



Wir schaffen Wissen – heute für morgen



Laboratory for  
Nuclear Materials

Nuclear Energy  
and Safety

## **MEGAPIE Project Overview PART III** *(Focus on MEGAPIE PIE)*

Reported by Yong Dai (PSI)

# Acknowledgement

## MEGAPIE PIE Team

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**SCK-CEN:** M. J. Konstantinović, E. Stergar, M. Lambrecht, S.  
Gavrilov

**CNRS, EANA, KAERI**

## Ø Non-destructive test (NDT)

- Gamma mapping of the Al-calotte
- Visual inspection of the T91 calotte
- Thickness measurement of the T91 calotte

## Ø Analysis of LBE samples

## Ø Surface analyses

- EPMA analysis
- SEM observation
- SIMS analysis

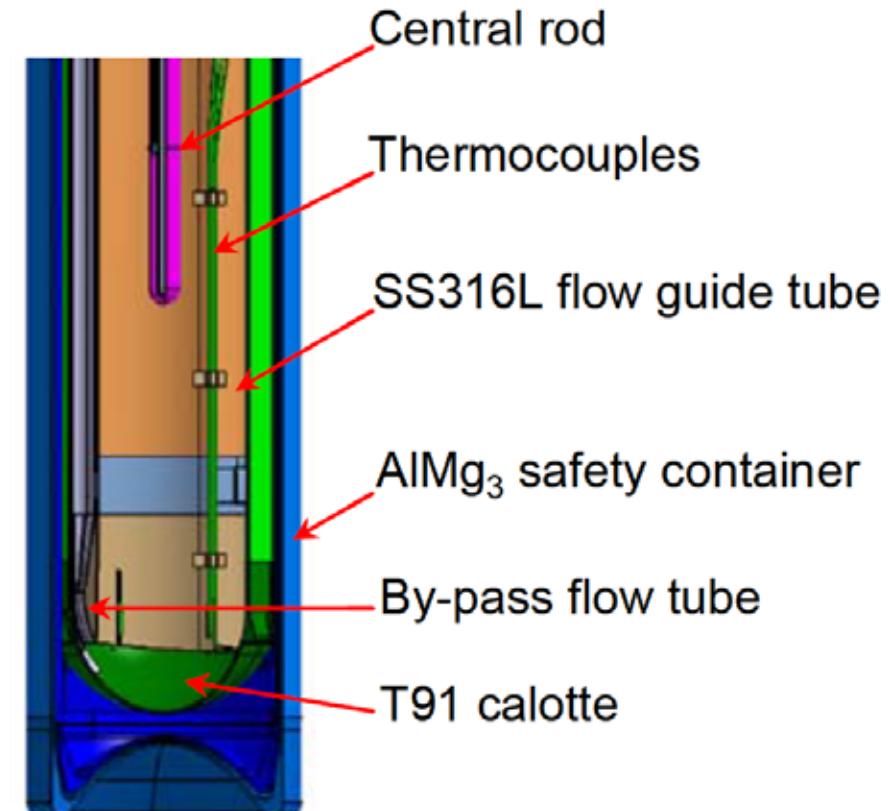
## Ø Microstructural analyses

- TEM observation

## Ø Mechanical test

- Tensile test
- Bend test
- Hardness measurement

## Ø Summary



- ∅ **Non-destructive test (NDT)**
  - Gamma mapping of the Al-calotte
  - Visual inspection of the T91 calotte
  - Thickness measurement of the T91 calotte
- ∅ **Analysis of LBE samples => D. Schumann**
- ∅ **Surface analyses**
  - EPMA analysis
  - SEM observation
  - SIMS analysis
- ∅ **Microstructural analyses**
  - TEM observation
- ∅ **Mechanical test**
  - Tensile test
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  - Hardness measurement
- ∅ **Summary**

## Ø **Non-destructive test (NDT)**

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## Ø **Surface analyses**

- EPMA analysis
- SEM observation
- SIMS analysis

## Ø **Microstructural analyses**

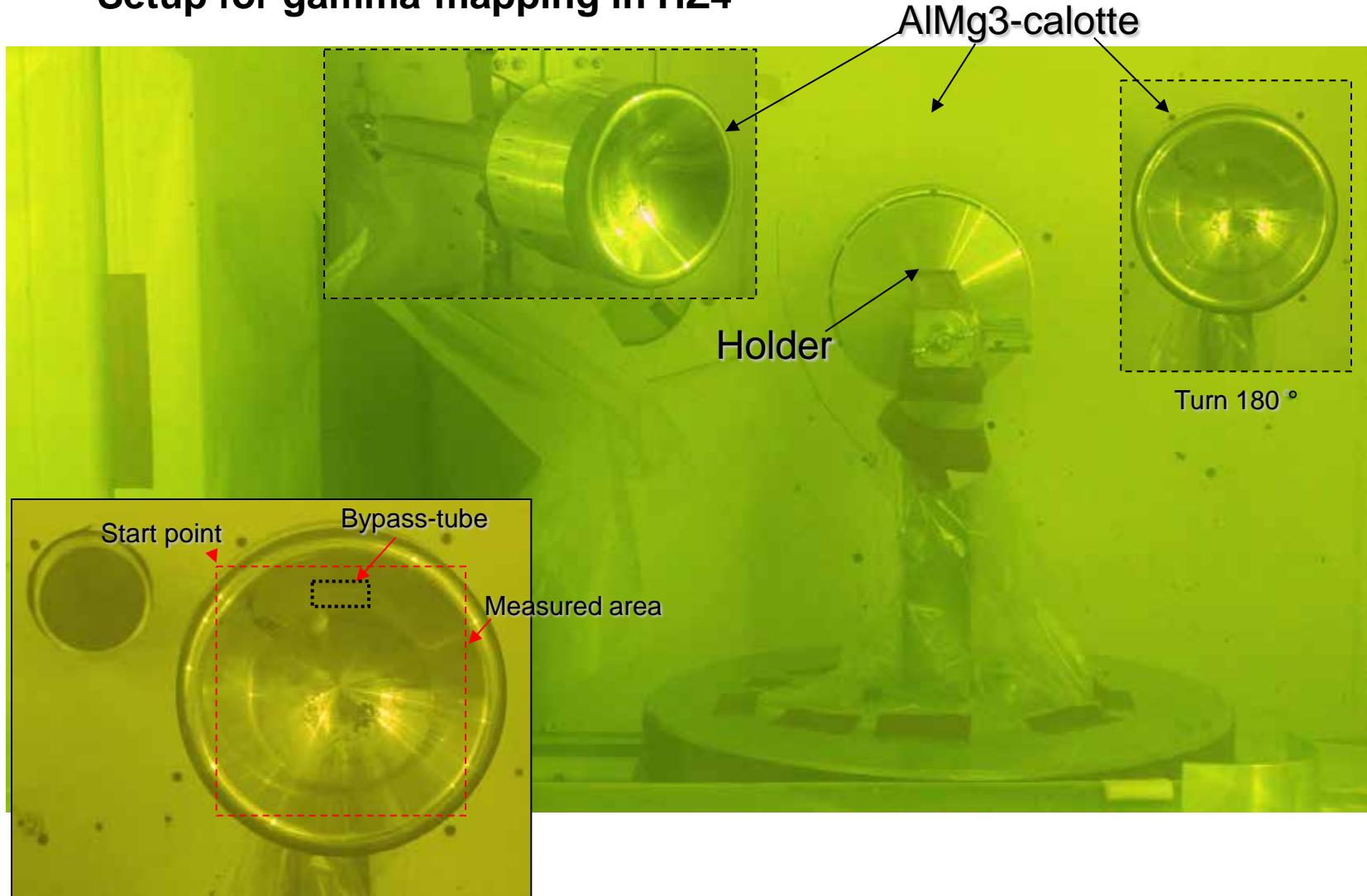
- TEM observation

## Ø **Mechanical test**

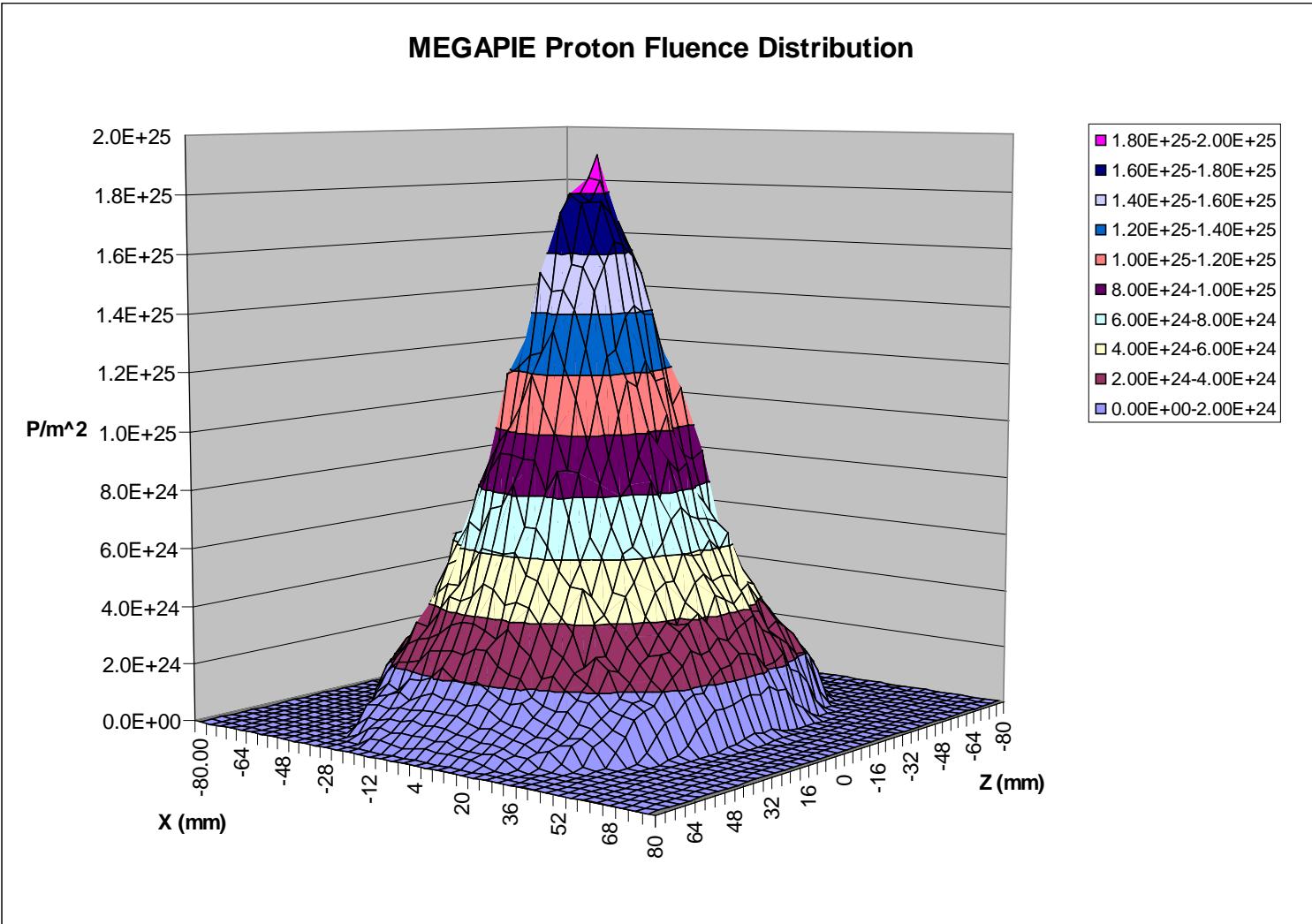
- Tensile test
- Bend test
- Hardness measurement

## Ø **Summary**

## Setup for gamma-mapping in HZ4



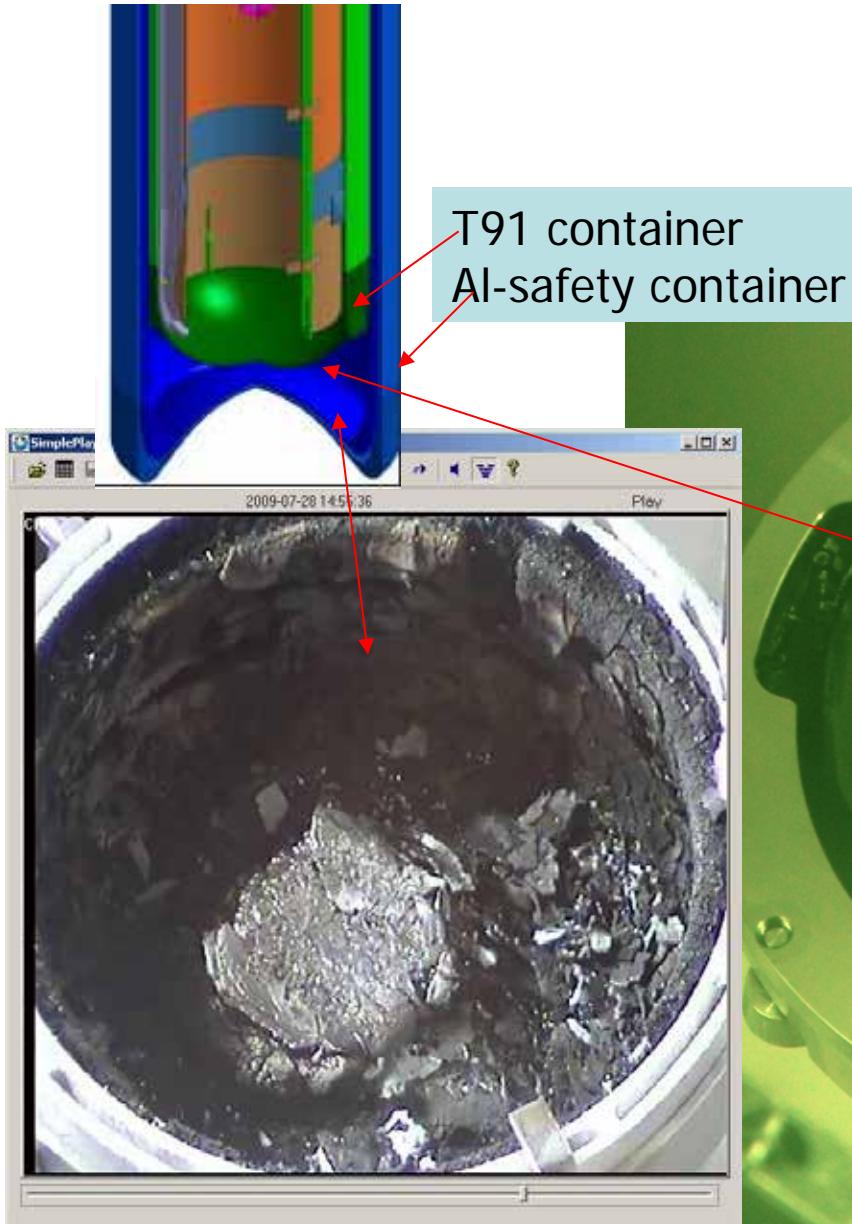
# NDT – Gamma mapping



**Max fluence:  $1.9 \times 10^{25} \text{ p/m}^2$**   
**=> 7.1 dpa**

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- ∅ **Summary**

# NDT – Visual inspection

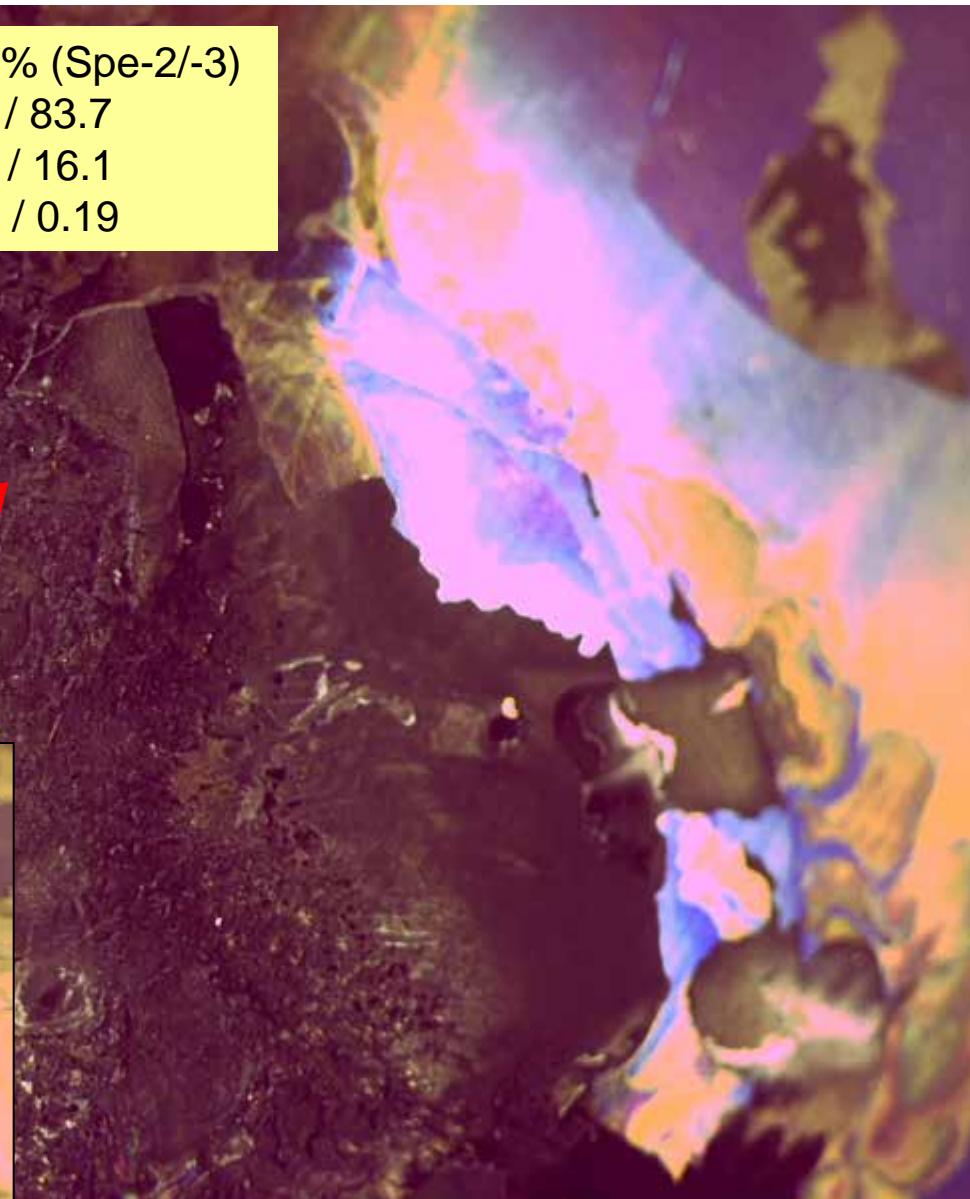


**T91-calotte after irradiation**

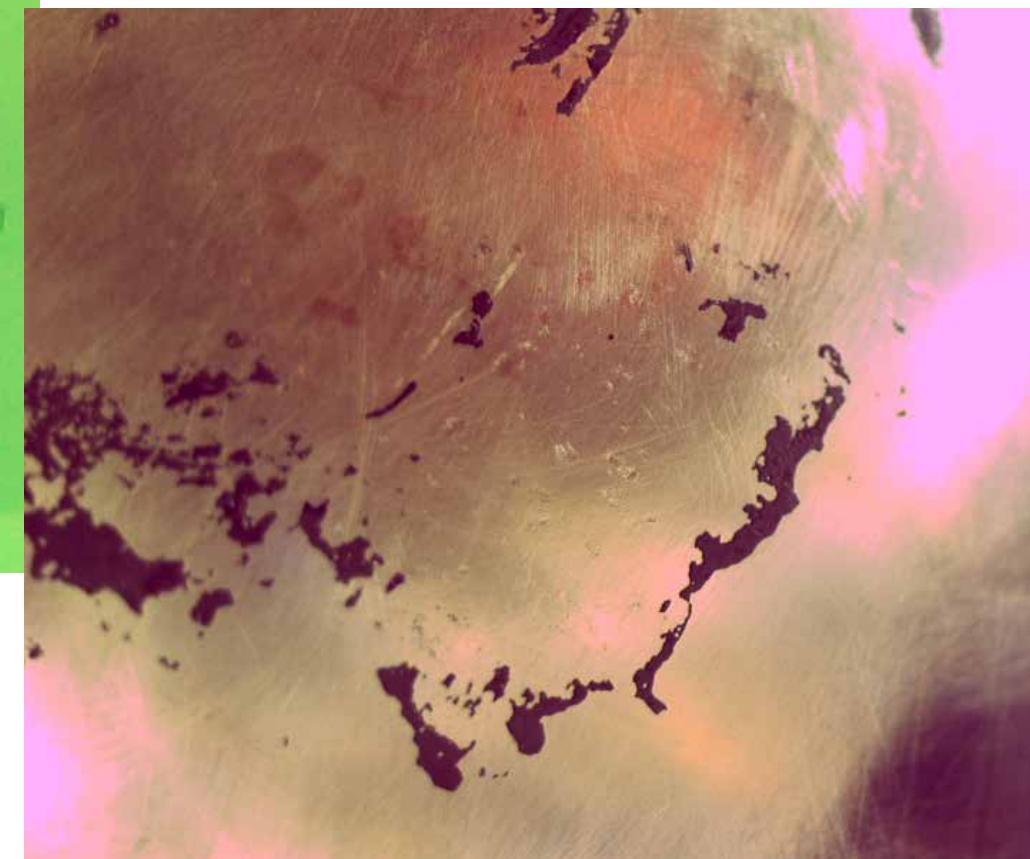
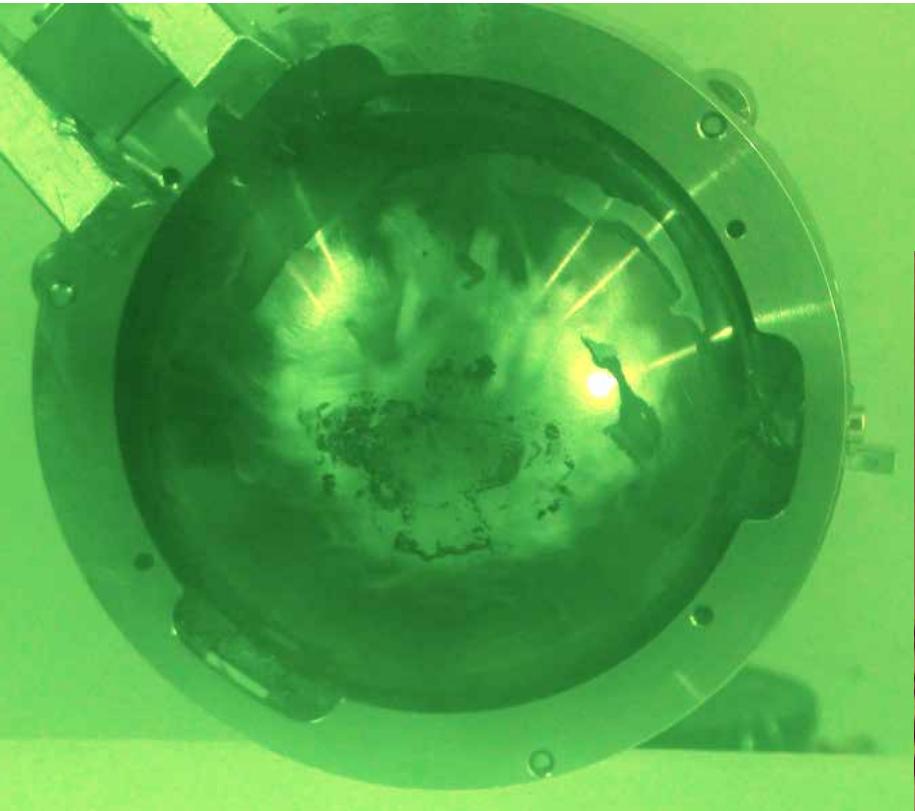
# NDT – Visual inspection

Element	Weight% (Spe-2/-3)	Atomic% (Spe-2/-3)
C	77.4 / 79.2	82.1 / 83.7
O	22.2 / 17.7	20.3 / 16.1
Si	0.37 / 0.17	0.42 / 0.19

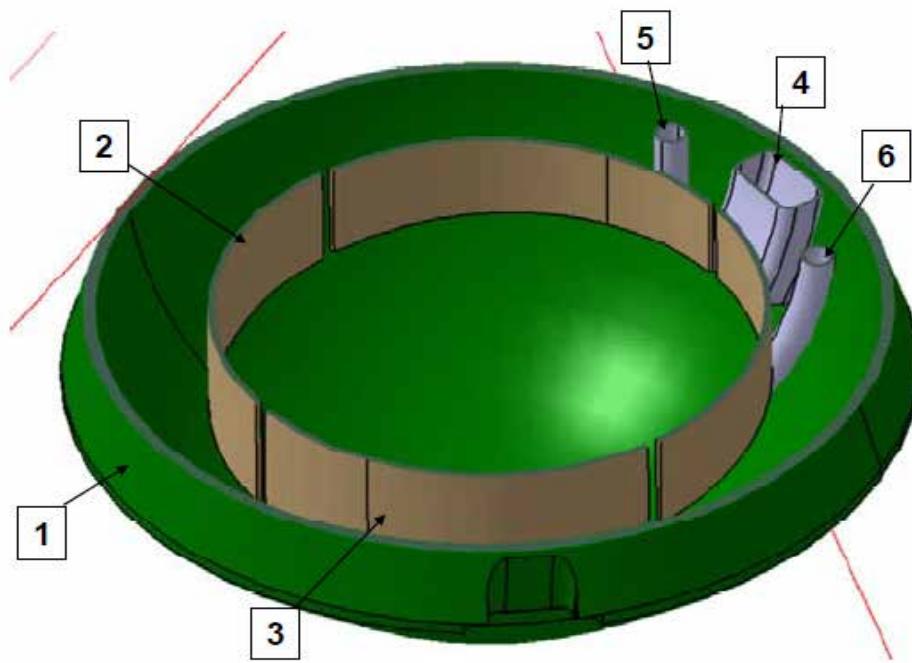
Element	Weight%	Atomic%
C	4.5	7.1
O	53.2	63.0
F	4.1	4.1
Si	38.3	25.9



**T91-calotte after removing deposited materials on surface**



# NDT – Visual inspection



## Ø **Non-destructive test (NDT)**

- Gamma mapping of the Al-calotte
- Visual inspection of the T91 calotte
- Thickness measurement of the T91 calotte

## Ø **Analysis of LBE samples**

## Ø **Surface analyses**

- EPMA analysis
- SEM observation
- SIMS analysis

## Ø **Microstructural analyses**

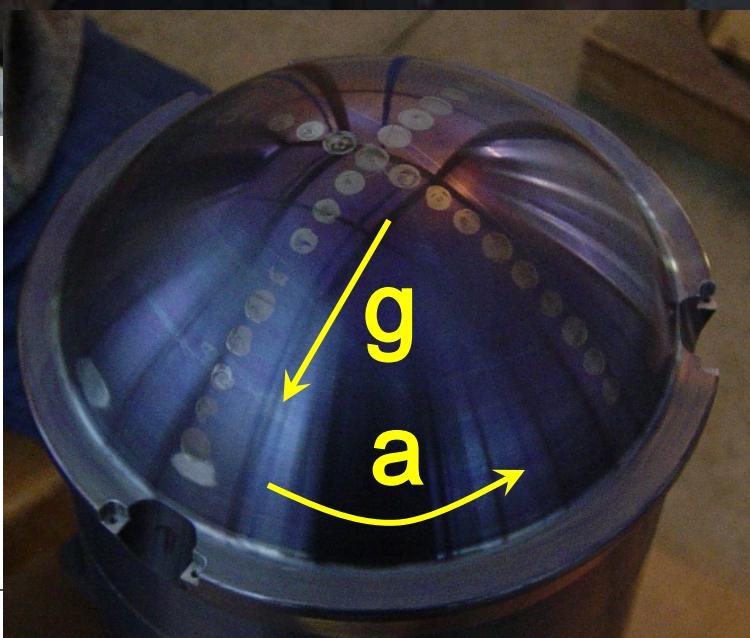
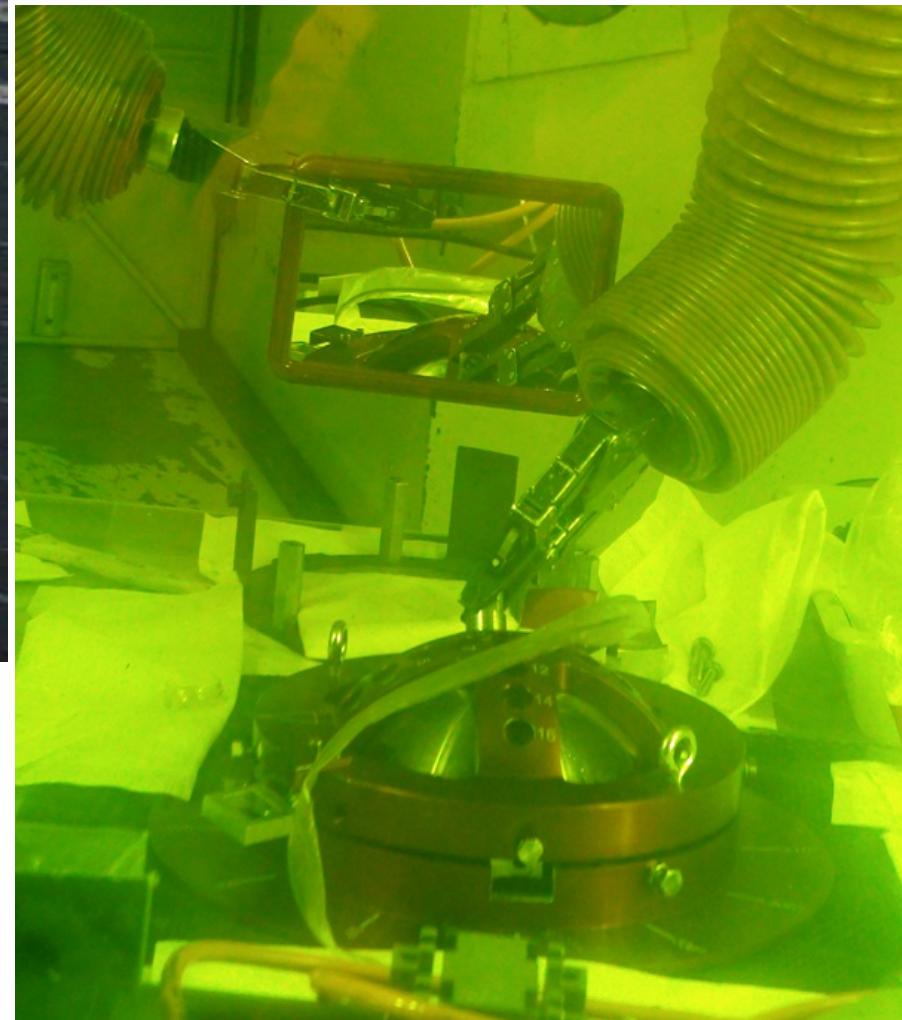
- TEM observation

## Ø **Mechanical test**

- Tensile test
- Bend test
- Hardness measurement

## Ø **Summary**

# NDT – Thickness measurement of the T91 calotte



# NDT – Thickness measurement of the T91 calotte

## Thickness before and after irradiation

Conf. Angle a	0°		30°		60°		90°		120°	
Vertical Angle g	Bef. Irr. Thickness (mm)	After Irr. Thickness (mm)	Bef. Irr. Thickness (mm)	After Irr. Thickness (mm)	Bef. Irr. Thickness (mm)	After Irr. Thickness (mm)	Bef. Irr. Thickness (mm)	After Irr. Thickness (mm)	Bef. Irr. Thickness (mm)	After Irr. Thickness (mm)
0	1.428	1.408	1.432	1.409						
6	1.452	1.438	1.452	1.436	1.451	1.431	1.451	1.438	1.452	1.435
12	1.501	1.479	1.500	1.482	1.501	1.475	1.501	1.475	1.505	1.463
18	1.530	1.508	1.526	1.507	1.526	1.497	1.526	1.498	1.525	1.501
24	1.545	1.519	1.541	1.527	1.539	1.529	1.539	1.511	1.537	1.519
30	1.572	1.551	1.578	1.561	1.575	1.553	1.575	1.547	1.571	1.548
36	1.598	1.575	1.607	1.589	1.603	1.589	1.603	1.571	1.602	1.581
42	1.641	1.621	1.656	1.635	1.650	1.627	1.650	1.618	1.643	1.621
48	1.680	1.661	1.697	1.662	1.682	1.662	1.682	1.652	1.681	1.665

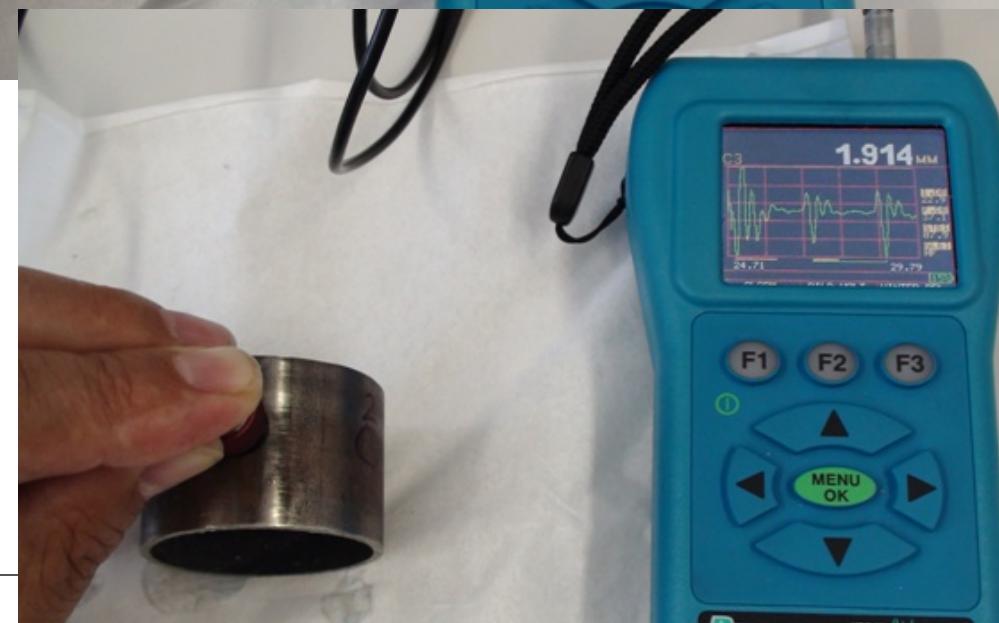
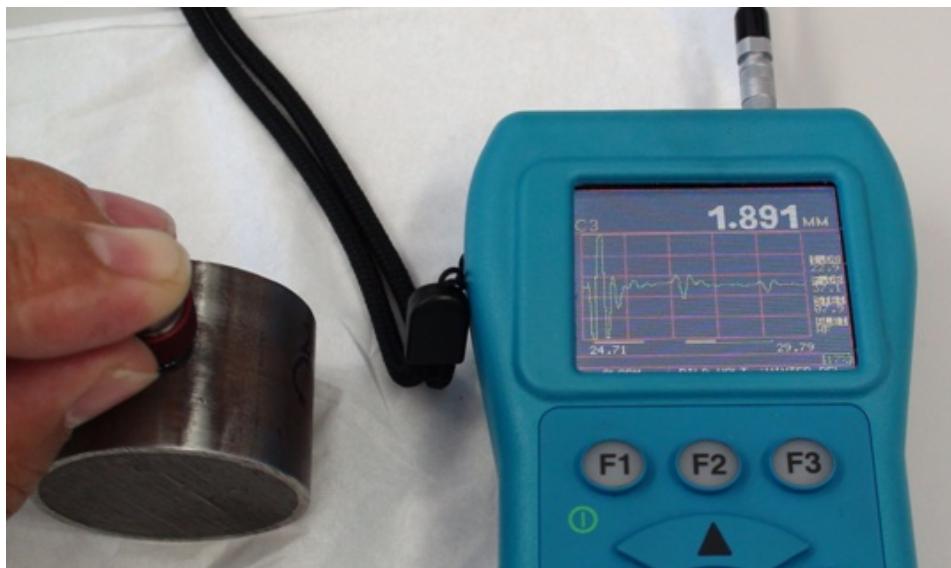
## Difference before and after irradiation

Vertical Angle g	Difference BI & AI	Average difference				
0	0.020	0.023				0.022
6	0.014	0.016	0.020	0.013	0.017	0.017
12	0.022	0.018	0.026	0.026	0.042	0.023
18	0.022	0.019	0.029	0.028	0.024	0.022
24	0.026	0.014	0.010	0.028	0.018	0.018
30	0.021	0.017	0.022	0.028	0.023	0.022
36	0.023	0.018	0.014	0.032	0.021	0.022
42	0.020	0.021	0.023	0.032	0.022	0.023
48	0.019	0.035	0.020	0.030	0.016	0.020
Av. Dt(mm)	0.021	0.020	0.021	0.027	0.023	0.021

The difference is almost constantly 20 mm, implying no evident dissolution corrosion effects considering very different LBE flow velocity and temperature etc at different positions.

Is the 20 mm a systematic error? from which source?

# NDT – Thickness measurement of the T91 calotte





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- Thickness measurement of the T91 calotte



## **Analysis of LBE samples**



## **Surface analyses**

- EPMA analysis
- SEM observation
- SIMS analysis



## **Microstructural analyses**

- TEM observation

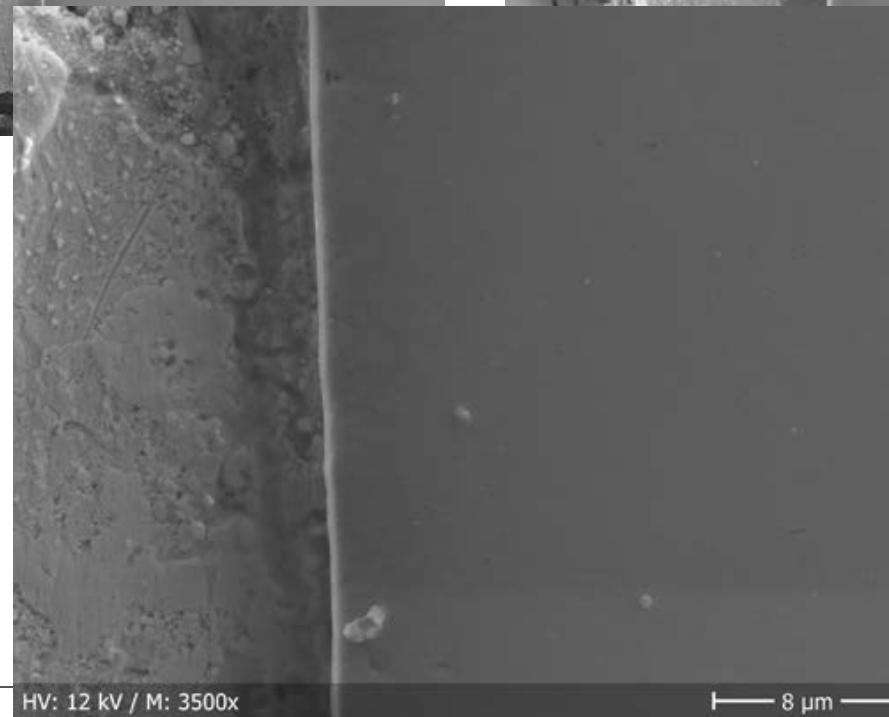
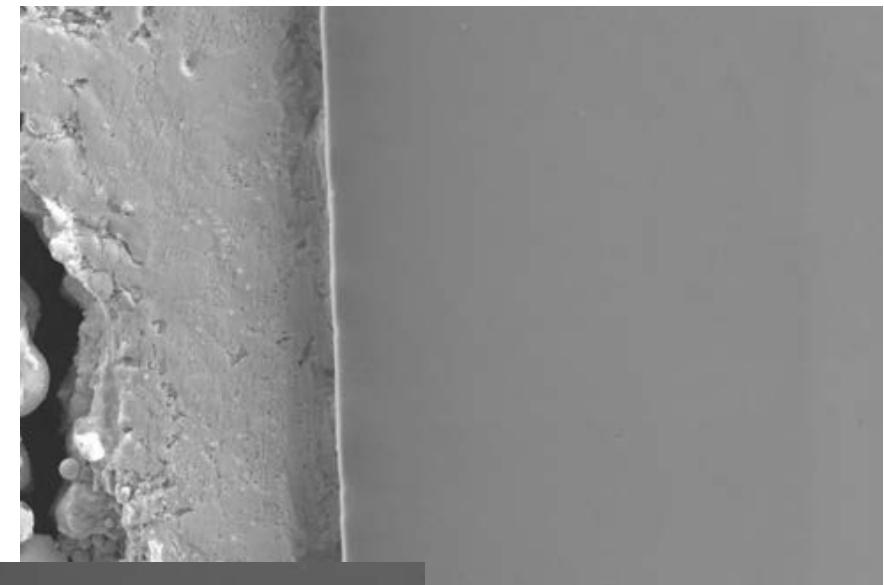
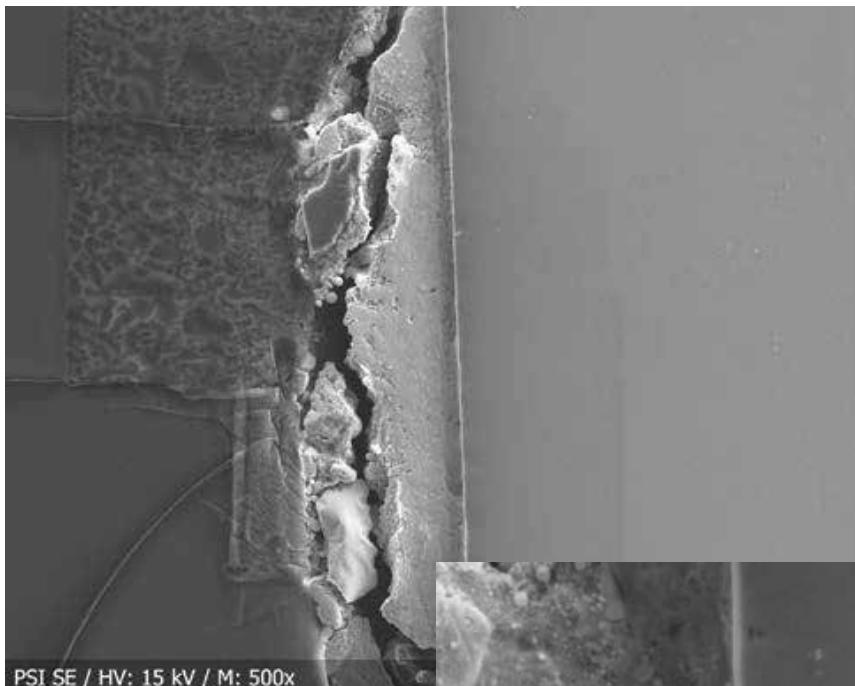


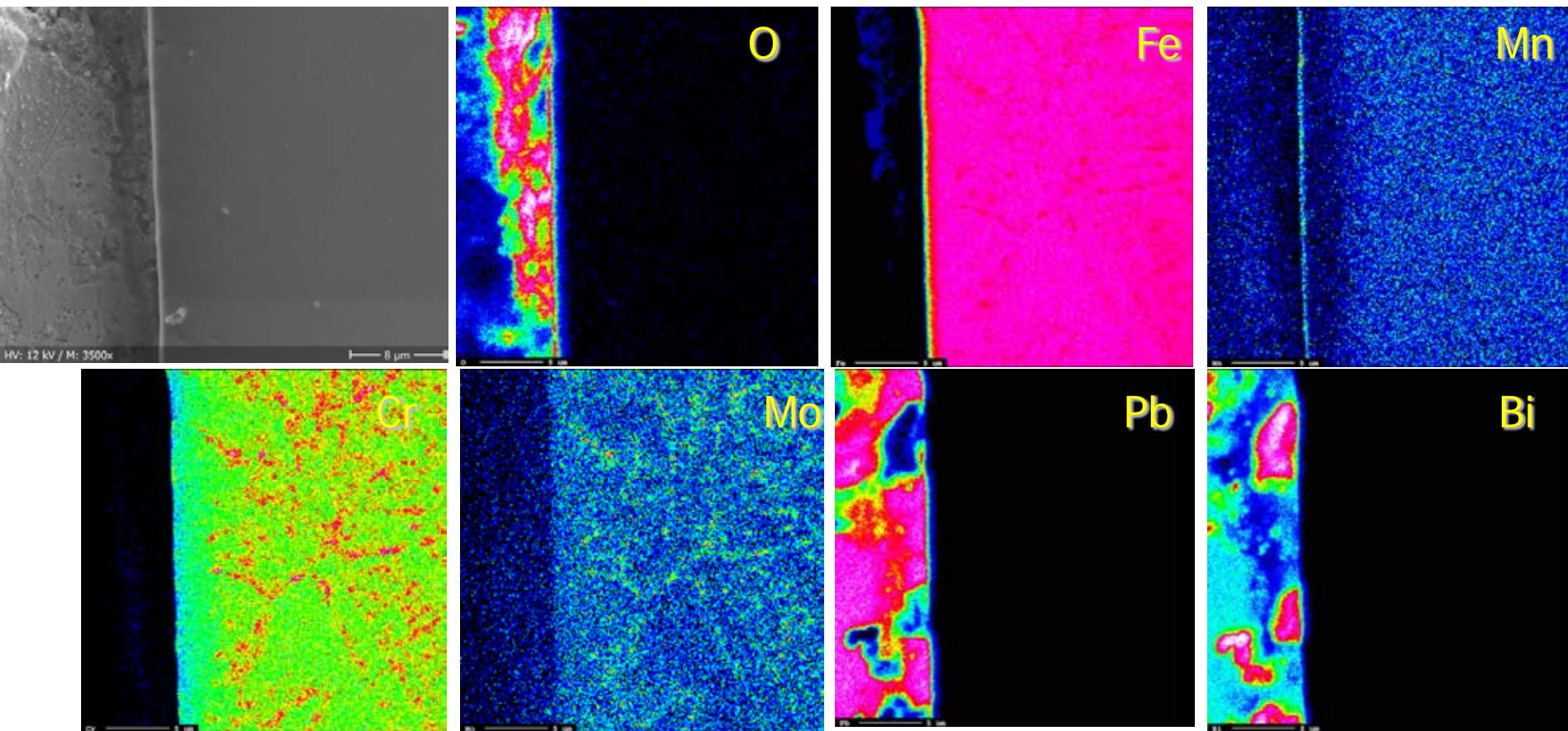
## **Mechanical test**

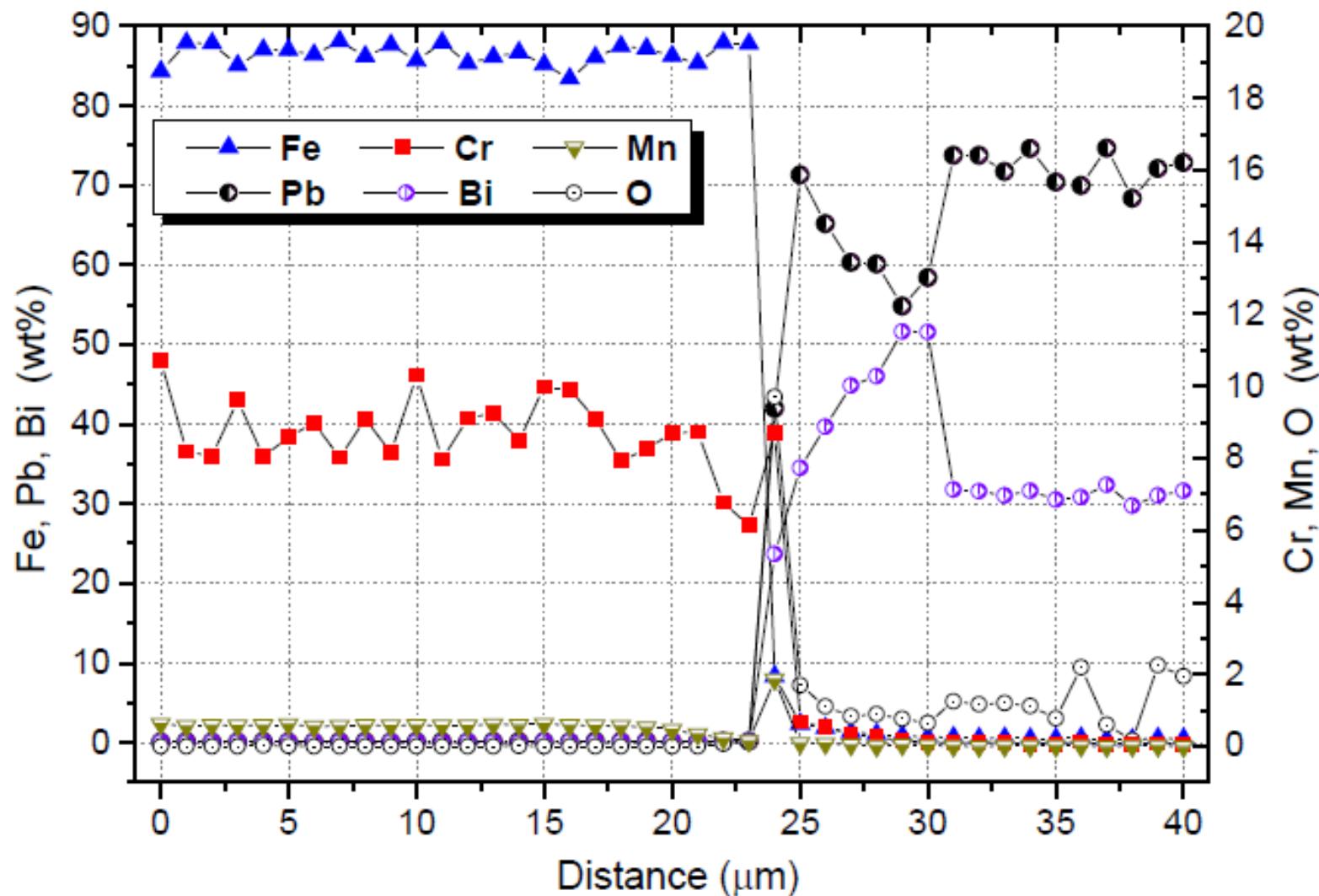
- Tensile test
- Bend test
- Hardness measurement



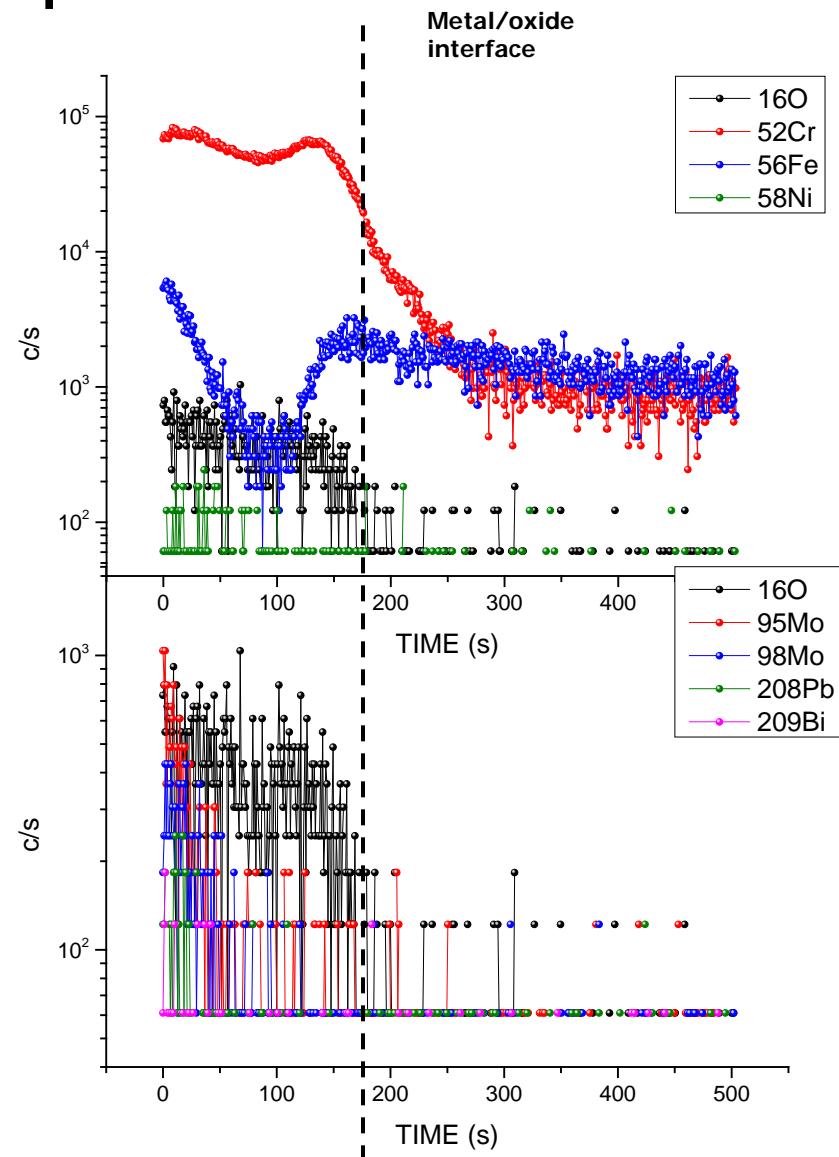
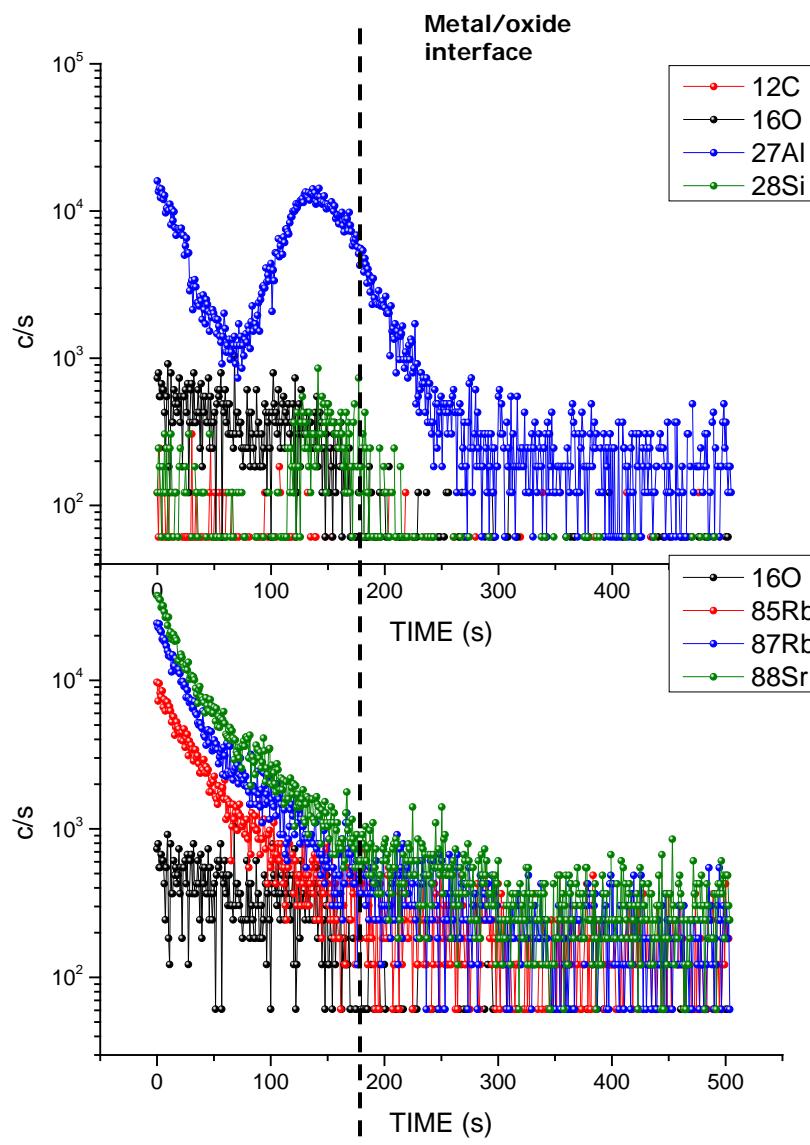
## **Summary**

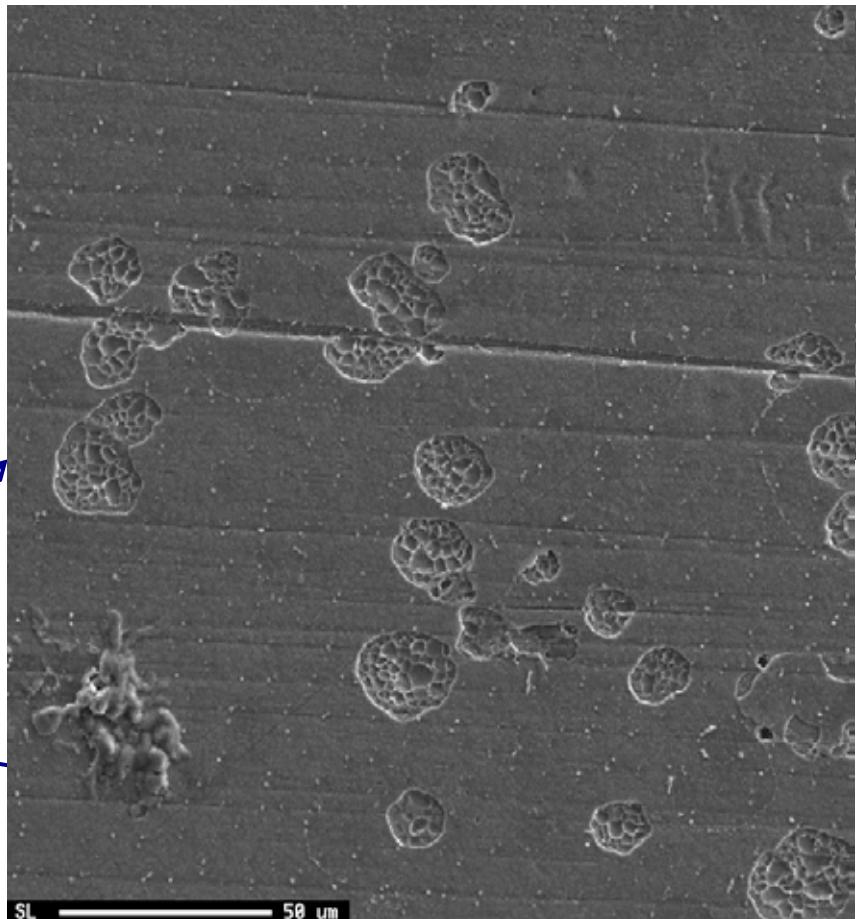
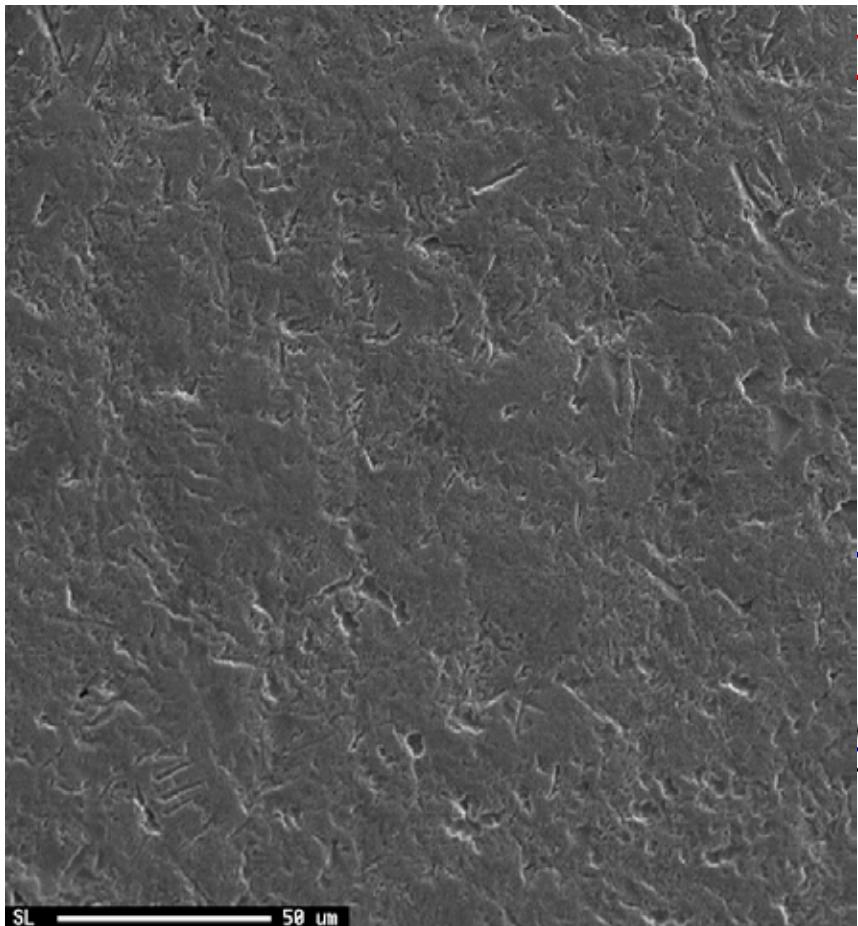




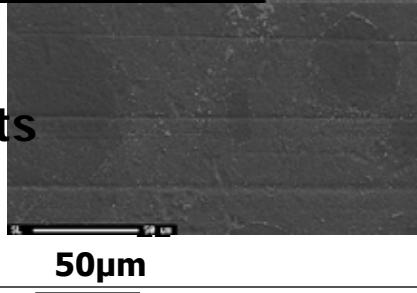
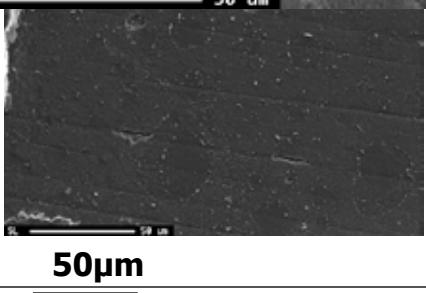


### Depth profile





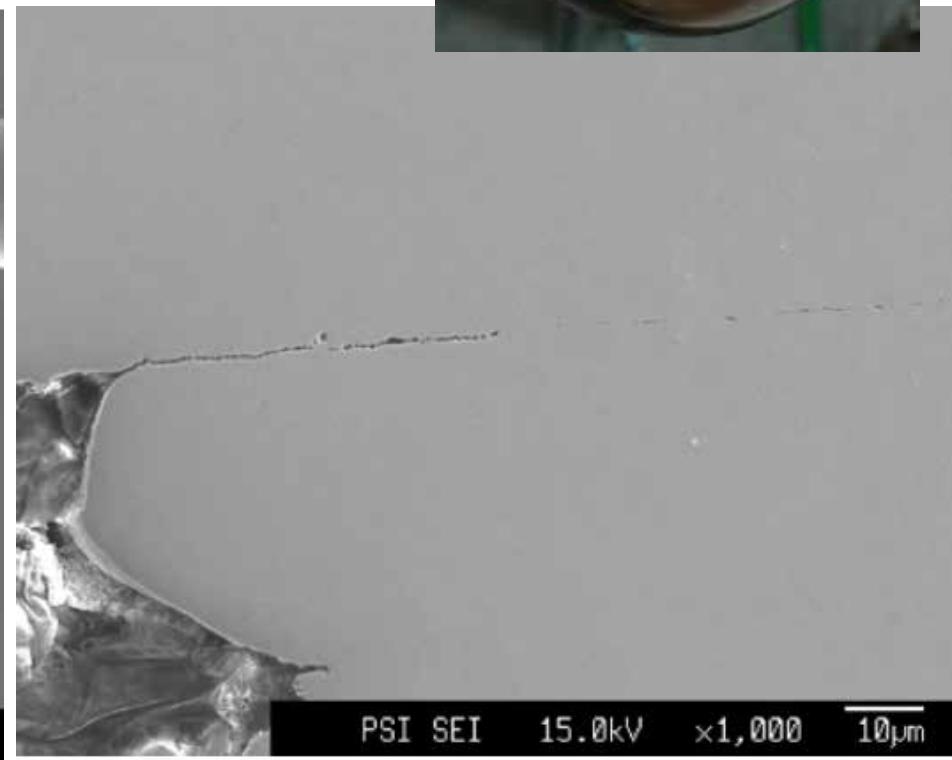
Small number of pits



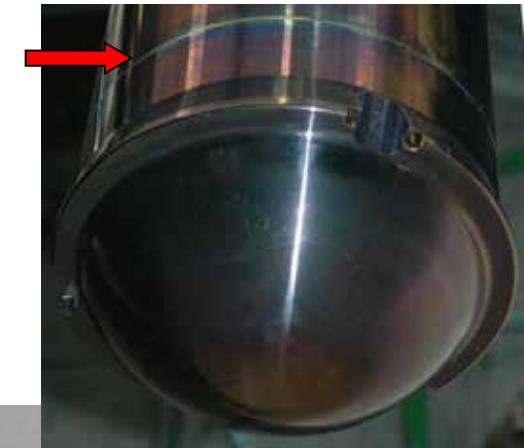
October 22, 2014

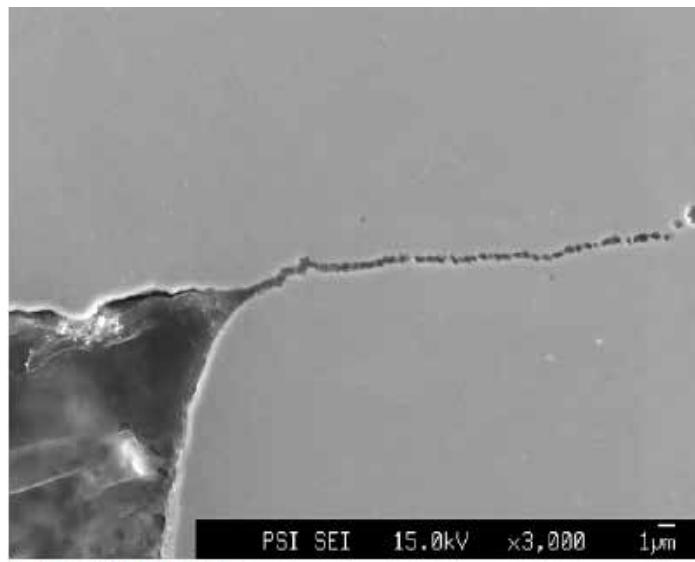


P2, Weld position (inside is on the left)

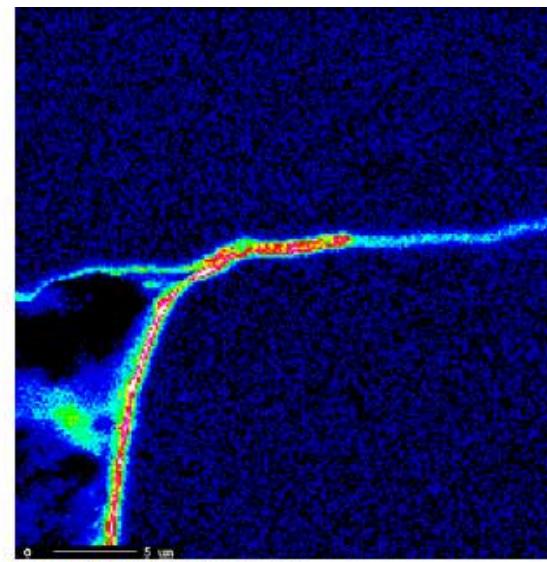


P2, section of left SEM (note: microcrack)

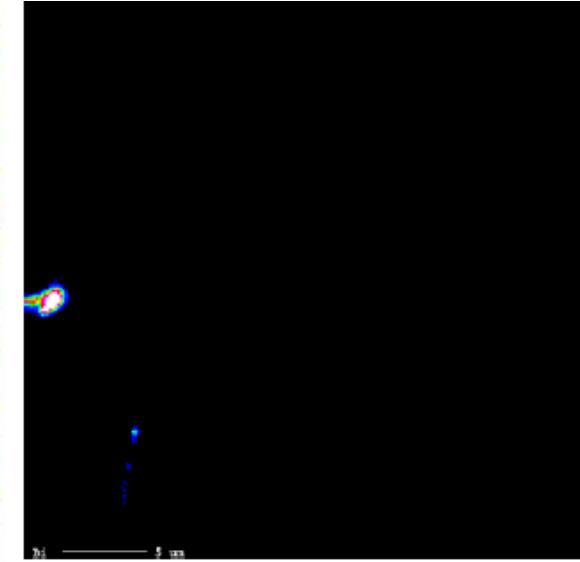




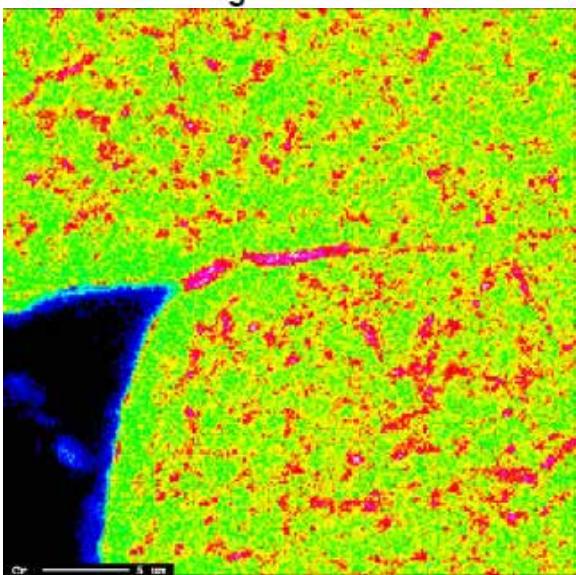
P2 : SEM-image



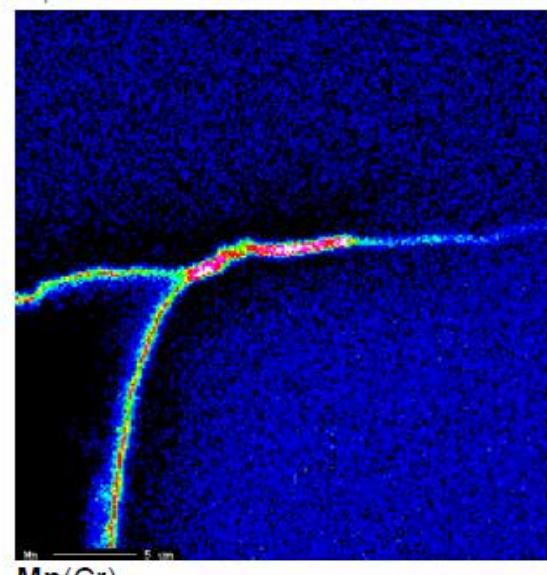
O, oxidation in crack



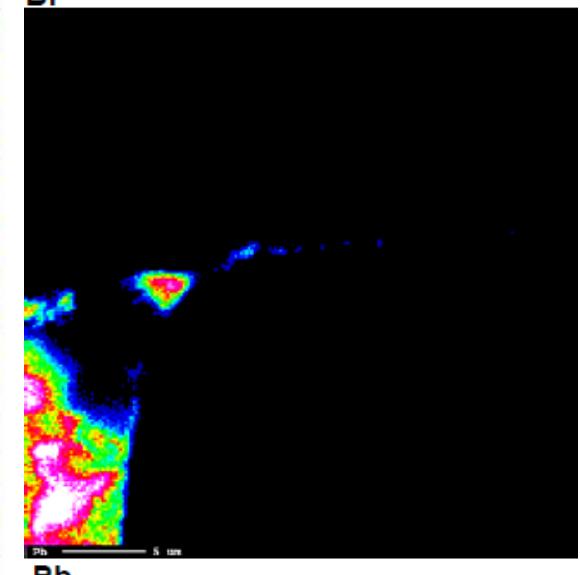
Bi



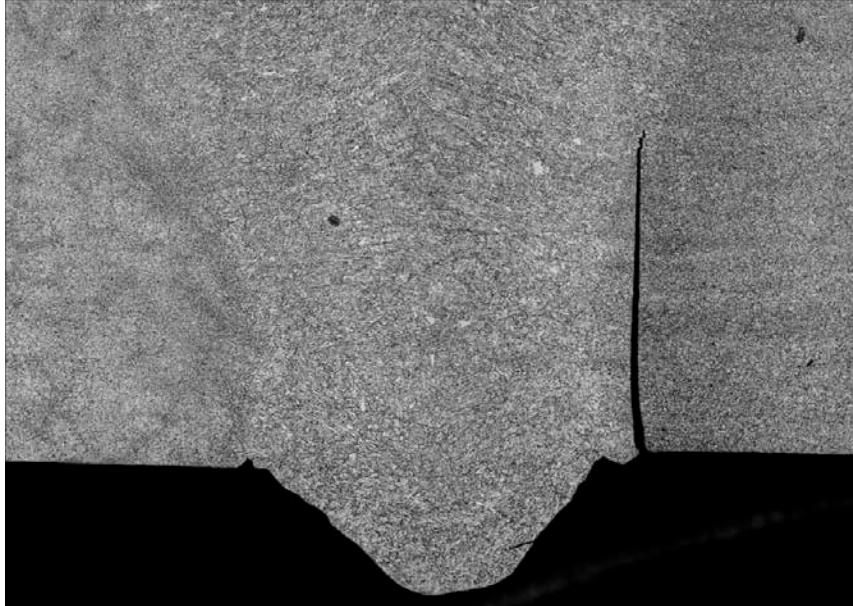
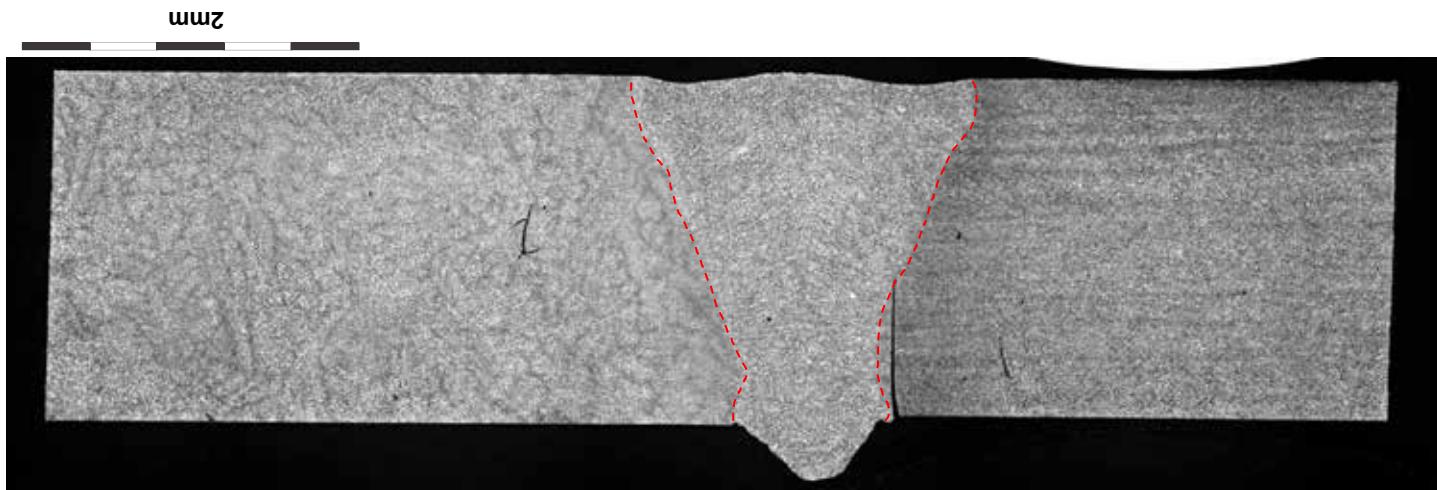
Cr



Mn(Cr)

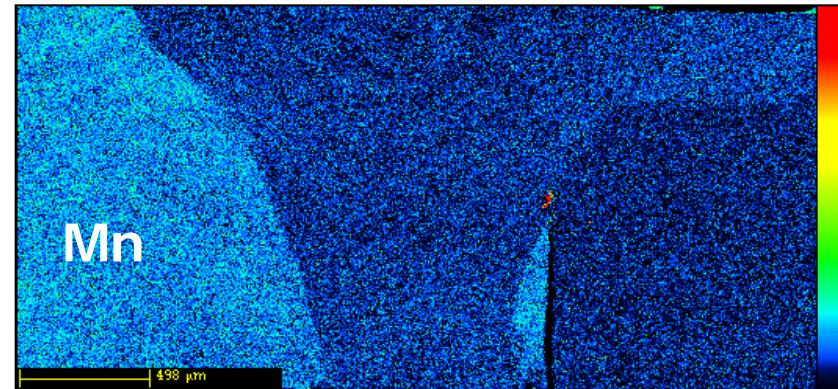
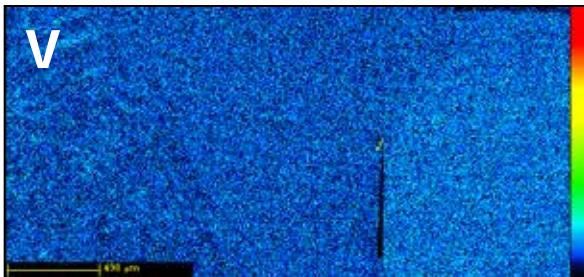
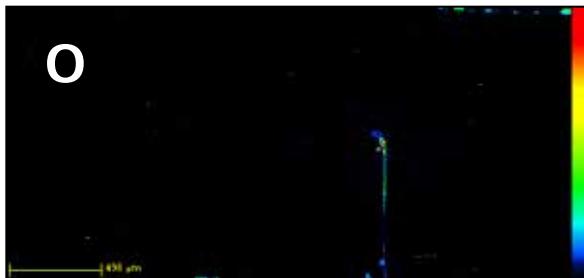
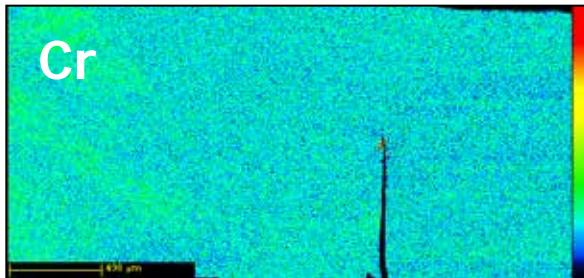


Pb

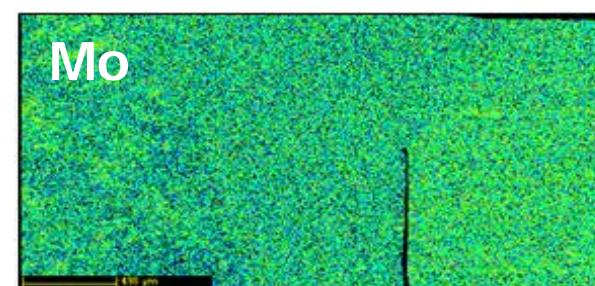
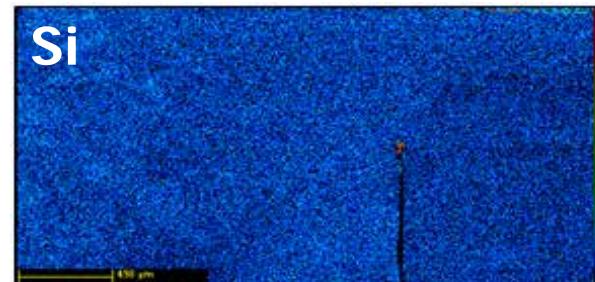


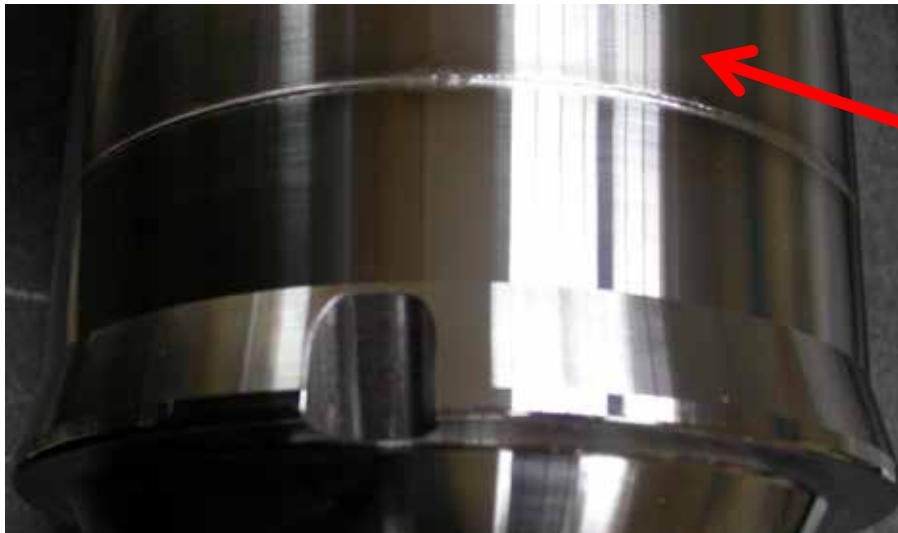
§ « crack »: two opposite surfaces ; electron beam deviated due to magnetic steel?

§ no evidence of propagation !



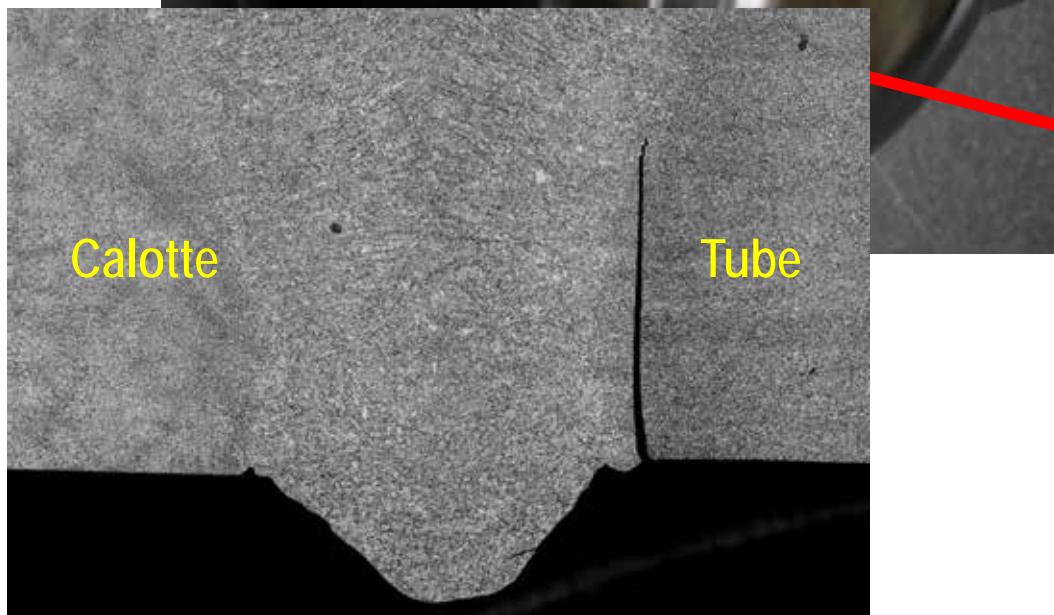
Why is the composition different on both sides of the « crack »?





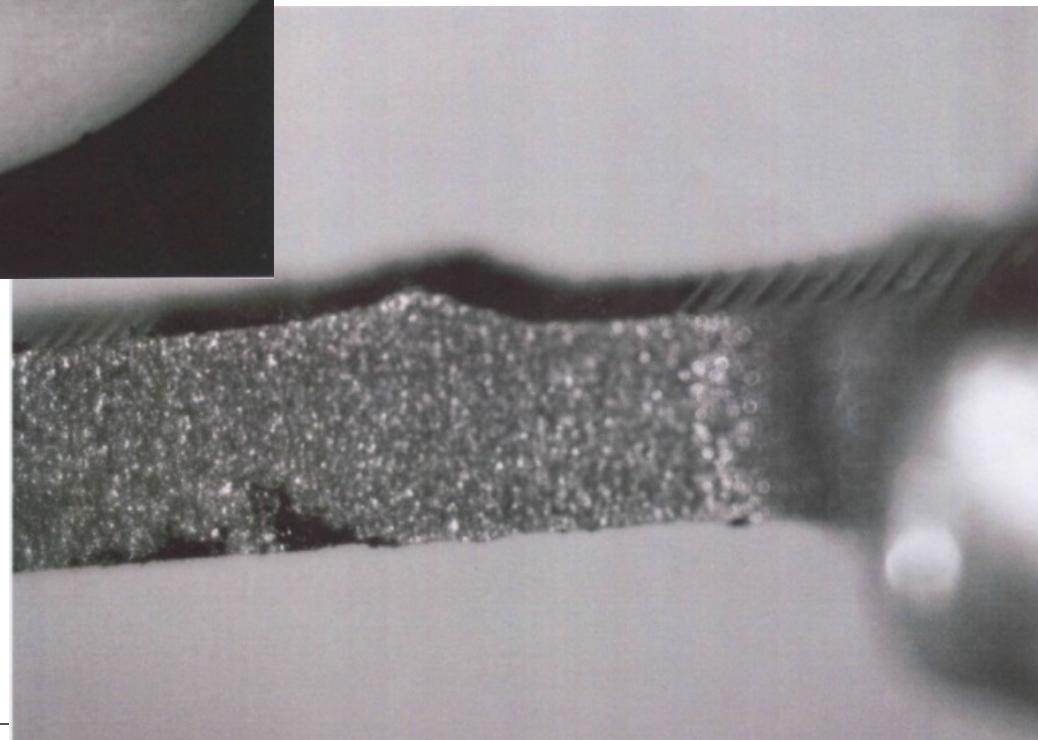
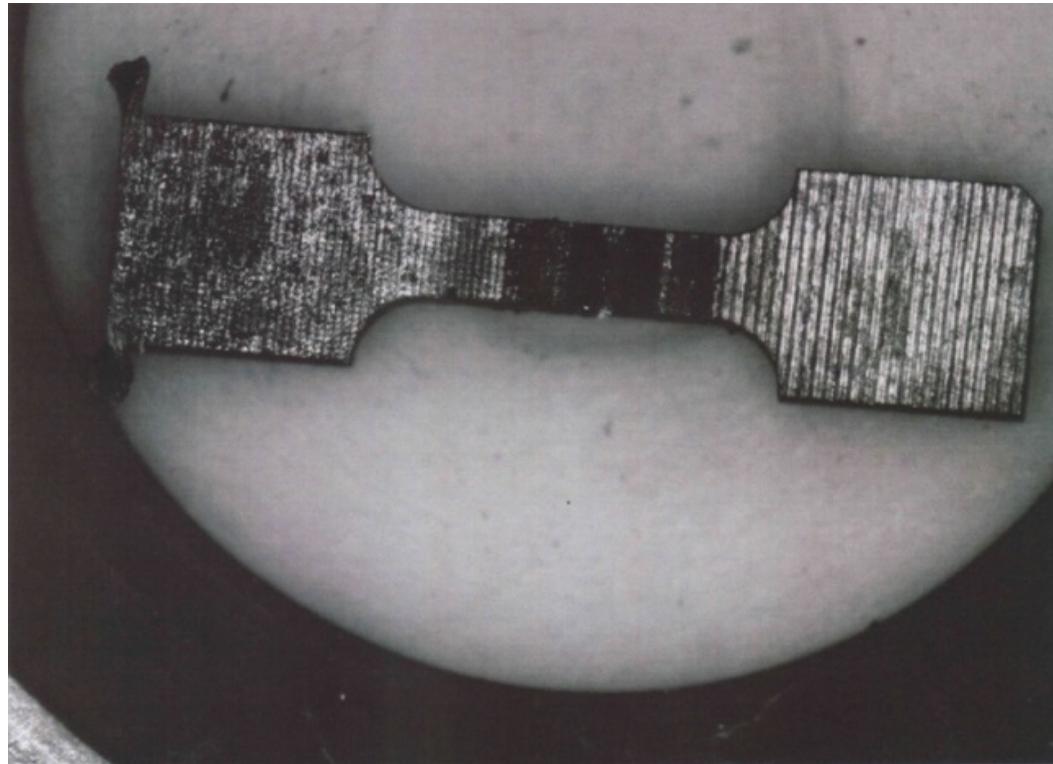
**T91 Tube**  
**USINOR/Vallourec-**  
**Mannesmann**

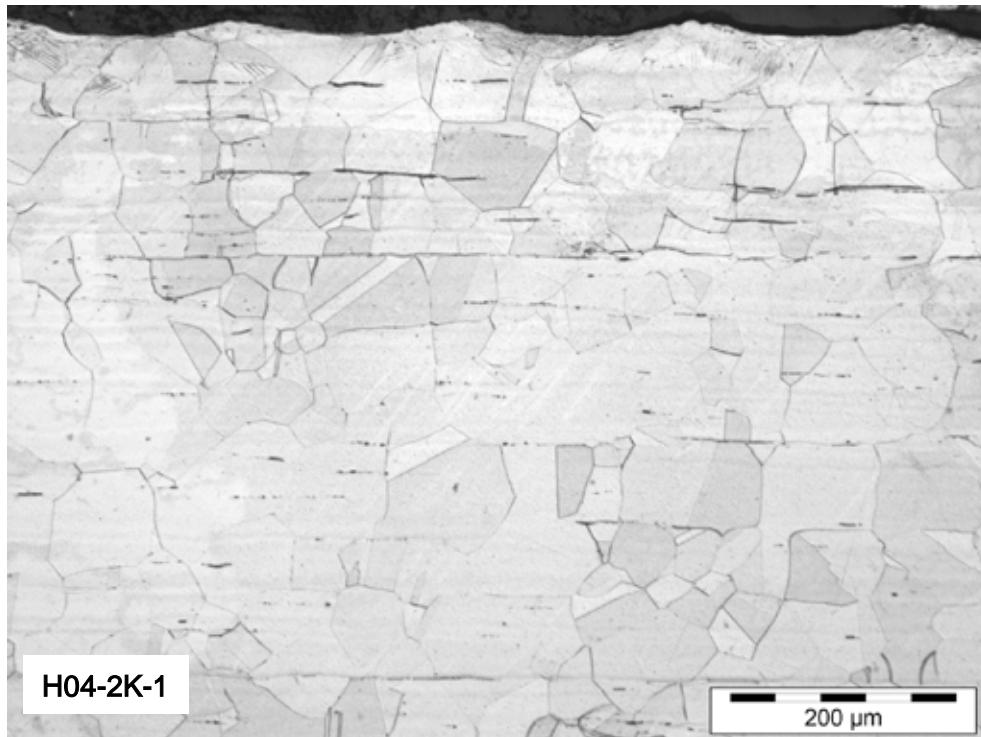
Cr	Mo	Mn	V	Si
8.54	0.93	0.40	0.21	0.29



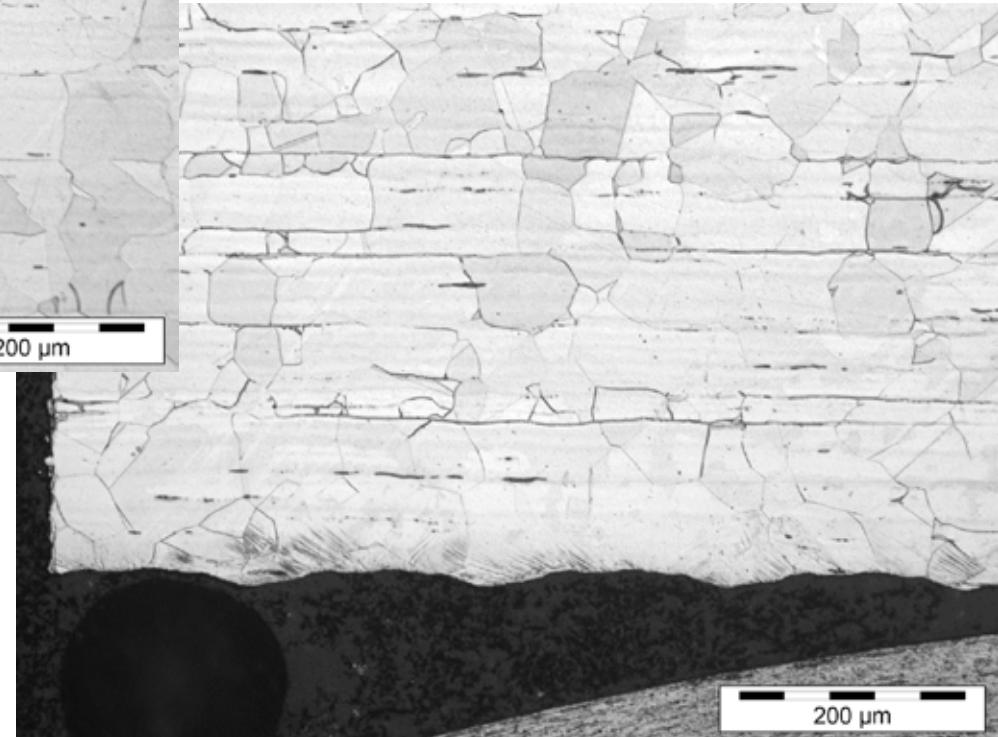
**T91 INDUSTEEL**  
**(Arcelor)**

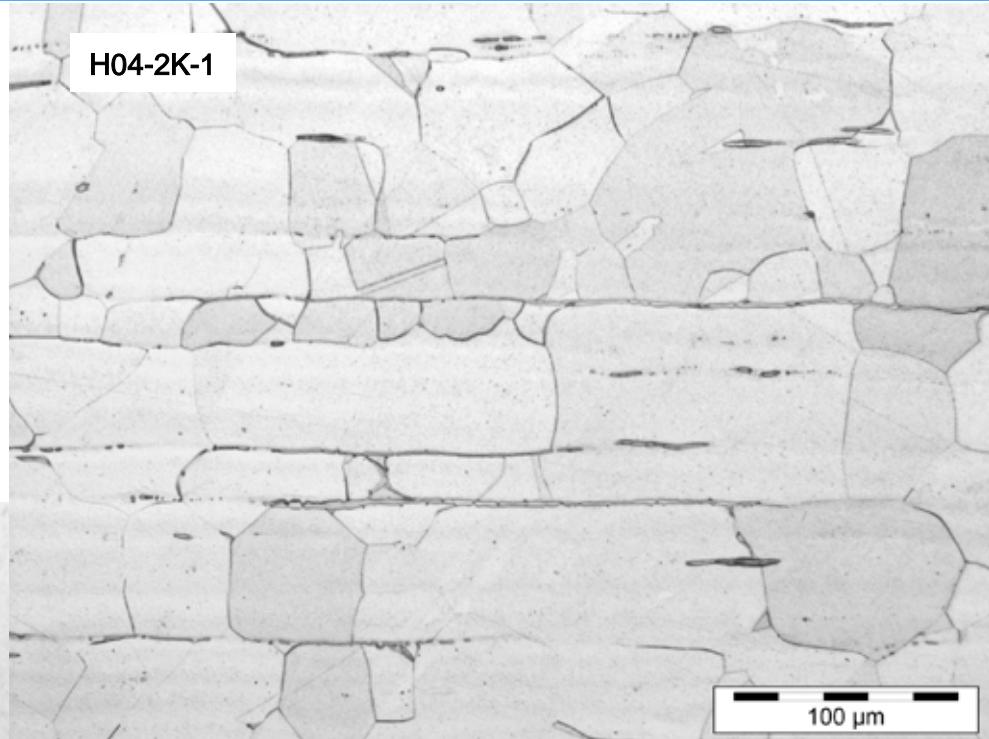
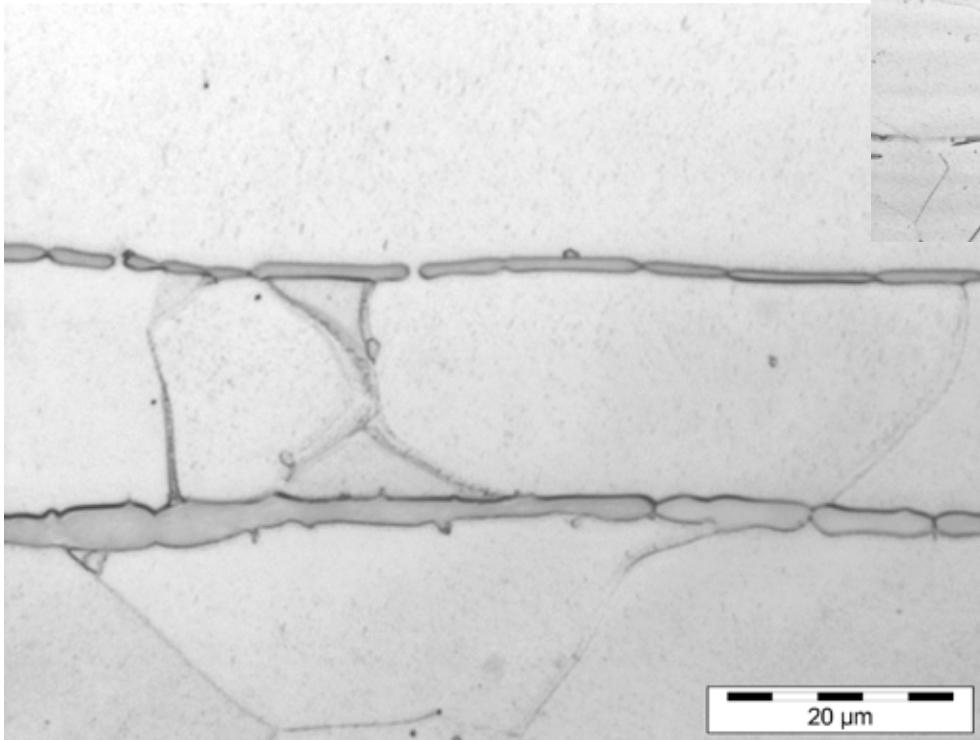
Cr	Mo	Mn	V	Si
8.76	0.86	0.60	0.186	0.317



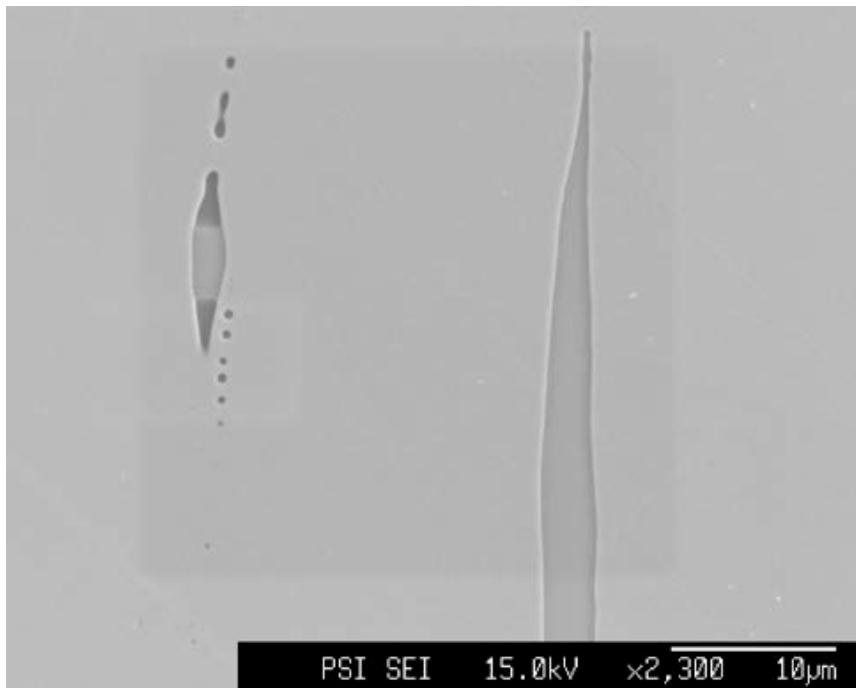


Grooves on inside  
and outside of FGT

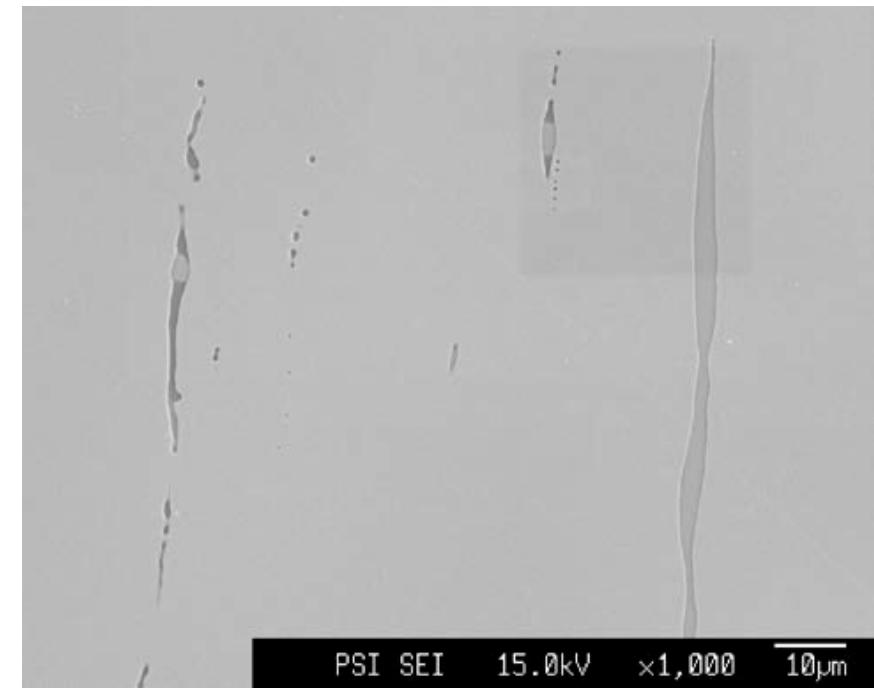




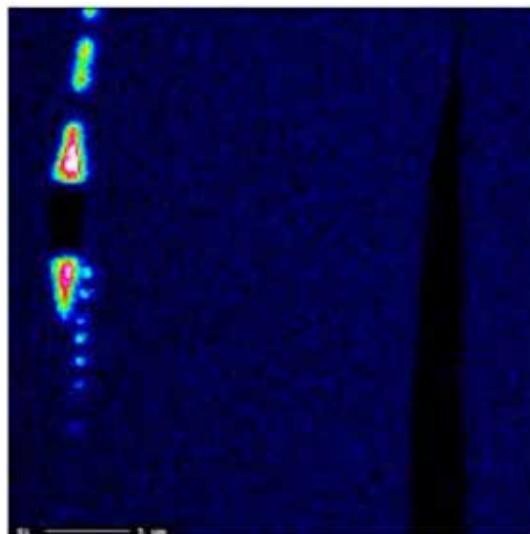
Heavy precipitation,  
character TBD



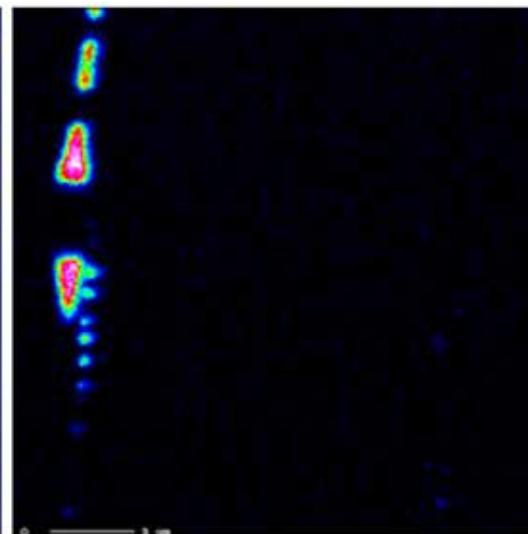
PSI SEI 15.0kV  $\times 2,300$  10 $\mu\text{m}$



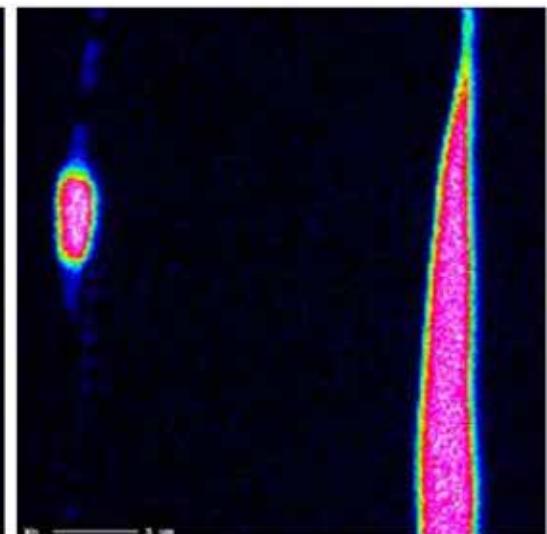
PSI SEI 15.0kV  $\times 1,000$  10 $\mu\text{m}$



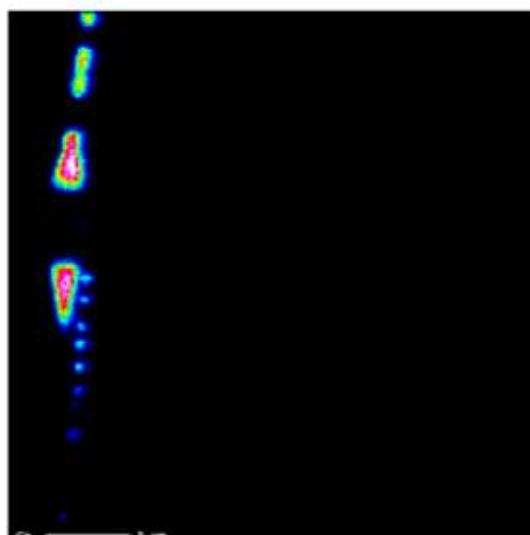
O



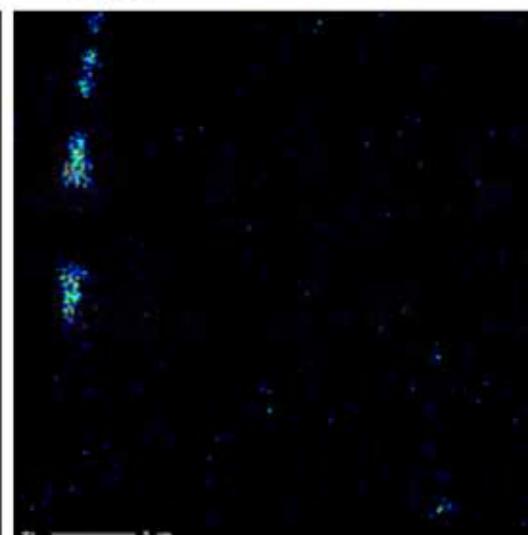
Ca



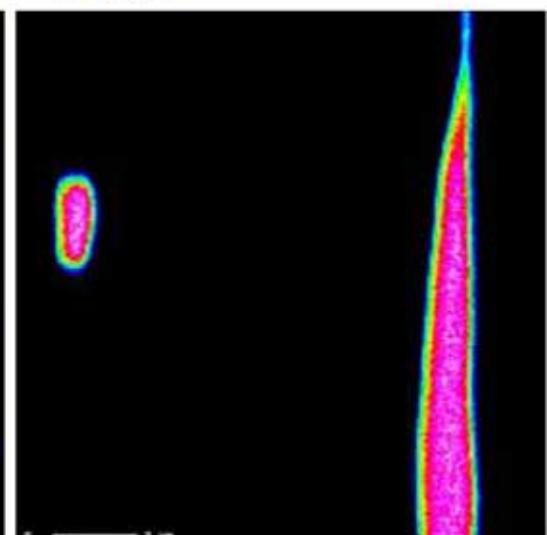
Mn



Si



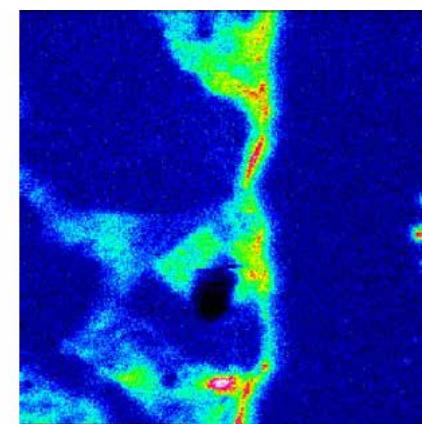
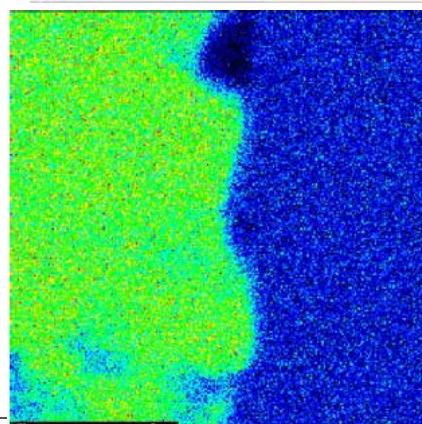
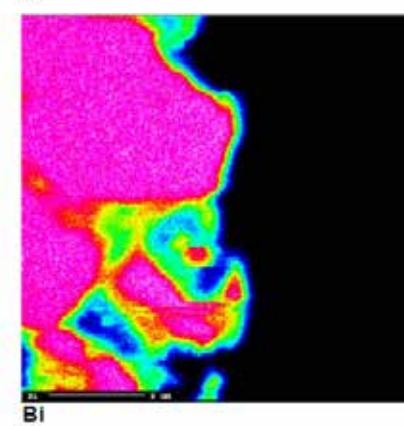
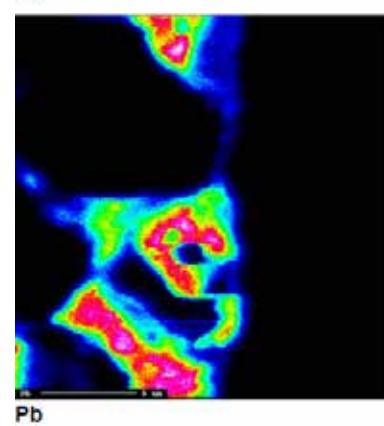
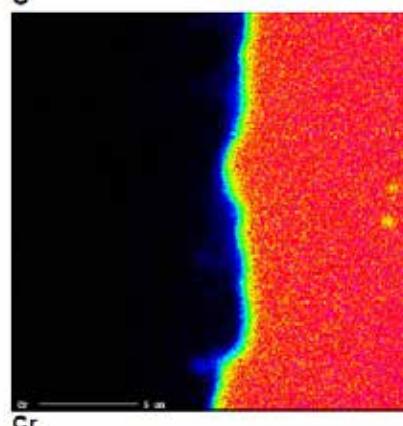
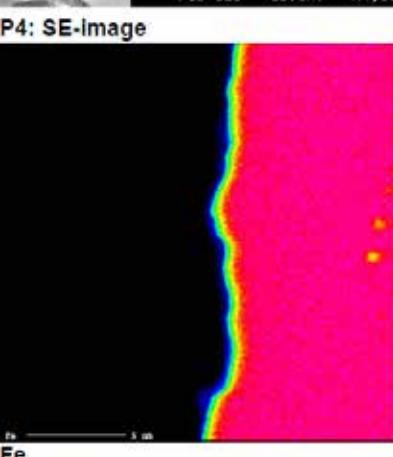
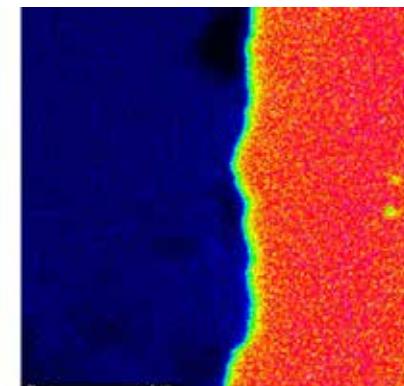
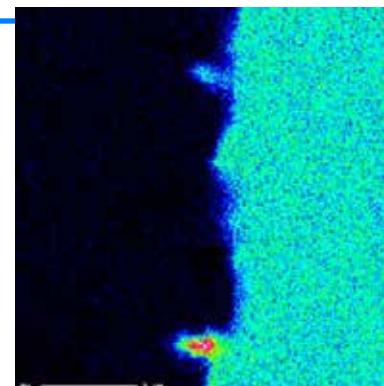
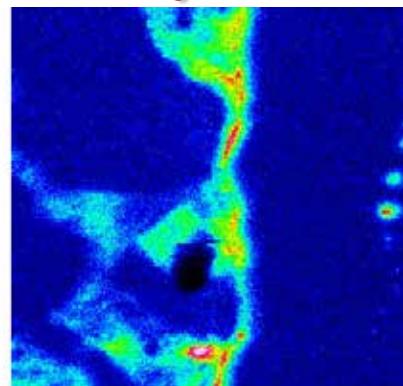
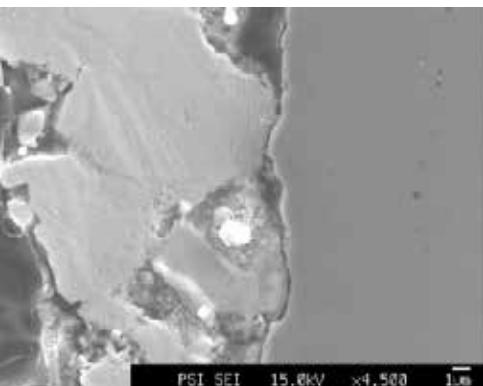
Ti



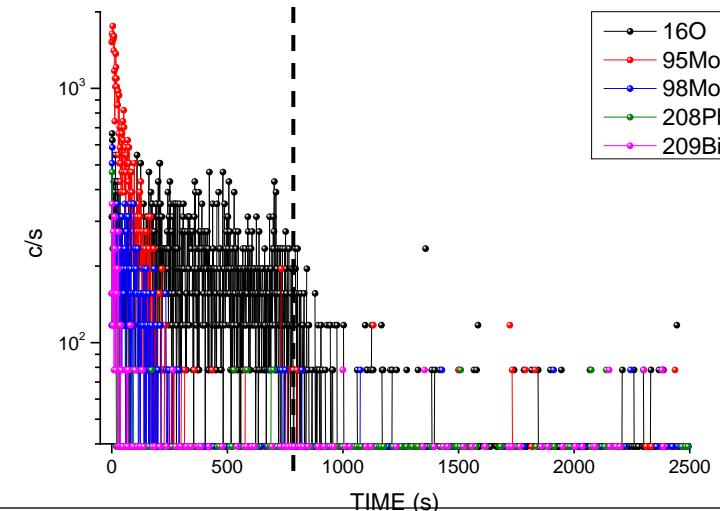
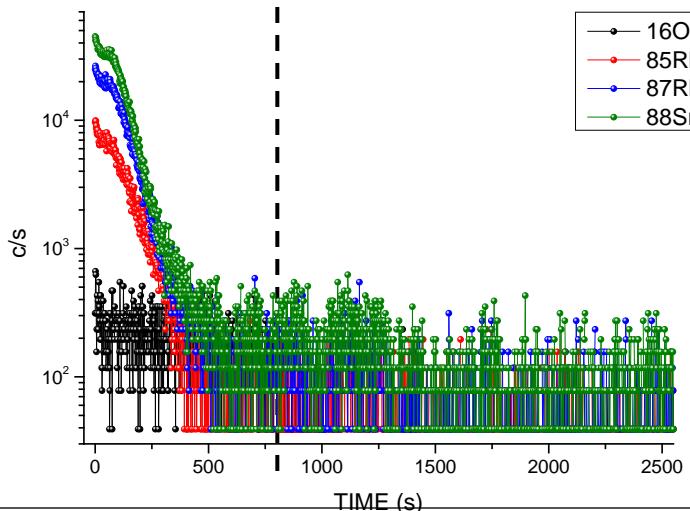
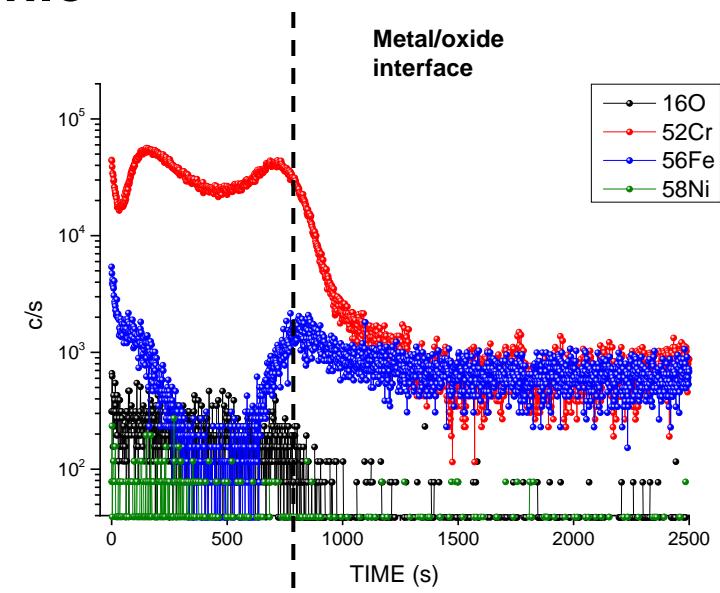
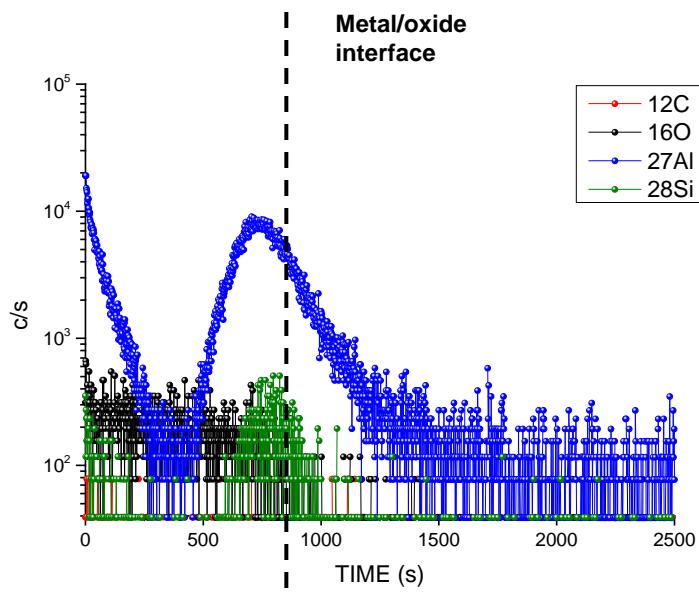
S

# Surface analyses – EPMA

**SS 316 FGT**



## Depth profile



## Ø **Non-destructive test (NDT)**

- Gamma mapping of the Al-calotte
- Visual inspection of the T91 calotte
- Thickness measurement of the T91 calotte

## Ø **Analysis of LBE samples**

## Ø **Surface analyses**

- EPMA analysis
- SEM observation
- SIMS analysis

## Ø **Microstructural analyses**

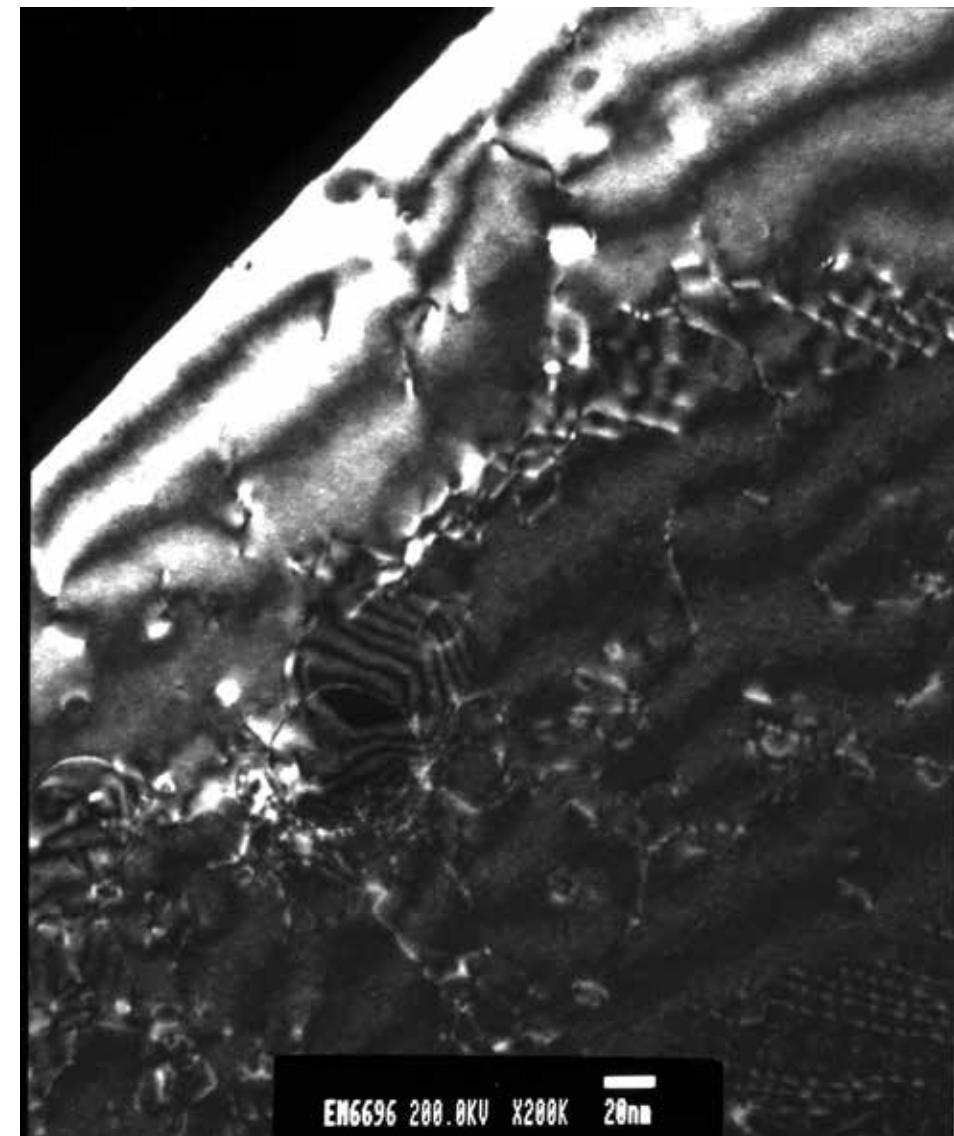
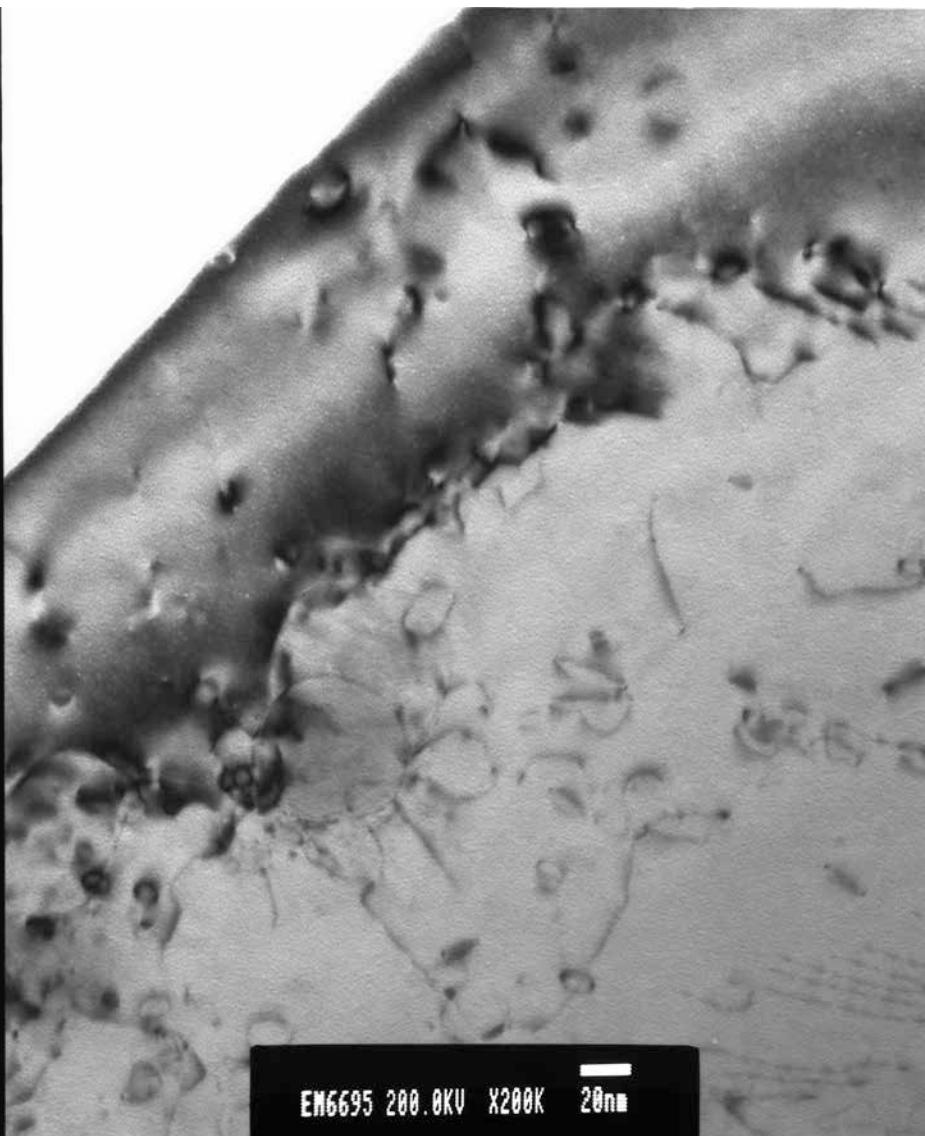
- TEM observation

## Ø **Mechanical test**

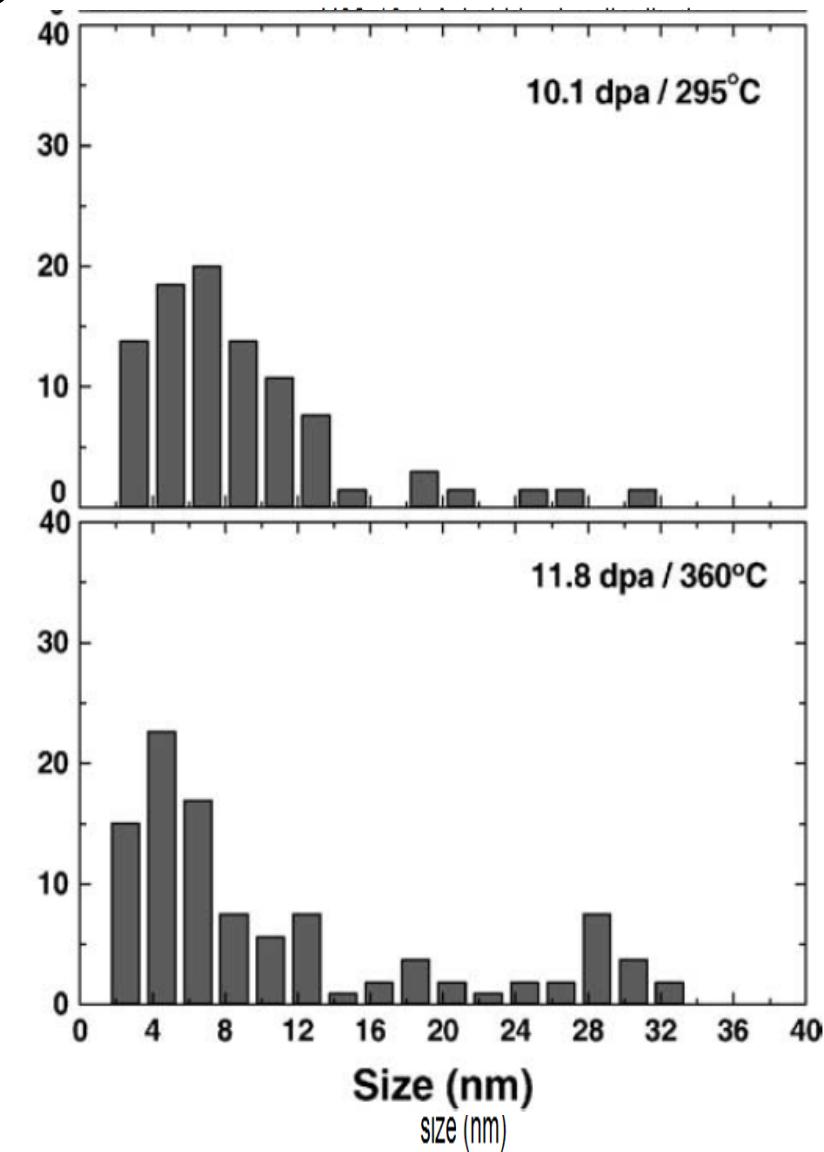
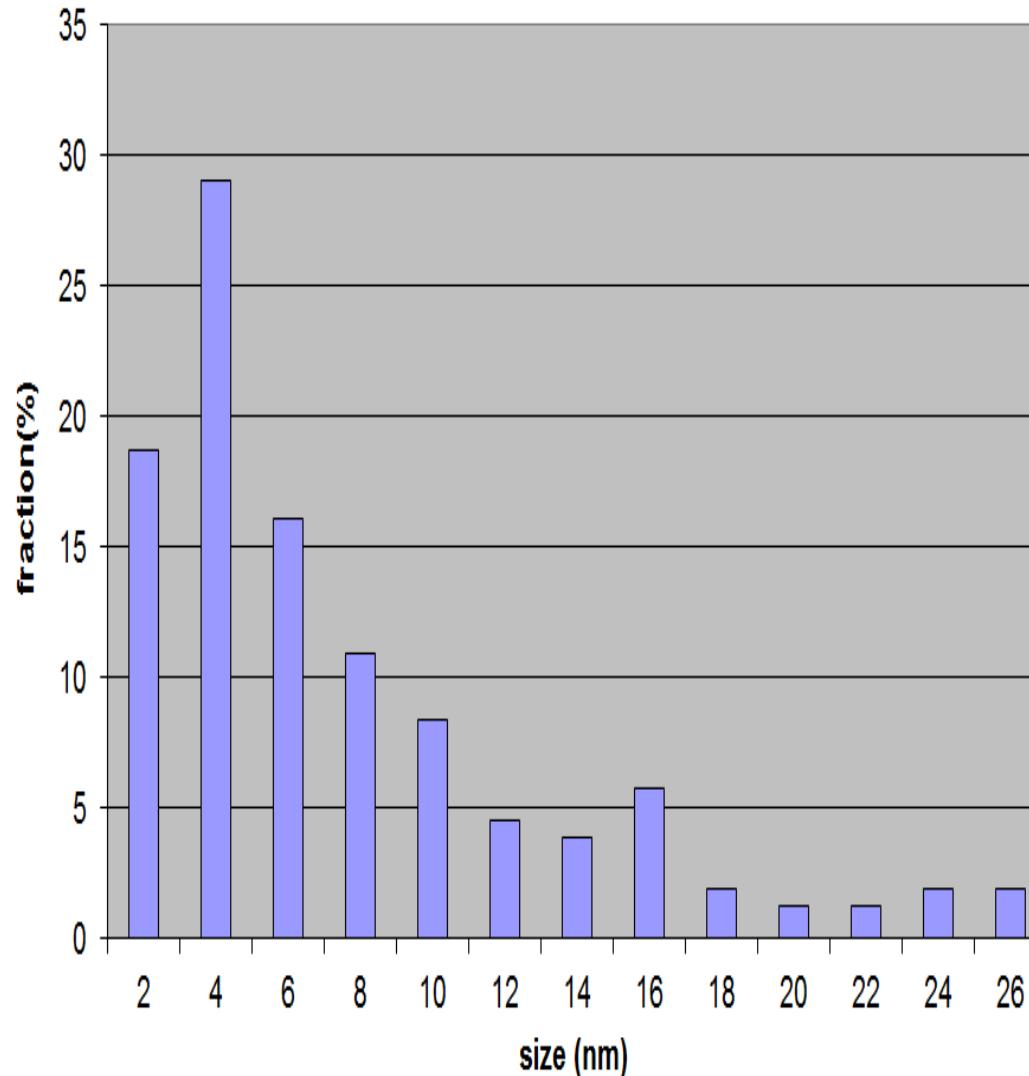
- Tensile test
- Bend test
- Hardness measurement

## Ø **Summary**

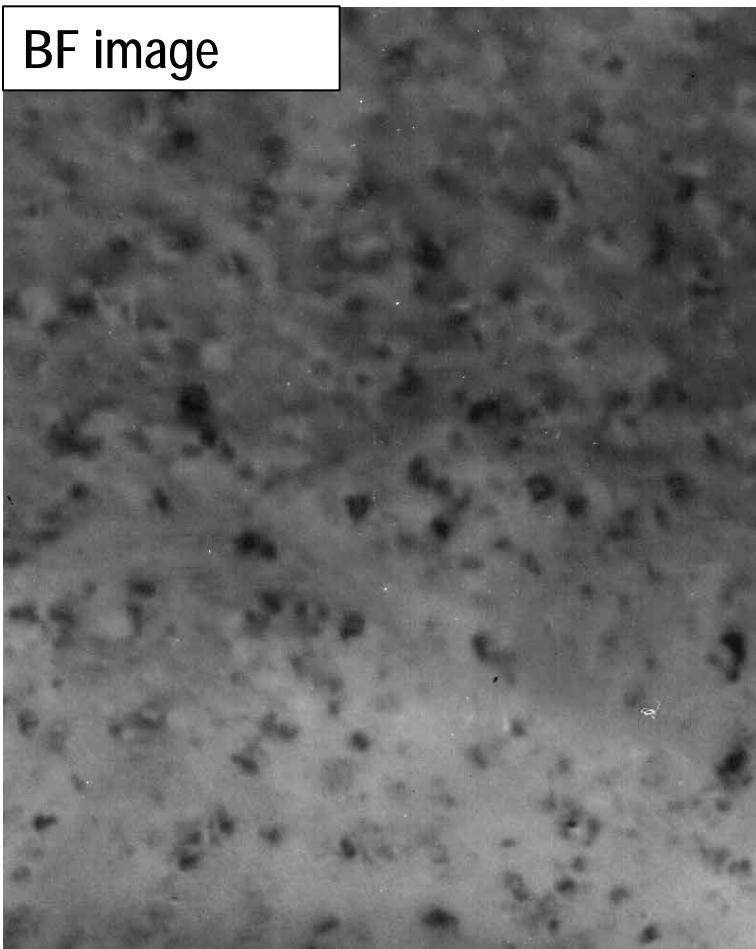
# TEM observation: T91 calotte (6.5 dpa, 325 ° C)



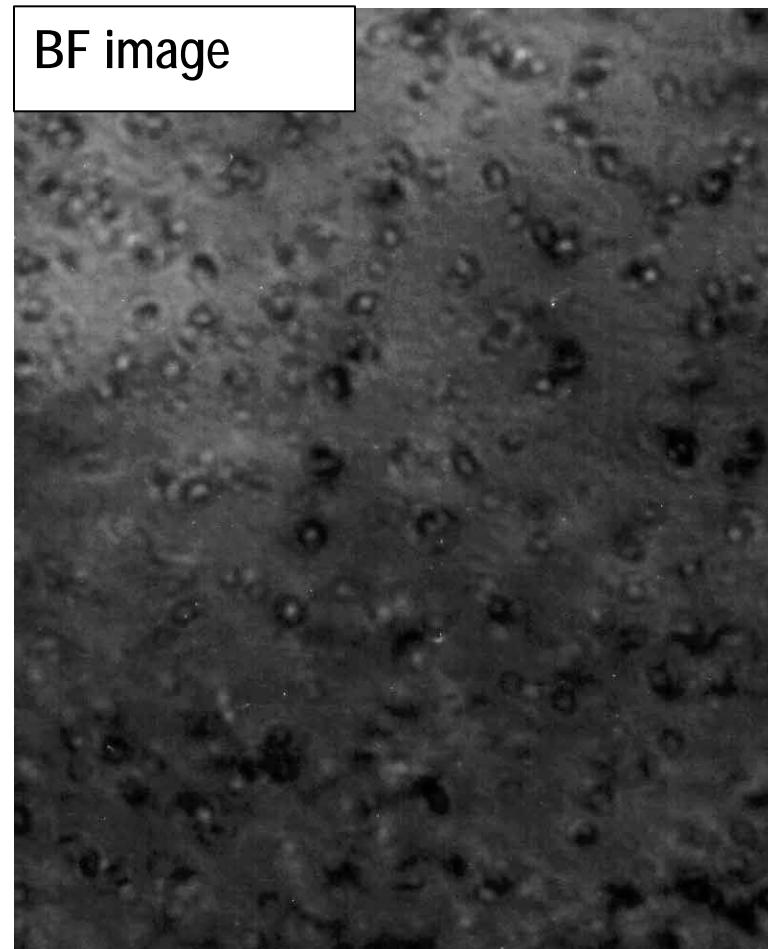
## Size distribution of loops and bubbles



Frank loops



Cavities



100nm

- High number density Frank loops and cavities.



## **Non-destructive test (NDT)**

- Gamma mapping of the Al-calotte
- Visual inspection of the T91 calotte
- Thickness measurement of the T91 calotte



## **Analysis of LBE samples**



## **Surface analyses**

- EPMA analysis
- SEM observation
- SIMS analysis



## **Microstructural analyses**

- TEM observation



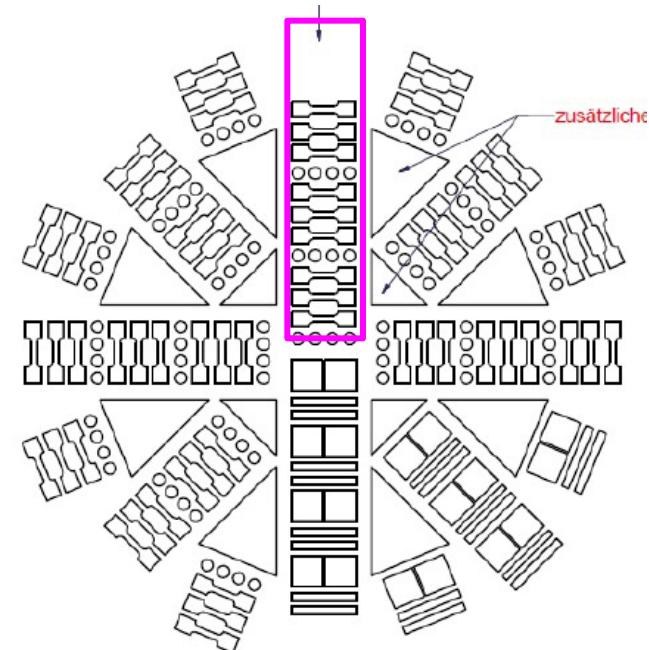
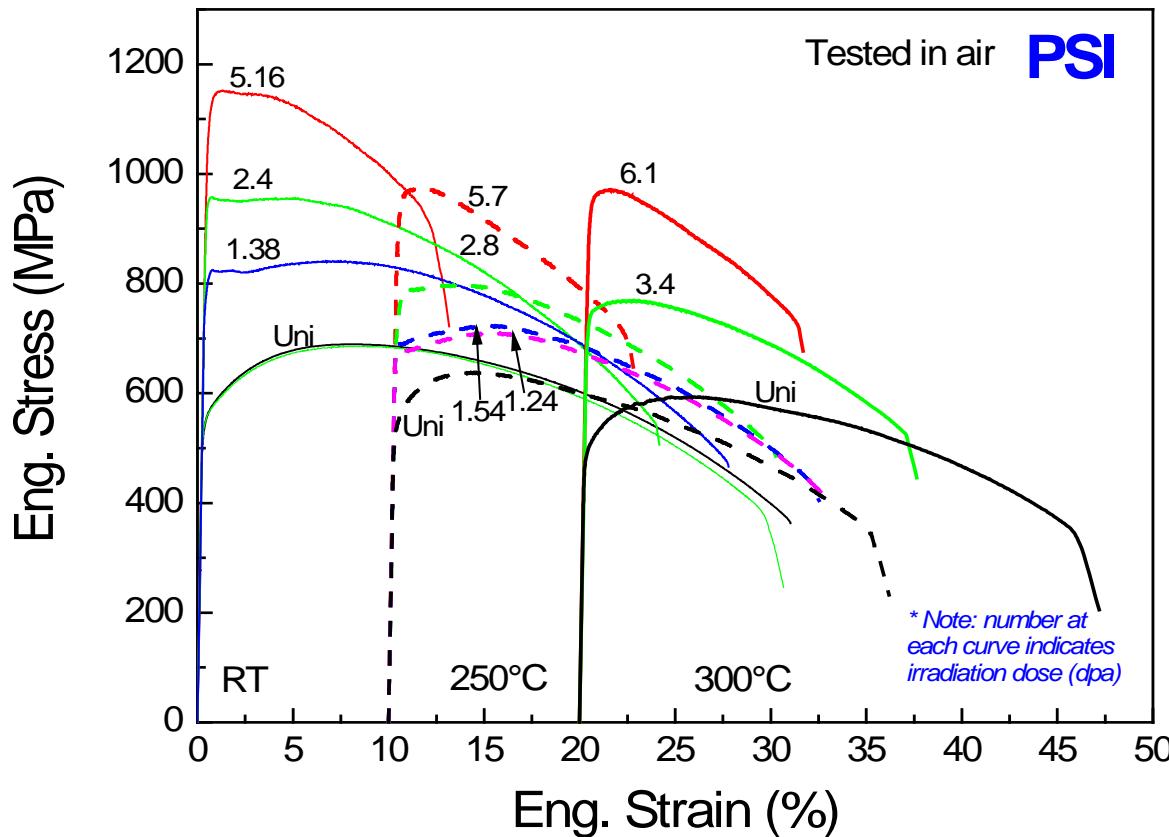
## **Mechanical test**

- Tensile test
- Bend test
- Hardness measurement



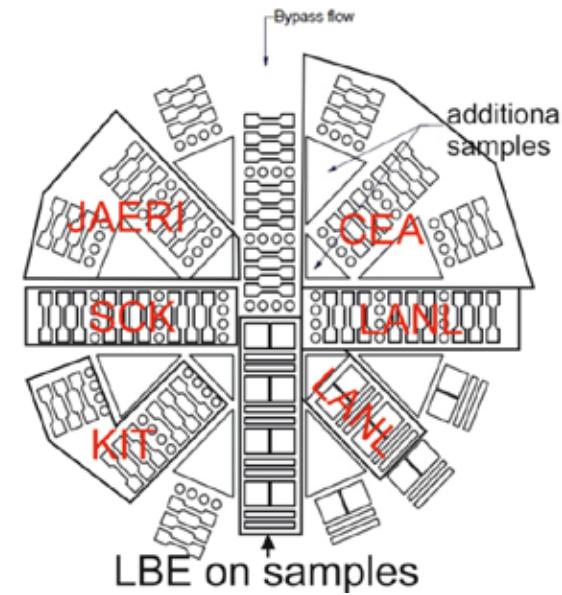
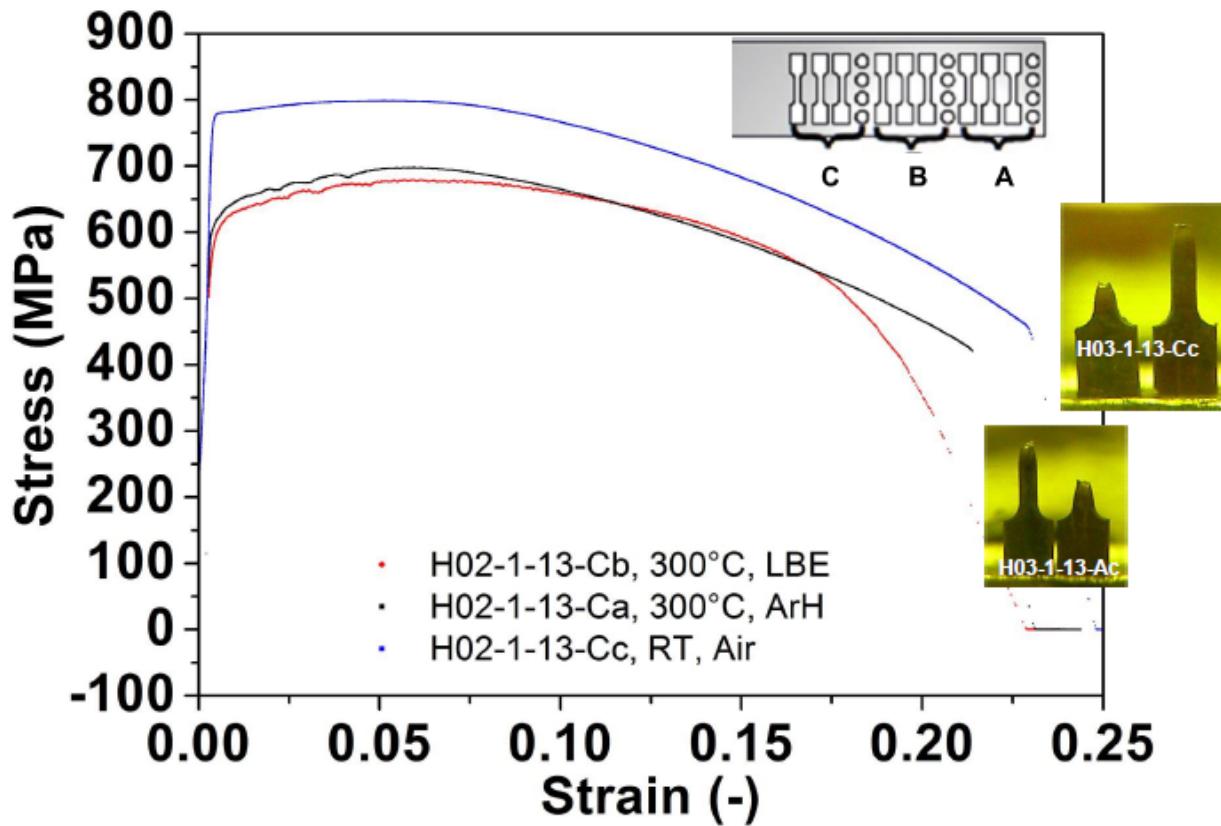
## **Summary**

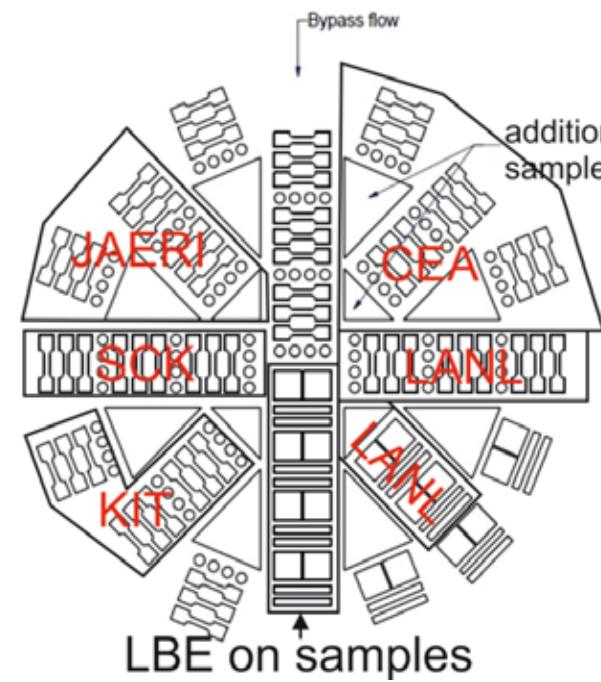
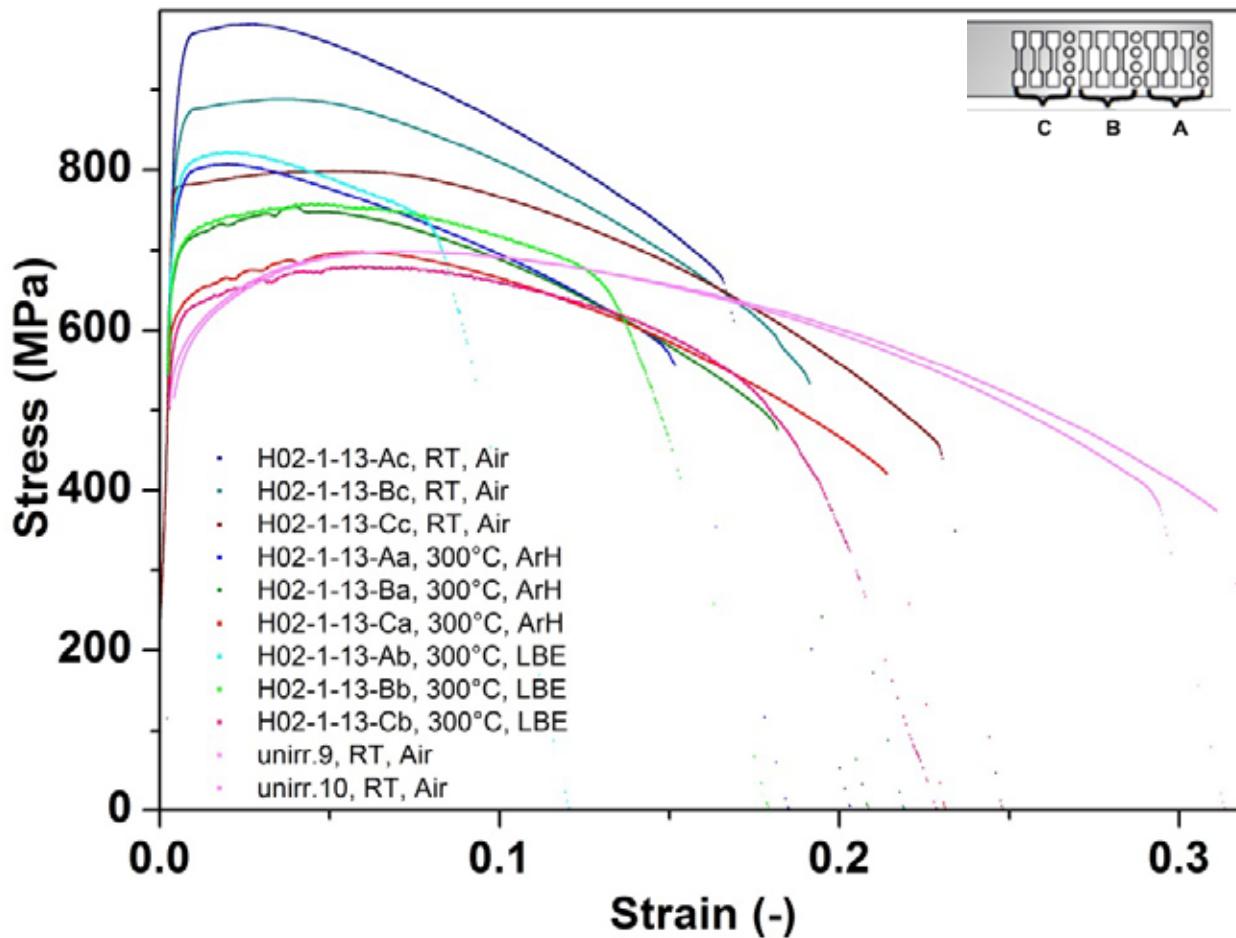
Eng. strain-stress tensile curves of specimens of MEGAPIE T91 Calotte

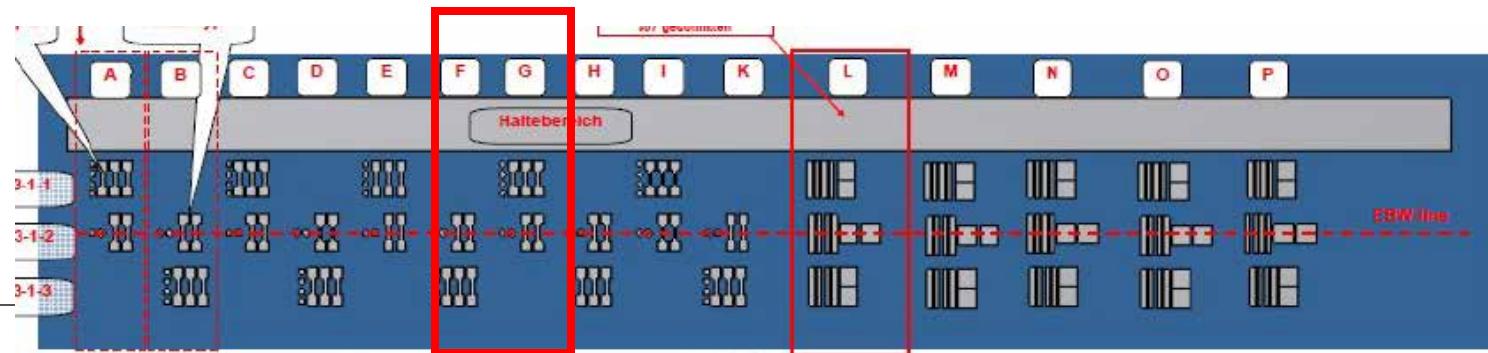
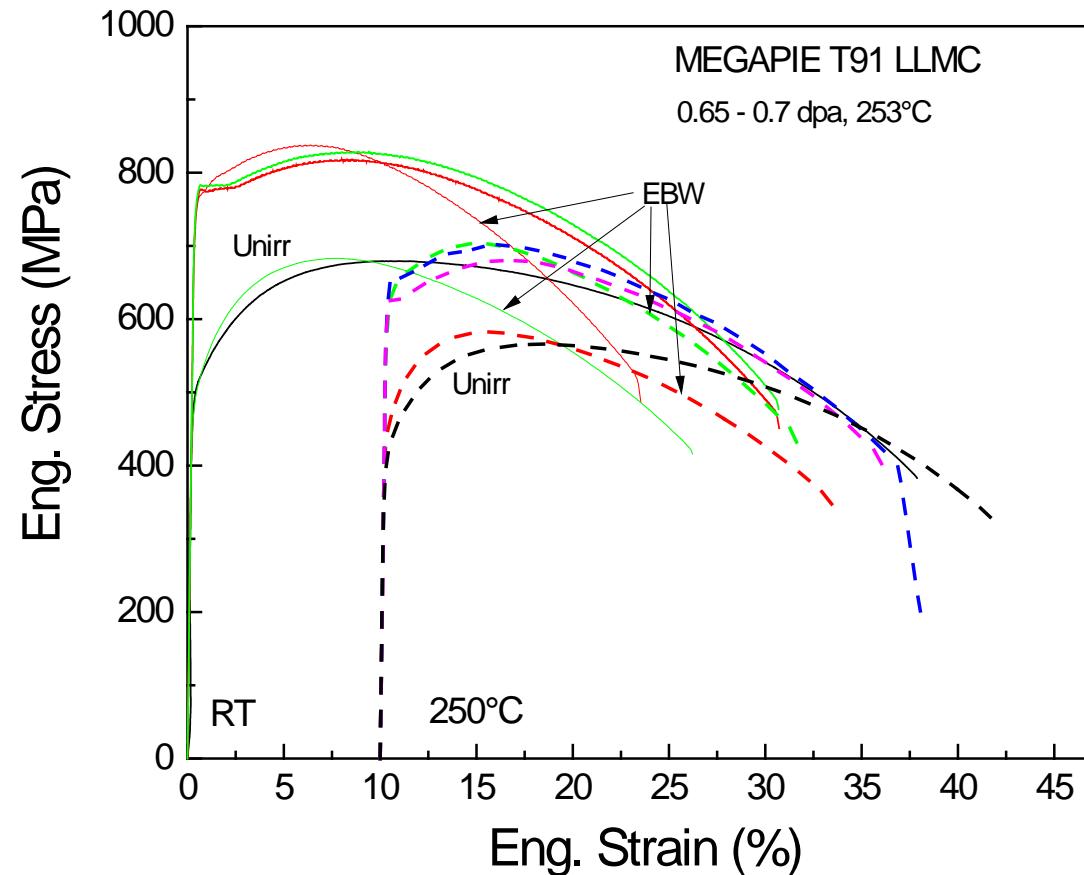


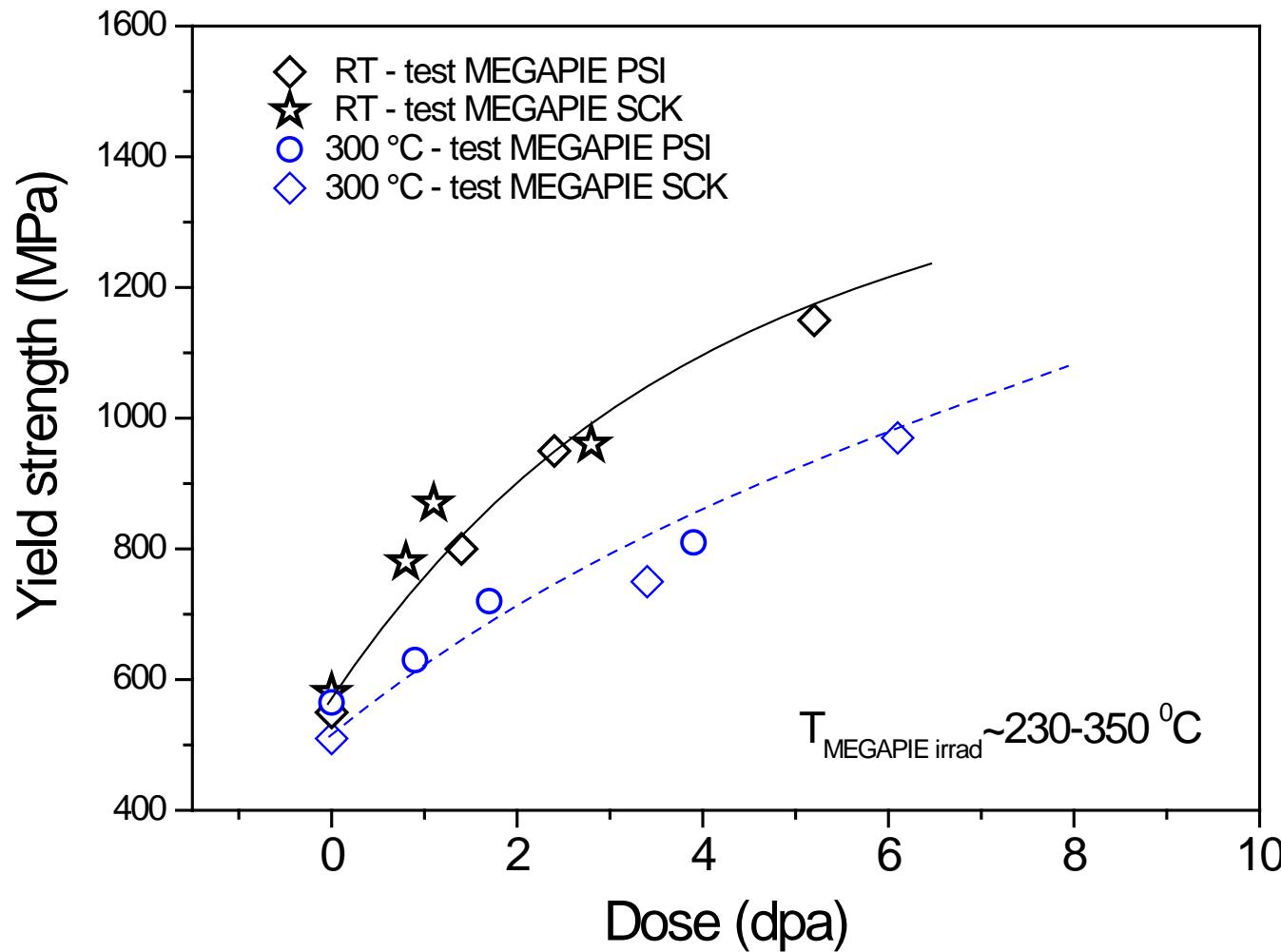
## Tensile test

## T91 Calotte (after cleaning LBE)



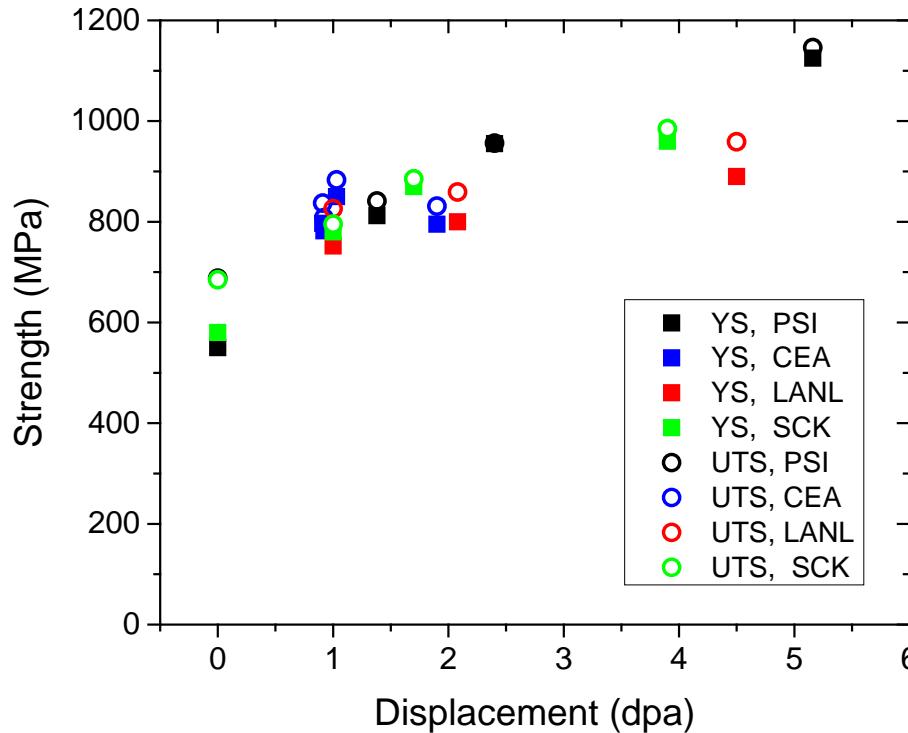




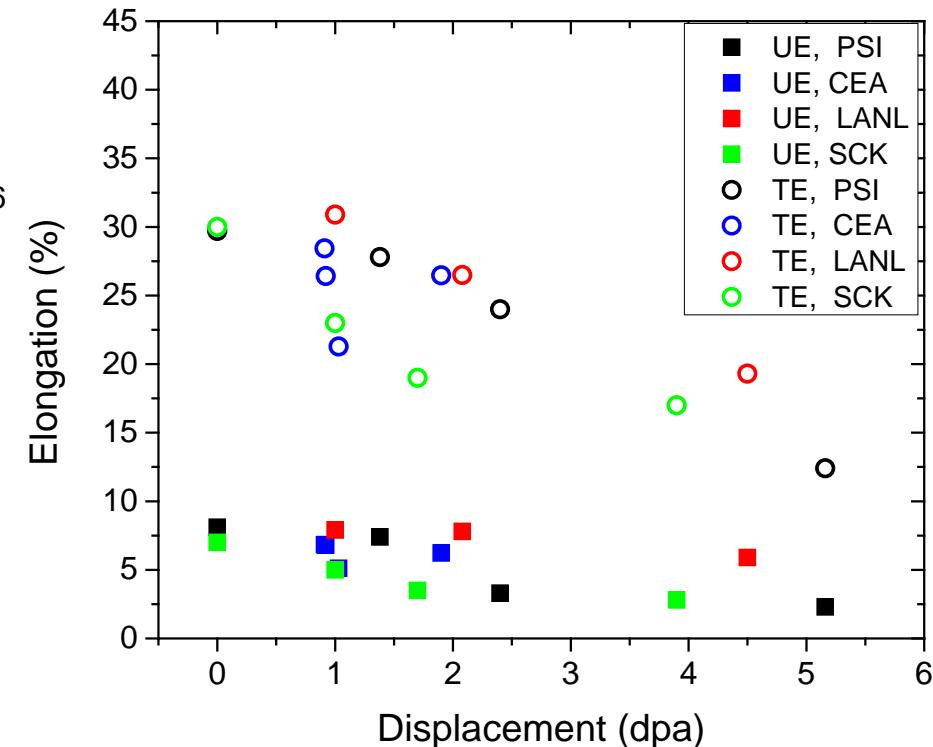


# Tensile test

T91 Calotte (after cleaning LBE)

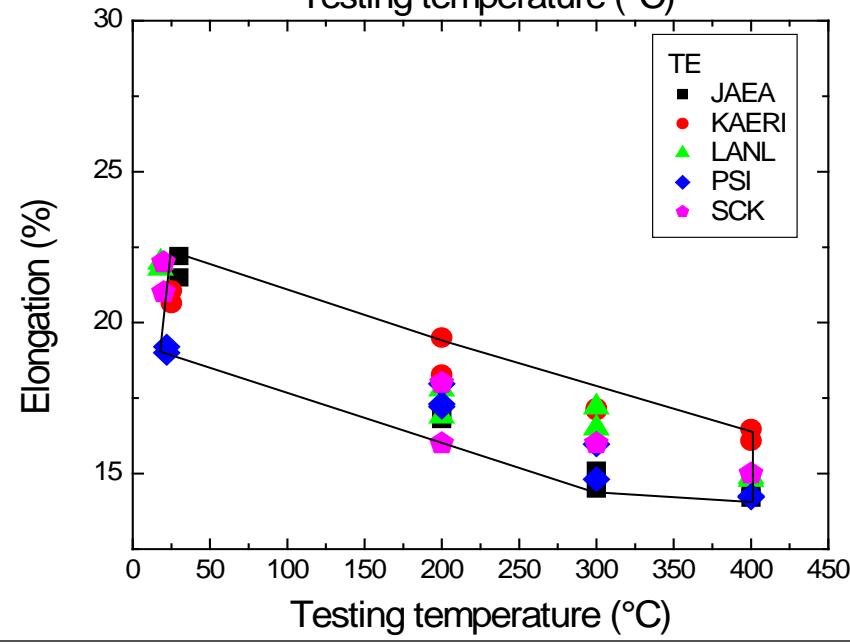
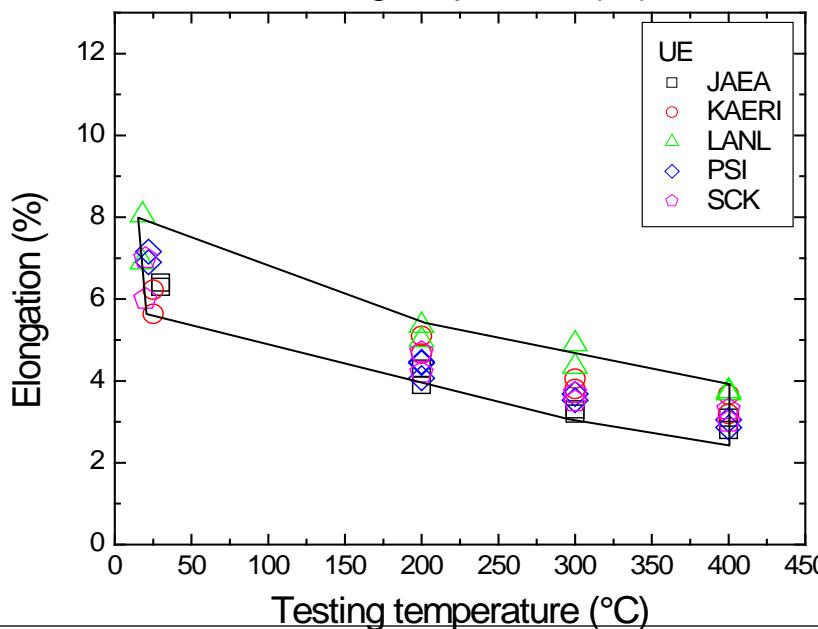
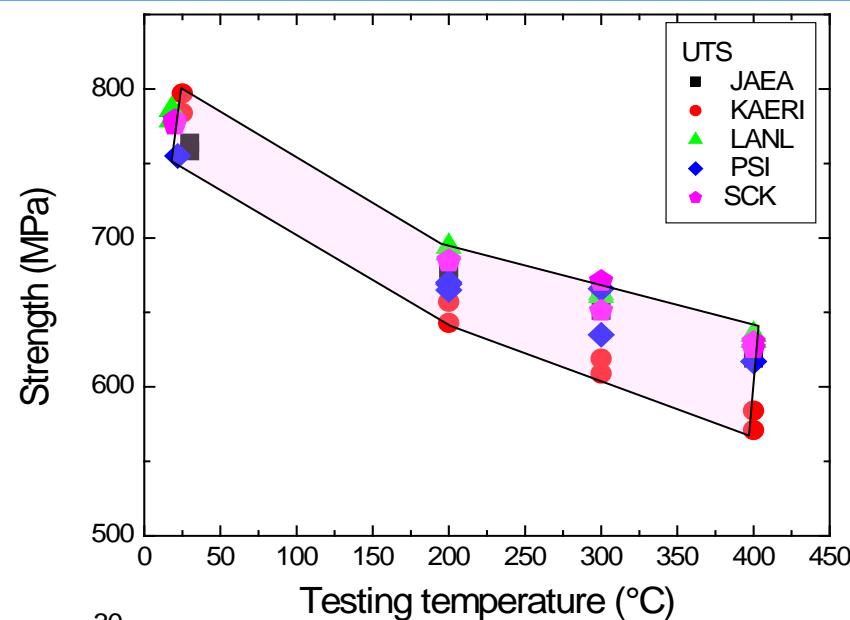
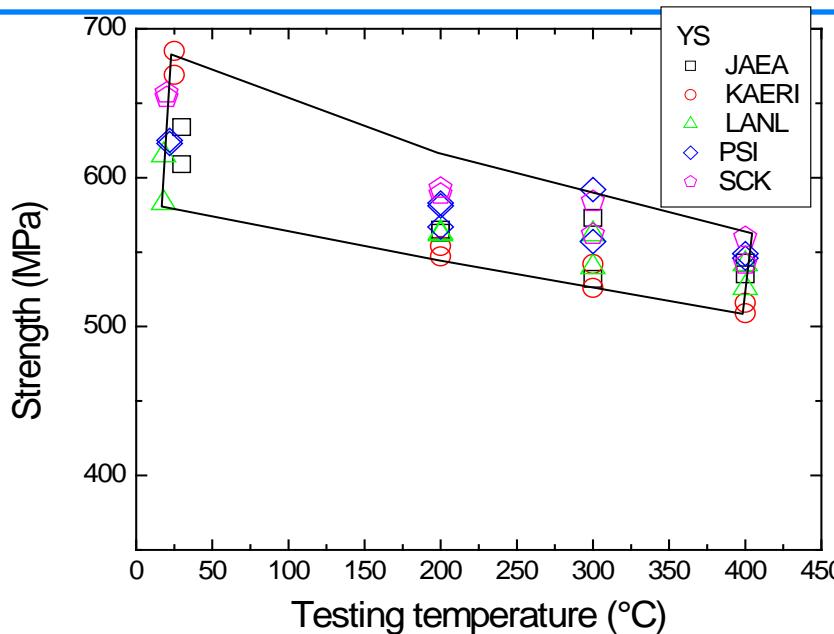


Tensile results obtained at RT

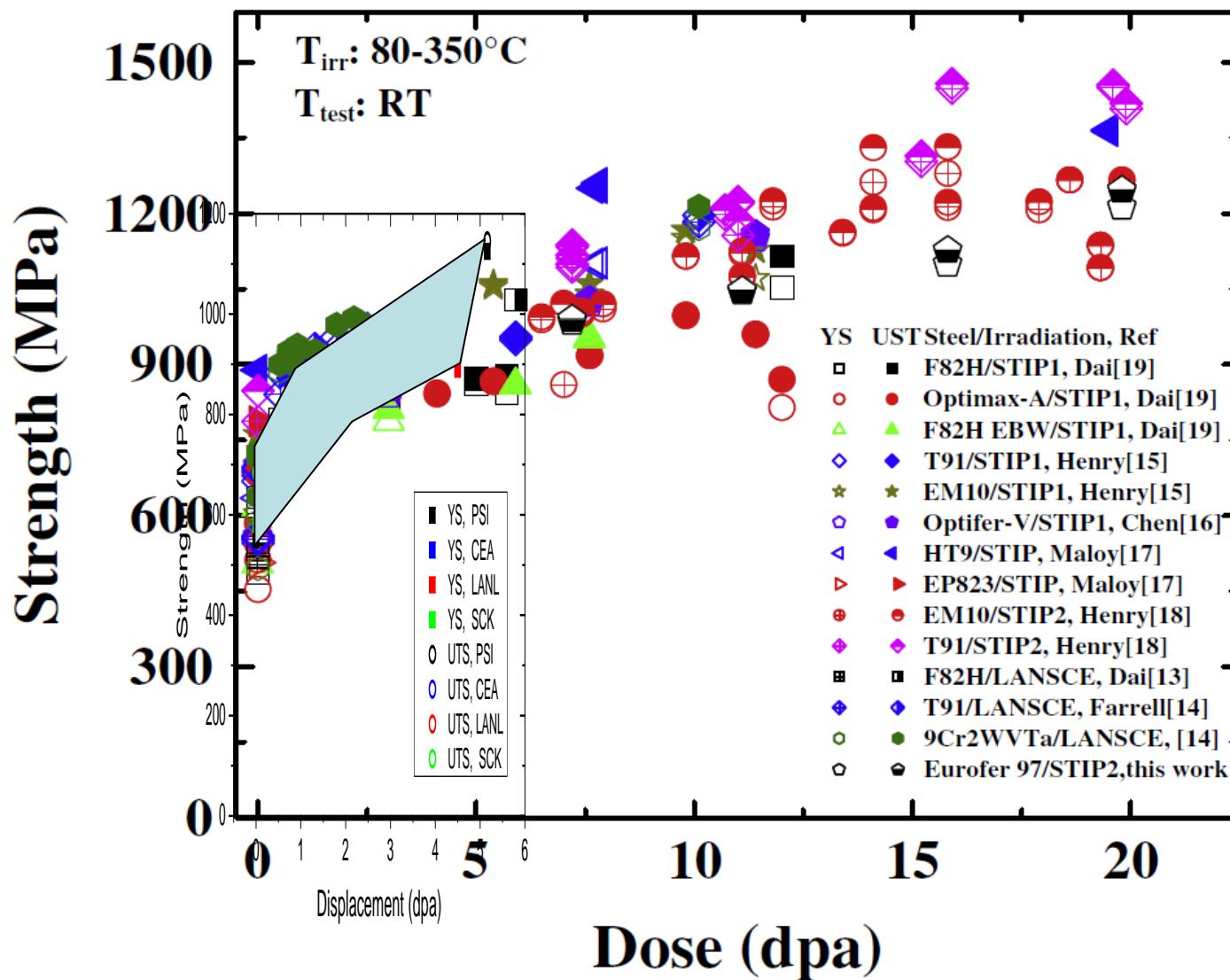


# Tensile test

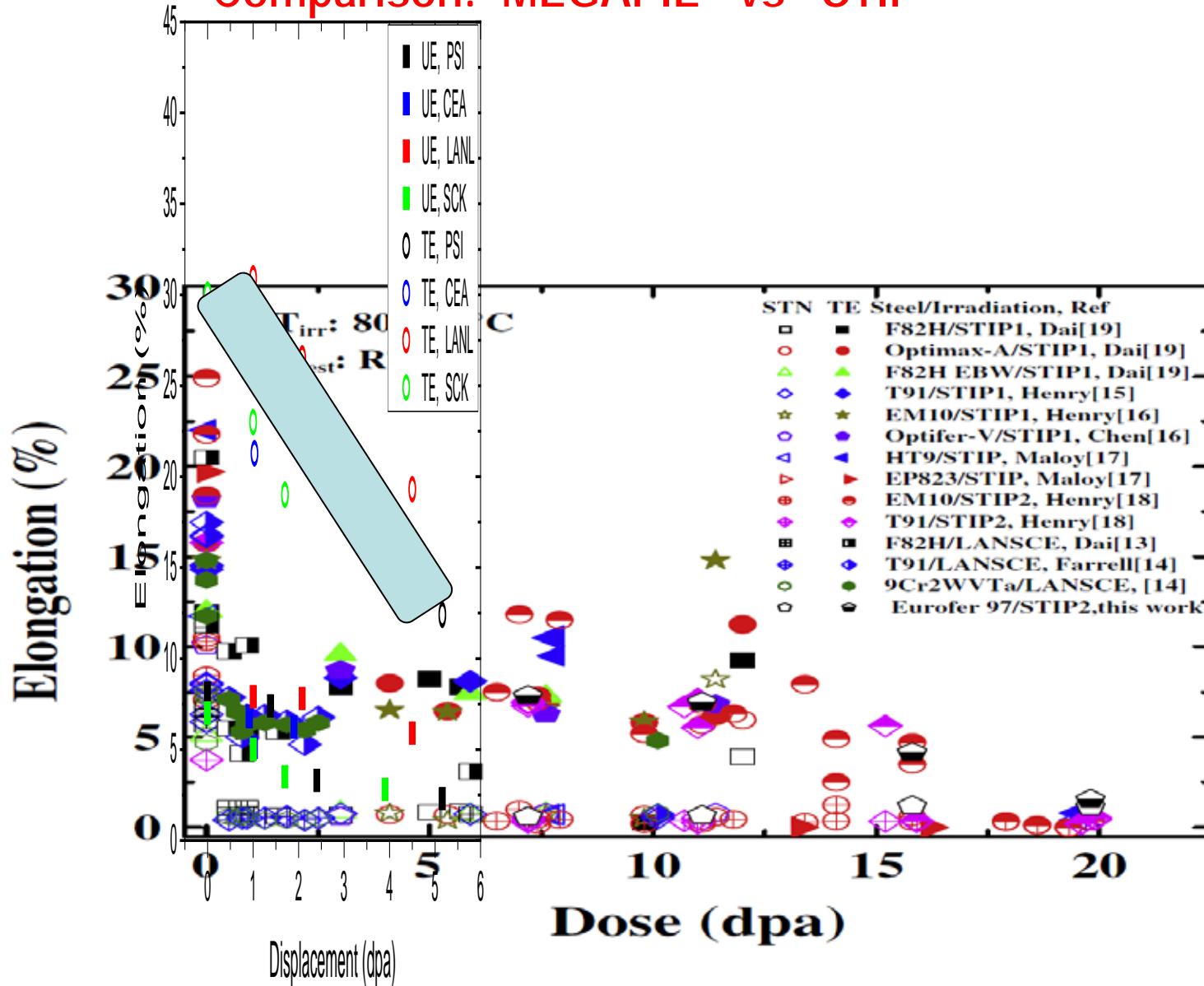
2<sup>nd</sup> RRTT on ref MEGAPIE T91

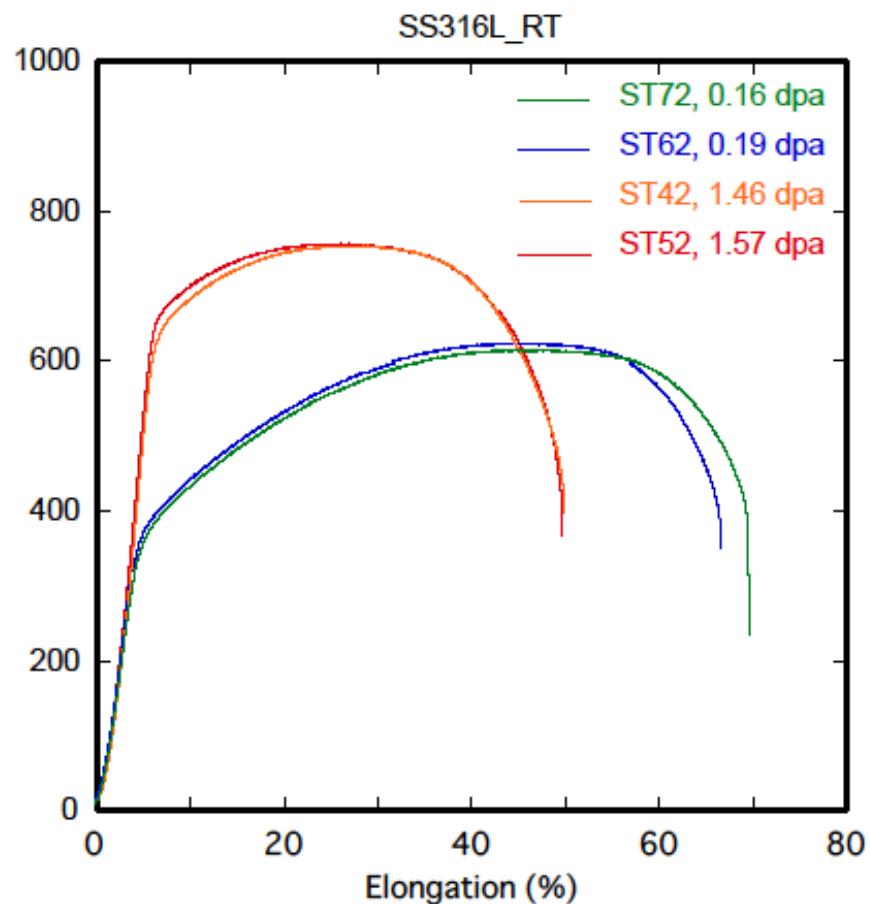
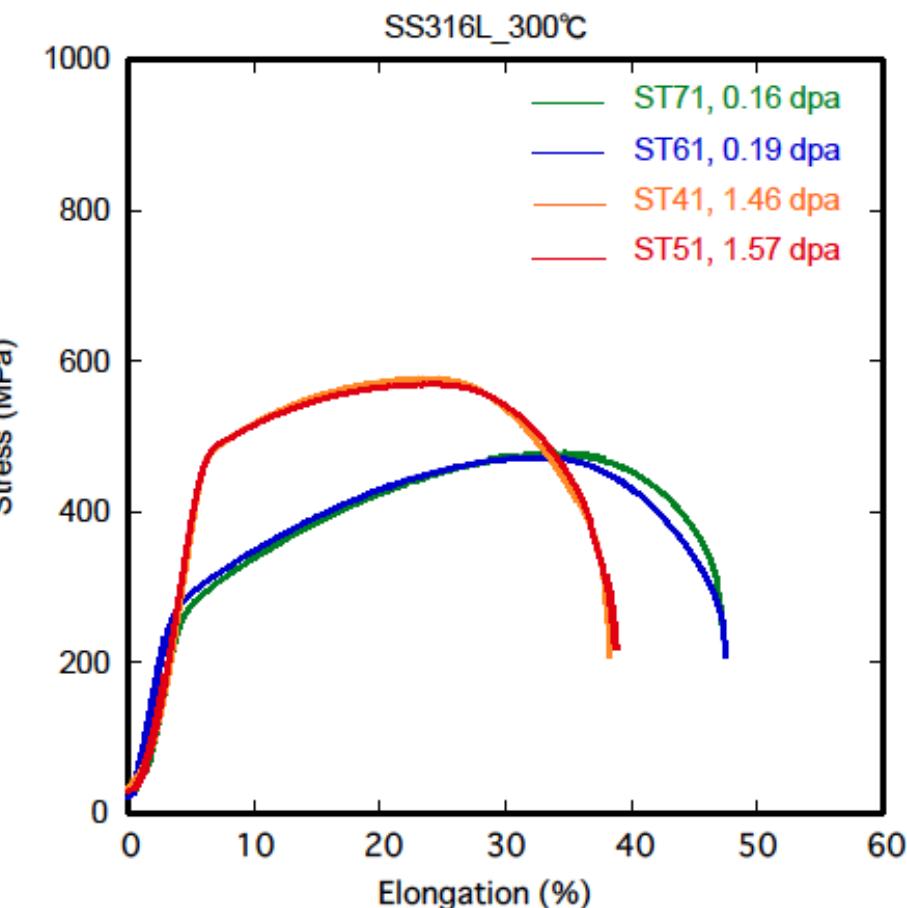


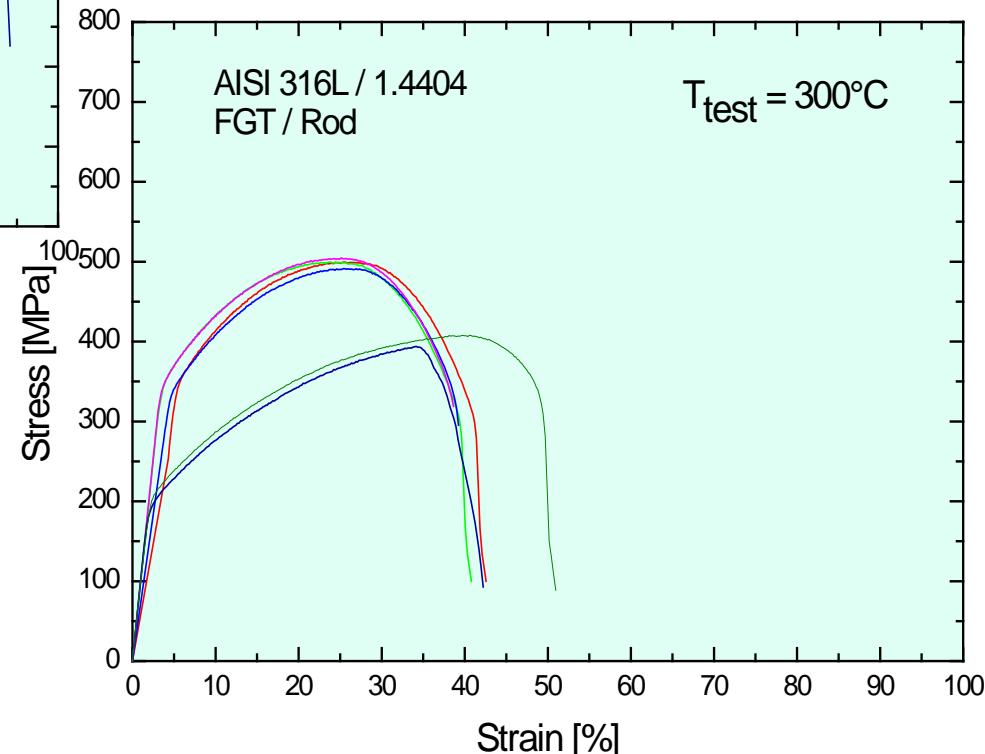
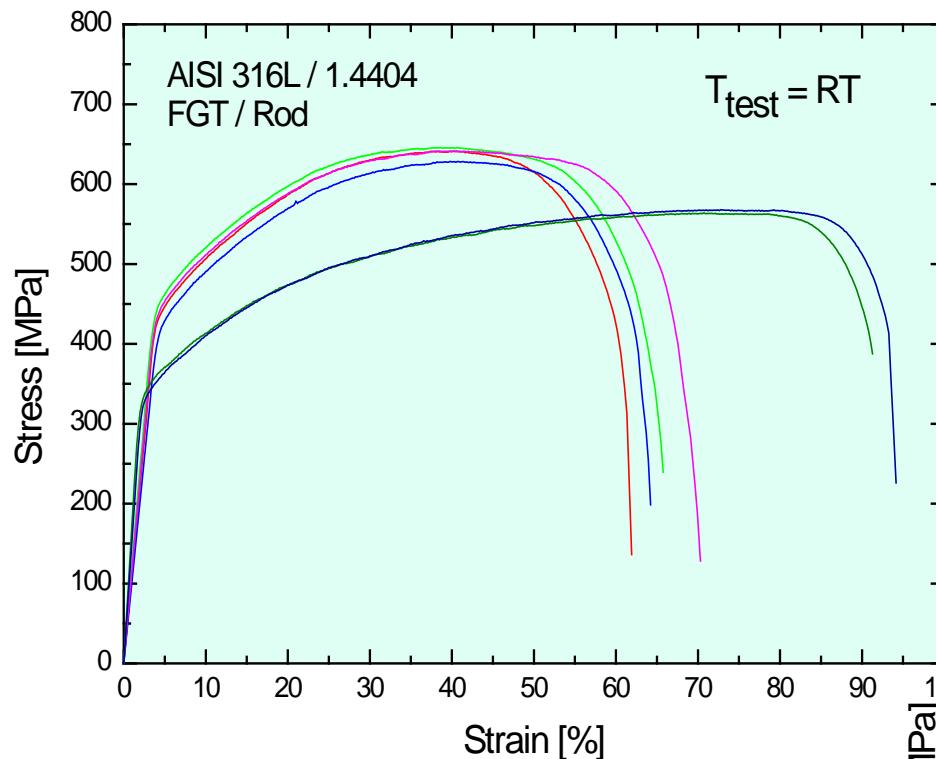
Comparison: MEGAPIE vs STIP

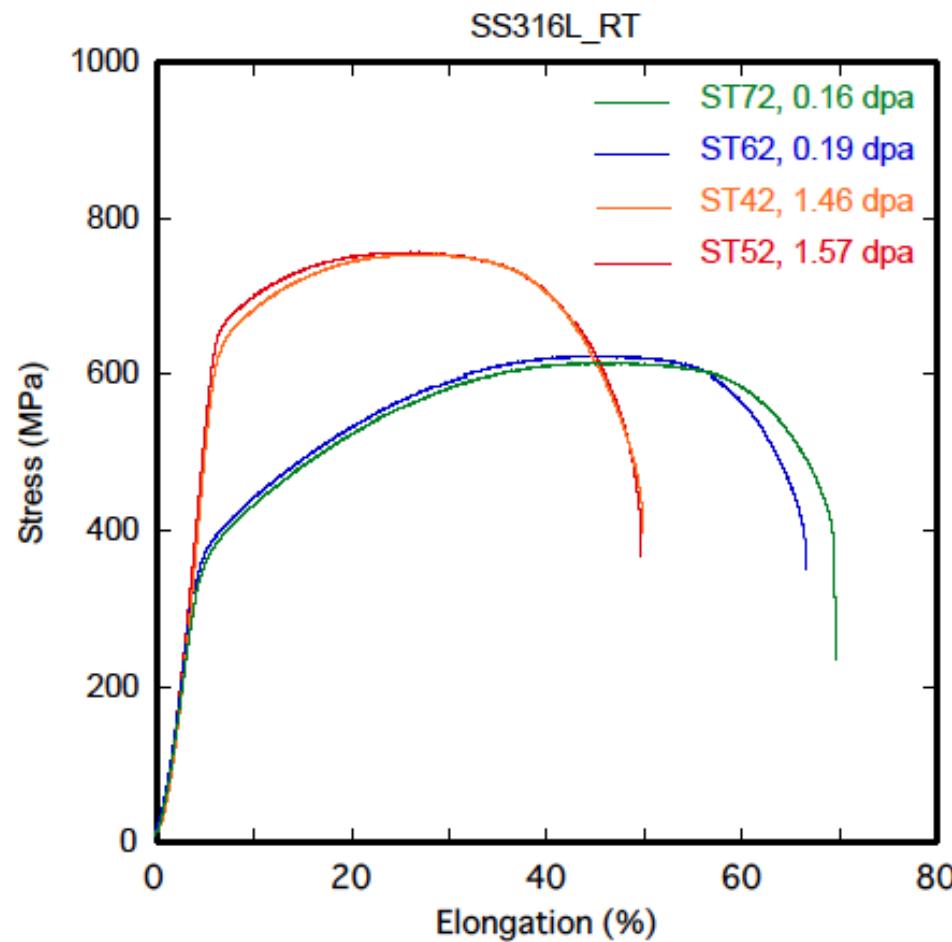
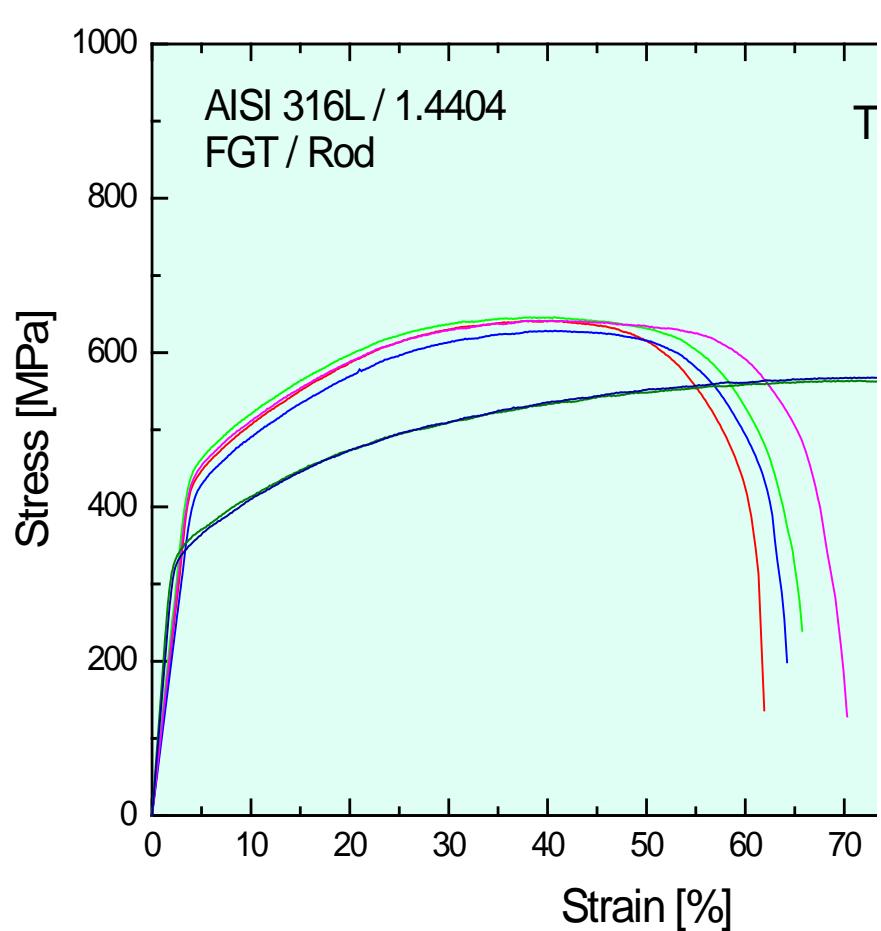


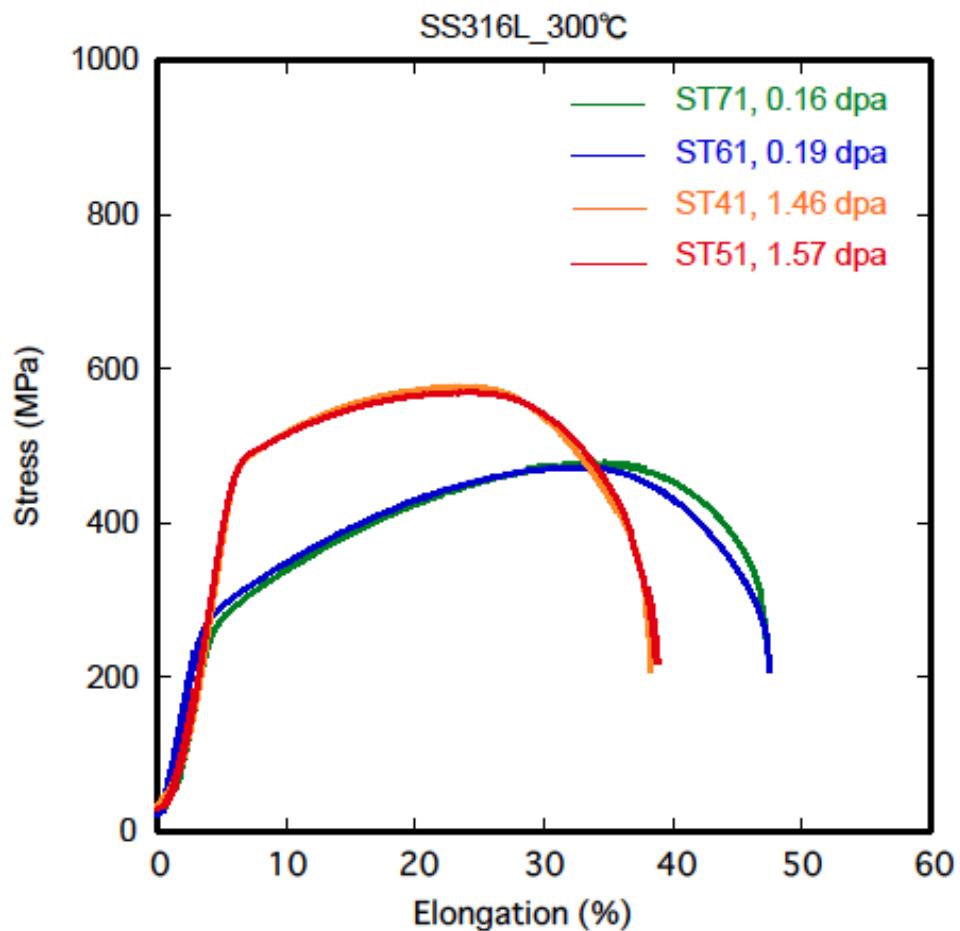
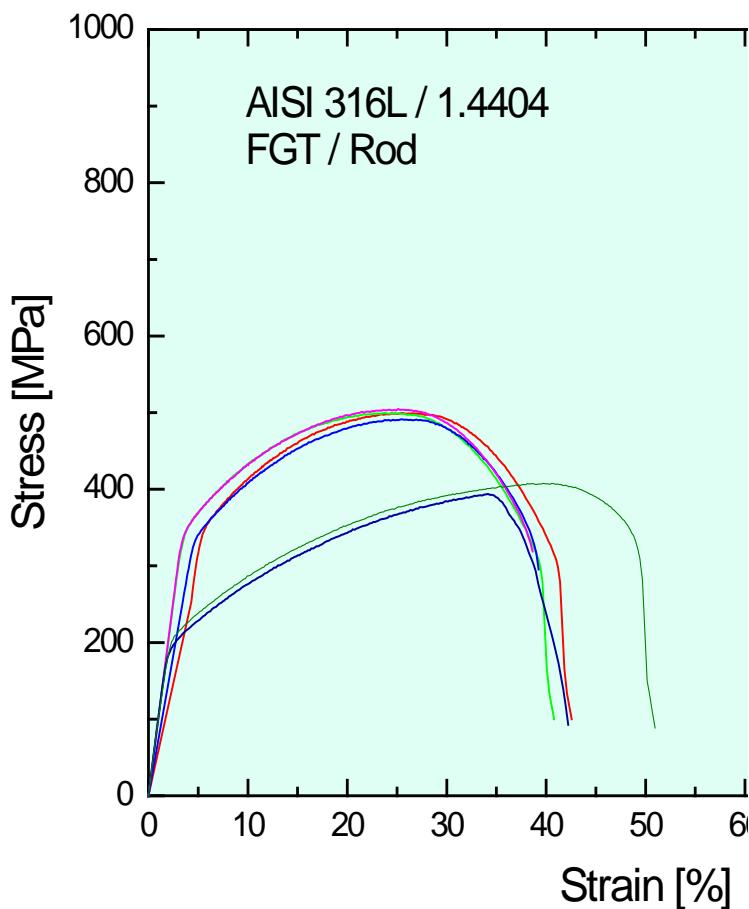
### Comparison: MEGAPIE vs STIP



RT tests300°C tests









## **Non-destructive test (NDT)**

- Gamma mapping of the Al-calotte
- Visual inspection of the T91 calotte
- Thickness measurement of the T91 calotte



## **Analysis of LBE samples**



## **Surface analyses**

- EPMA analysis
- SEM observation
- SIMS analysis



## **Microstructural analyses**

- TEM observation



## **Mechanical test**

- Tensile test
- Bend test
- Hardness measurement



## **Summary**

The PIE has been performed on the structural materials, T91 and SS 316L steels, irradiated in the lower part of the MEGAPIE target. The conclusions are:

- No evident failure and detectable change in thickness in the beam window area were observed.
  - Slight corrosion damage was detected on the inner surface of the beam window.
  - A slight misalignment was observed at the EBW of the lower liquid metal container.
  - Results of tensile test show the T91 steel in the beam window area persists significant ductility after irradiation.
- 
- Some additional PIE (mechanical (bend) tests and TEM investigations) are still needed.

# Report



**Grant Agreement N°: FP7-212175**

## DELIVERABLE D3.5

### Report on the MEGAPIE Target PIE

Y. Dai, V. Boutellier, R. Grabherr, B. Hammer, H.P. Linder, M. Martin,  
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*SCK-CEN, Boeretang 200, 2400 Mol, Belgium*

# Acknowledgement

MEGAPIE PIE is the last phase of the MEGAPIE project. Any results of the PIE include essentially the contributions of those who ever worked on the MEGAPIE project. There is an extensive list of contributors and we do not think that we could have a complete one. Nevertheless, we are appreciative of the invaluable contributions from: G. Bauer, F. Barbagallo, R. Brun, K. Clausen, S. Dementjev, E.M. Donegani, M Dubs, T. Dury, R. Erne, D. Gavillet, K. Geissmann, H. Glassbrenner, F. Groeschel, S. Hahl, G. Hauswirth, H. Heyck, F. Heindrich, A. Janett, X. Jia, S. Joray, A. Kalt, D. Kuster, A. Lagotzki, H. Leu, W. Leung, J. Ledermann, B. Long, E. Manfrin, P. Ming, J. Patorski, C. Perret, A. Rehmann, M. Reuning, K. Samec, R. Schwarz, H. Schweikert, B. Sigg, B. Smith, A. Spahr, P. Stiller, A. Strinning, P. Suter, S. Teichmann, R. Thermer, K. Thomsen, D. Viol, G. Von Holzen, W. Wagner, H. Wiese, L. Zanini, U. Zimmermann, C. Zumbach who work or previously worked at PSI; L. Cahhon, J.-L. Courouau, G. Laffont, S. Leray, P. Roubin, A. Terlain who work or previously worked at CEA; T. Auger, A. Cadiou, Y. Foucher, J. M. Gauthier, D. Gorse, A. Guertin, T. Kirchner, M. Krauth, A.C. Mueller, P. Pariset, I. Serre, J.S. Stutzmann, J.-B. Vogt who work or previously worked at CNRS; P. Agostini, A. Ciampichetti, A. Gessi, D. Pellini, P. Turroni, A. Zucchini who work or previously worked at ENEA; Y. Ikeda, K. Kikuchi, Y. Kurata, S. Saito who work or previously worked at JAEA; K. Kim, J.W. Park, T.Y. Song who work or previously worked at KAERI; H. Jacobs, J. Knebel, J. Konys, R. Stieglitz who work or previously worked at KIT; F. Goldner, S.A. Maloy, E. Pitcher, T. Sahel, K. Woloshun who work or previously worked at DOE / LANL. A. Almazouzi, P. D'hondt, M. Dierckx, P. Kupschus who work or previously worked at SCK-CEN. We would also like to thank the colleagues who work at ZWILAG for cutting the MEGAPIE target, the colleagues in IPUL (Latvia) for manufacturing the electro-magnetic pumps and the colleagues in ATEA for manufacturing the MEGAPIE target.



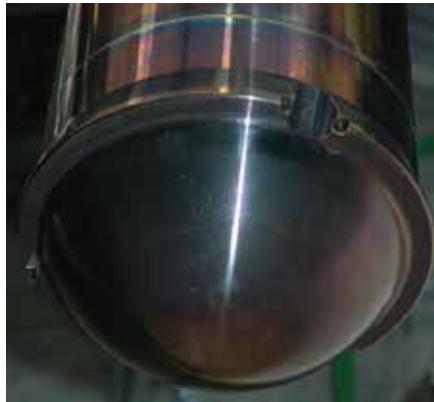
Thank you !

Good  
results  
?!

What  
do you  
think ?

Well, I  
am not  
sure.

# PIE on beam window



	Corro-sion + erosion	Embrittle-ment (irr., He, LBE)	Mechani-cal change	Microstru-ctural change	Chemic-al change	Association (number of conditions)
OM + m hardness + SEM/EPMA	X	X (mhard-ness)				CEA (2 cond) LANL (2 cond) PSI (2 cond) JAEA (2 cond), KAERI
TEM / FEGSTEM		X	X	X	X	CEA (2 cond) FZK (3 cond) PSI (2 cond) JAEA (2 cond), KAERI
H and He analyses		X				PSI (3 cond)
SIMS / XPS	X				X	PSI (SIMS:2cond) SCK (XPS:1cond)
XRD	X				X	CEA (2 cond)
Tensile test		X	X			CEA (2 cond) LANL (2 cond) PSI/ENEA(3cond)* KAERI
Bending test		X	X			LANL (3cond) PSI (3 cond)*
Small punch test **		X	X			CEA (2 cond) PSI (2 cond) CNRS (3 cond)*

\* Tests to be done in both Ar (or Air) and LBE environments.

\*\* CNRS will use 8.9 mm diameter discs and the others will use 3 mm diameter discs.

Sample H02-1-1-Zug1, 6.1 dpa, 310-333 ° C, tested at 300 ° C

