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Chromium depleted zone in nickel base alloy : experimental study of chromium diffusion between 350°C and 500°C

J. Nguejio, J. Crepin, C. Duhamel, C. Guerre, F. Jomard

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HAL Authorization

Josiane NGUEJIO ^{1,2}, Jérôme CREPIN ², Cécilie DUHAMEL ², Catherine GUERRE ¹, François JOMARD ³

¹ : Den-Service de la Corrosion et du Comportement des Matériaux dans leur Environnement (SCCME), CEA, Université Paris-Saclay, F-91191, Gif-sur-Yvette, France

² : MINES Paristech, PSL - Research University, Mat - Centre Des Matériaux, CNRS UMR 7633, BP 87, 91003 Evry, France

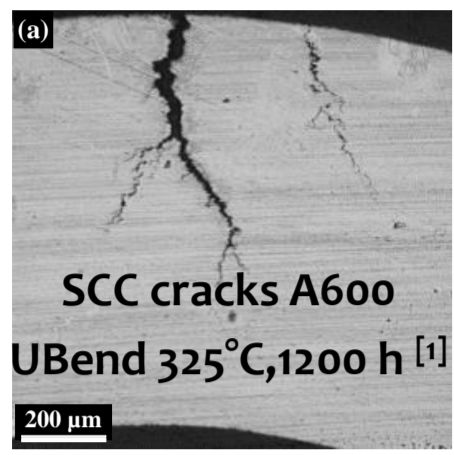
³ : Laboratoire GEMaC, UMR 8635 CNRS - Université de Versailles - 45 Avenue Des Etats-Unis, 78035 Versailles Cedex, France

CONTEXT AND OBJECTIVES

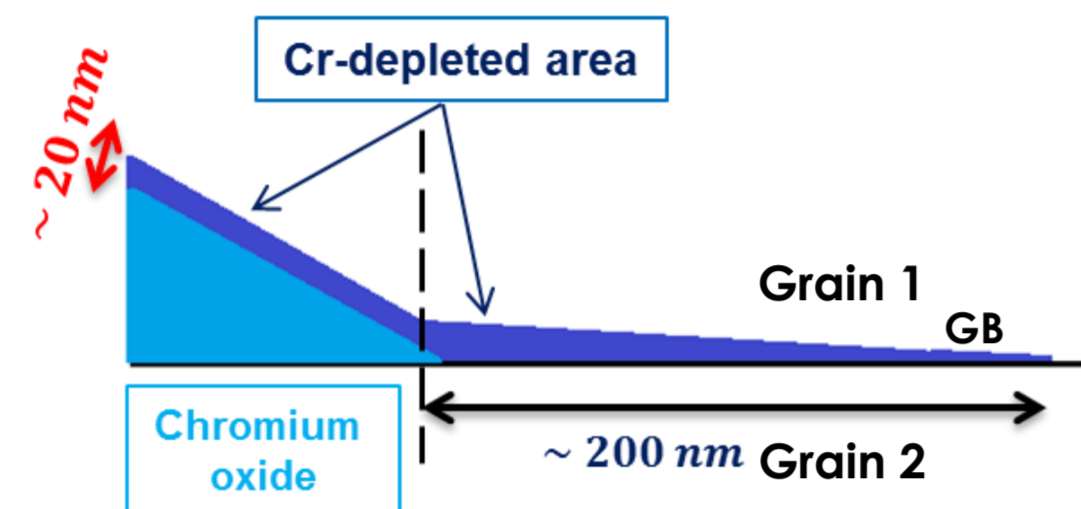
Industrial context

Stress Corrosion Cracking (SCC)

Steam generator tubes : Nickel Base Alloy A600 -> Ni-15Cr-Fe

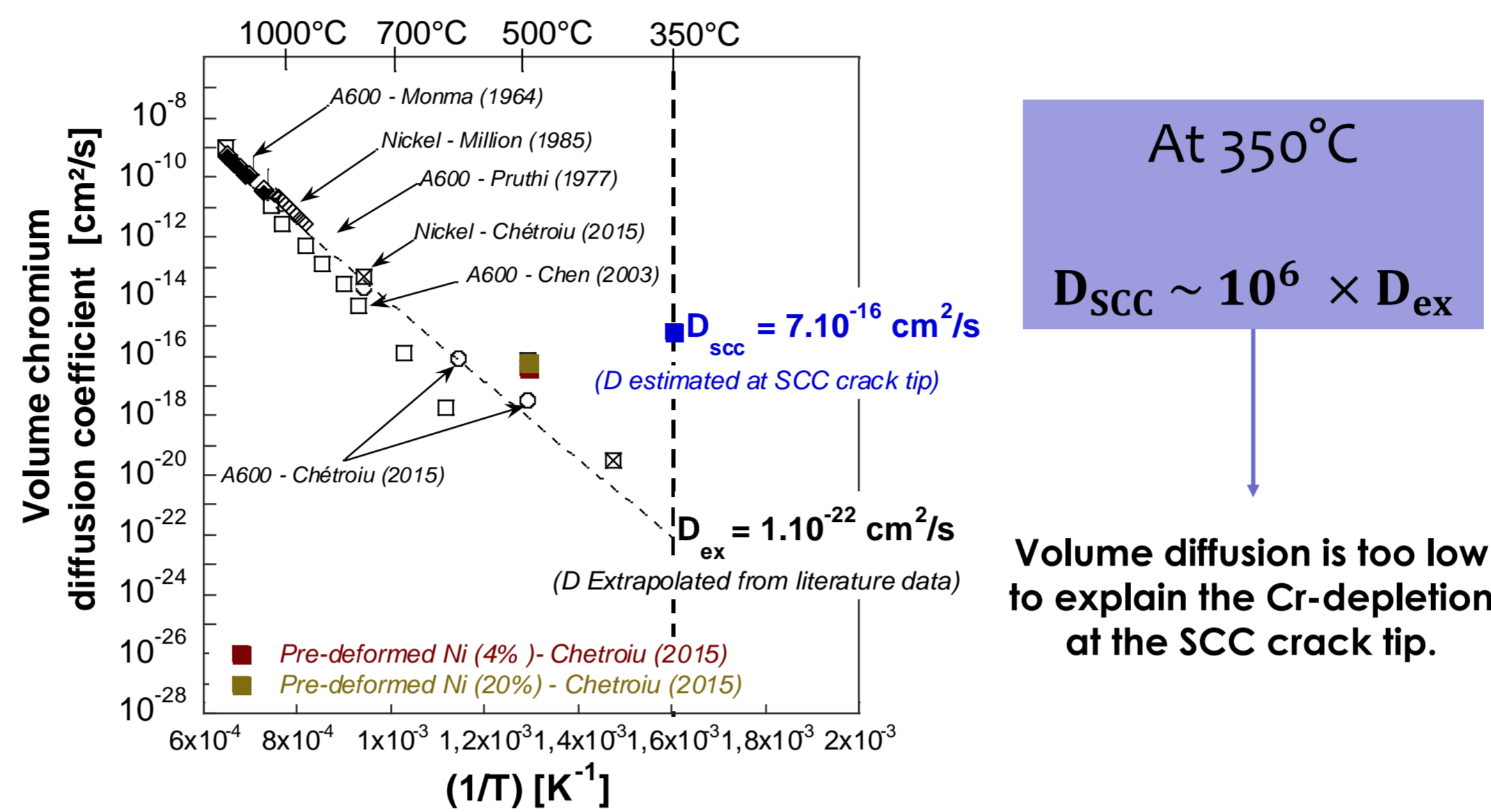


SCC crack tip [1] : Oxidation and/or Cr-depleted area leads to grain boundary embrittlement. The crack propagates until the end of the depleted zone.



Scientific context

Chromium diffusion : a key step of the SCC mechanism



At 350°C
 $D_{SCC} \sim 10^6 \times D_{ex}$

Volume diffusion is too low to explain the Cr-depletion at the SCC crack tip.

Objectives

- ✓ To determine which mechanism explain the formation of the chromium-depleted area in the vicinity of grain boundary at low temperatures (350°C), in order to understand the propagation mechanism of the SCC crack.

Assumptions

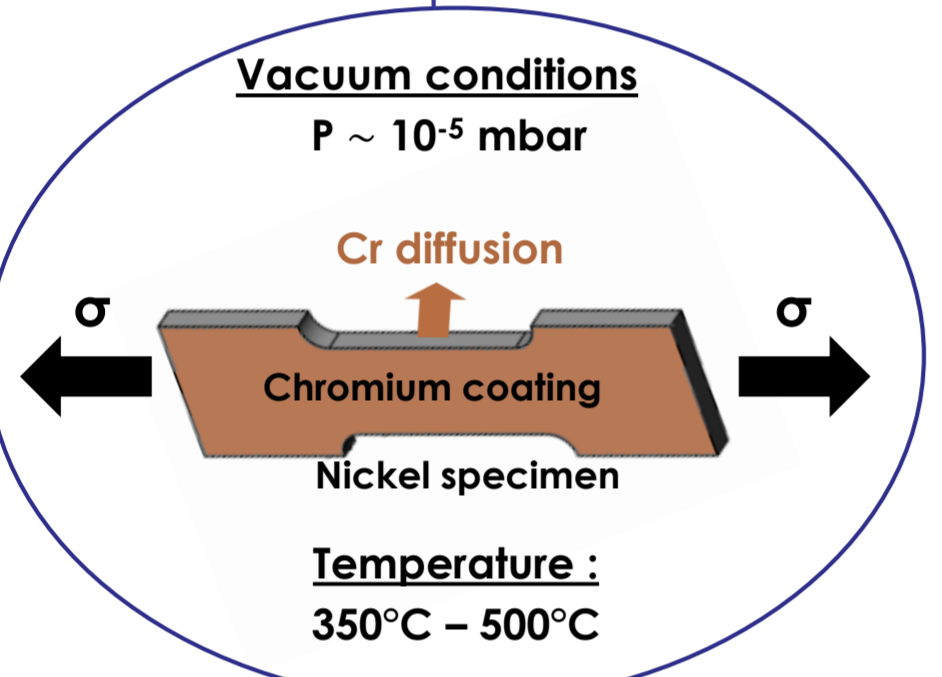
1. Plasticity-enhanced diffusion
2. Diffusion induced grain boundary migration (DIGM).

PLASTICITY ENHANCED DIFFUSION OF CHROMIUM IN PURE NICKEL

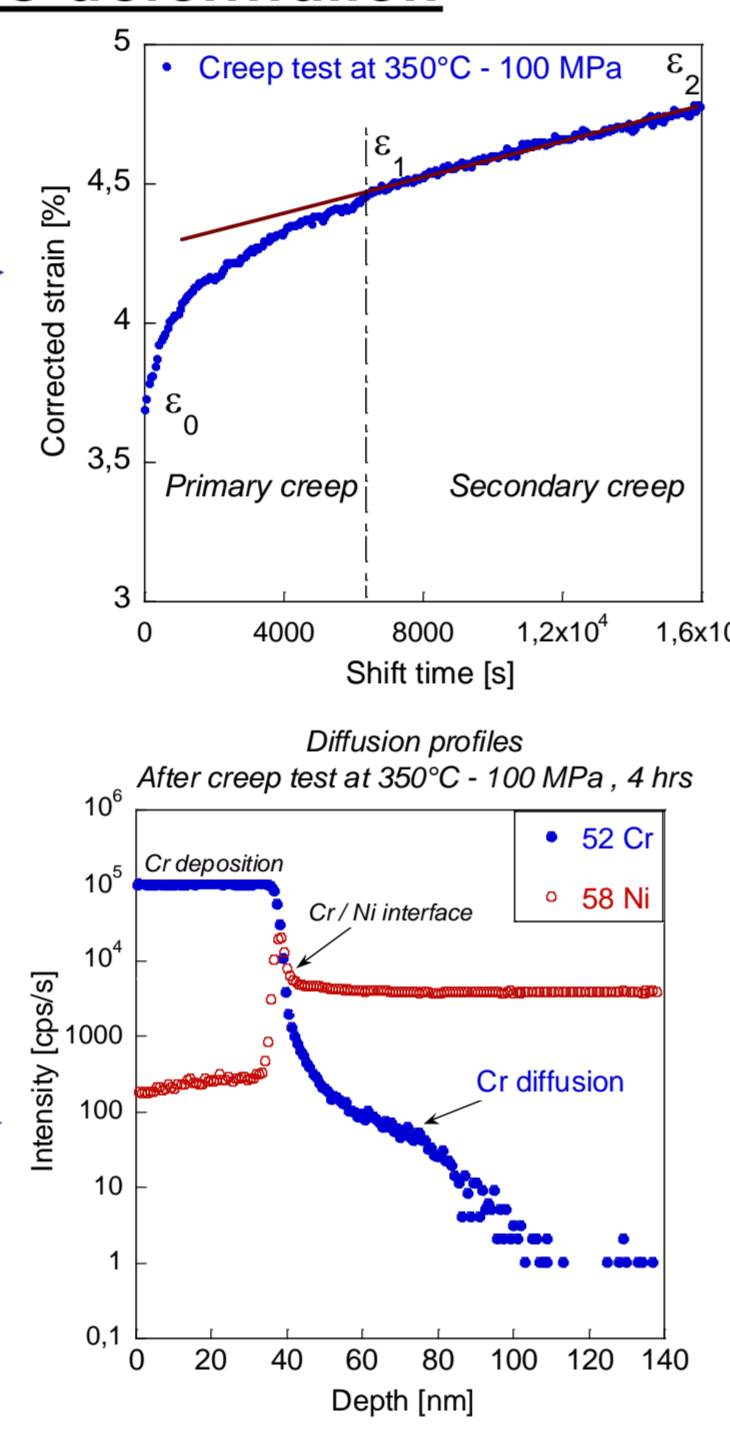
Experimental approach

Both diffusion and plastic deformation

Mechanical measurements during creep tests (Average strain rate)



Diffusion profiling by SIMS (CAMECA IMS 7F - GEMaC laboratory)

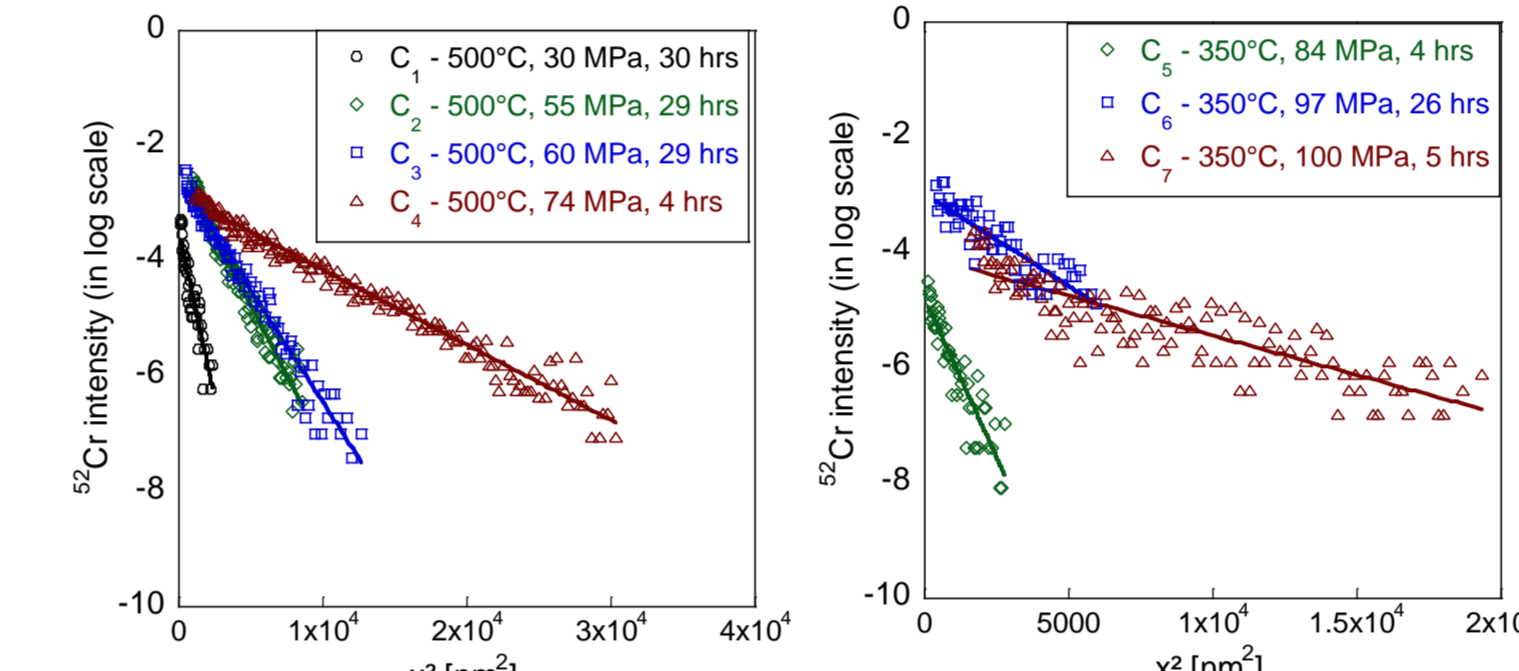


Results

Strain-enhanced diffusion

$$\frac{\partial C}{\partial t} = D_p \frac{\partial C}{\partial x^2} - \dot{\epsilon} x \frac{\partial C}{\partial x}$$

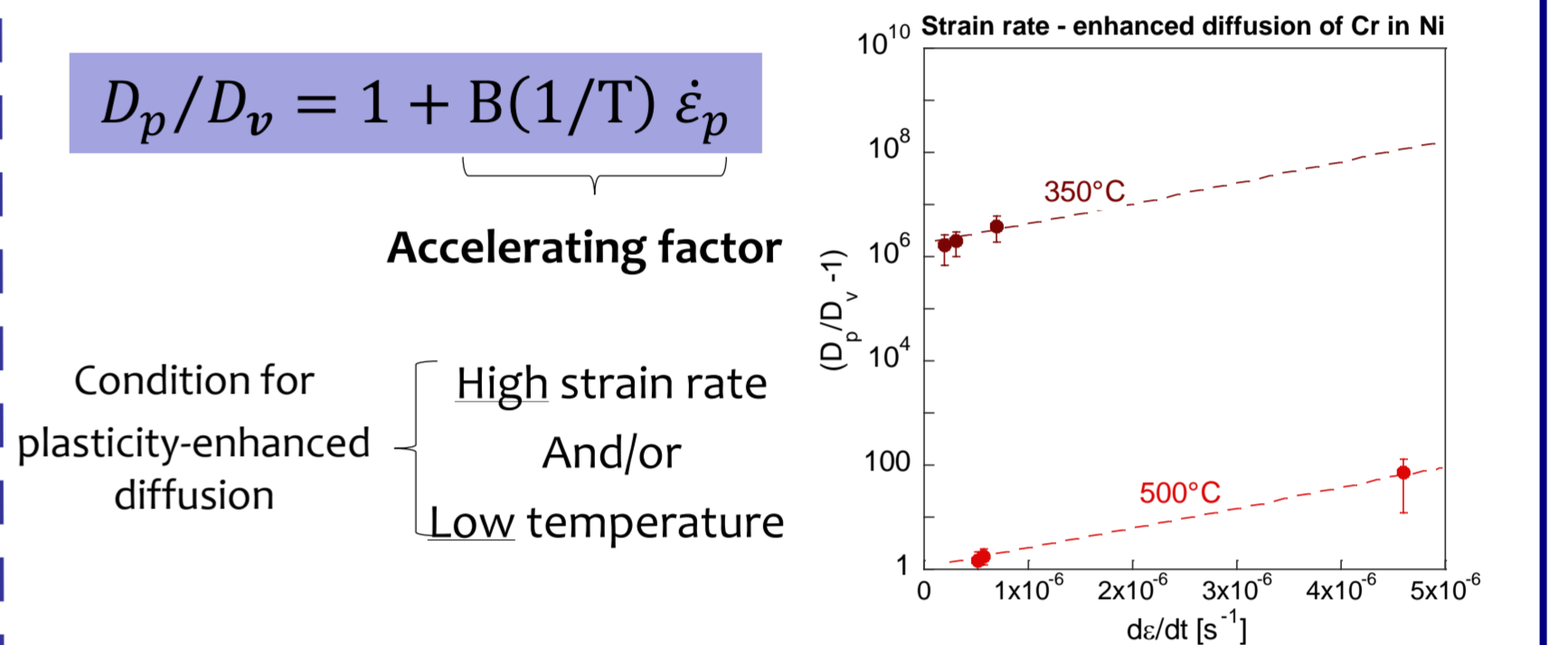
$$\frac{\partial \ln C}{\partial x^2} = \frac{1}{4 D_p t} \left[\frac{2\epsilon}{\exp(2\epsilon) - 1} \right]$$



Mechanical state	Diffusion	Final strain ε [%]	Average strain rate [10 ⁻⁷ s ⁻¹]	D [cm ² /s]	Enhanced diffusion D _p /D _v
Without plasticity	500°C, 30 hrs	-	-	(3.0 ± 1) · 10 ⁻¹⁷	1.0
Below the yield stress	500°C, 30 hrs	2.5	4.7	(4.6 ± 1) · 10 ⁻¹⁷	1.5
	500°C, 29 hrs	5.0	5.2 ± 0.7	(7.6 ± 1) · 10 ⁻¹⁷	2.5
	500°C, 29 hrs	5.4	5.7 ± 0.1	(8.3 ± 1) · 10 ⁻¹⁷	2.8
Plasticity-enhanced diffusion	500°C, 4 hrs	7.5	46 ± 13	(2.2 ± 1) · 10 ⁻¹⁵	73
	350°C, 4 hrs	1.4	1.9	(1.7 ± 1) · 10 ⁻¹⁶	1 × 10 ⁶
	350°C, 26 hrs	5.9	3.1 ± 2.0	(2.0 ± 1) · 10 ⁻¹⁶	2 × 10 ⁶
	350°C, 5 hrs	4.8	7.0 ± 3.6	(3.9 ± 1) · 10 ⁻¹⁶	4 × 10 ⁶

Interpretation

- ✓ Diffusion is enhanced by plasticity at 350°C and 500°C
- ✓ Diffusion depends on the strain rate and temperature.



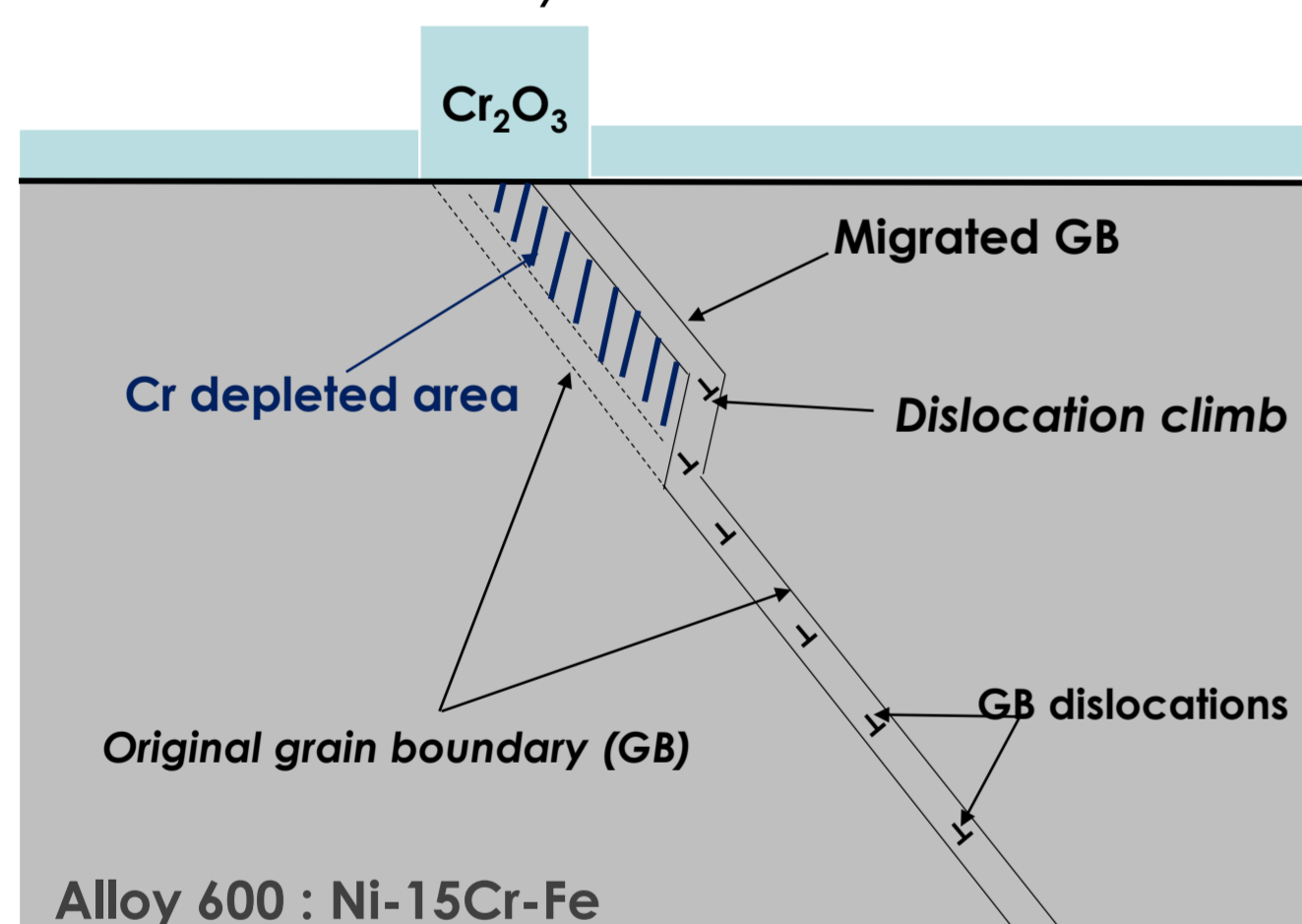
Mechanism : Dislocation-assisted diffusion along moving short-circuiting.

Consistent with the Hart model assuming that the dislocations pipes move and facilitate the diffusion.

DIFFUSION INDUCED GRAIN BOUNDARY MIGRATION (DIGM)

DIGM Mechanism

Grain boundary dislocation climb [4]

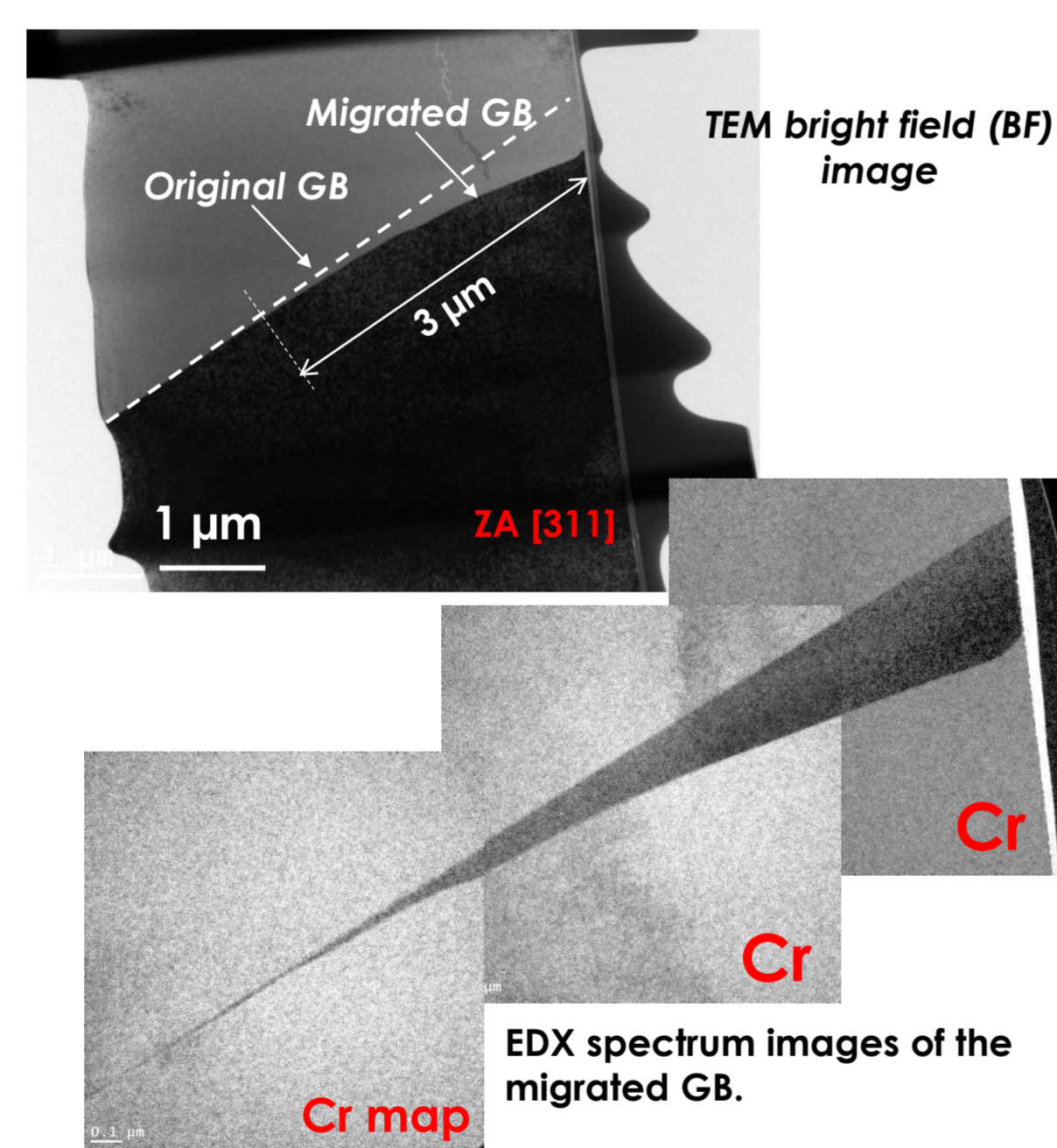


Steps of mechanism :

1. Intergranular diffusion
2. Kirkendall effect (vacancies flow) along the GB
3. Lead to the self-sustaining climb of GB dislocations
4. Steps of climb cause the GB migration.

DIGM characterizations

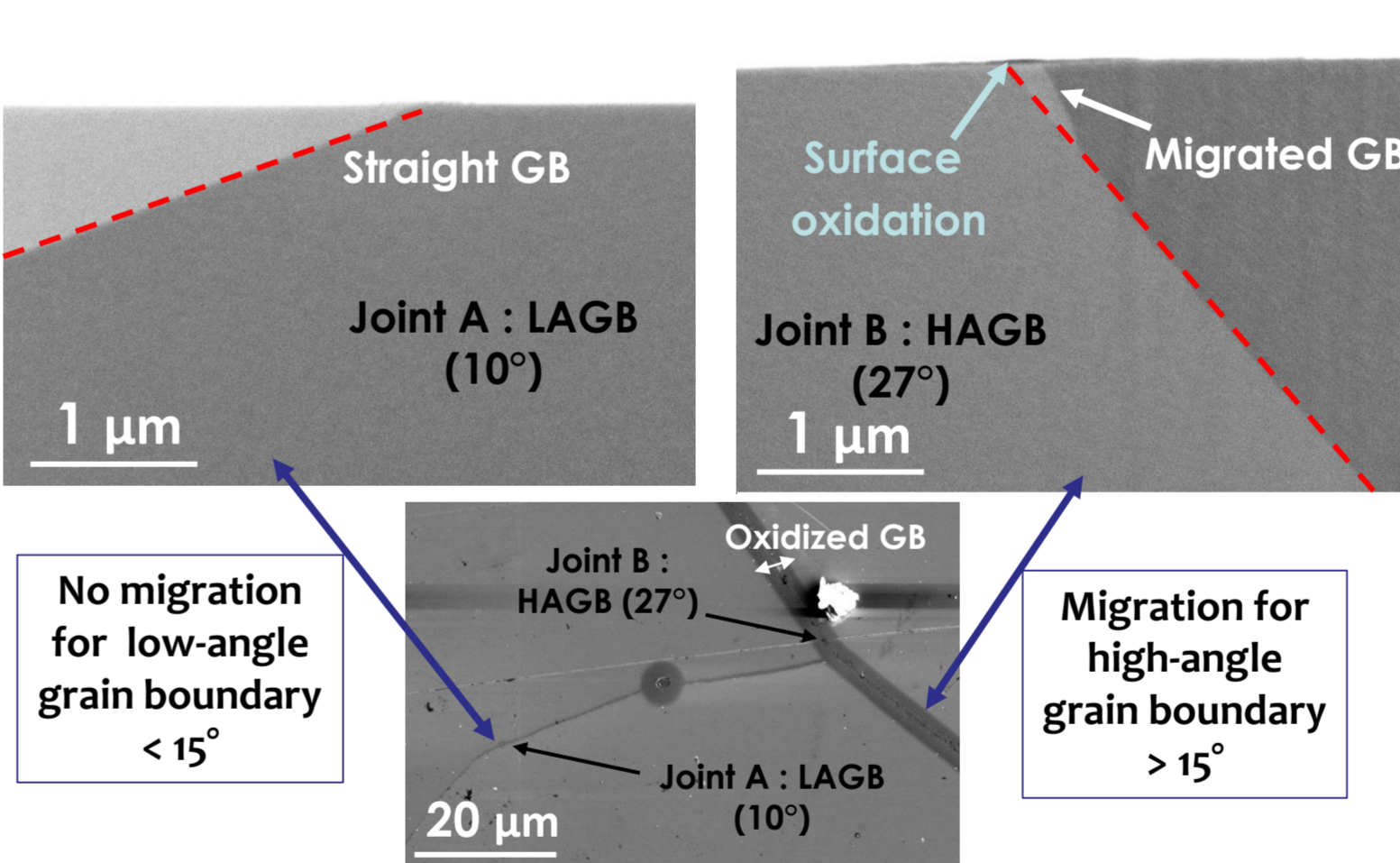
Heat treatments at 360°C and 500°C on A600 (under vacuum).



Migrated grain boundary leaves in its wake a Cr-depleted area.

Effect of grain boundary misorientation on DIGM

Results obtained on A600 sample after 30 hrs at 500°C, in vacuum (P = 10⁻⁶ mbar)

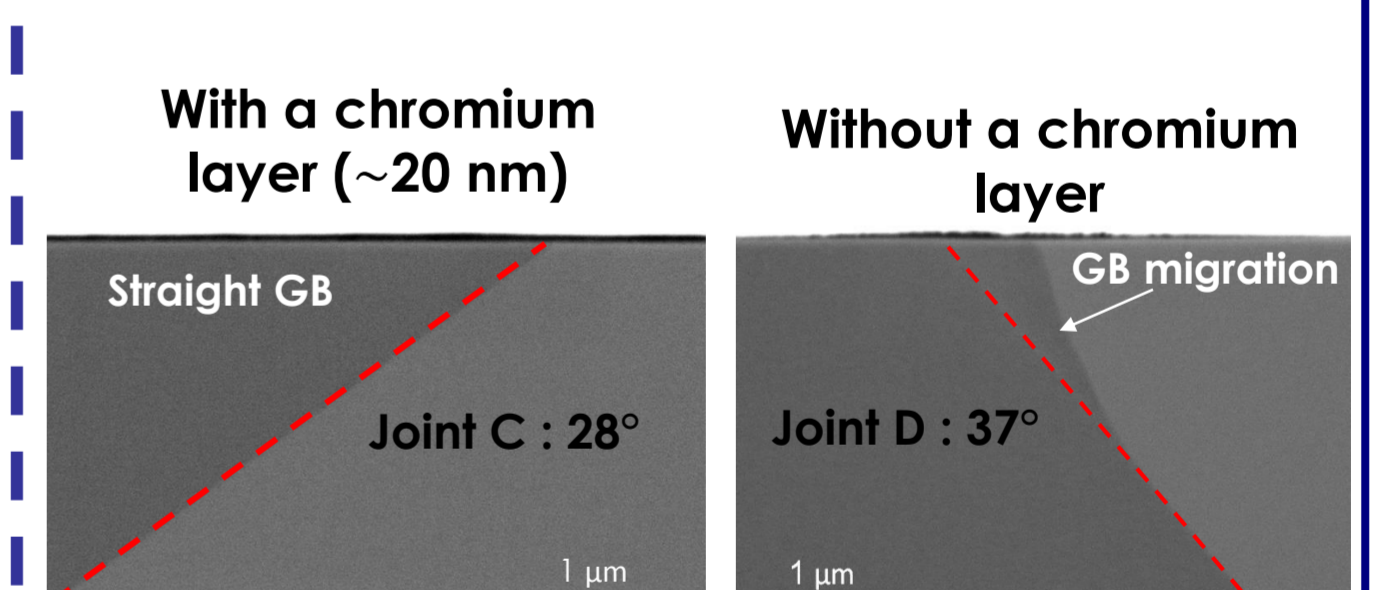


The effect is observed only for high angle grain boundaries ; It is not observed for low-angle grain boundaries (less than about 15°) [4].

BSE images of the cross-sections obtained using FIB/SEM (Equipex MATMECA) by Fabrice Gaslain (Centre des Matériaux...)

DIGM : a result of oxidation process

Results obtained on A600 sample after heating at 500°C, 30 hrs in vacuum.



Chromium deposition = No GB migration

GB migration is possible only if the oxidation contribute to the intergranular diffusion mechanism.

CONCLUSION

First assumption

Enhanced diffusion by plastic deformation

At 350°C : $D_p \sim D_{SCC}$
Calculated Cr-depleted area \Leftrightarrow 30 nm

A dynamic strain rate could accelerate the chromium diffusion at the SCC crack tip and could explain the formation of the Cr-depleted area.

Second assumption

Diffusion induced grain boundary migration

At 500°C :
Measured Cr-depleted area \Leftrightarrow 600 nm

Without any external stresses, GB migration could explain the remarkable extent of Cr depletion on A600 at low temperatures (PWR primary water)

PERSPECTIVES

- To perform the same tests (diffusion during creep) on Alloy 600
- To model the mechanism of dislocation-assisted diffusion
- To highlight the DIGM phenomenon in PWR conditions.

References

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