

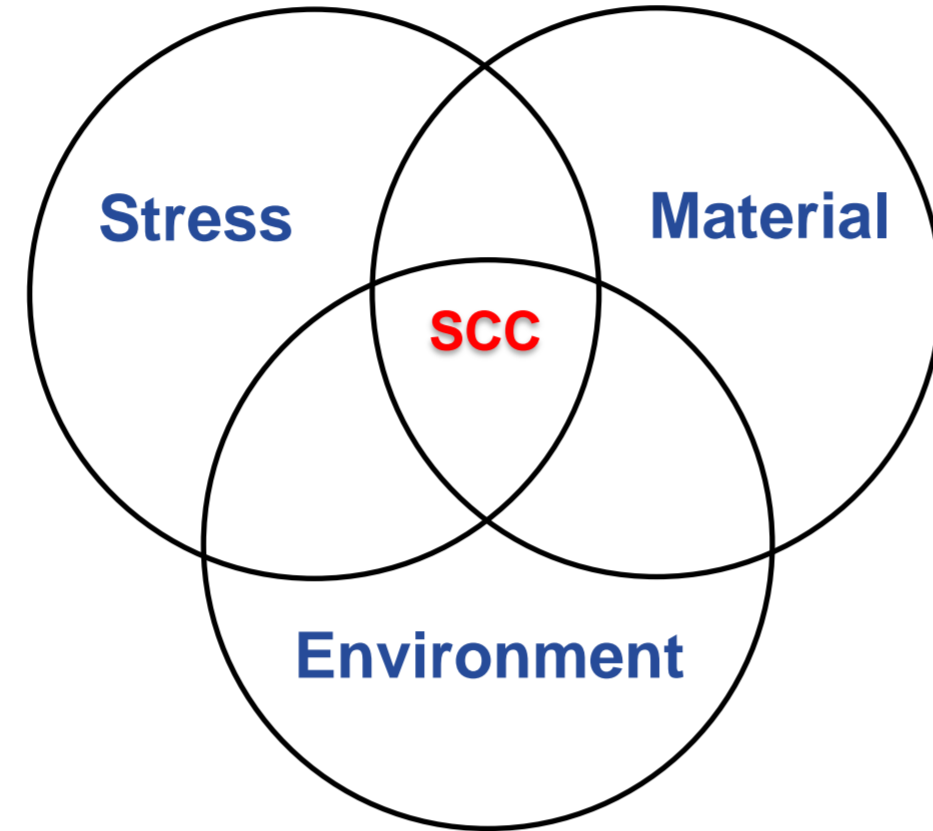
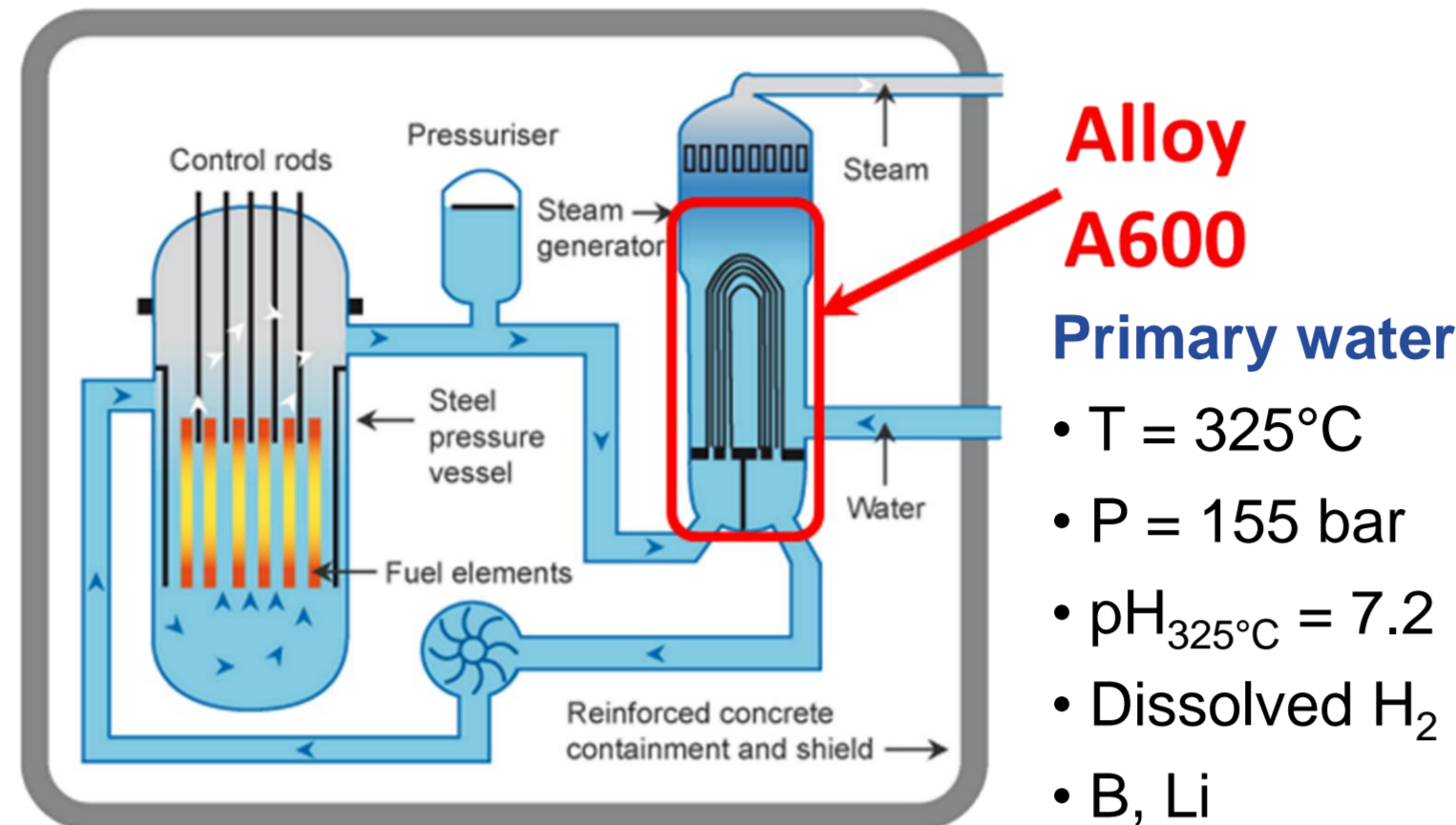
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² CEA, DEN, DPC, SCCME, Laboratoire d'Etude de la Corrosion Aqueuse, FR-91191 Gif-sur-Yvette, France

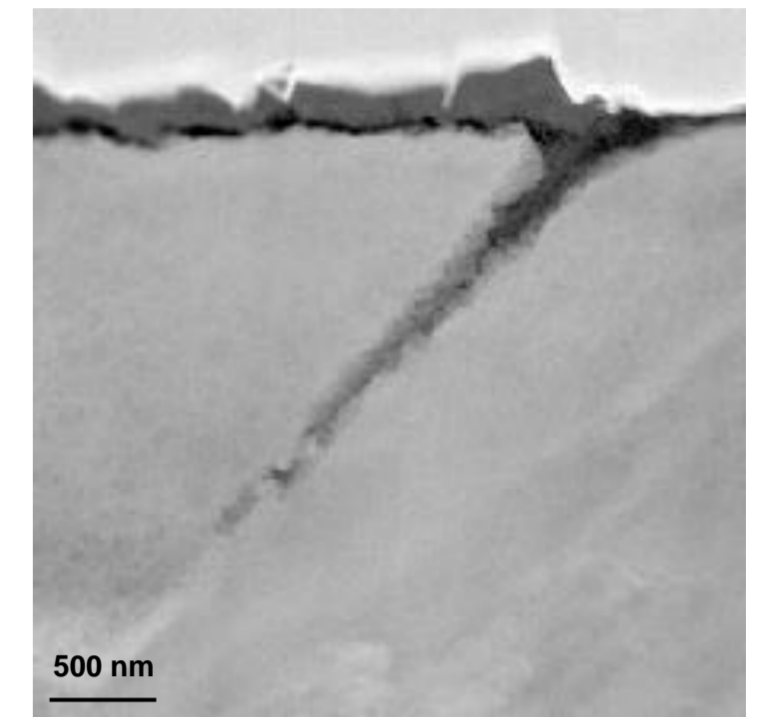
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Context : - There are plans to extend the lifetime of pressurised water reactors (PWRs) of French nuclear power plants up to 60 years.
- Alloy 600 (Ni-16Cr-10Fe) provides a good overall corrosion resistance, but is susceptible to stress corrosion cracking (SCC).

A Typical Pressurized Water Reactor (PWR)



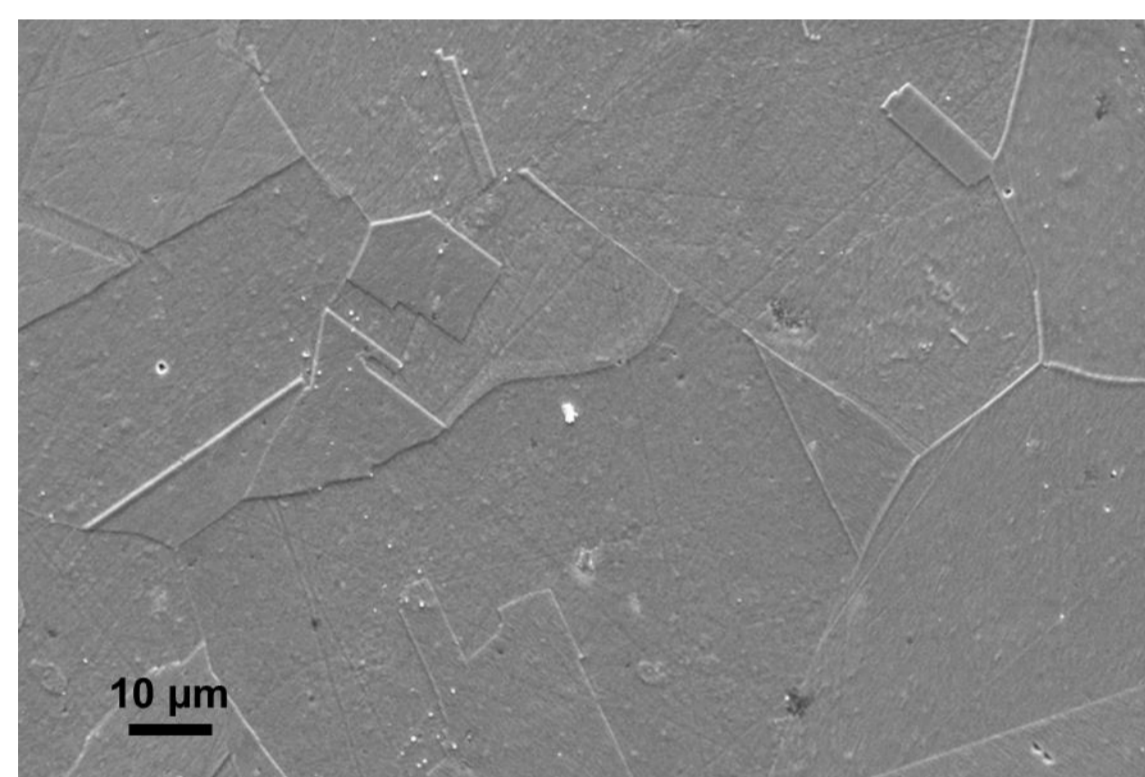
- SCC is the main degradation phenomenon of PWRs.
- Intergranular oxidation is a key step in SCC initiation.
- Chromium carbides at the grain boundaries showed themselves beneficial in terms of resistance to SCC by reducing the oxidation progress.



Objective : Understanding the effect of intergranular Cr carbides on intergranular oxidation kinetics

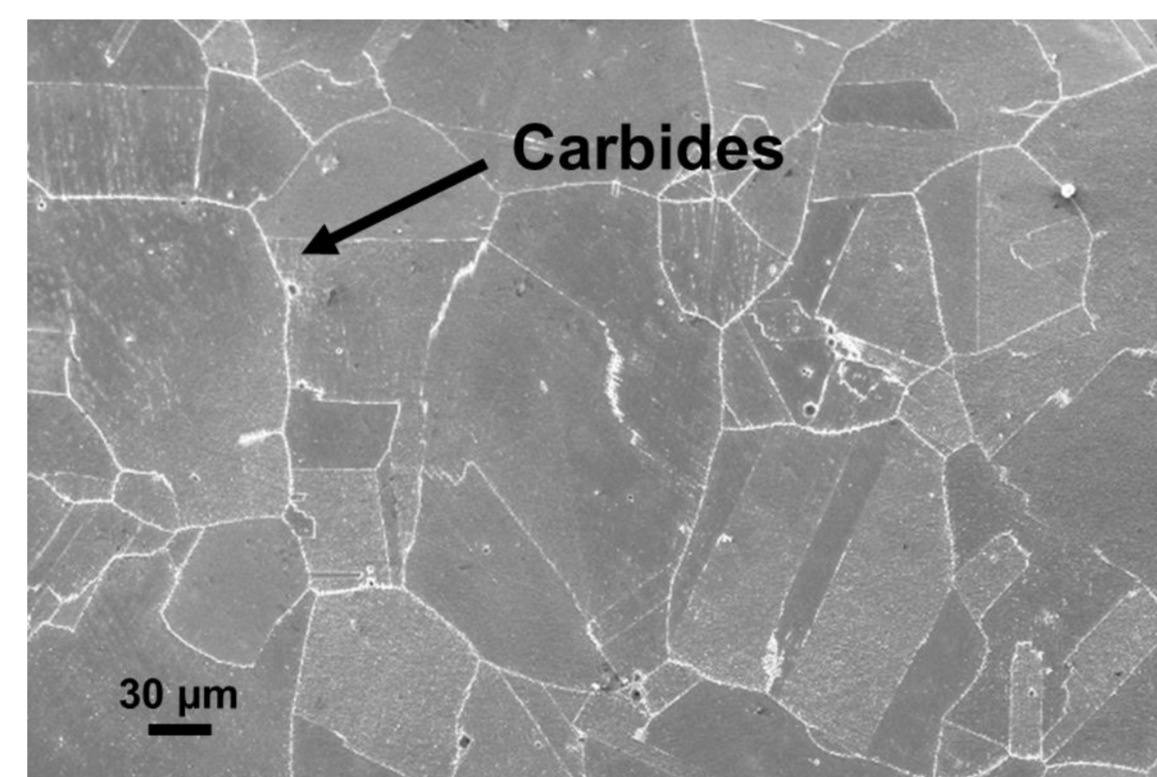
Materials : 2 model samples have been prepared from WF675

Sample SA "Solution Annealed"



1050 °C for 1 h + water quenching

Sample SA carbides (with an important intergranular precipitation)



1050 °C for 1 h + water tempering + tempering at 700 °C for 16 h

Grain boundary oxidation for 1400 h in autoclave :

P = 120 bar, 325 °C, [H₂] = 30 mL (TPN)/Kg H₂O (200 mbar à 325 °C), H₃BO₃ (1000 ppm), LiOH (2.2 ppm)

3D SEM/FIB electron tomography :

Image acquisition :

Oxidation depth and path along grain boundaries have been characterised by 3D electron tomography using a FEI Helios 660 SEM/FIB with AutoSlice & View G3.

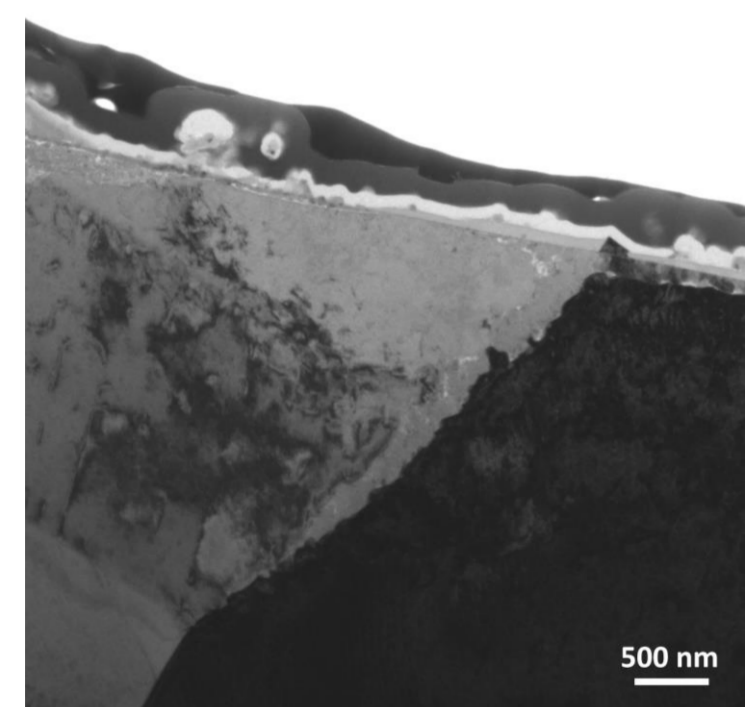


	Sample SA	Sample SA carbides
Number of slices	640	800
Voxel (nm)	13x13x20	10x10x10
Image acquisition parameters	HV 5 kV, beam current 0.1 nA, Non-immersion mode, Everhart-Thornley detector.	HV 5 kV, beam current 0.2 nA, Immersion mode, TLD detector in SE mode.
Image type		

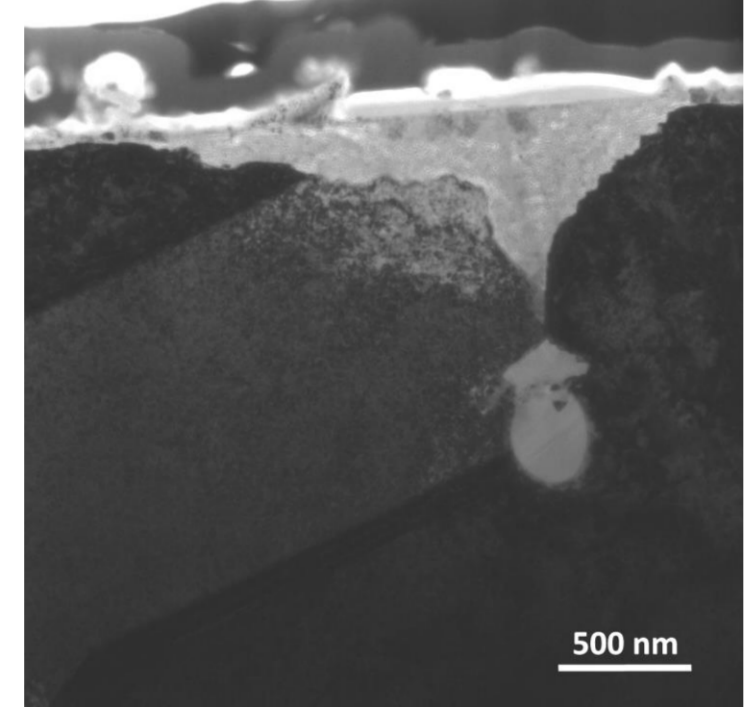
Analytical transmission electron microscopy :

Performed on FIB-lamellae, using a FEI Tecnai F20-ST TEM.

TEM



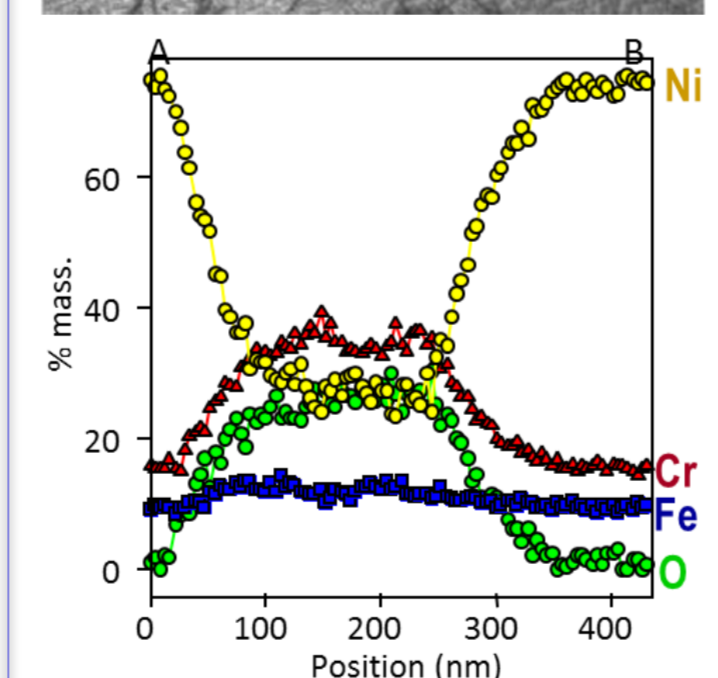
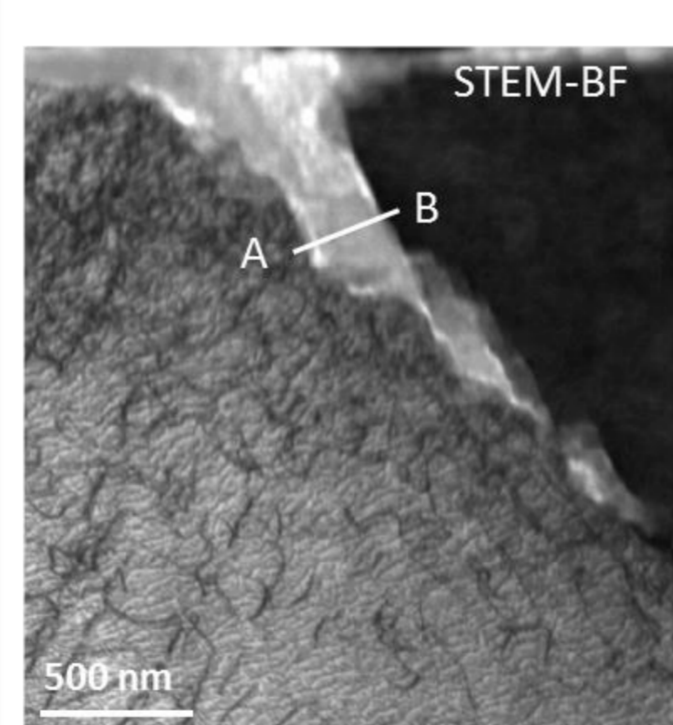
Oxide penetration without Cr carbide



Oxide penetration with Cr carbide

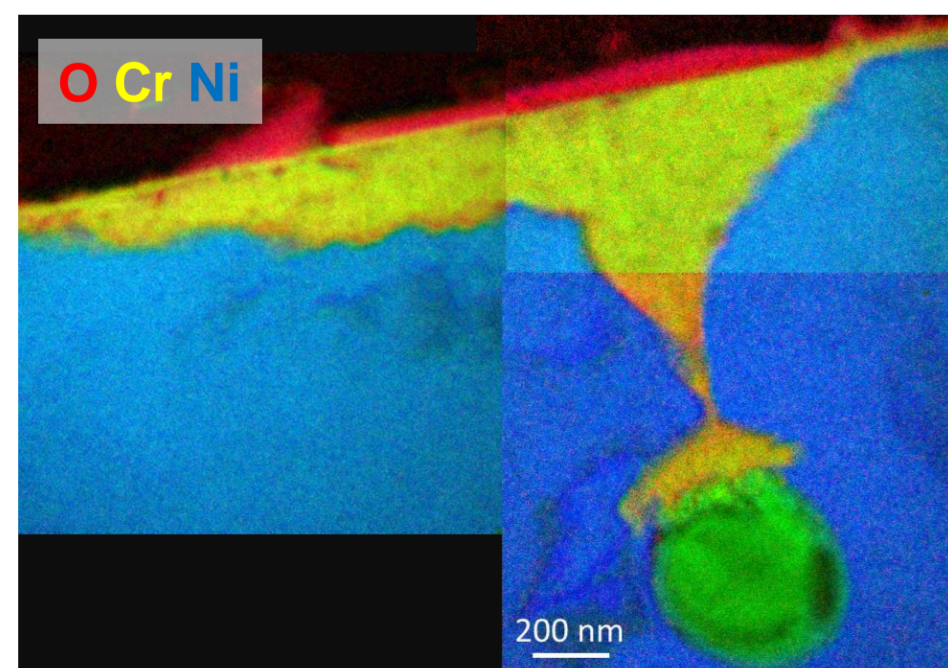
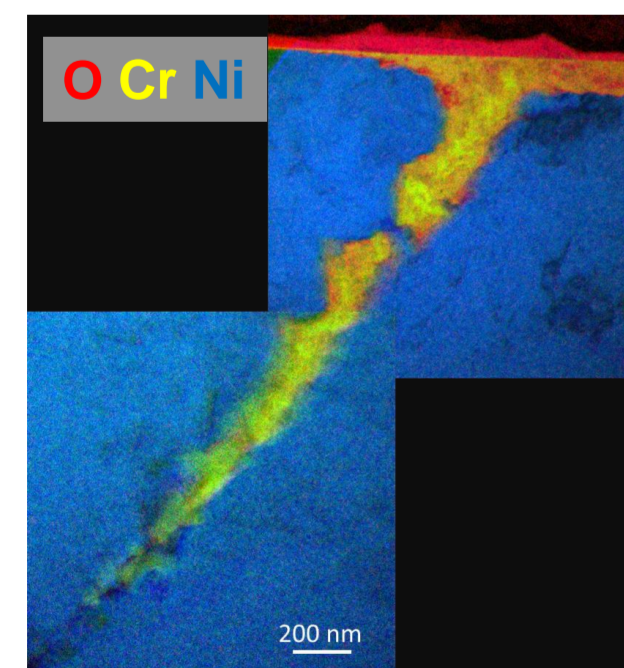
[011] zone axis in right hand side grain

STEM-EDS



Carbides : mostly Cr₇C₃
Oxides : Cr-rich in the penetration

EFTEM

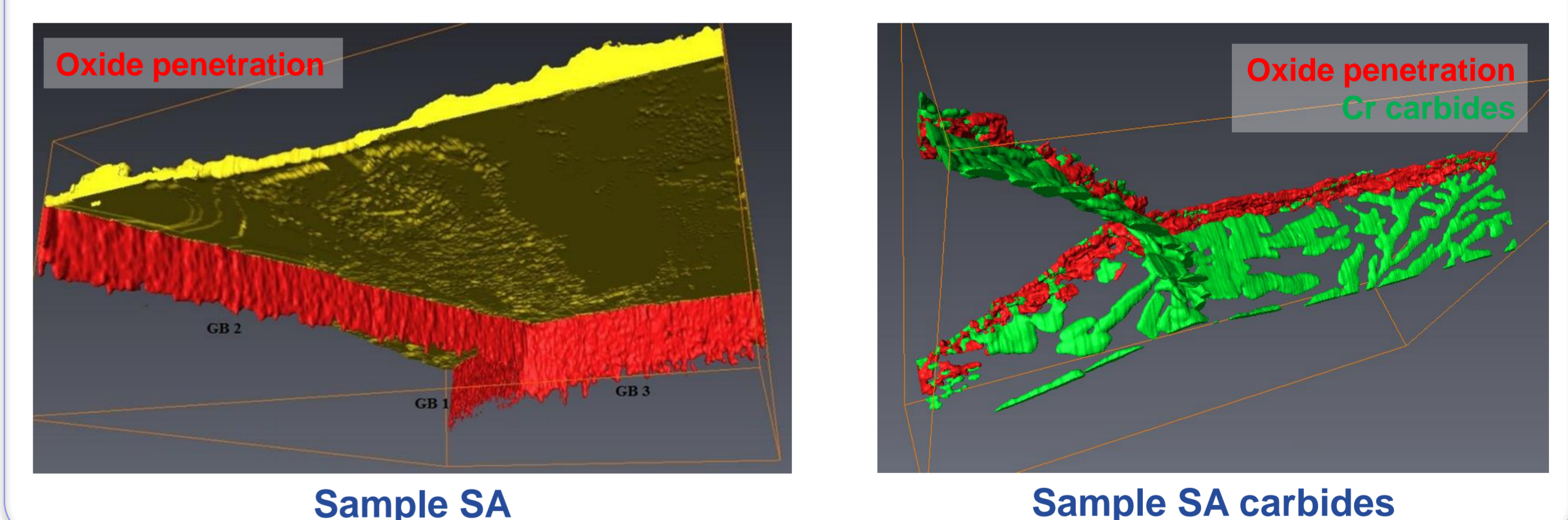


Conclusion :

- Sample SA : long slim Cr-rich oxide penetration of ~ 2 μm.
- Sample SA carbides : broad Cr-rich oxide penetration stopped by the Cr₇C₃ carbides and forming a cap.

3D reconstruction in Avizo Fire :

Alignment, tilt correction, contrast/brightness normalisation, filtering (smoothing, edge enhancement) followed by segmentation.



Conclusion :

- Sample SA : oxide depths along grain boundaries are even and continuous of ~ 2 μm. GB2 is the shallowest oxide penetration, while GB1 and GB3 have the same depth. GB1 oxide penetration is porous and discontinuous.
- Sample SA carbides : oxide depths are much shallower, uneven and wider along grain boundaries than the previous sample. Cr carbides stop the ingress of oxide penetration.

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