RÉPUBLIQUE FRANÇAISE
Liberté
Égalité
Fraternité

IRSN
INSTITUT DE RADIOPROTECTION ET DE SÛRETÉ NUCLÉAIRE

Burst and oxidation tests on bare and pre-oxidized claddings

C. Duriez, Q. Ferrand, J. Desquines, M. Kpemou, T. Taurines

IRSN

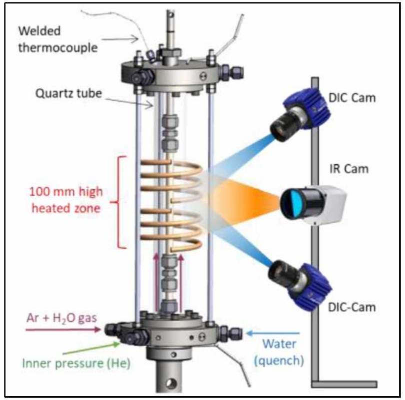
PSN-RES/SEREX
Centre de Cadarache, 13115 St Paul-Lez-Durance
France

27th International QUENCH Workshop, Karlsruhe, September 27-29, 2022.

MEMBRE DE ETSON

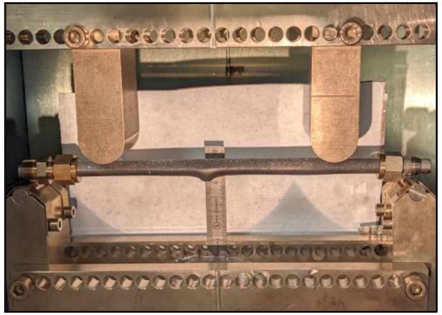
Content

STEP 1
Ballooning/burst tests in steam

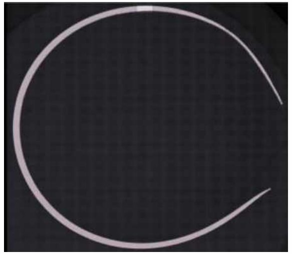


STEP 2
High temperature steam oxidation

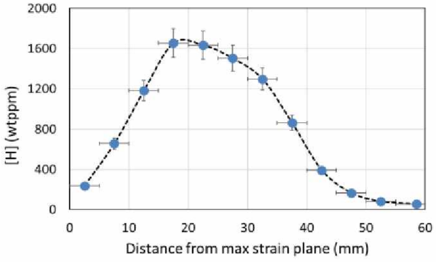
STEP3
4 points bending tests



STEP 4
Post-test examinations

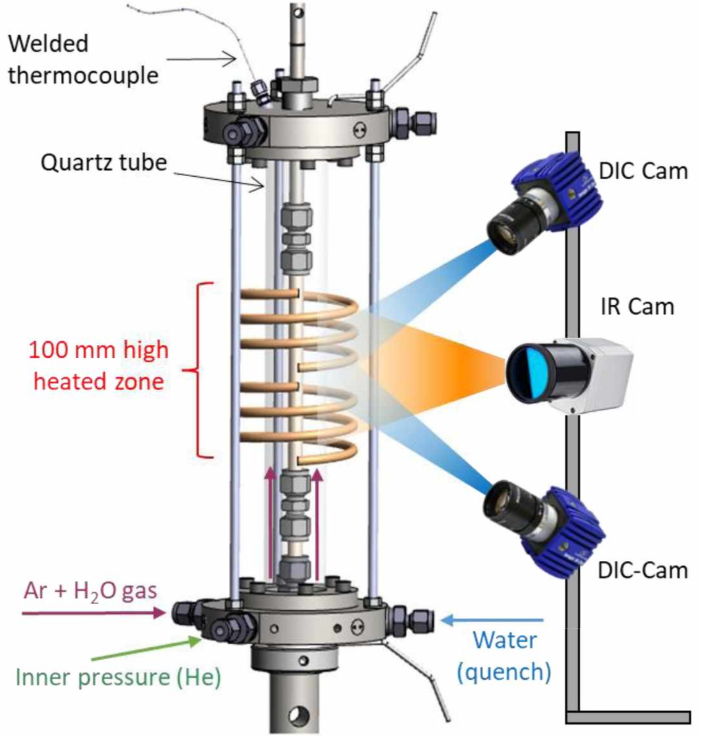


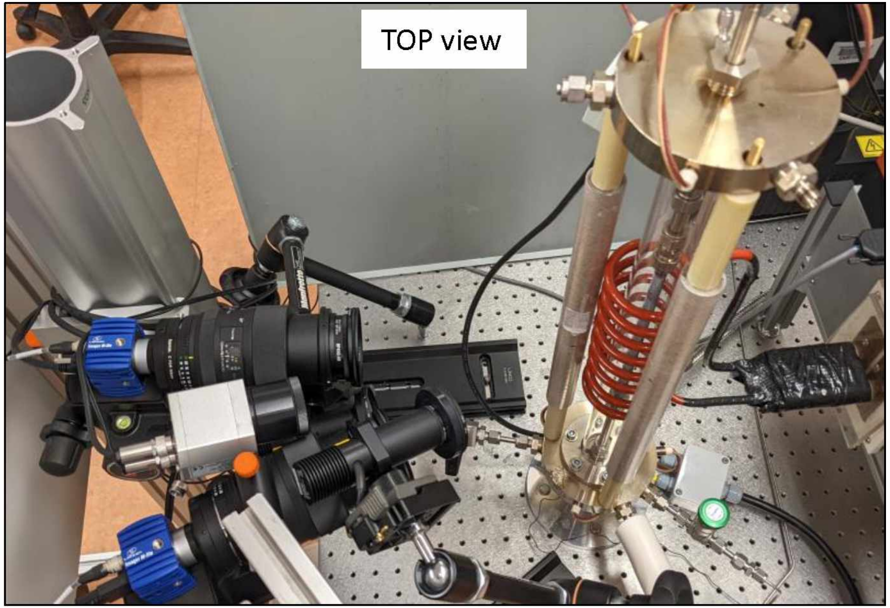
Hydrogen concentration profile



Step 1
ballooning/burst tests in steam

Device for ballooning/burst tests in steam





Bottom

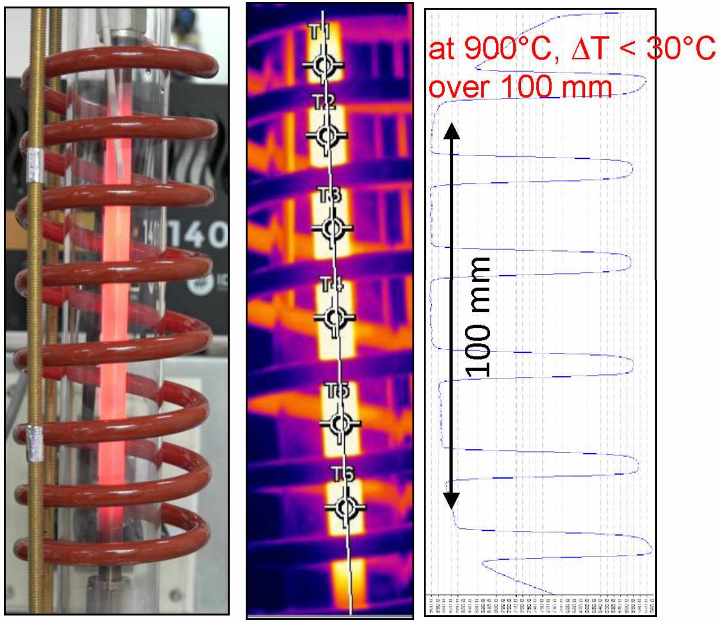
Top

250 mm

Al_2O_3 pellets Ø 8.2 mm → gap ~ 100 µm

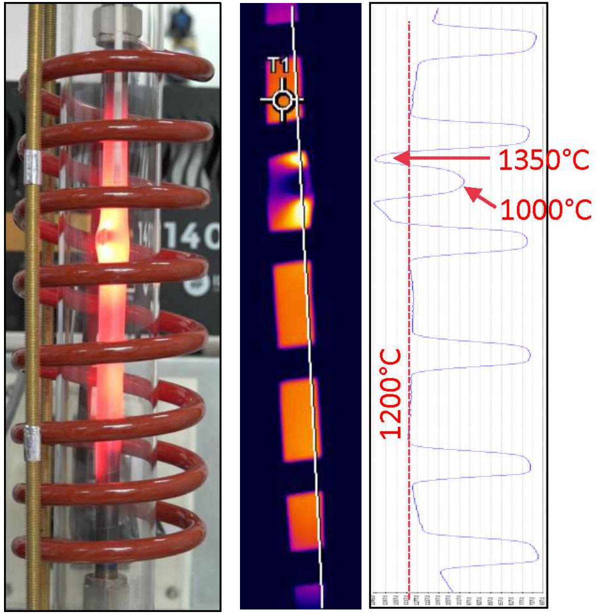
Induction heating

- Fast heating, high T
- Good control of the temperature axial profile
- The specimen is viewable → T from IR cam

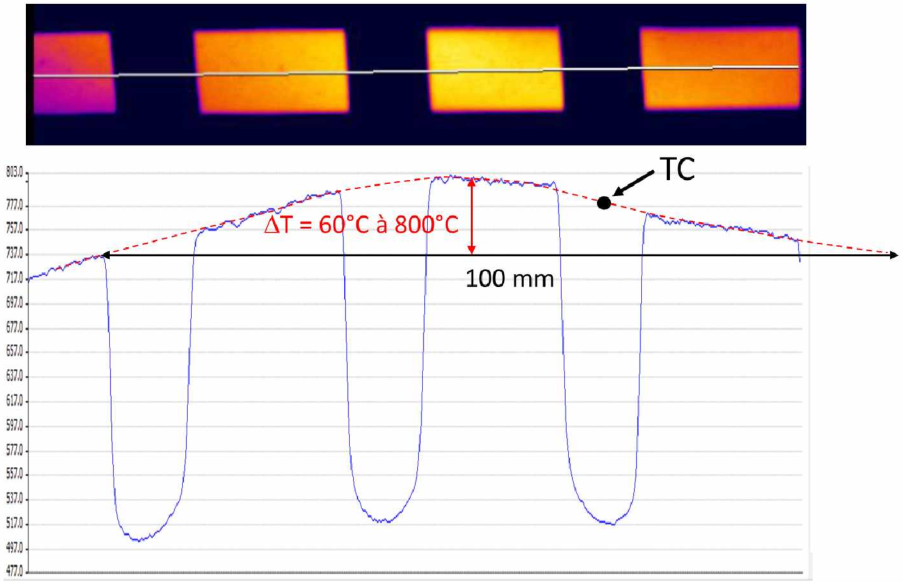


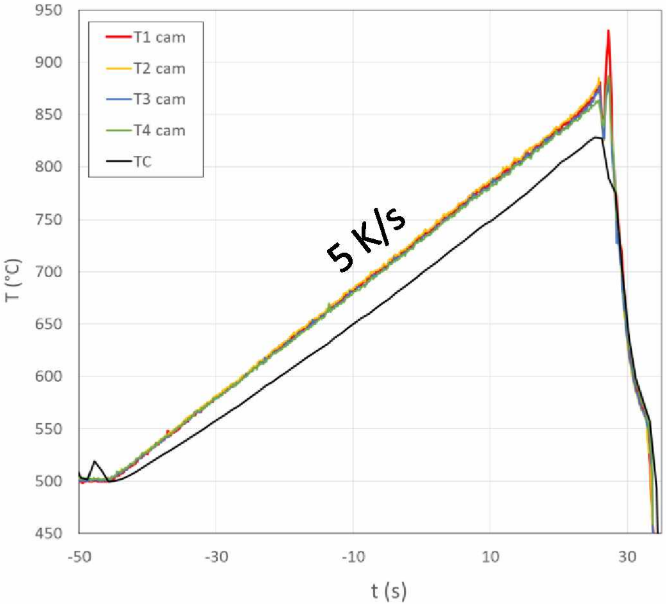
After burst, heating is not anymore homogeneous !

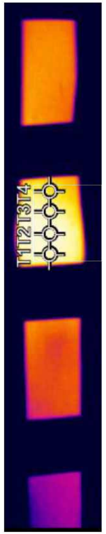
- Cold zone in the burst opening region
- Hot spots on both sides of the opening



Temperature profile for burst tests







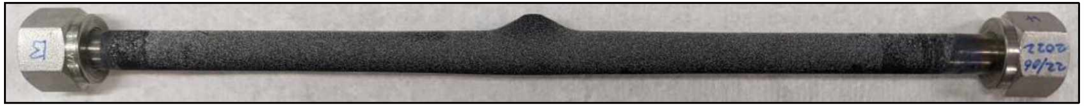
- ΔT = 60°C over 100 mm (for non ballooned tubes).
- T ramp regulated from IR cam measurement in the balloon.
- Thermocouple welded close to the balloon to calibrate the IR cam.

Burst test matrix

Alloy	Initial state	Inner pressure	Ramp rate
Zy4	As-received	20, 30, 50 bar	1 K/s 5 K/s
	Pre-ox 40/30µm, [H] ~ 250 wtppm	10, 20, 30, 50 bar	5 K/s
M5 FRAMATOME	As-received	20, 50 bar	5 K/s 10 K/s
	Pre-ox 10/0 µm, [H] = 19 ± 5 wtppm Pre-ox 10/10 µm, [H] = 40 ± 5 wtppm	20, 50 bar	5 K/s

Pre-oxidation was performed at 425°C in $O_2 + H_2O$

→ 25 burst tests
20 tests at 5 K/s

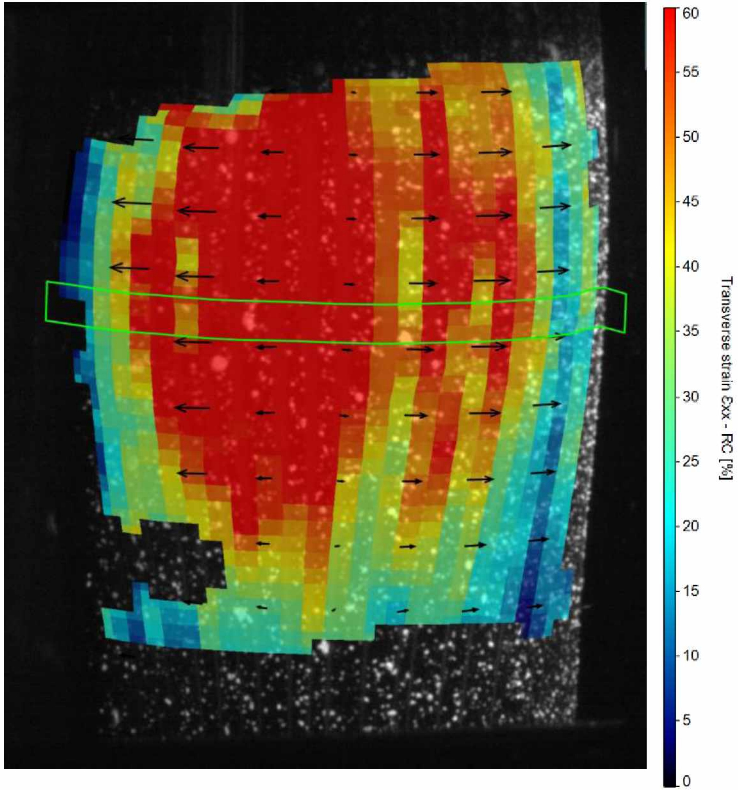


As-received M5, 20 bar, 5 K/s

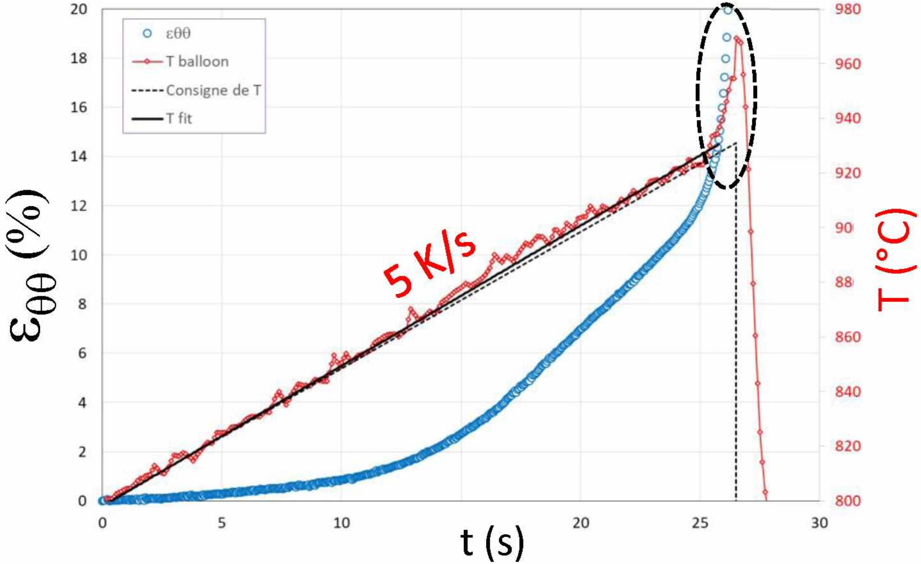
Pre-ox 10/0µm M5 , 50 bar, 5 K/s

⇨ At 50 bar, slight bending likely due to gas ejection at burst

The use of 3D-DIC for ballooning/burst tests



Burst test at 20 bar, 5 K/s

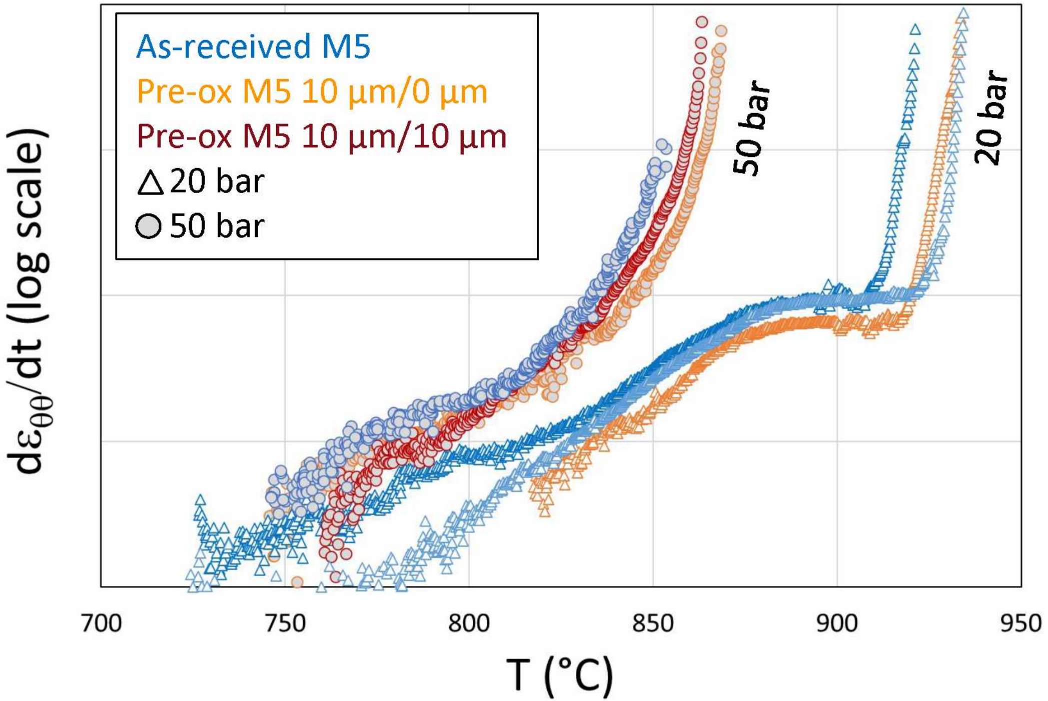


- From DIC → $\varepsilon_{\theta\theta}$ = f(T)
- From IR cam → T = f(T)

→ $\dot{\varepsilon}_{\theta\theta}$ = f(T)

- T follows the setup ramp, except for the last two seconds.

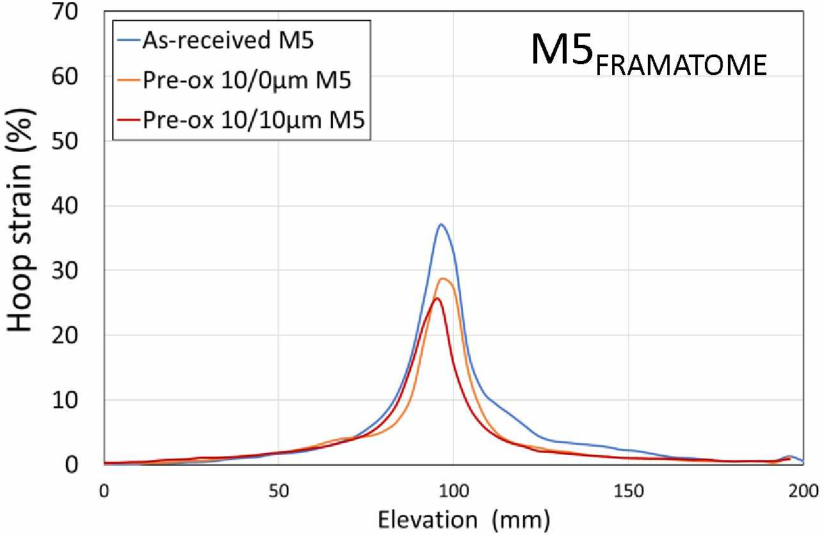
Creep rate at 5K/s, 20 and 50 bar, on $M5_{FRAMATOME}$

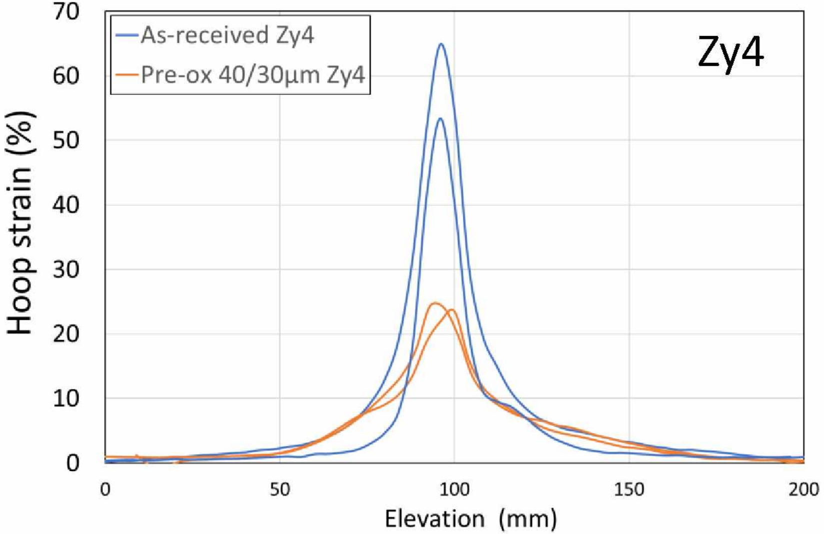


- The creep rate slows down as the β-Zr fraction increases.
- Limited influence of pre-oxidation.

Post-test examinations: 3D laser scanning → Final strain axial profiles

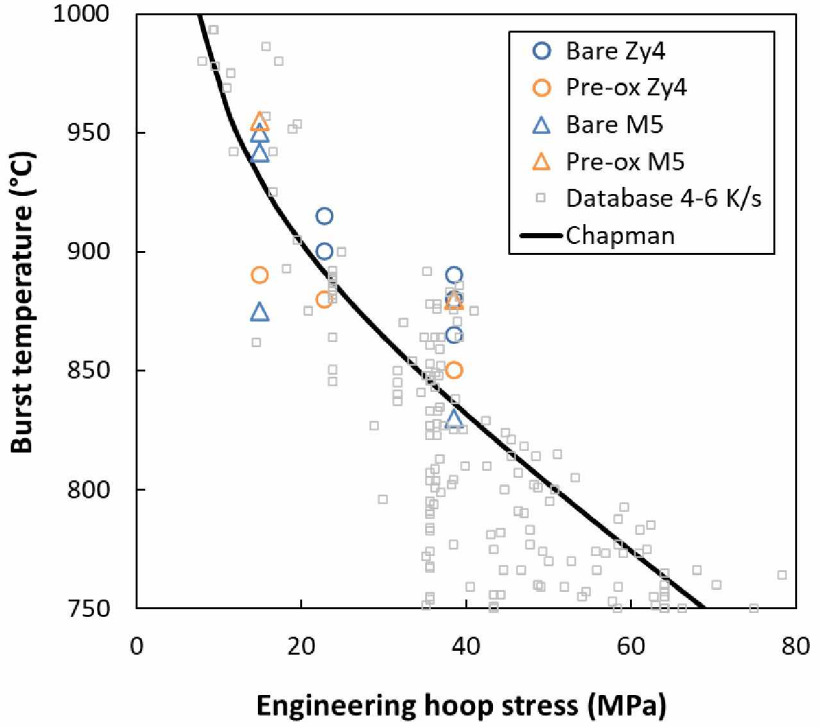
Results at 50 bar and 5 K/s





- Lower strain for $M5_{FRAMATOME}$ than for Zy4
- Limited effect of a thin pre-oxidation scale (M5)
- Significant influence of thick pre-oxidation scales (Zy4)

Burst temperature at 5 K/s



R.H. Chapman et al., from US-NRC NUREG-0630, 1980.

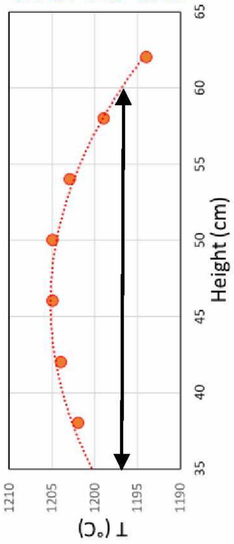
For Zy-4, burst temperatures are lower for pre-oxidised rods

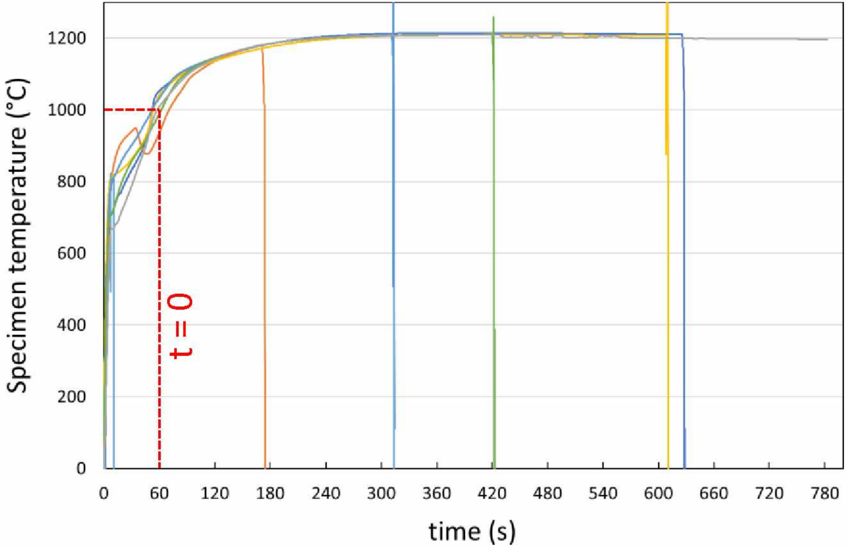
→ probable effect of hydrogen (~250 wtppm)

Step 2
High temperature steam oxidation

10% Ar
90% Steam (500 g/h)

1200 ± 5°C
over 25 cm





- Heat-up rate is rather slow, due to thermal inertia of Al_2O_3 pellets → no overshoot !
- ECR_{BJ} in the balloon is calculated considering double side oxidation, and taking into account the thermal history, the strain and the pre-oxidation

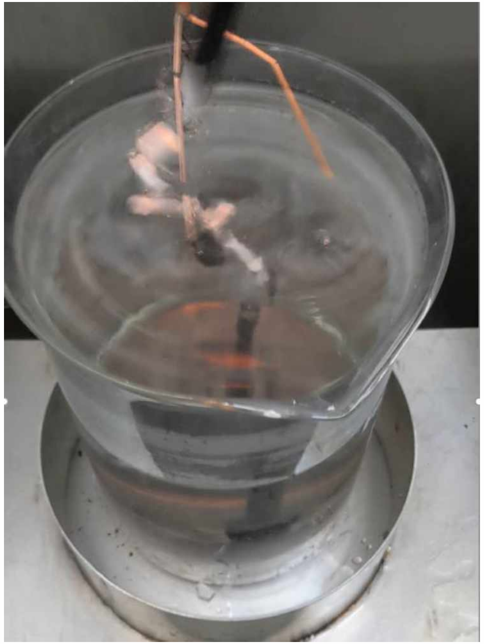
Δt = 90 s to 12 min → ECR_{BJ} in the range 10-40%

Spontaneous rupture during quench

At very high ECR (ECR-BJ ≈ 40%, i.e. ECR-CP ≈ 30%), rupture without applied load occurs in the secondary hydriding region during quench.
This was observed both for M5 and Zy4 rods

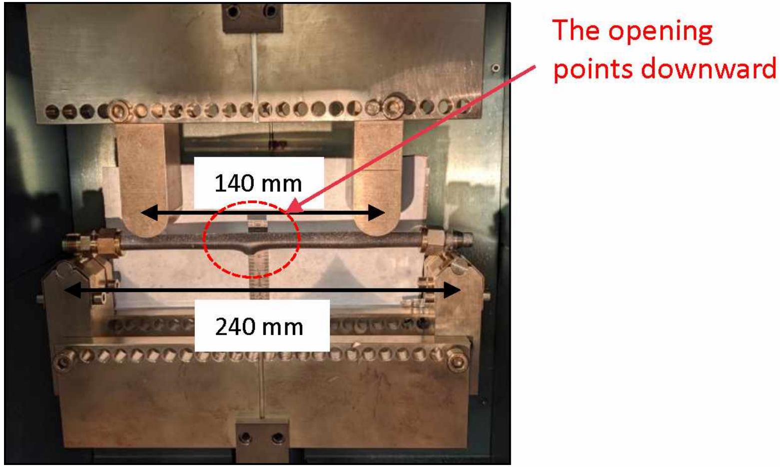
Quench @ t = 0

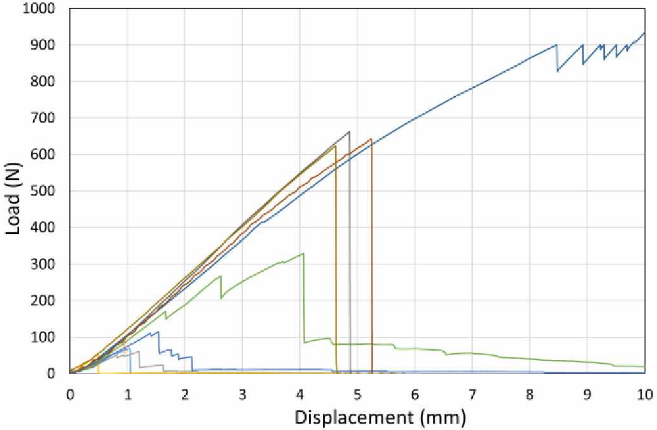
Rupture @ t = 23s

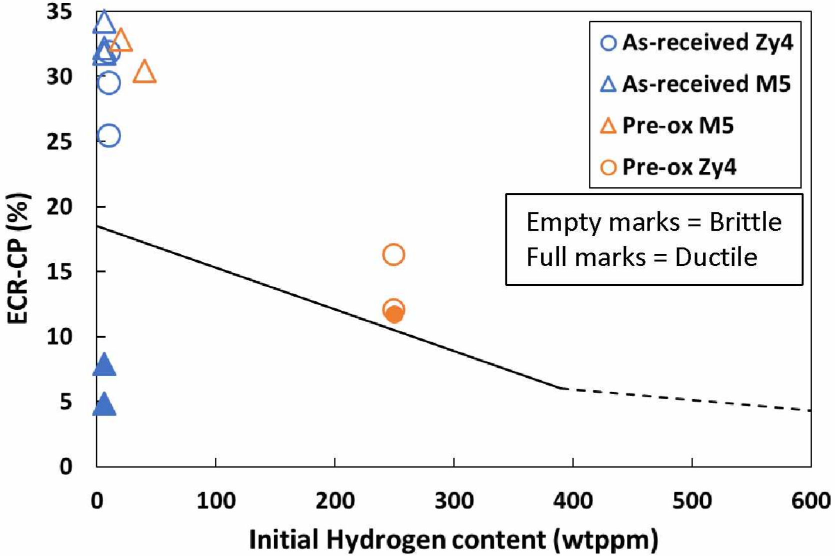


Step 3
4 points bending tests

Four-points bending tests (4PBT) at 135°C



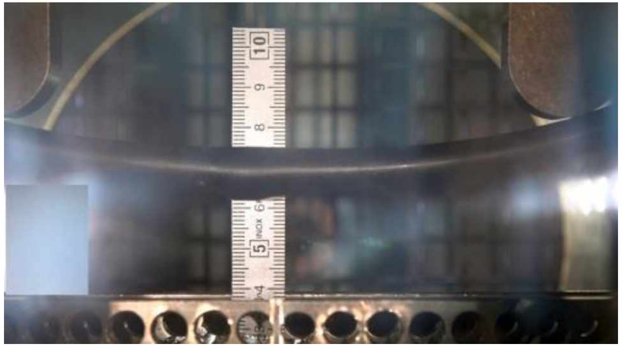




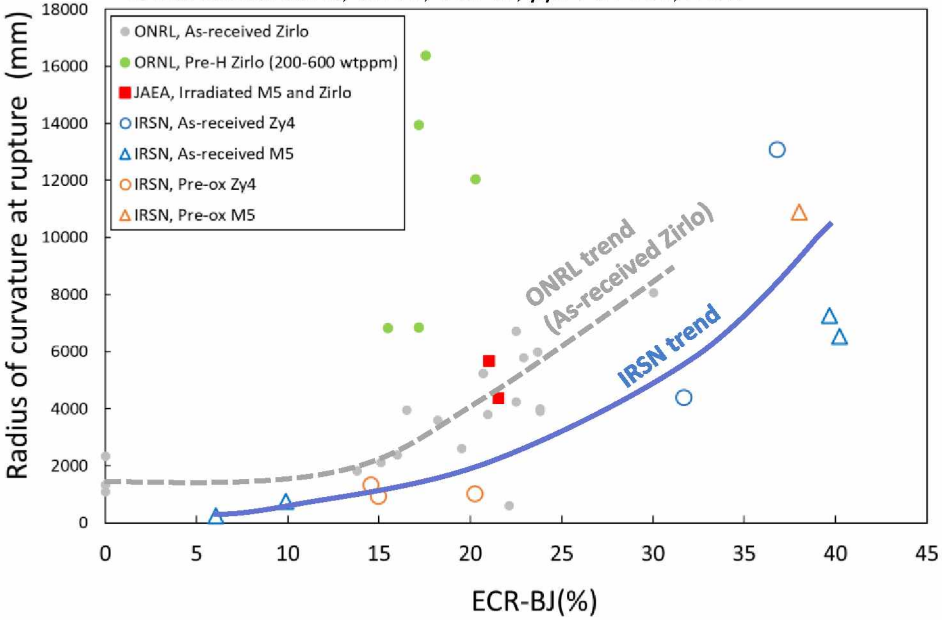
- Most of the rods were brittle, results are consistent with the ductile/brittle limit derived from Argonne RCTs.
- All rupture during 4PBT occurred in the balloon, even for rods having high H content.

Four point bending tests at 135°C

Radius of curvature at rupture is calculated from the rod deflexion at rupture



M. C. Billone et Al. NUREG-7219, 2016.
T. Narukawa et Al., JNST, vol. 57, pp. 782-791, 2020

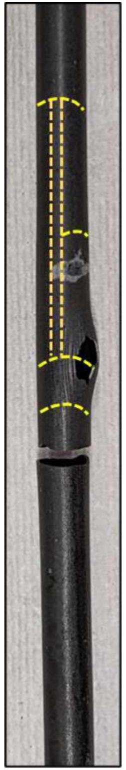


Better behaviour than for as-received Zirlo ?

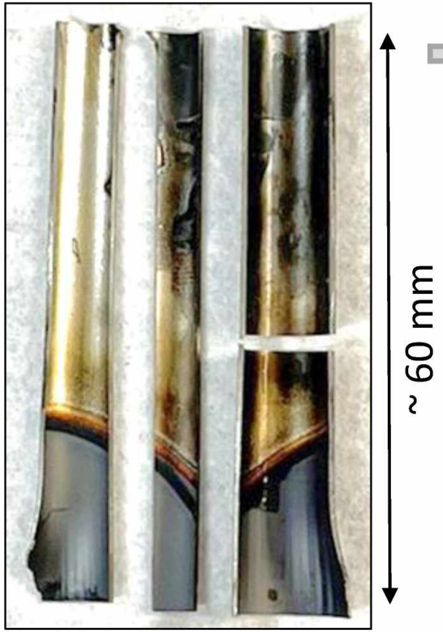
Step 4
Post-test examinations

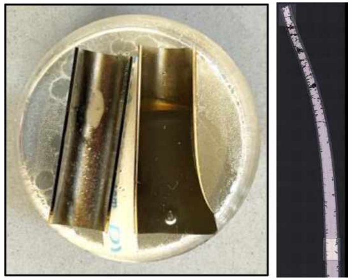
Thanks to Alice Viretto and Gaëlle Villevieille for the post-test exams

Post-test destructive examinations

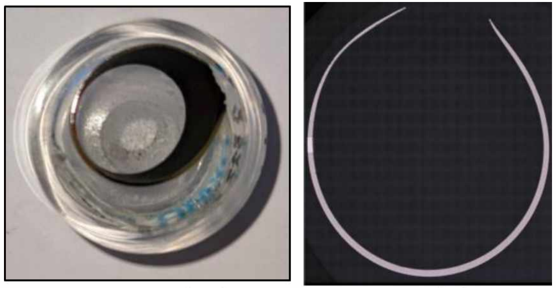


Inner surface





Axial cross-section



Radial cross-section(s)

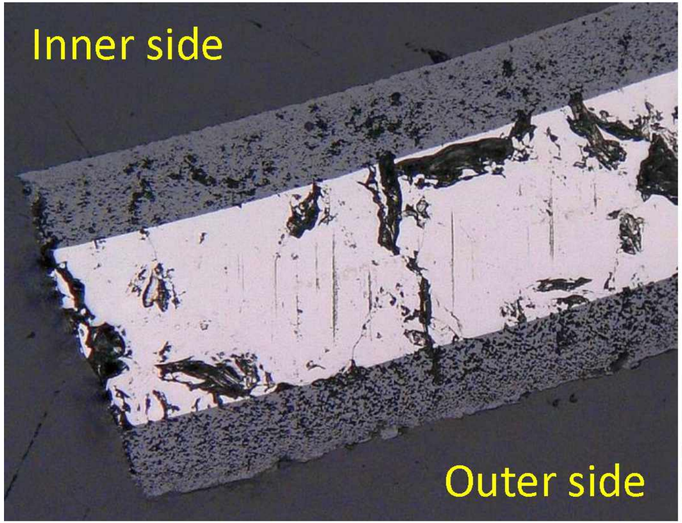
- At balloon center
- At failure location

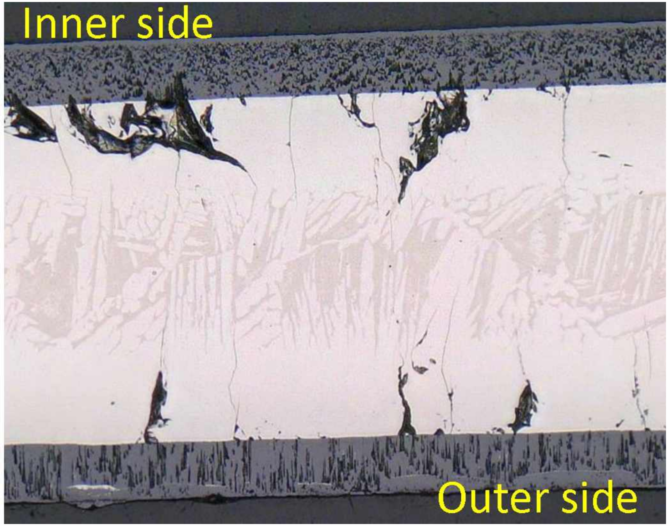
Band cut at the opposite side of
the opening for [H] measurements
by hot vacuum extraction (LECO)

→ Axial Hydrogen distribution

Impact of the painting on the HT oxidation ?

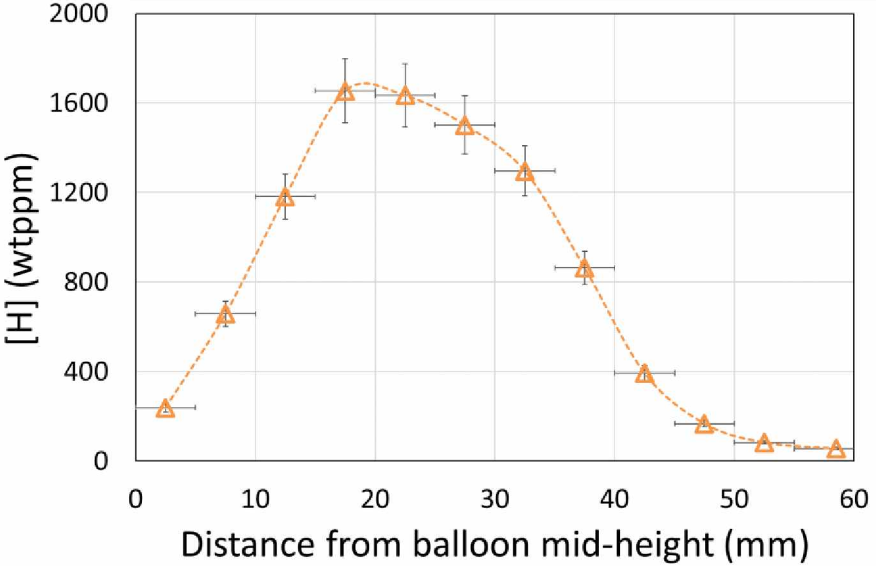
As-received M5, ECR_{BJ} = 40.2%

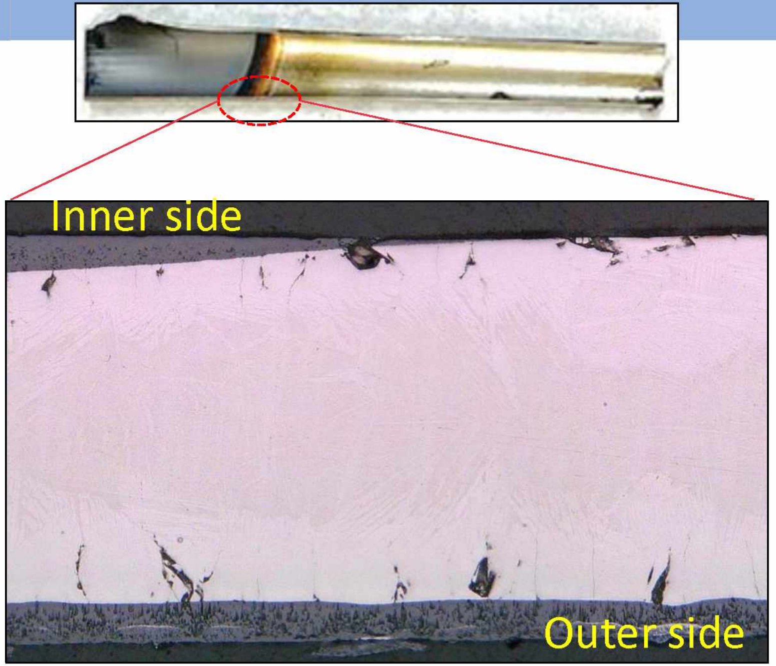




⇨ Close to the opening, same oxide thickness outside and inside → no or limited influence of the painting.

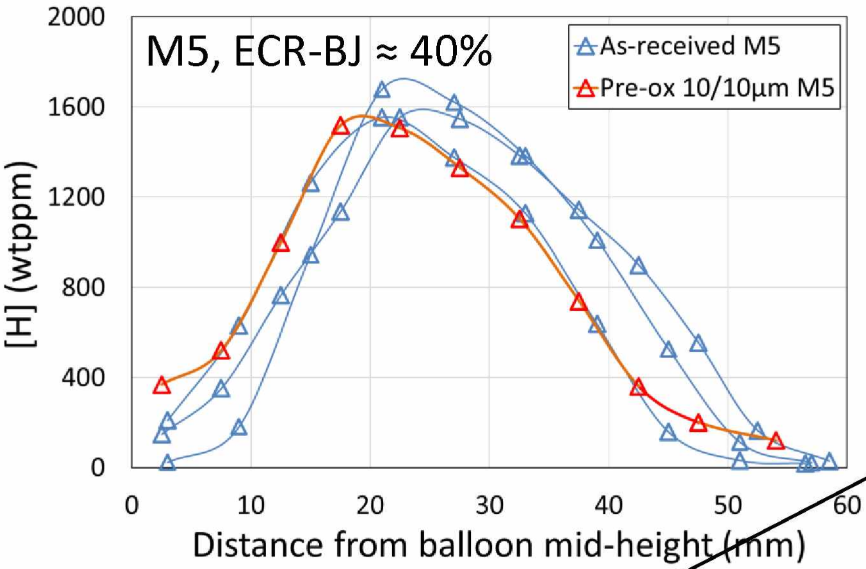
Hydrogen axial distribution

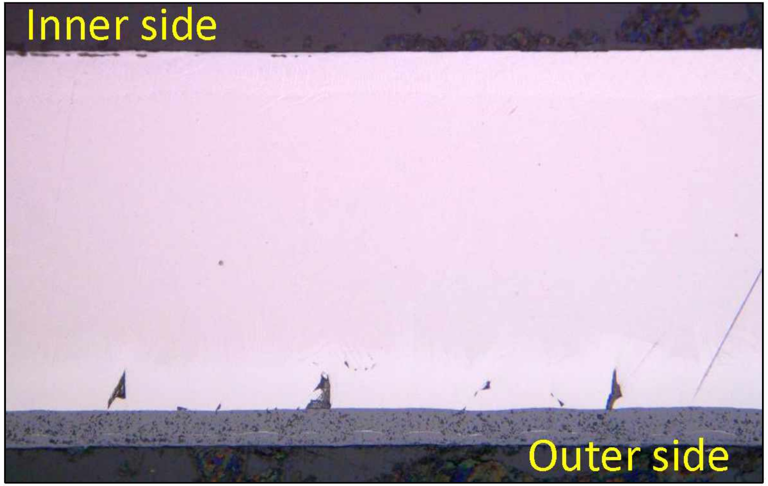


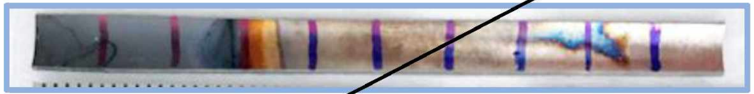


- The colour change corresponds to the end of the inner oxide layer.
- Clear link between the colour change and the hydrogen axial distribution.
- H peak maximum is located few mm from the end of the inner oxide layer.

Effect of the inner pre-oxydation on H pick-up

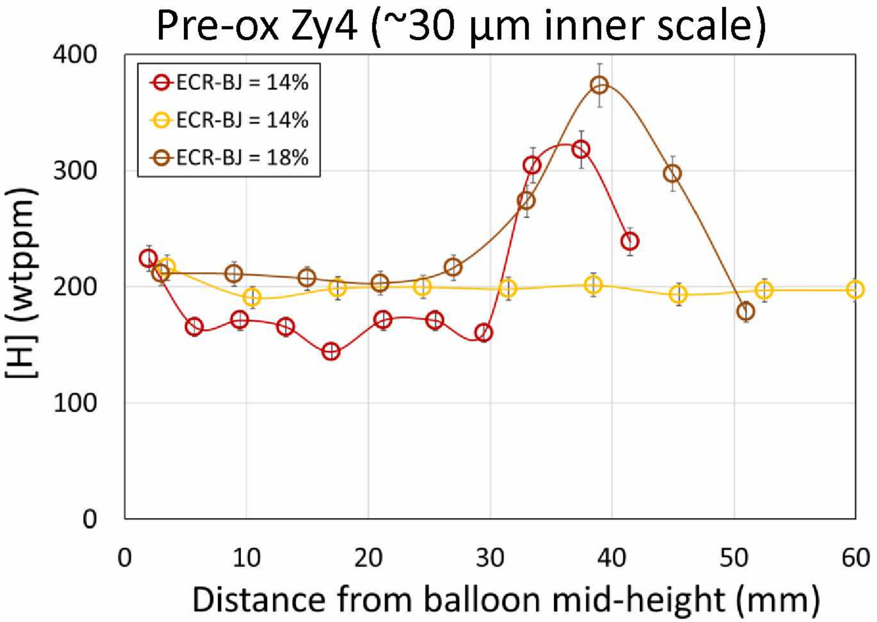




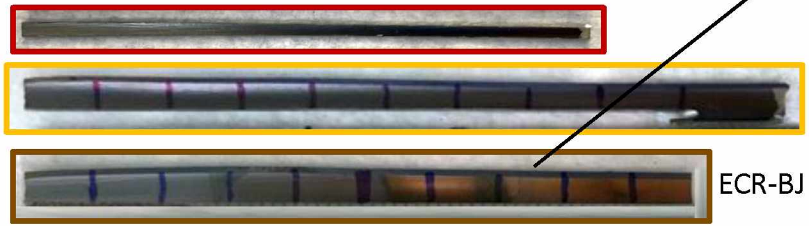


- For high ECR, a thin (10 µm) inner pre-oxide scale dissolves and doesn't prevent secondary hydriding.

Effect of the inner pre-oxidation on H pick-up



ECR-BJ = 18%

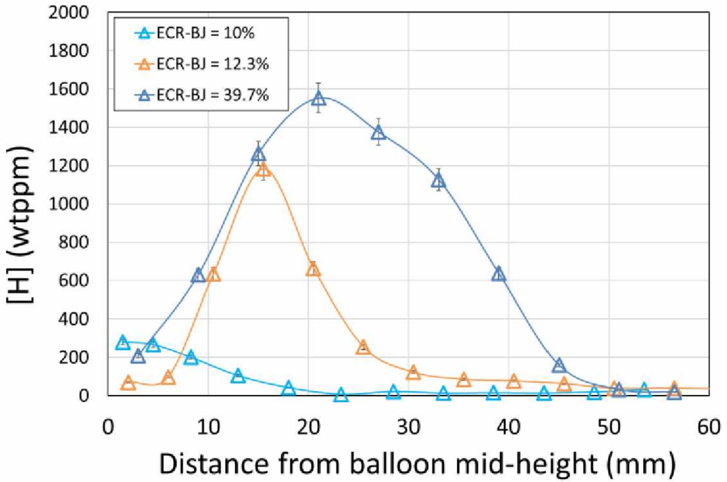


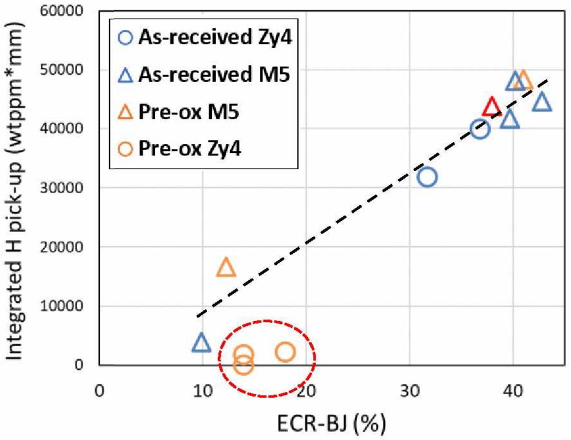
ECR-BJ = 18%

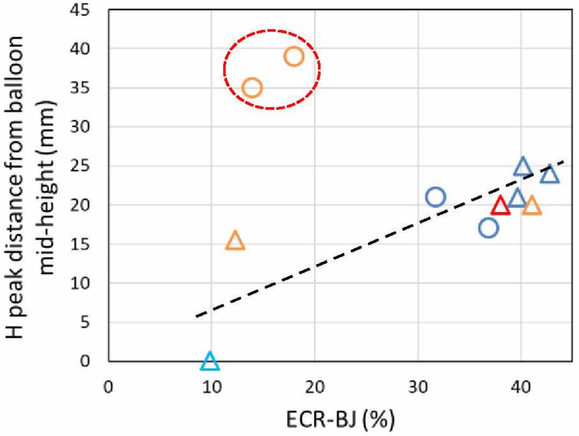
For Pre-ox Zy4 (30 µm inner scale):

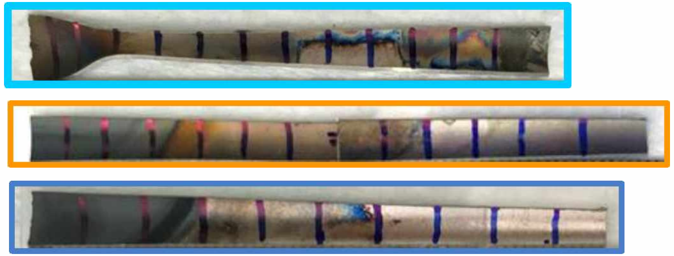
- Moderate (but non zero) H-pick-up.
- Pick-up occurred away from the opening.
- Inner zirconia scale is darker (but not fully dissolved) were H pick-up occurred.

Amount of H picked-up and H-peak/balloon distance









= thick inner pre-oxide scale

- The amount of incorporated hydrogen increases with ECR.
- The H-peak maximum may shift away from the balloon mid-height as ECR increases.

Conclusions (preliminary)

<u>Regarding the strain in the ballooned region</u>

- Lower strain for $M5_{Framatome}$ than for Zy4,
- Thick pre-oxide layers (40/30µm) significantly decreases the strain (Zy4),
- Thin pre-oxidation layers (10/0 or 10/10µm) have much lower influence ($M5_{Framatome}$).

<u>Regarding rupture after HT oxidation</u>

- At very high ECRs, rupture without applied load sometimes occurred in the secondary hydriding region during quench.

<u>Regarding secondary hydriding</u>

- A thin inner pre-oxide scale can be dissolved and doesn't prevent secondary hydriding.
- The amount of H incorporated increases with ECR (i.e. with exposure time),
- The H-peak maximum shifts away from the opening as exposure time increases.

More data needed, specifically at intermediate ECRs.

We are ready to test Cr-coated ATF claddings !

Thank you for your attention