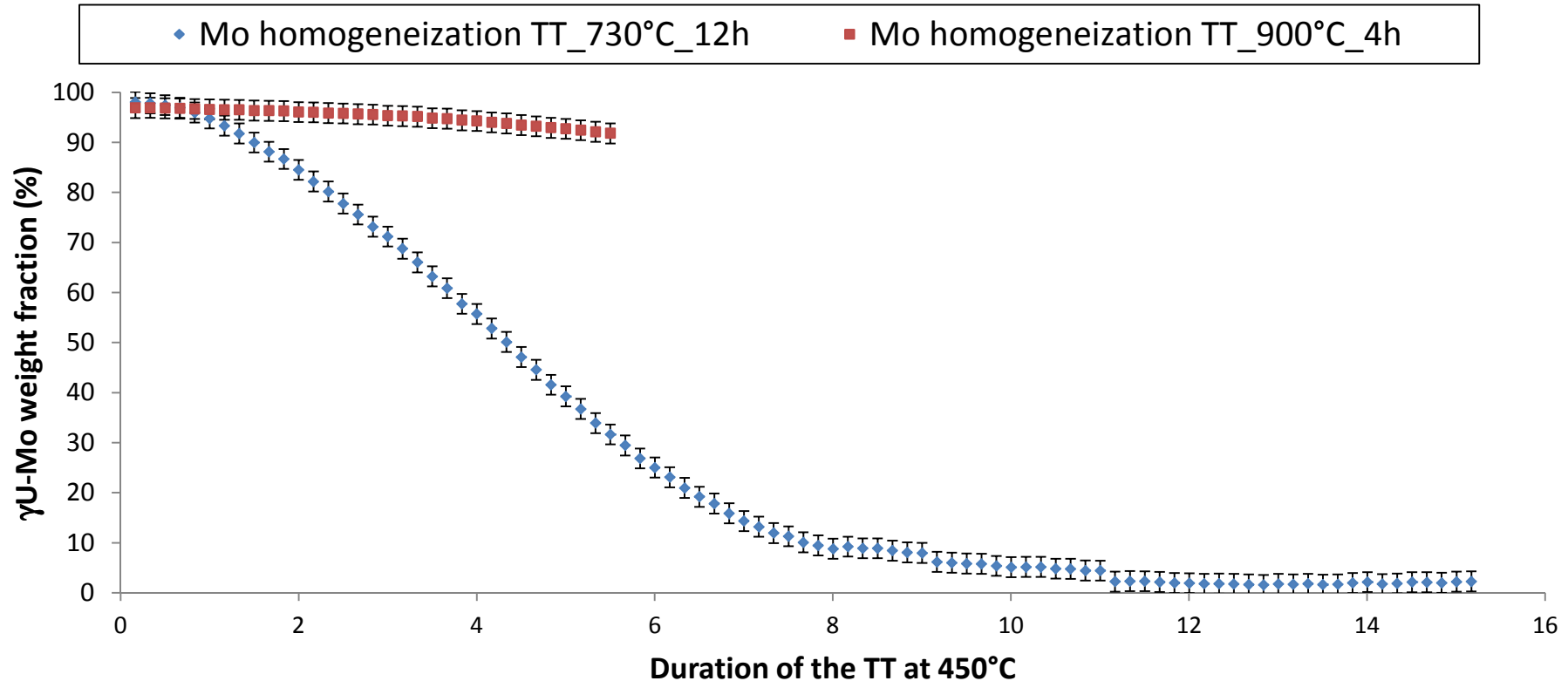
DESTABILIZATION KINETICS AT 450°C



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DESTABILIZATION KINETICS AT 450°C



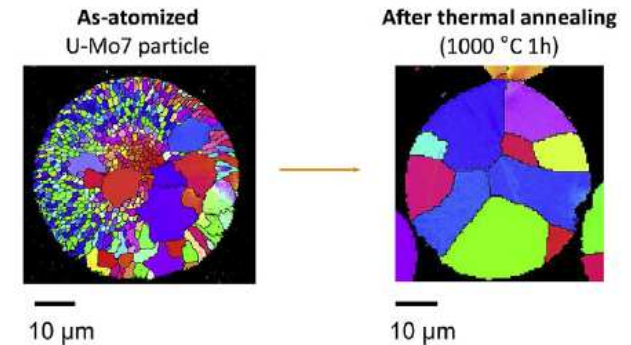
Destabilization kinetics are different for both powders whereas they exhibit the same initial Mo homogenization level - Interpretation ? **Grain size effect**

In the reference case (homogenization at 1000°C during 1h), a very low destabilization level in the particles is expected after fuel plate fabrication

Two high temperature “processes” were investigated on as-atomized U-7Mo

1. Grain growth analysis using EBSD

- EBSD is a very powerful technique after sample surface preparation optimization
- Validation of the reference TT conditions for grain coarsening: reproducibility assessed

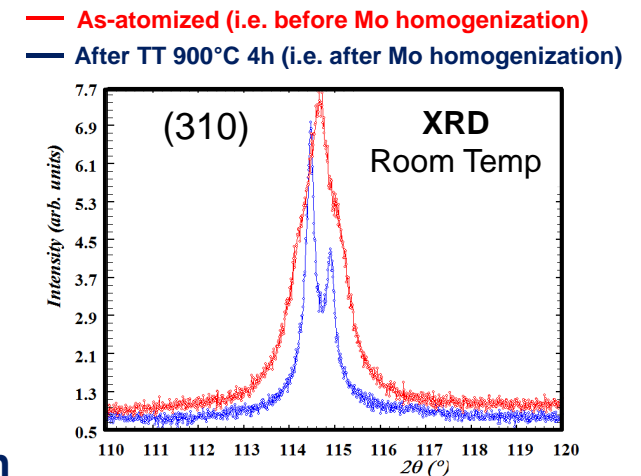


X. Iltis *et al.*, J. Nucl. Mater. **495** (2017), 249-266.

2. Mo-homogenization using *in situ* neutron diffraction

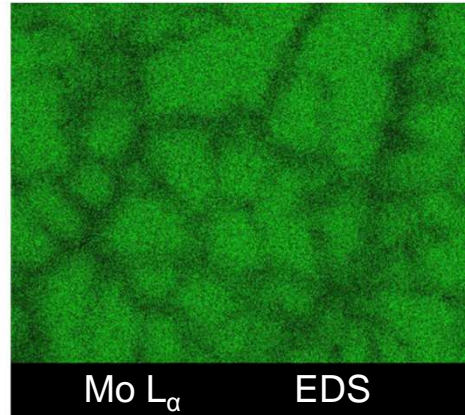
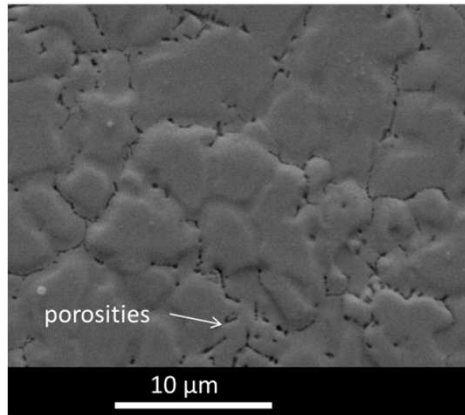
- Preliminary step (further confirmation work in progress)
- Mo-homogenization at $T > 730^\circ\text{C}$ is a swift process: it is completed during the temperature ramp
- Mo-homogenization completed in the reference conditions (1000°C , 1h)
- $\gamma\text{U-Mo}$ destabilization kinetics influenced by grain size

Mo-homogenization kinetics are not correlated to grain growth

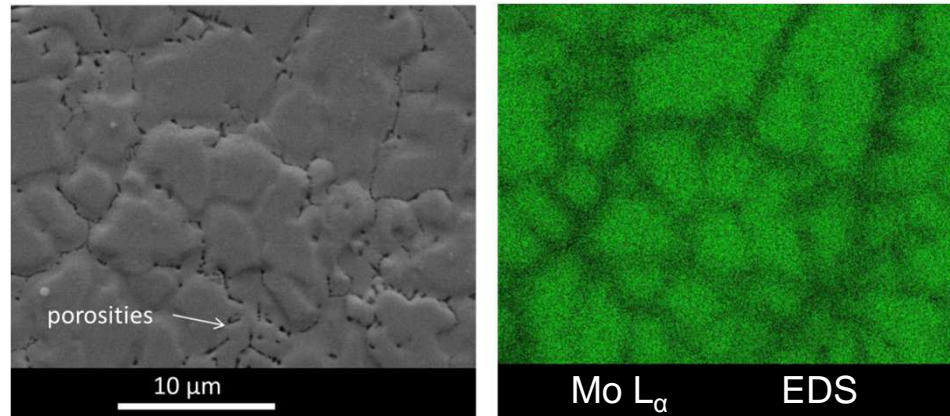


**THANK YOU FOR YOUR
ATTENTION !!!**

AS-ATOMIZED U-MO PARTICLES: MICROSTRUCTURE

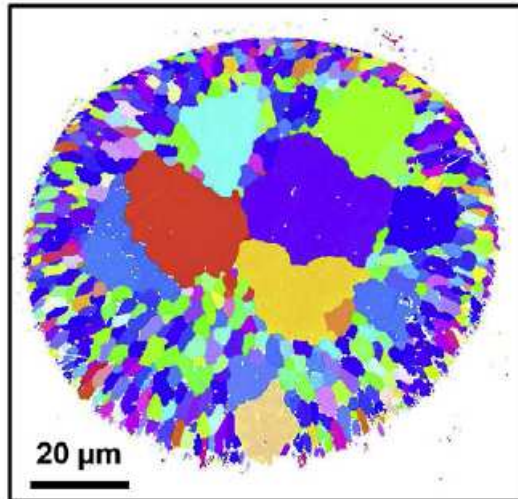


**Crystallographic relationships
between Mo-rich and Mo-depleted
areas (core and grain boundary areas)**



**Crystallographic relationships
between Mo-rich and Mo-depleted
areas (core and grain boundary areas)**

Dendrites are single crystals



Grain orientation map

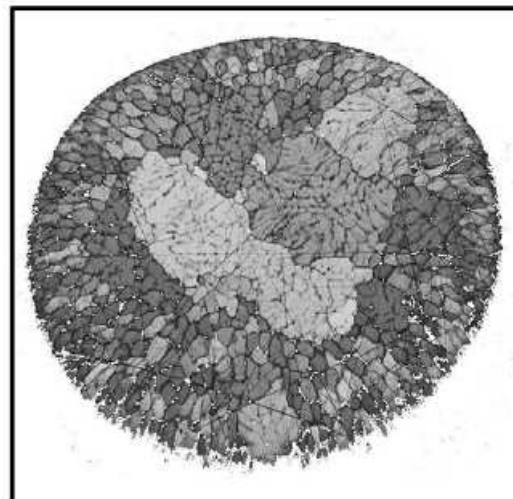
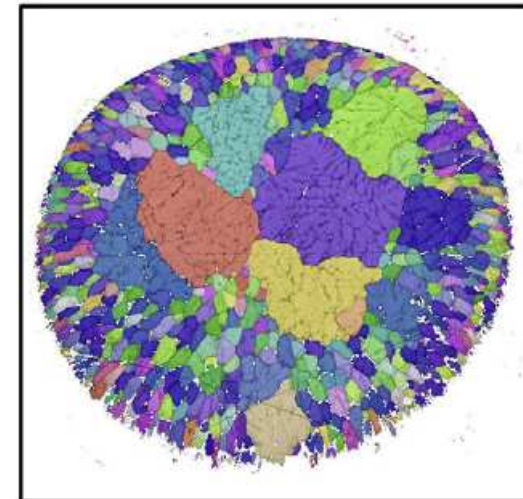


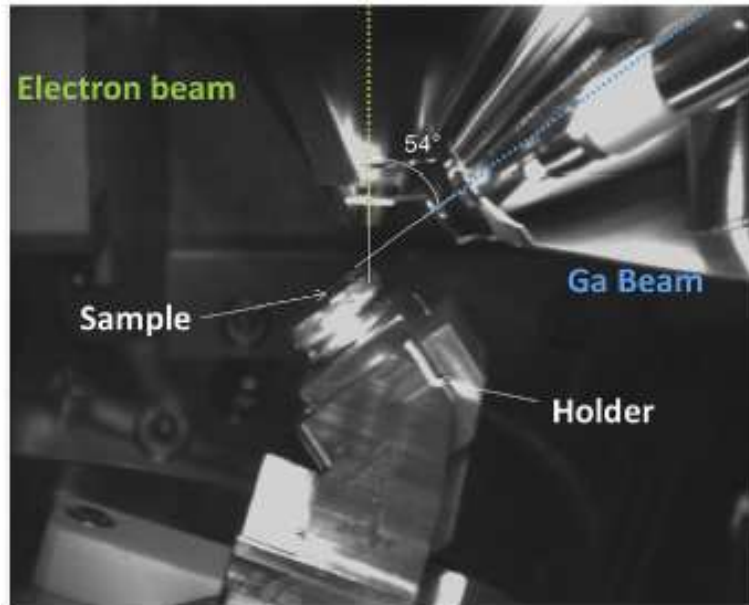
Image quality map



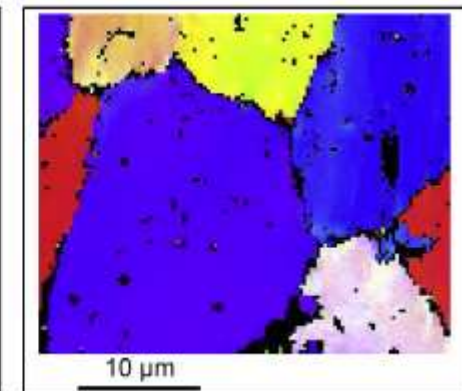
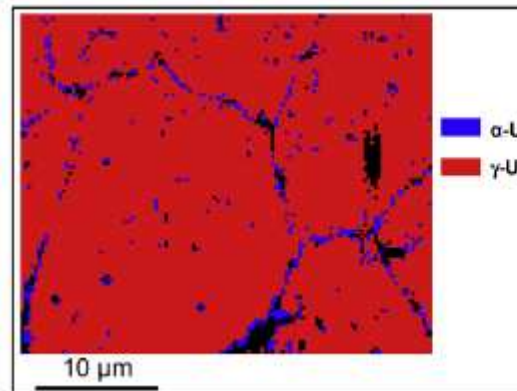
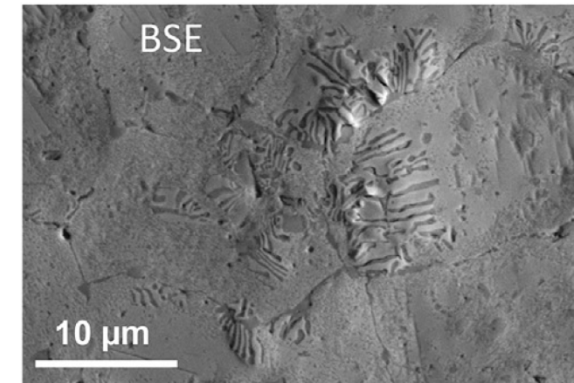
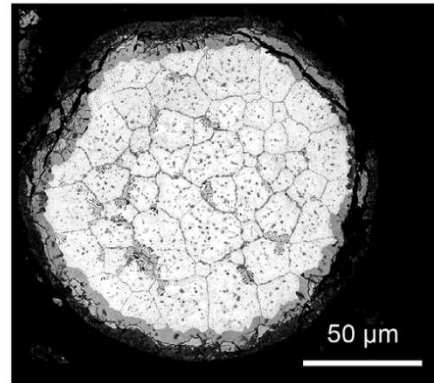
Overlay

G. Champion, Ph.D. thesis
Rennes University, 2013.

X. Iltis *et al.*,
J. Nucl. Mater. **495** (2017), 249-266.



KOMO-5 UMo₇(Si)/Al rod:
Evidence for destabilized areas



Successful sample preparation using FIB milling