



Effect of powder outgazing conditions

D. Sornin, P.-F. Giroux, D. Fabrègue, P. Mas, R. Soulas, D. Hamon

► To cite this version:

D. Sornin, P.-F. Giroux, D. Fabrègue, P. Mas, R. Soulas, et al.. Effect of powder outgazing conditions: On mechanical and microstructural properties of oxides dispersed strengthened steel. THERMEC'2016 - International Conference On Processing and Manufacturing of Advanced Materials, May 2016, Graz, Austria. cea-02509731

HAL Id: cea-02509731

<https://cea.hal.science/cea-02509731>

Submitted on 17 Mar 2020

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.

DE LA RECHERCHE À L'INDUSTRIE



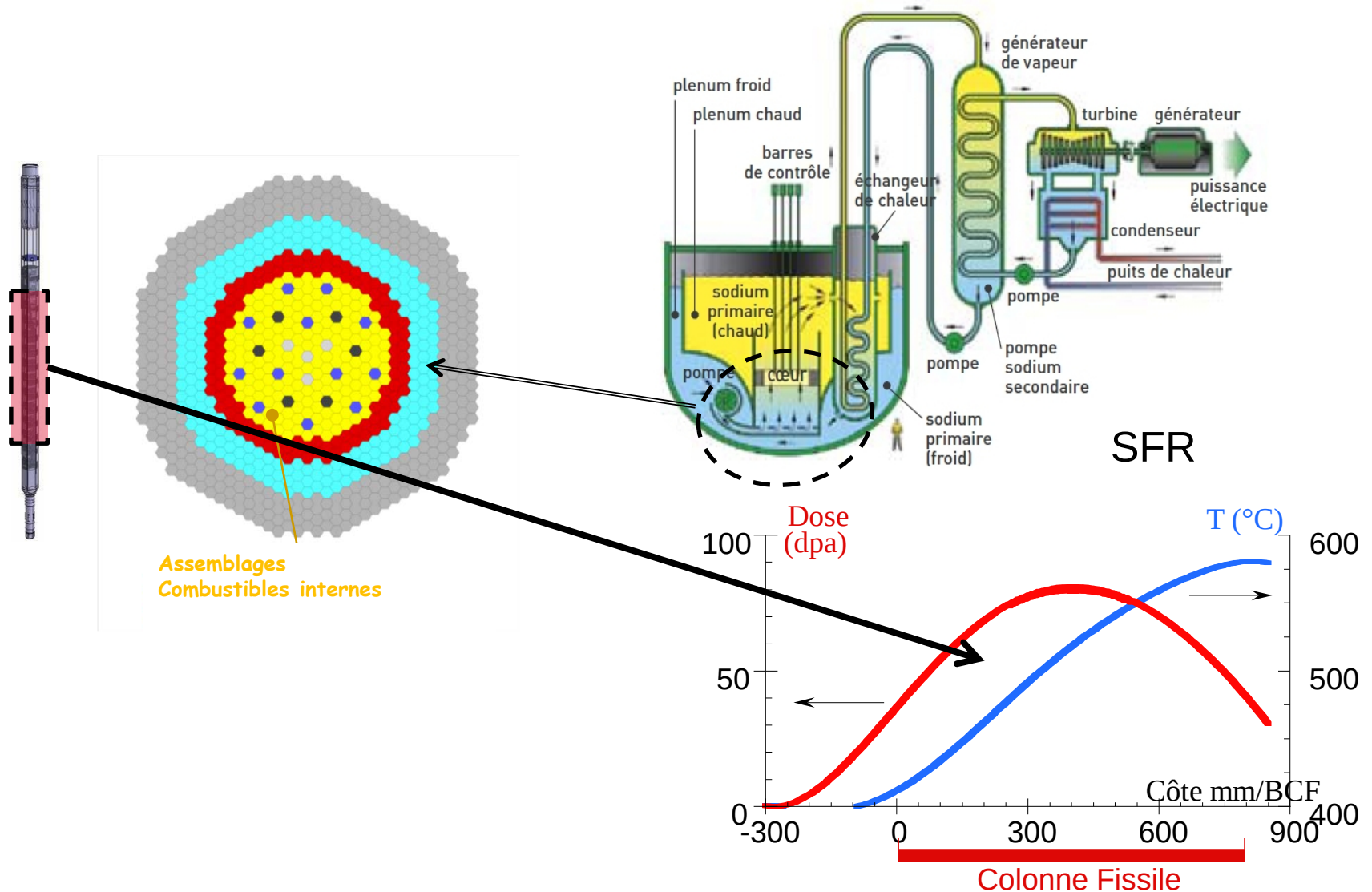
EFFECT OF POWDER OUTGAZING CONDITIONS ON MECHANICAL AND MICROSTRUCTURAL PROPERTIES OF **OXIDES DISPERSED** **STRENGTHENED STEEL**

THERMEC-2016 | May 29 - June 3. GRAZ | Denis Sornin, P-F. Giroux, D. Fabrègue, P. Mas, R. Soulas, D. Hamon.

www.cea.fr

JUNE 2, 2016 SESSION: G7
MATERIALS UNDER EXTREME CONDITIONS 3

ODS STAINLESS STEEL ODS FOR NUCLEAR APPLICATIONS



ODS = Oxide Dispersion Strengthened

- **Application** : fuel cladding for sodium cooled fast reactors (Gen. IV fission reactor) in view to reach very high burn-up
- **Definition** : ferritic/martensitic stainless steels (**Fe-9/18% Cr**) reinforced by an homogeneous dispersion of nano-sized particles (Y, Ti, O)
- **Properties** :
 - ➔ Resistance under irradiation (low swelling of bcc matrix)
 - ➔ Resistance to corrosion
 - ➔ Good behavior under creep strength thanks to nano-oxides.



Nano-oxides
(radius ~ 2-4 nm)

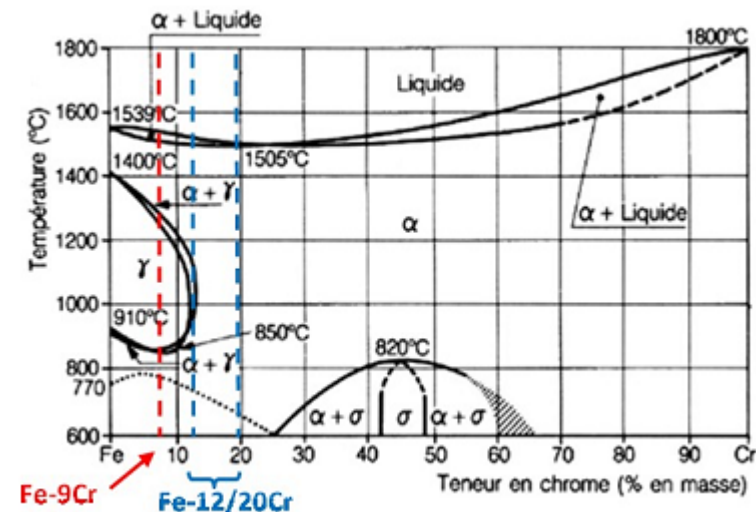
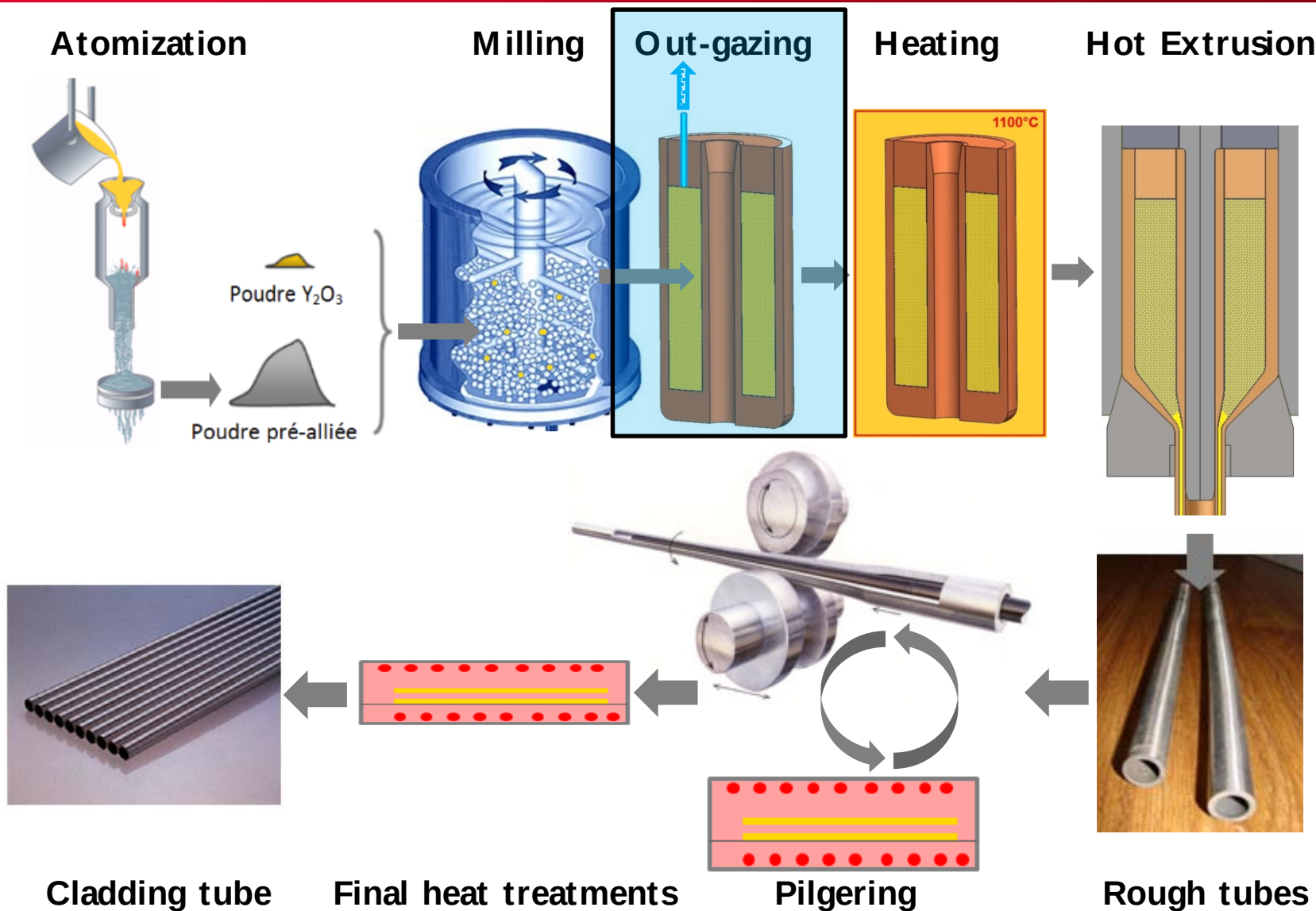


Figure I-5 : Binary diagram Iron-Chrome.

ODS STEELS MANUFACTURING BASED ON POWDER METALLURGY



Goal of the study

Does outgazing conditions impacts or not the chemical composition ?

Does outgazing conditions impacts the mechanical behavior ?

Is there any metallurgical phenomenon occurring during this stage ?

Does micro-structure is dependent on the outgazing conditions ?

Does improvement of ODS properties could be achieved by an optimization of outgazing cycles ?

EXPERIMENTAL SET-UP

Chemical composition

Stainless Steel Nominal in wt%: Fe-14Cr-1W-0,3Ti-0,25Y₂O₃

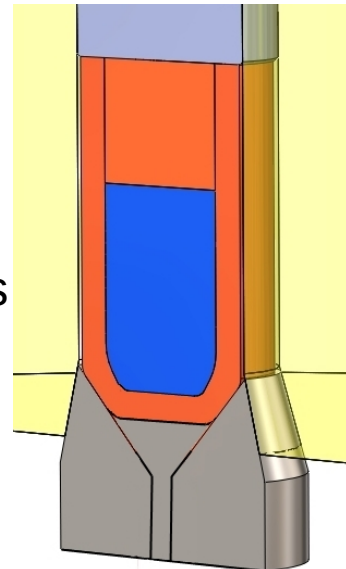
Measured in wt%: $\approx$ Fe-13Cr-0,6W-0,26Ti-0,3Y

The same powder for all samples

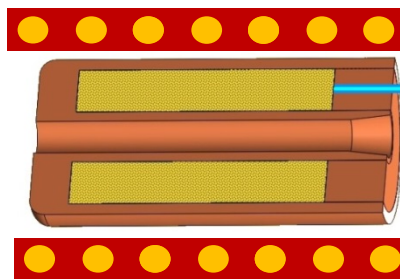
Processing

All forming parameter are equivalent expect the outgazing conditions

Hot Uni-axial Pressing (9000 bars) at 1100°C after 1 h dwell time in the furnace



Outgazing Conditions



Void pumping ($\approx 10^{-4}$ mBar)

References	Conditions	
	Temperature (°C)	Time
L59	130°C	24h
L60	400°C	2h
L61	Optimized	Optimized
L62	1100°C	1h

Minor “light” elements

References	O ₂ (wt%)	N ₂ (wt%)	C (wt%)
130°C/24h	0,11	0,017	0,035
400°C/2h	0,12	0,021	0,04
Optimized (L61)	0,10	0,017	0,03
1100°C/1h	0,11	0,017	0,044

Measured by FRCT (nitrogen), FRIR (oxygen) and GD-OES (carbon)

→ Poor impact on the minor “light” elements concentration

X-Ray Tomography

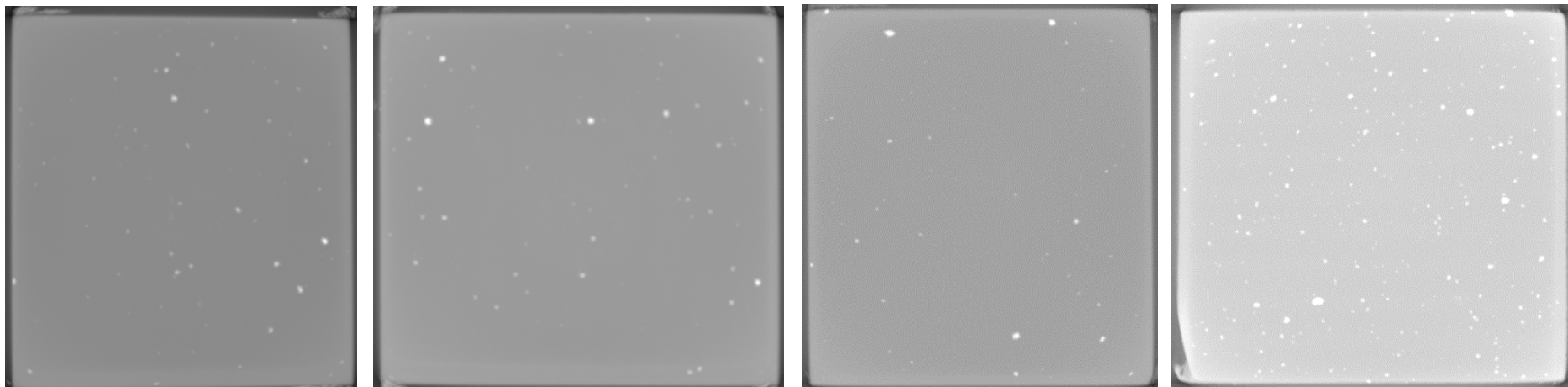


130°C/24h

400°C/2h

L61

1100°C/1h

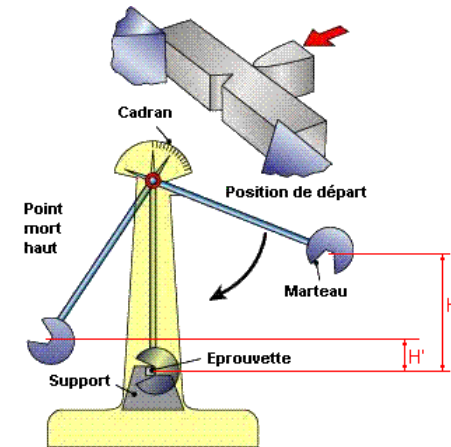
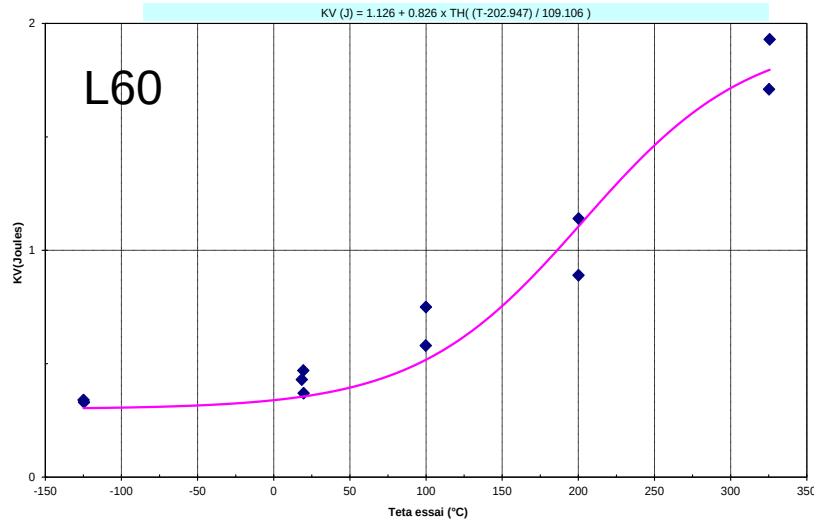


Sommation of the slices 1000*1000 μm

- All material are fully consolidated and dense
- The second phase density depends on the outgazing conditions
- The second phase composition is not determined yet due to it's very low volume fraction

Charpy tightness testing

Conditions	Upper Shelf		DBTT (°C)		Part. density from XR Tomo.
	Energy KV (J)	Lat. Exp. (mm)	Energy	Exp. Lat. (mm)	
130°C/24h	2,4	0,22	166	215	0.0021 %
400°C/2h	2	0,18	183	226	0.0034 %
Optimized	3,3	0,4	170	214	0.0022 %
1100°C/1h	2,7	0,32	207	239	0.0100 %



Conditions	Hardness
	HV5
130°C/24h	379
400°C/2h	372
Optimized	362
1100°C/1h	355

- All material presents a low upper shelf energy. (linked to the low deformation state)
- Outgazing conditions impact more the tightness properties than the hardness

MICROSTRUCTURE AND PRECIPITATION

130°C/24h

400°C/2h

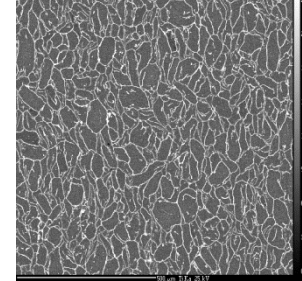
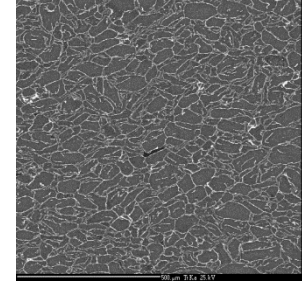
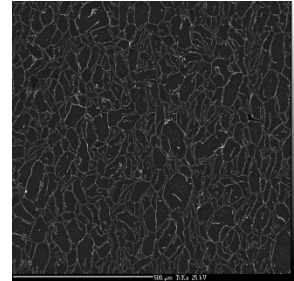
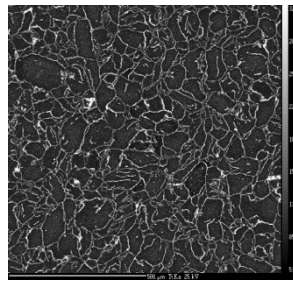
Optimized

1100°C/1h

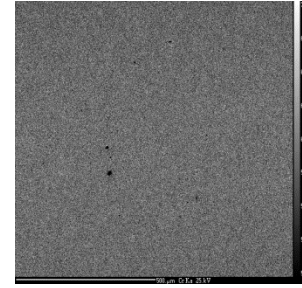
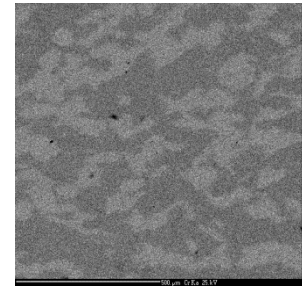
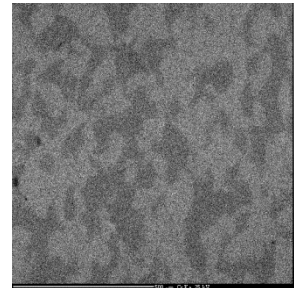
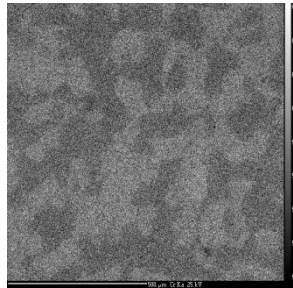
EPMA characterization

1000*1000µm window

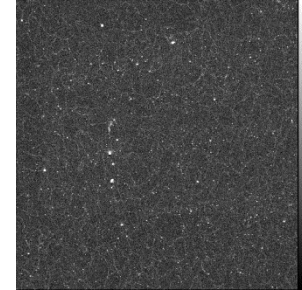
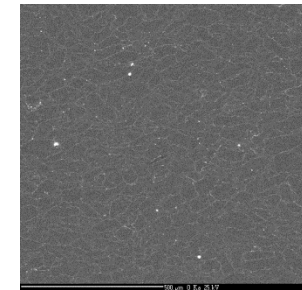
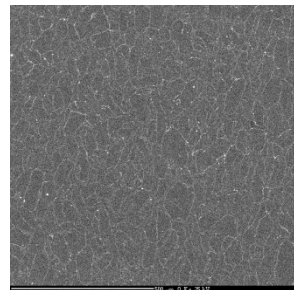
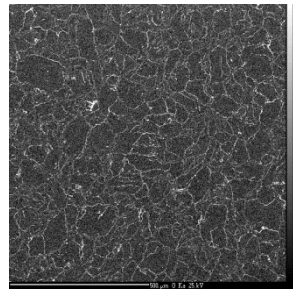
Ti



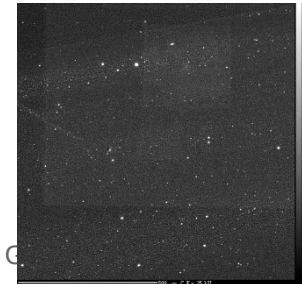
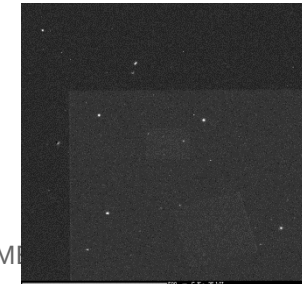
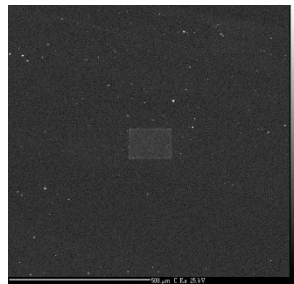
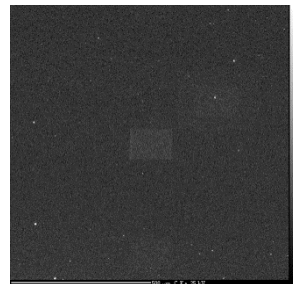
Cr



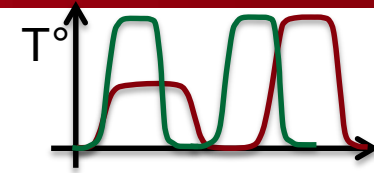
O



C



EPMA characterization



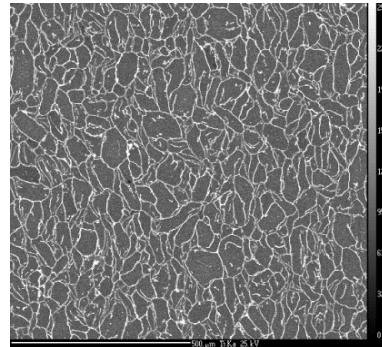
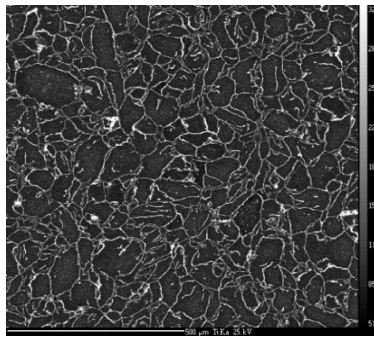
130°C/24h

1100°C/1h

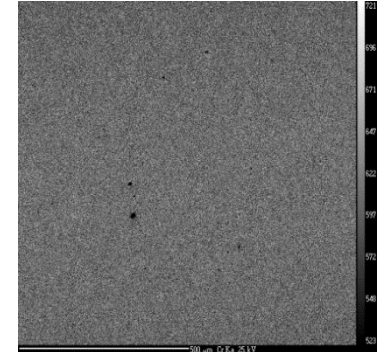
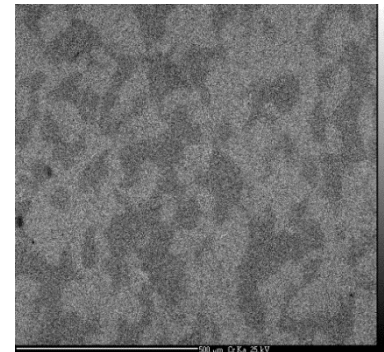
400°C/2h

1100°C/1h

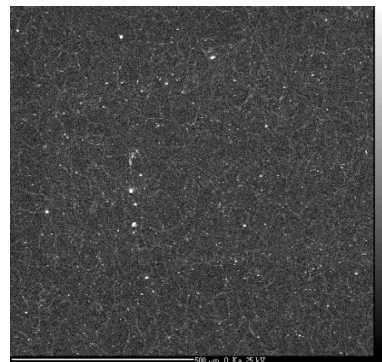
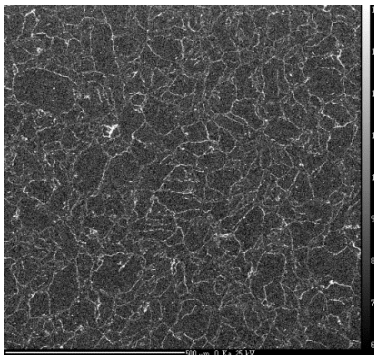
Ti



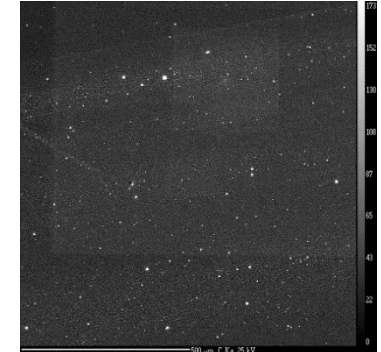
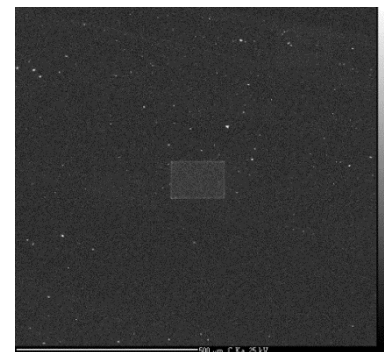
Cr



O



C



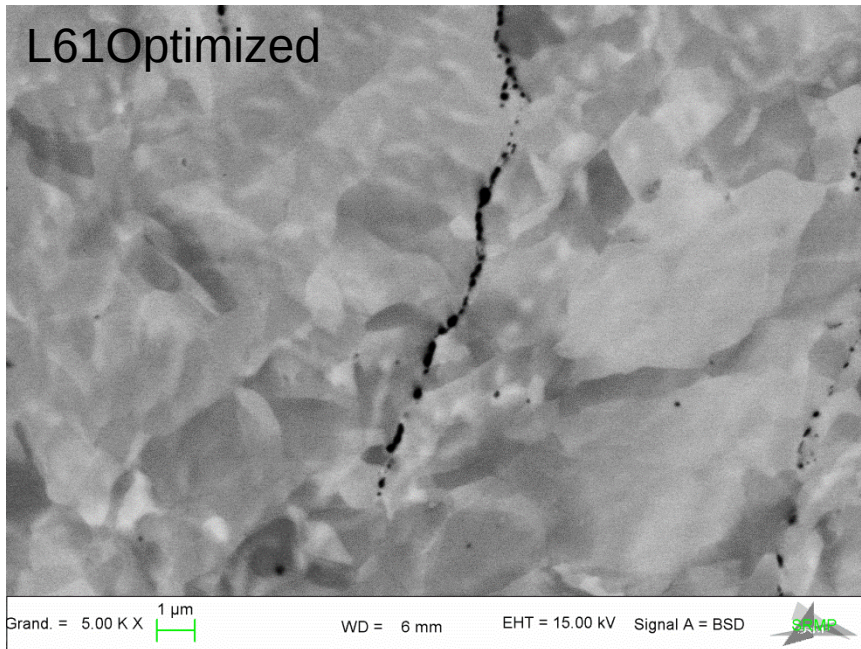
→ Titanium segregation on powder Prior Particles Boundary (PPB) Coupled to Oxygen.

→ Cr segregation for lower outgazing temperature. Aggregates bigger than prior particles ??? + no correlation with Carbon.

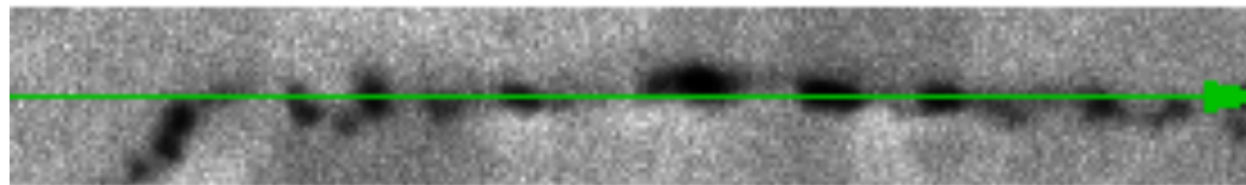
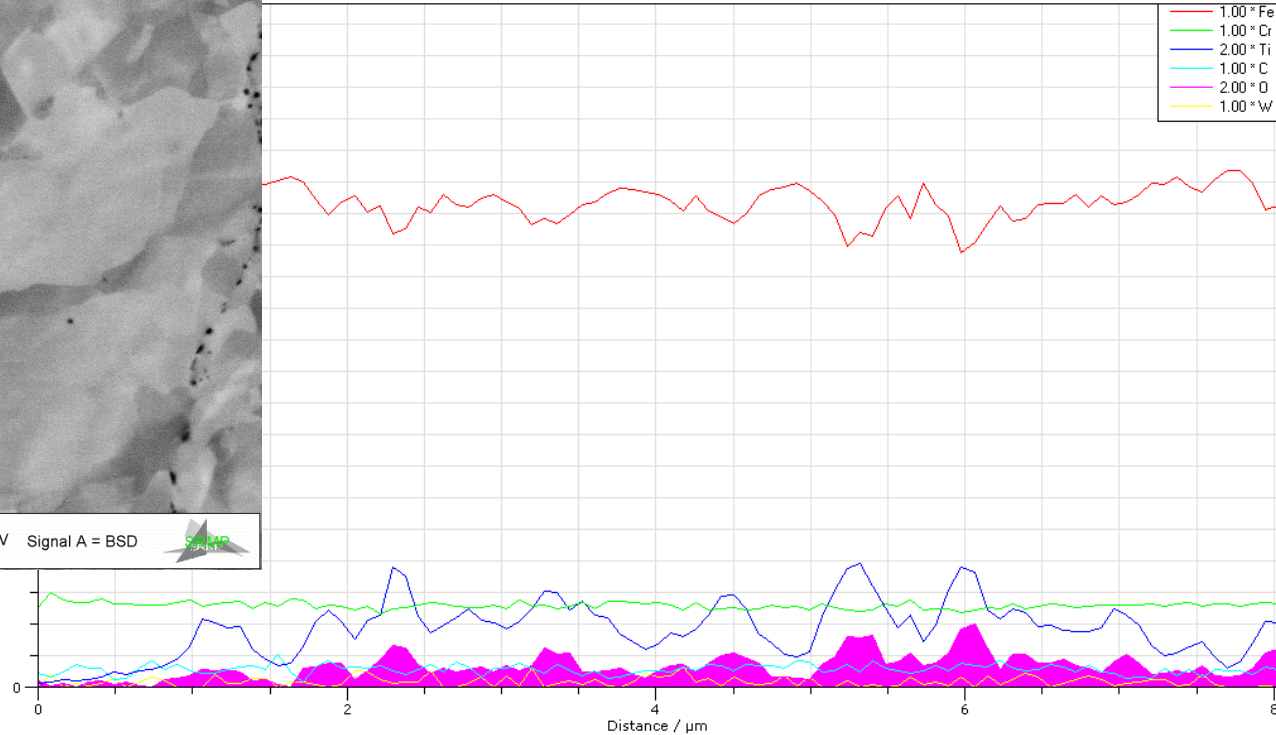
PRECIPITATION

Microstructure by SEM + EDX

L61Optimized

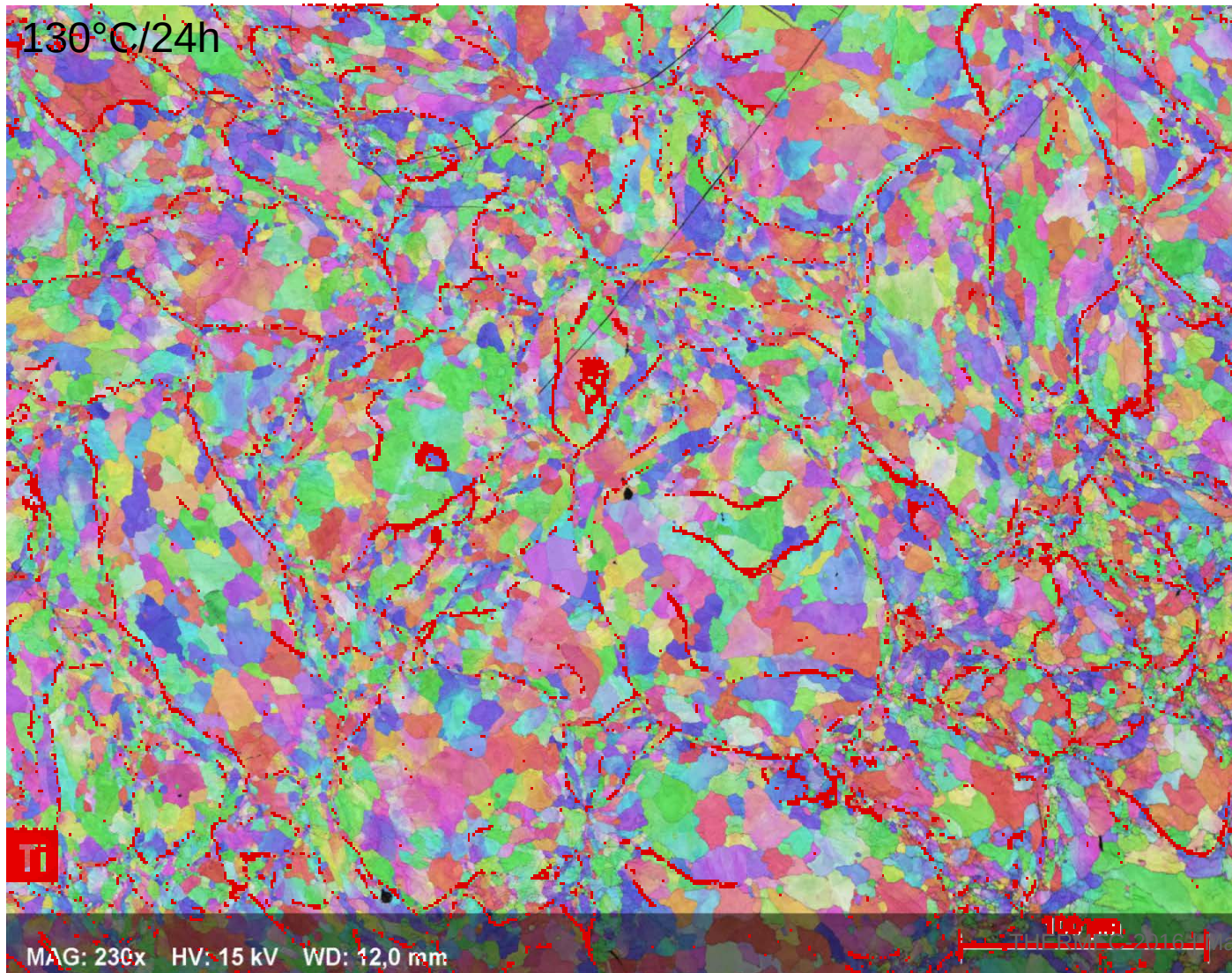


→ Prior Particles Boundary
precipitation is probably
 TiO_2 particles of about 200
nm

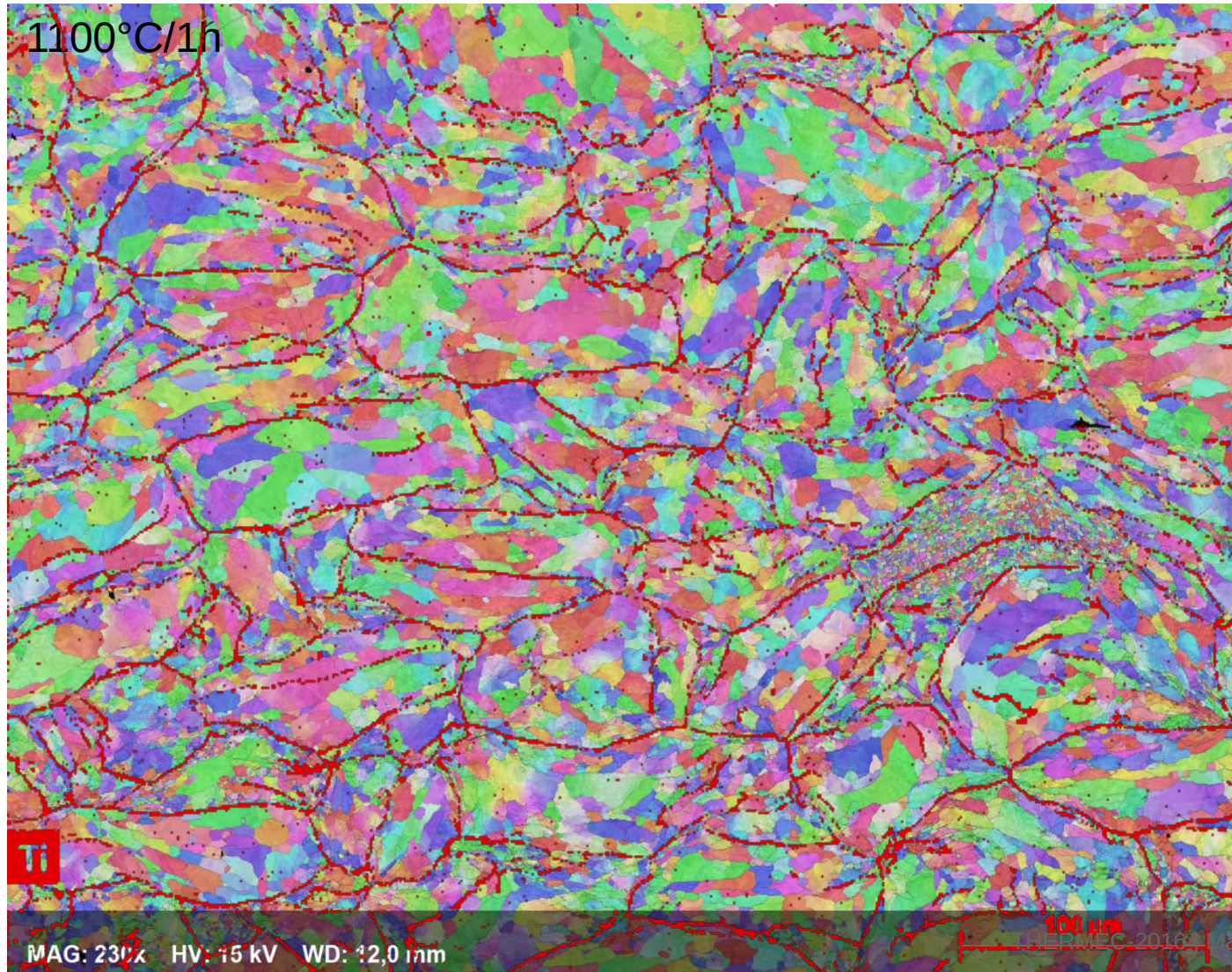


MICROSTRUCTURE AND PRECIPITATION

Microstructure by SEM EBSD + EDX Ti



Microstructure by SEM EBSD + EDX Ti



MICROSTRUCTURE AND PRECIPITATION

Microstructure by SEM EBSD +EDX Ti

- Titanium segregation (TiO_2) on powder Prior Particles Boundary (PPB) is clearly evidenced. It seems that precipitates pins GB.
- Microstructure is poorly dependent on the outgazing conditions.

CONCLUSIONS

- ➡ Outgazing has a poor impact on “light” minor element concentration in the consolidated material
- ➡ Outgazing conditions can impact mechanical properties of tightness (USE and DBTT)
- ➡ Those properties appear linked to the density of large second phases observed by X-Ray tomography
- ➡ A strong segregation of Ti and O₂ riche precipitates is evidence on PPB.
- ➡ A Cr segregation is observed on domains larger than the previous particles for outgazing conditions under low temperatures.
- ➡ Outgazing conditions as a poor impact on grain size and shape

PROSPECTS

- ➡ Identify composition of second phases observed by X-Ray tomography
- ➡ Check tightness after HE ($\neq$ from HUP) of the various samples
- ➡ Identify precisely composition and size of Ti+O rich particles on PPB on various samples.
- ➡ Evidence the phenomenon of quick Ti migration on PPB
- ➡ Evidence the reason of Cr segregation observed in case of low ($<1000^{\circ}\text{C}$) temperature outgazing conditions
- ➡ Characterization of outgazed powder particles before consolidation
- ➡ Perform chemical analysis of the outgas species

Questions ?

Commissariat à l'énergie atomique et aux énergies alternatives
Centre de Saclay | 91191 Gif-sur-Yvette Cedex
T. +33 (0)1 69 08 30 07 | F. +33 (0)1 69 08 71 67

DEN/DANS
DMN
SRMA

Etablissement public à caractère industriel et commercial | RCS Paris B 775 685 019