



## Effect of powder outgazing conditions

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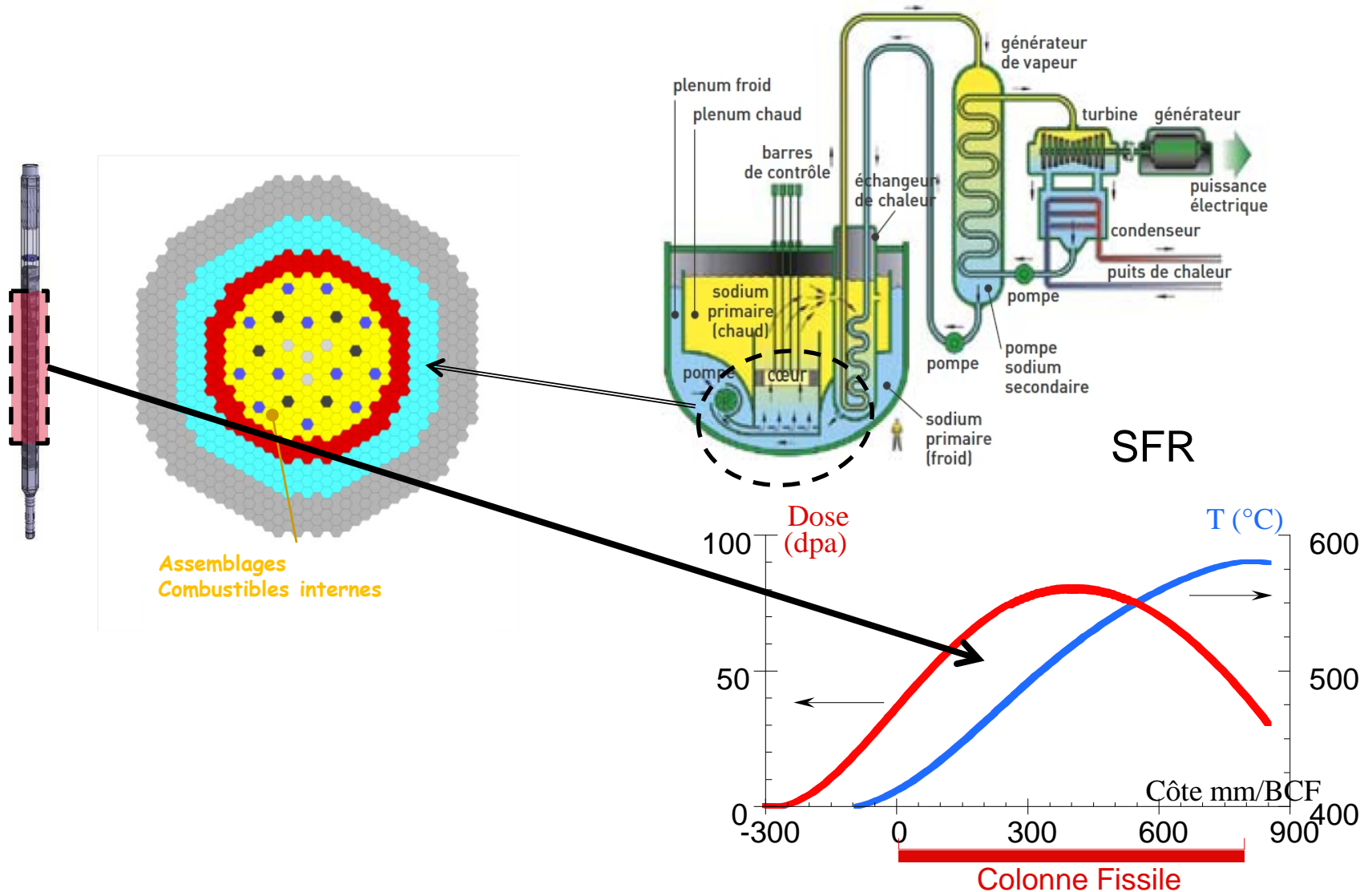
# EFFECT OF POWDER OUTGAZING CONDITIONS ON MECHANICAL AND MICROSTRUCTURAL PROPERTIES OF **OXIDES DISPERSED** **STRENGTHENED STEEL**

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

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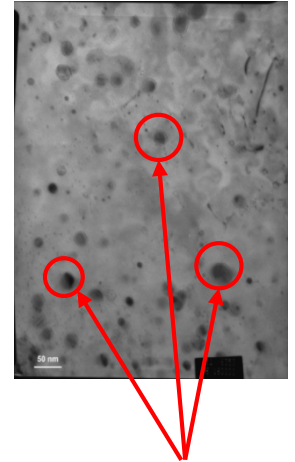
JUNE 2, 2016    SESSION: G7  
MATERIALS UNDER EXTREME CONDITIONS 3

# 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)

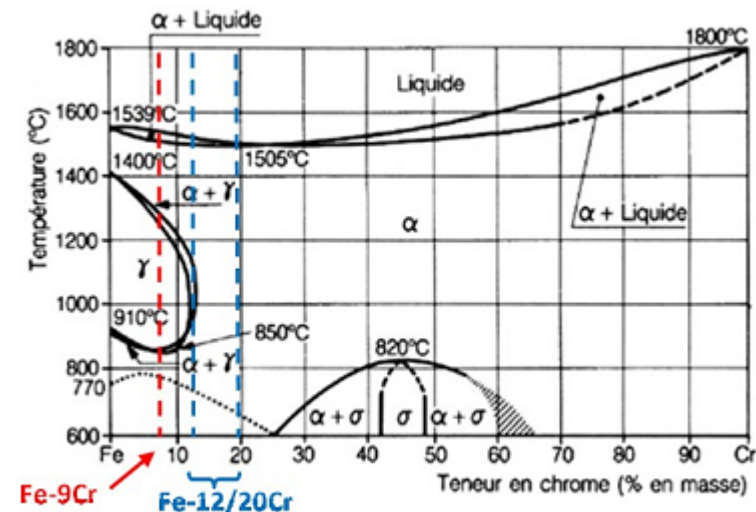
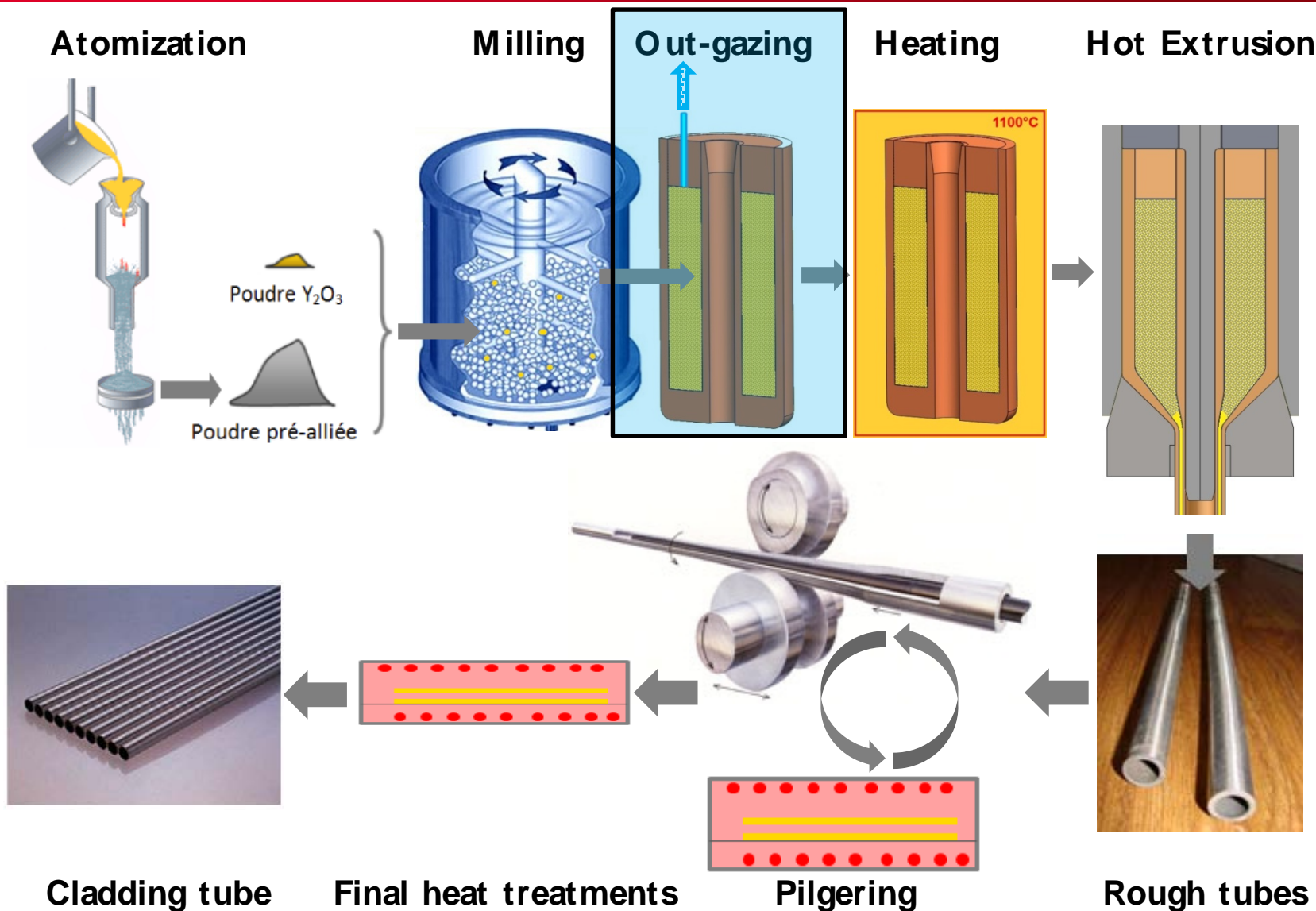


Figure I-5 : Binary diagram Iron-Chrome.

# 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-0,25Y<sub>2</sub>O<sub>3</sub>

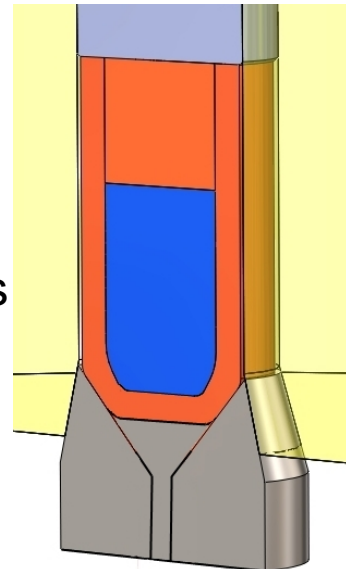
Measured in wt%:  $\approx$  Fe-13Cr-0,6W-0,26Ti-0,3Y

The same powder for all samples

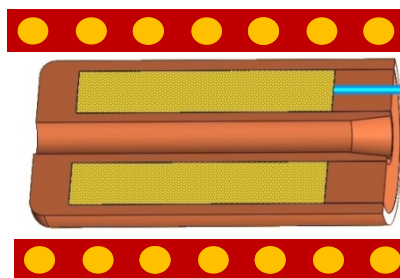
### Processing

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 <sub>2</sub> (wt%)	N <sub>2</sub> (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

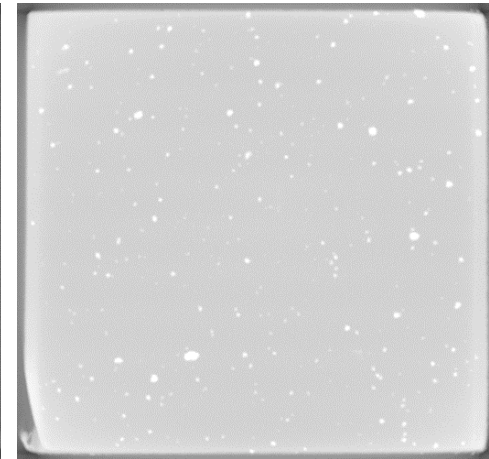


130°C/24h

400°C/2h

L61

1100°C/1h

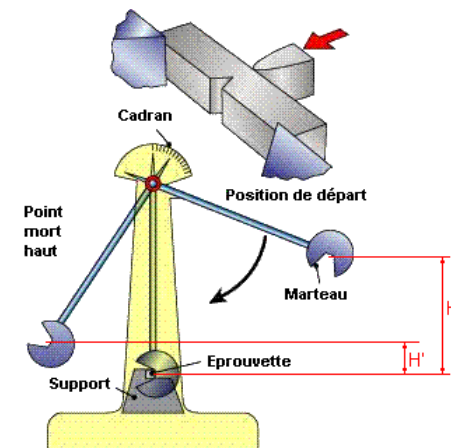
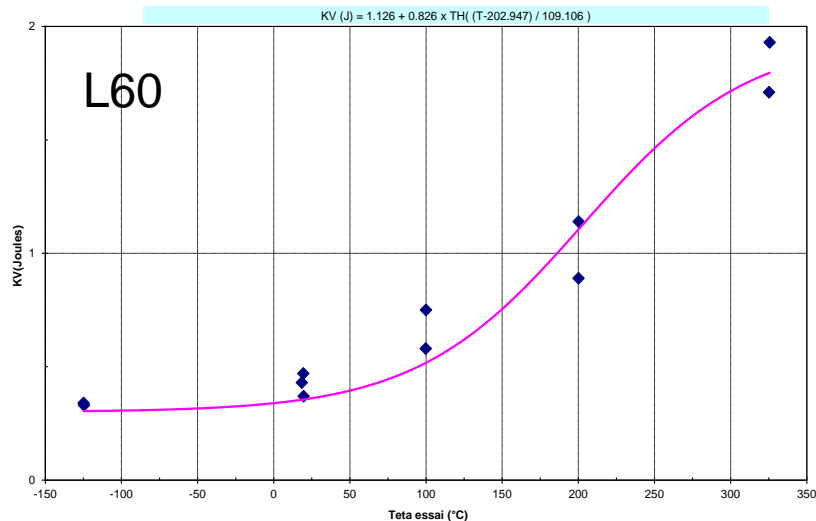


Sommation of the slices 1000\*1000  $\mu\text{m}$

- 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

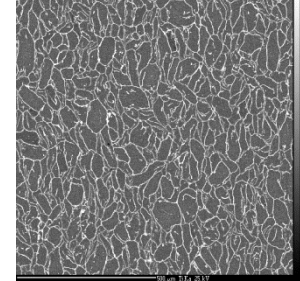
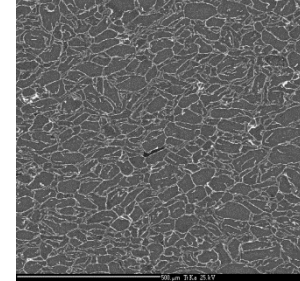
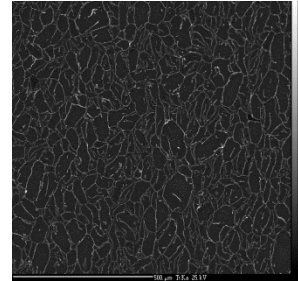
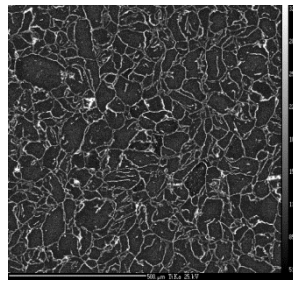
Optimized

1100°C/1h

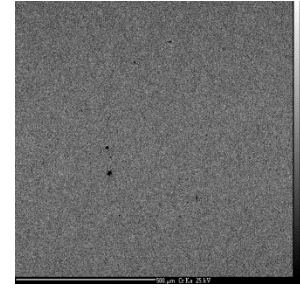
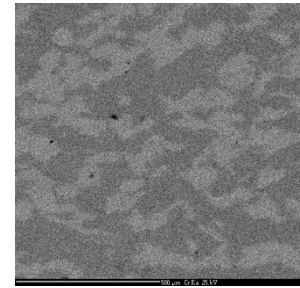
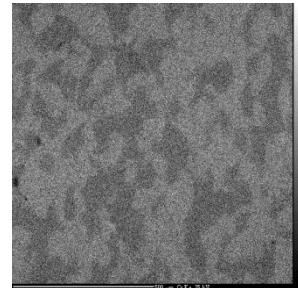
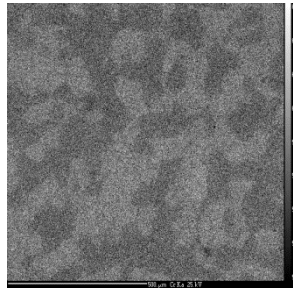
## EPMA characterization

1000\*1000µm window

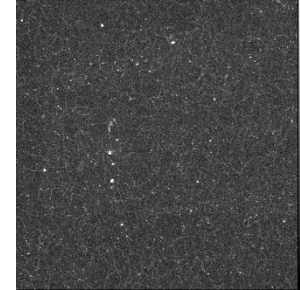
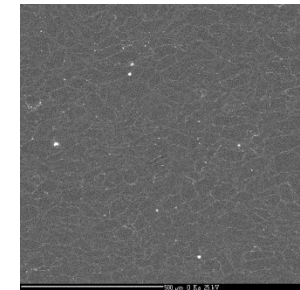
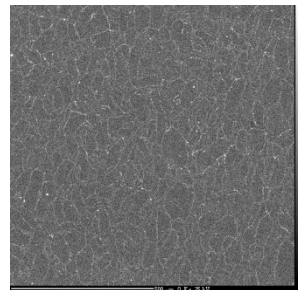
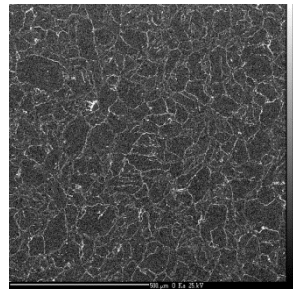
Ti



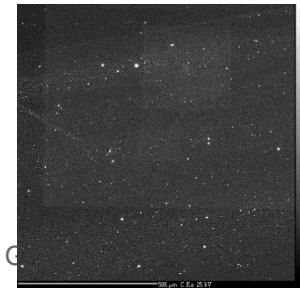
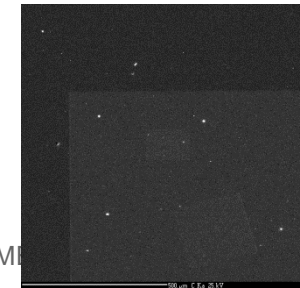
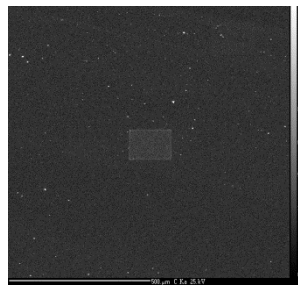
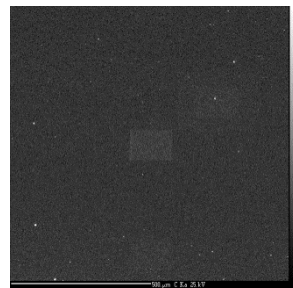
Cr



O



C



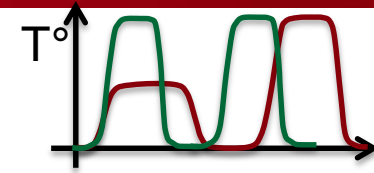
RM

S. C



# MICROSTRUCTURE AND PRECIPITATION

## EPMA characterization



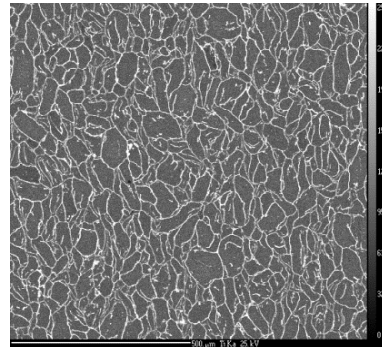
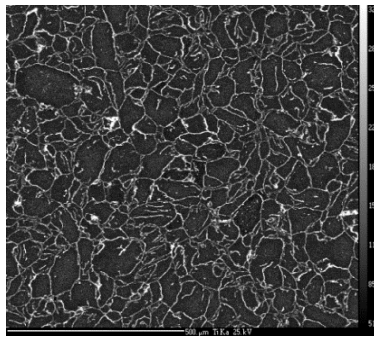
130°C/24h

1100°C/1h

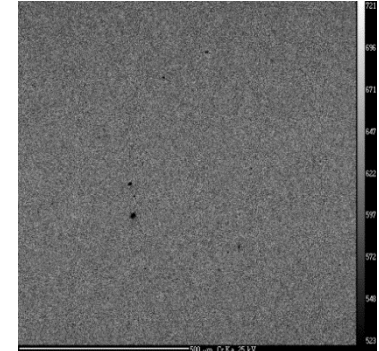
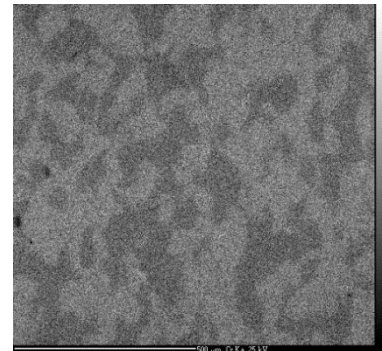
400°C/2h

1100°C/1h

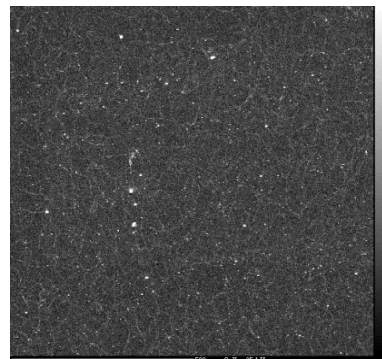
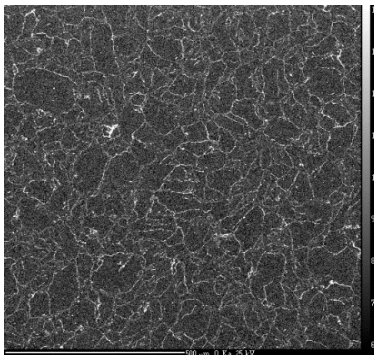
Ti



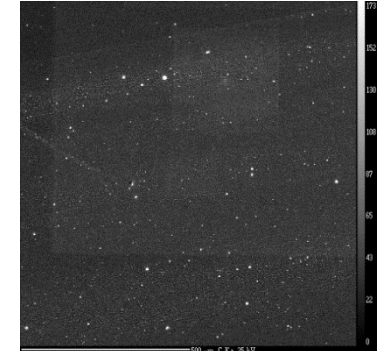
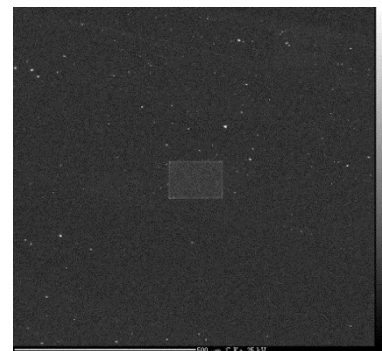
Cr



O



C

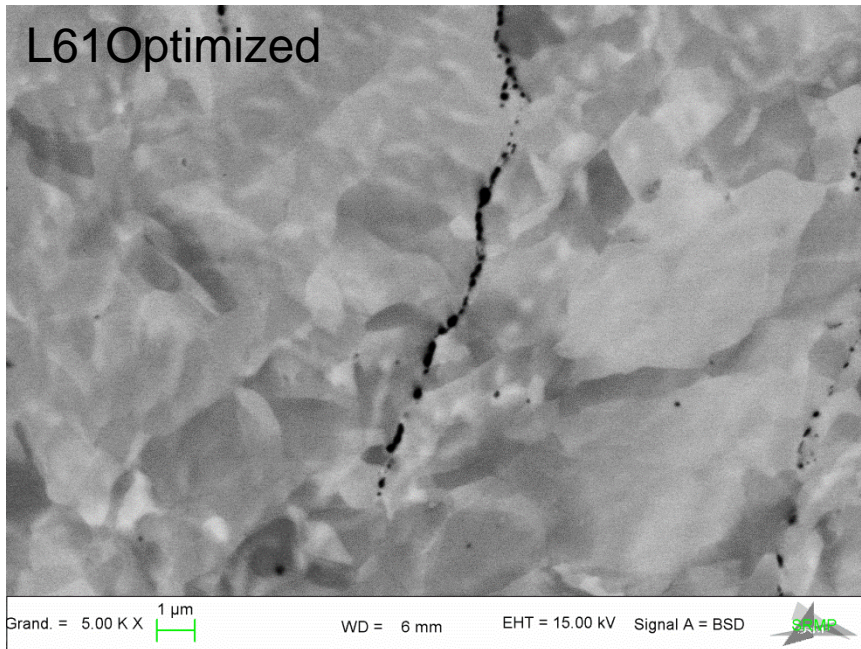


→ Titanium segregation on powder Prior Particles Boundary (PPB) Coupled to Oxygen.

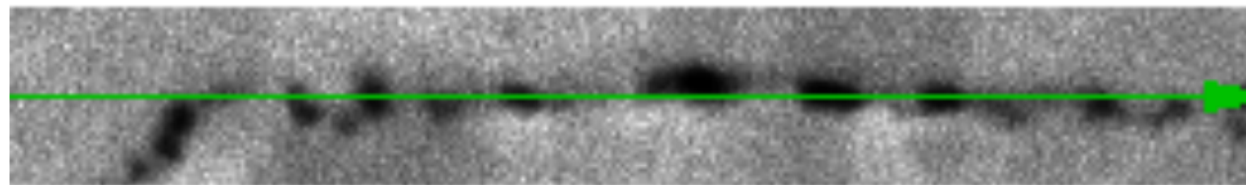
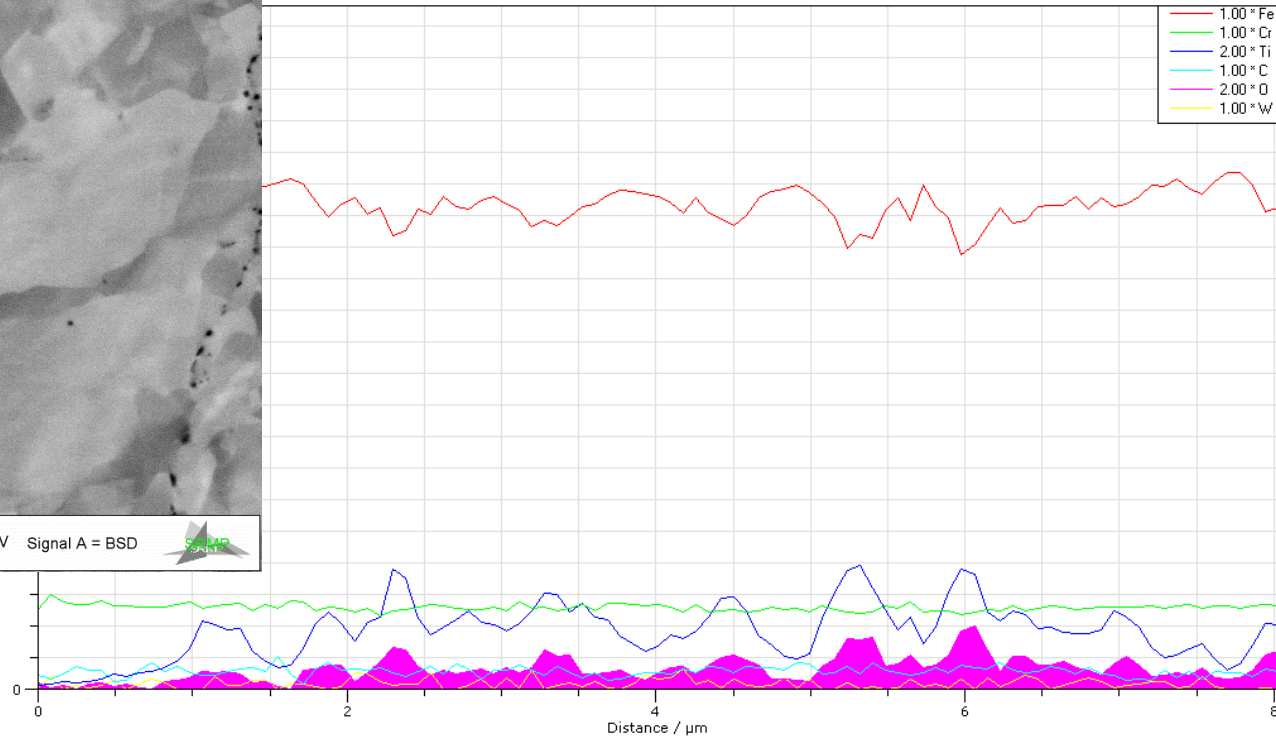
→ Cr segregation for lower outgazing temperature. Aggregates bigger than prior particles ??? + no correlation with Carbon.

## Microstructure by SEM + EDX

L61Optimized

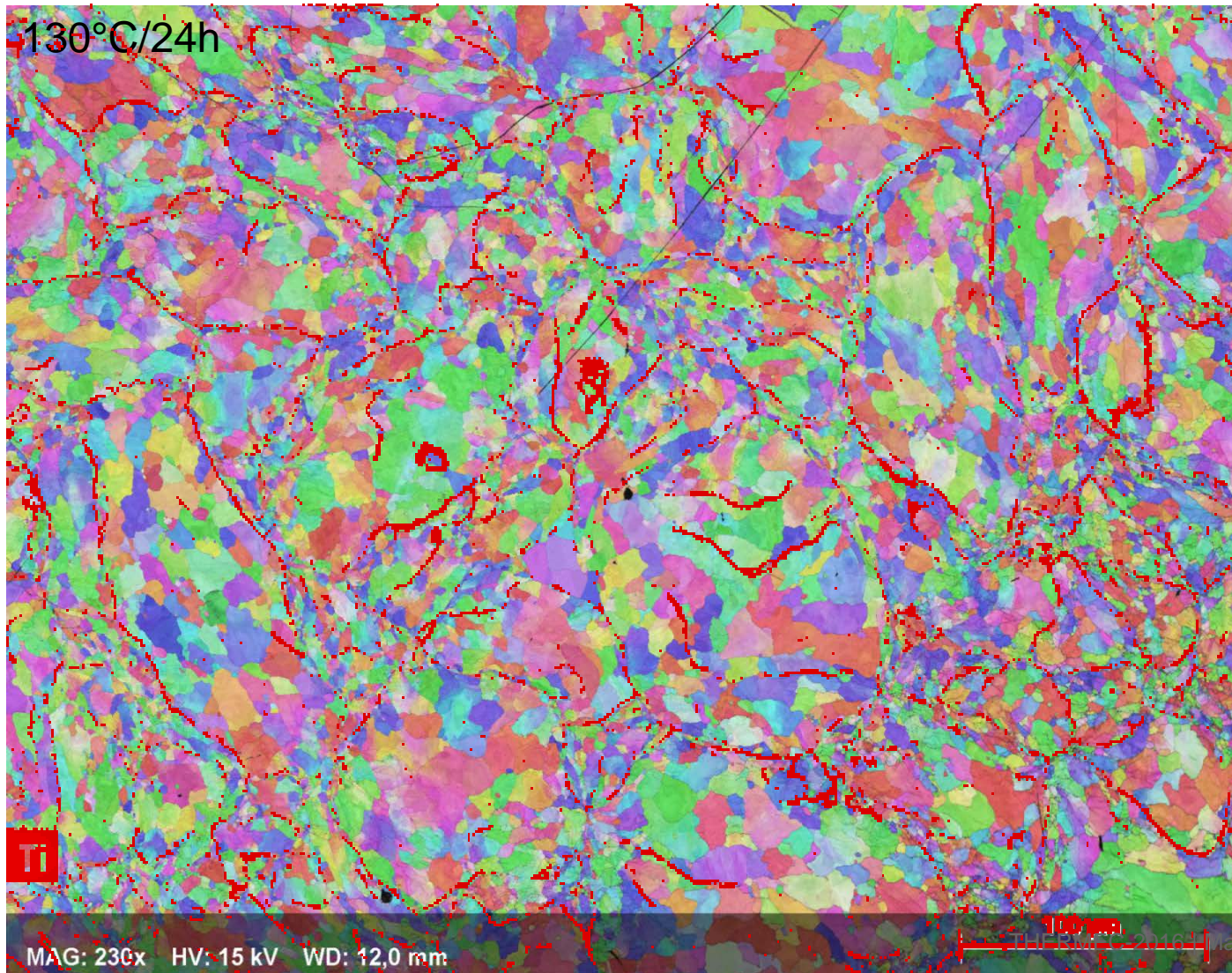


→ Prior Particles Boundary  
precipitation is probably  
 $\text{TiO}_2$  particles of about 200  
nm



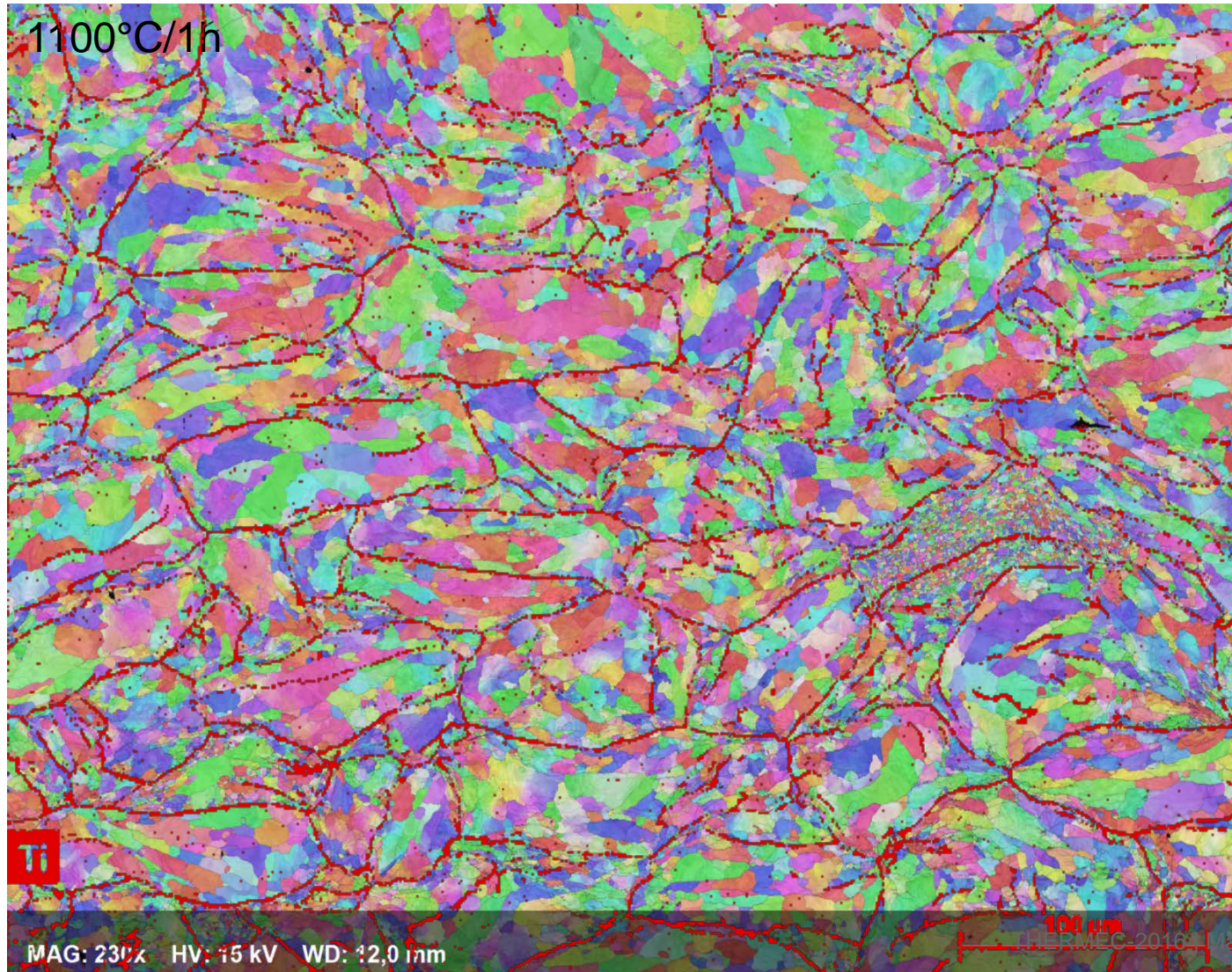


## Microstructure by SEM EBSD + EDX Ti





## Microstructure by SEM EBSD + EDX Ti





## Microstructure by SEM EBSD +EDX Ti

- Titanium segregation ( $\text{TiO}_2$ ) 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 O<sub>2</sub> 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 ?

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