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## Assesment of potential swelling of Pressurized Water Reactor internals The GONDOLE experiment in Osiris reactor

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# Fontevraud 9

## *Assesment of potential swelling of Pressurized Water Reactor internals: The GONDOLE experiment in Osiris reactor*

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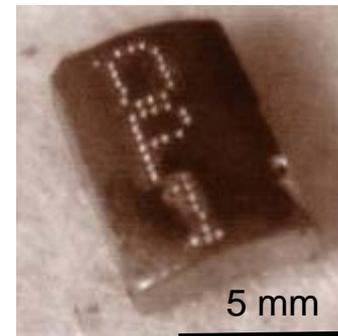
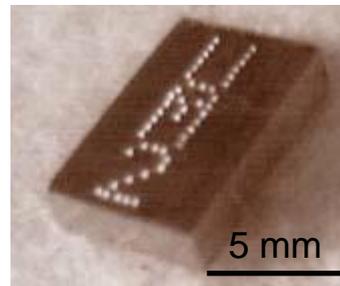
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  4. Density measurements experiments
  
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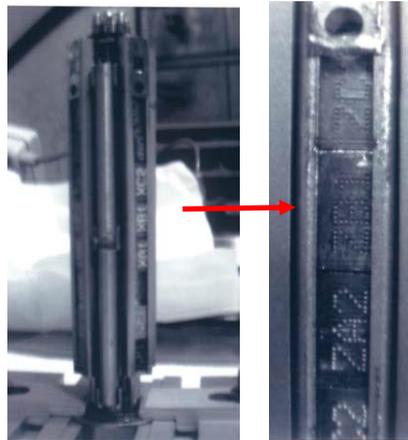
# 1.1. THE GONDOLE PROGRAM - OBJECTIVES

- Main goal: to evaluate potential swelling of PWR internals
  - by density measurements
  - PWR internals representative materials
  - Irradiation in a mixed spectrum (Osiris MTR) at 360°C
  - 30 cumulated dpa (15 dpa for initial program + 15 dpa for prolongation)
  
- Density specimens characteristics:
  - Nominally rectangular shape
  - Small size
  - $7.7 \times 5 \times 1.5 \text{ mm}^3$
  - Specimen reference engraved



## 1.2. THE GONDOLE PROGRAM – DENSITY SPECIMENS

- Virgin and pre-irradiated materials (reach higher doses)
- 304 / 316 type materials (metallurgical state and chemical composition), with in particular “PWR reference materials” (304 Solution Annealed and 316 Cold-Worked + 308 welds) – French irradiation programs, PWR core internals materials, archive materials
- Others materials (stabilized steels, “low activation” steel...)
- Specimens inserted in four barrels in two baskets in SEMI/CEA hot cells
- 64 specimens



# 1.3. THE GONDOLE PROGRAM – IRRADIATION CONDITIONS

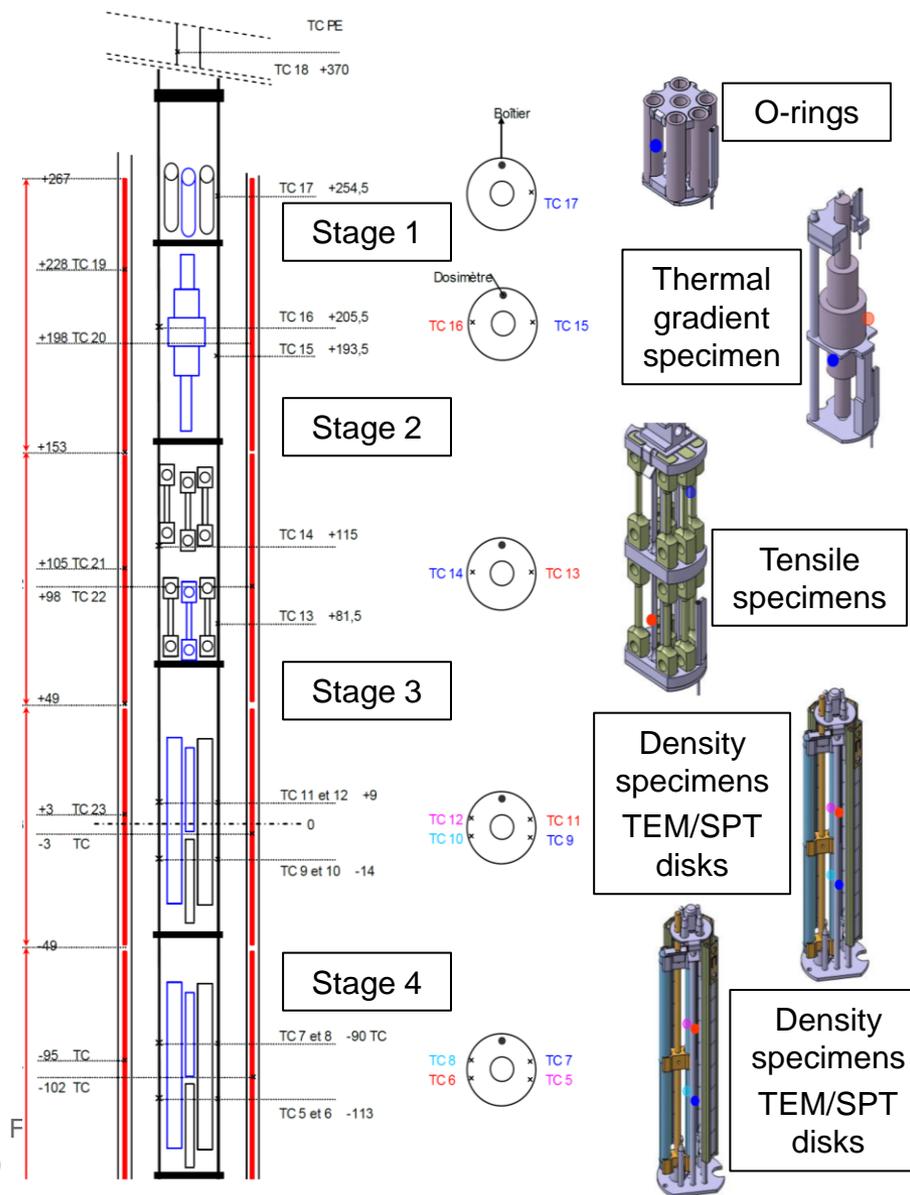
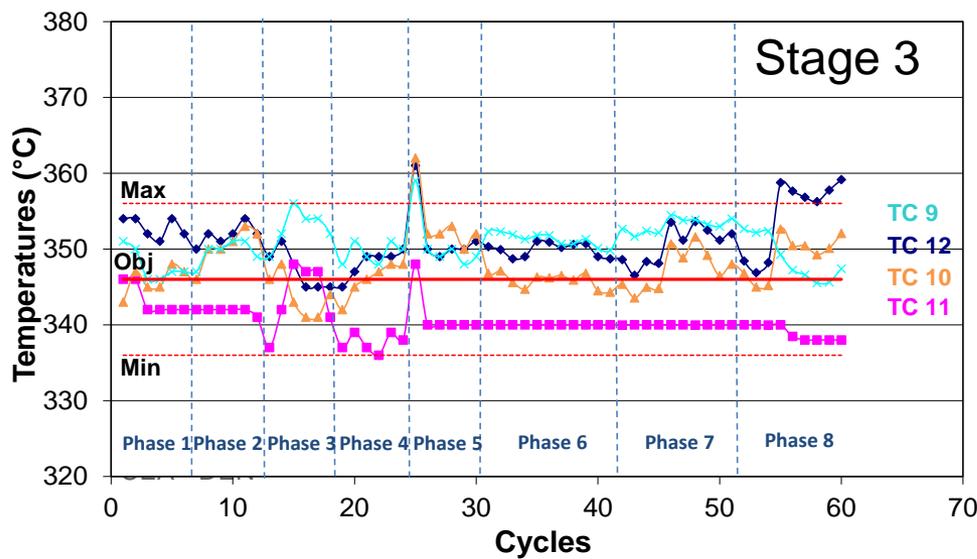
- Irradiation program: 2 parts
  - 2005-2010: 5 phases (6 cycles each)
  - 2011-2015: 3 phases (11, 10 and 9 cycles)
  - ~ 1206 Equivalent Full Power Days
  
- Irradiation in the core of Osiris reactor
  - Fast flux ~  $2 \times 10^{14}$  n/cm<sup>2</sup>/s
  - Thermal flux ~  $1.4 \times 10^{14}$  n/cm<sup>2</sup>/s
  
- Doses

	Phase 1	Phase 2	Phase 3	Phase 4	Phase 5	Phase 6	Phase 7	Phase 8	Cumulated dose (dpa)
Dose (dpa Fe)	2.50	2.41	3.25	3.05	2.82	5.75	5.13	4.65	29.56

- Gradient along the sample holder: results at max flux plane
- Final cumulated dose ~29.6 dpa
- Specimen changes at phase 1 and 2: only a part of the specimens cumulated the whole dose, but from phase 3 specimens were not changed (except one) (~24.7 dpa at the max flux plane)

# 1.3. THE GONDOLE PROGRAM – IRRADIATION CONDITIONS

- Sample holder immersed in NaK
- 4 stages:
  - Density specimens (stages 3 and 4)
  - Other specimens (stages 1 to 4)
- Heaters and thermocouples for temperature regulation
  - ~ 346°C on thermocouples for 360°C mean temperature on density specimens
- Activation foils for dosimetry measurements



# 1.4. THE GONDOLE PROGRAM – DENSITY MEASUREMENT EXPERIMENTS

- Density measurement principle
- Immersion density method by double weighing in air and phenyl bromide

$$\rho = \frac{(M1 - m1)}{(M1 - m1) - (M2 - m2)} \times (d_{\text{phenylBromide}} - d_{\text{air}}) + d_{\text{air}}$$

Mass in air →

↑  
Mass in liquid

- Immersion density measurements performed before GONDOLE irradiation and after each irradiation phase in hot cells
- Possibility to determine density evolution between phase 'j' and former phase 'i'

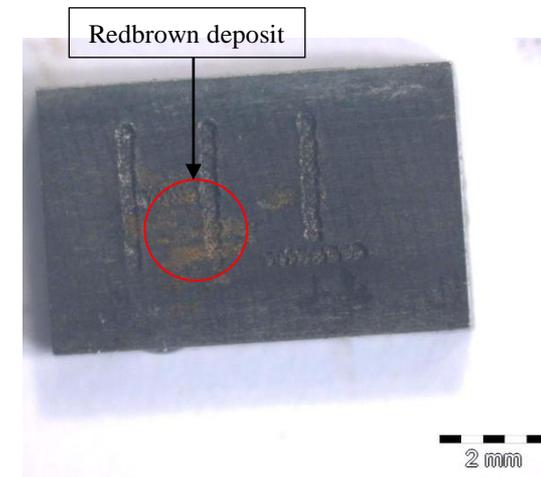
$$\Delta d(\%) = \frac{\rho_j - \rho_i}{\rho_i} \times 100$$



## 1.4. THE GONDOLE PROGRAM – DENSITY MEASUREMENT EXPERIMENTS

- Choice of the density specimen geometry is a compromise:
  - Size/mass of the specimens
  - Common geometry for pre-irradiated material machining in hot cells
  - Placing many specimens in the sample holder for material comparison
- Reduced weight of specimens (between 0.2 and 0.5 g) not optimal for density measurements (strong impact on uncertainty)

- Specimens were initially not checked
- Surface checked after phase 6
- Deposit confirmed on density specimens



- Enhancement of the density measurement protocol during the program
- Work to define the uncertainty as precisely as possible

## 1.4. THE GONDOLE PROGRAM – DENSITY MEASUREMENT EXPERIMENTS

- Density measurement procedure
  - Phase 0 to phase 4:
    - Specimen cleaning in US bath (alcohol, 5 min)
    - 2 double weighings** (taking account of  $T_{\text{phenylbromide}}$  and  $P_{\text{air}}$ )
    - If the two densities differ of  $> 0.02 \text{ g/cm}^3$  the measure is repeated
    - Scales precision: 0.1 mg**
  - Phase 5:
    - Similar protocol to that of phase 4 but with **4 double weighings** and a new set of scales (**precision: 0.01 mg**) to improve accuracy
  - Phase 6:
    - Specimen cleaning in US bath (alcohol, increased time 20 min), optical observation and weighing
    - Cleaning steps are repeated until mass stabilization
    - 4 double weighings** (30 specimens measured)
  - Phase 7:
    - Similar protocol to that of phase 6
    - Daily calibration of the scales and increased cleaning efforts
  - Phase 8:
    - Similar protocol to that of phase 7
    - 5 double weighings**

## 1.4. THE GONDOLE PROGRAM – DENSITY MEASUREMENT EXPERIMENTS

- Uncertainty determination

- 1 - Theoretical measurement uncertainty (precision of each measurement)

Accuracy limit on small samples (0.2 g)

Phase 1 - 4: +/- 0.40% (97.5% due to mass uncertainty)

Phase 5 - 7 : +/- 0.05% (80% due to mass uncertainty) – new scales

- 2 – Measurement scatter

Limited numbers of measurements : Student-Law approach

$$\mu = \pm t_{1-\frac{\alpha}{2}}^{n-1} \times \frac{\sigma}{\sqrt{n}}$$

**n**: number of measurements ( $n_{\text{phases 1-4}}=2$ ,  $n_{\text{phases 5-7}}=4$ ,  $n_{\text{phase 8}}=5$ )

**$\alpha$** : risk of error, taken equal to 5 %

**$\sigma$** : standard deviation for the n measurements

**$t_{1-\frac{\alpha}{2}}^{n-1}$** : Student law quantile for n-1 level of freedoms and with an error risk of  $\alpha/2$

$t_{\text{phases 1-4}} = 12.71$ ,  $t_{\text{phases 5-7}} = 3.182$ ,  $t_{\text{phase 8}} = 2.776$

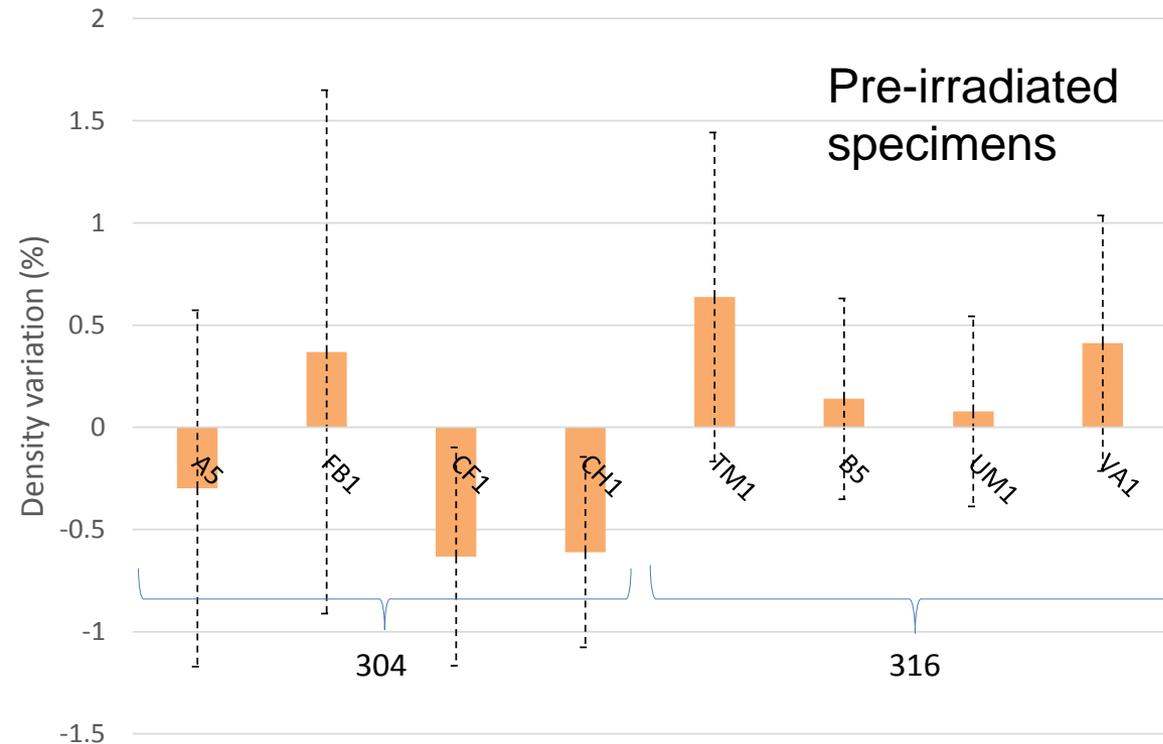
➤ Overall confidence interval = Precision + Student-Law interval

➤ Confidence interval reduced for last phases

➤  $\Delta d$  confidence interval = (Precision + Student-Law)<sub>Pi</sub> + (Precision + Student-Law)<sub>Pj</sub>

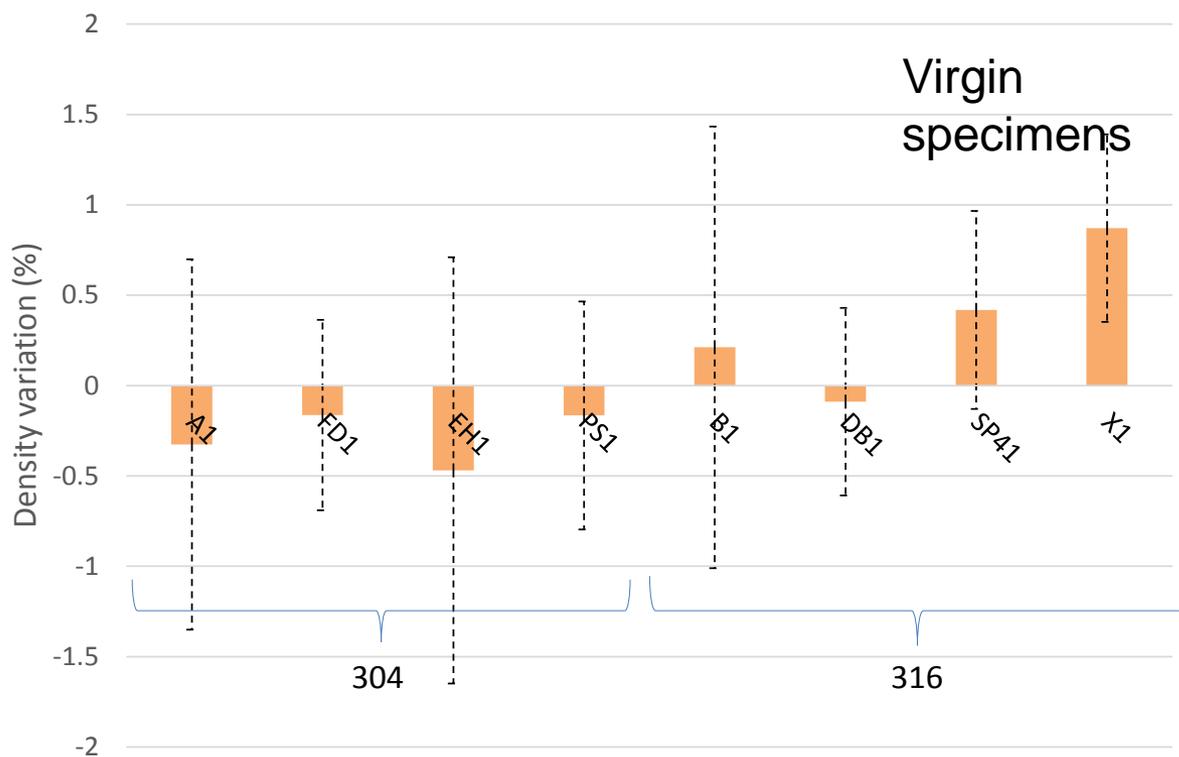
## 2.1. RESULTS – DENSITY MEASUREMENTS

- Evolution of density after GONDOLÉ irradiation program (comparison of density before GONDOLÉ irradiation and after the 8 phases of irradiation)
  - Positive values = densification
  - Negative values = swelling



- Positive and negative variations
- Limited variations in the uncertainty range
- Small variations on CF1 and CH1

## 2.1. RESULTS – DENSITY MEASUREMENTS

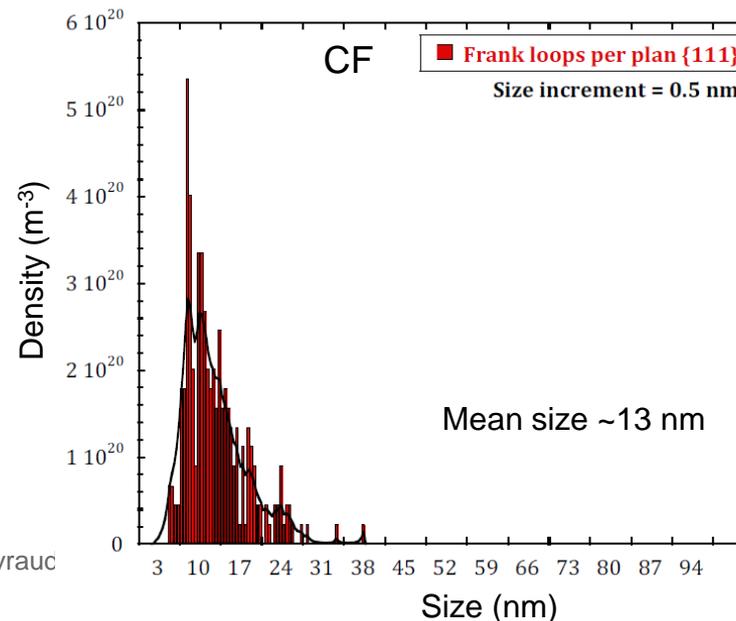
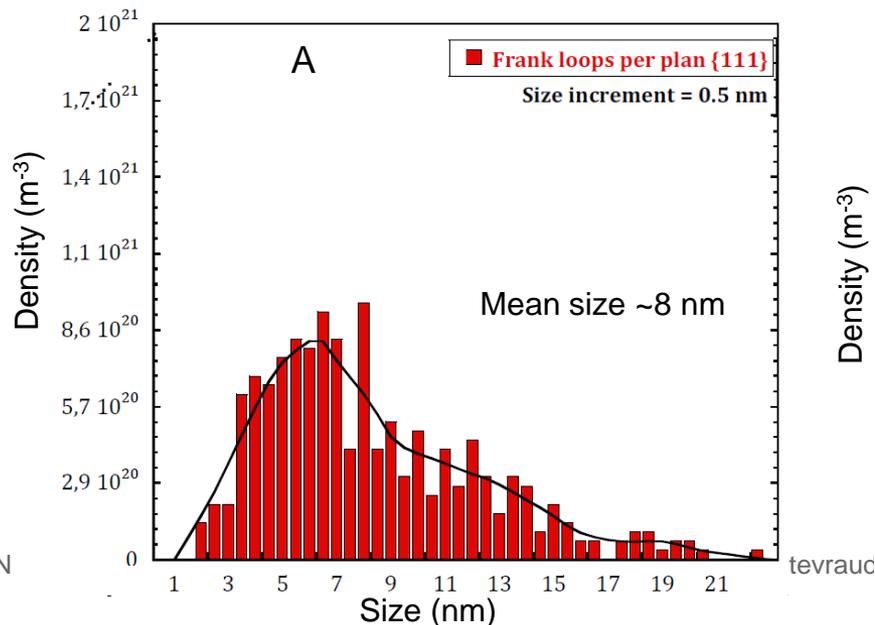


- Same results as pre-irradiated specimens
- Positive and negative variations
- Limited variations in the uncertainty range
- Small variations on X1

- Density measurements revealed that the density evolution are small and in the uncertainty range (virgin and pre-irradiated specimens)

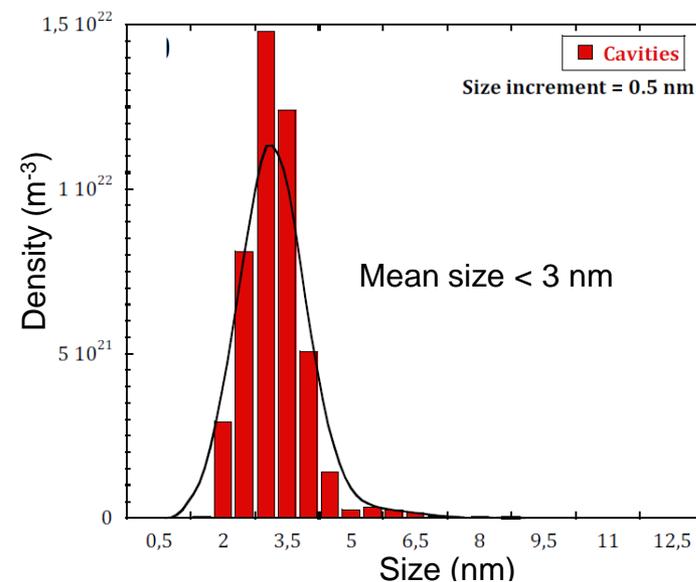
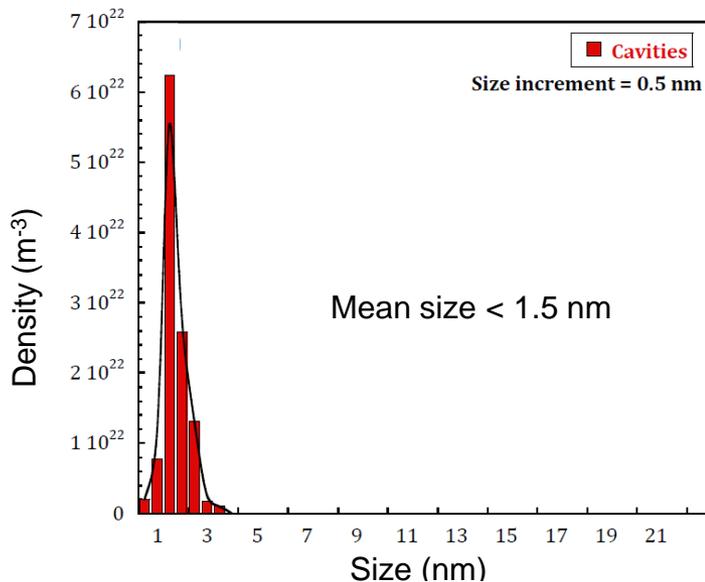
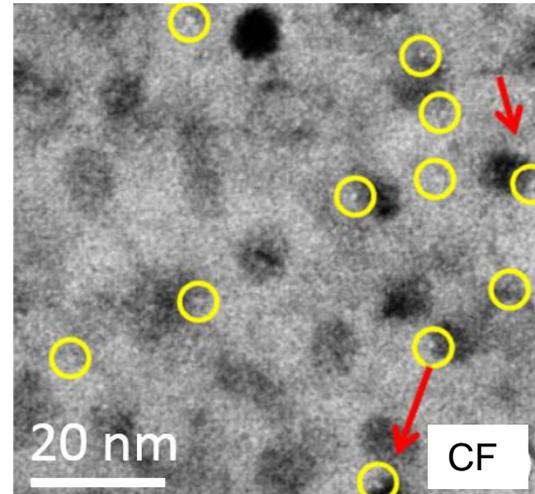
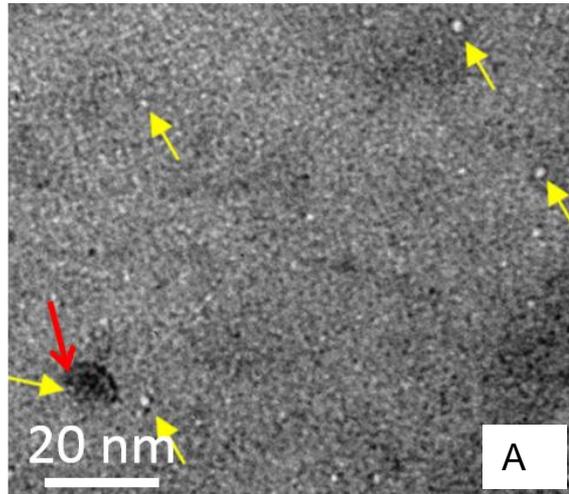
## 2.2. RESULTS – TEM CHARACTERIZATIONS

- At stages 3 and 4, specimens were also introduced as TEM disks in tubes
- TEM disks removed after part 1 for TEM characterizations:
  - A (pre-irradiated) specimen – 24 dpa – 375°C
  - CF (pre-irradiated) specimen – 44 dpa – 375°C
- Irradiated microstructure (both materials):
  - Frank loops
  - Segments of perfect dislocations
  - Precipitates
  - Cavities



## 2.2. RESULTS – TEM CHARACTERIZATIONS

### Cavities



- Small and numerous cavities
- Volume fraction limited (~0.04 and ~0.07%)

## 3. CONCLUSIONS AND PROSPECTS

### CONCLUSIONS

- Description of the GONDOLE irradiation program
- Assessment of potential swelling by density measurements
- Description of the protocol and uncertainty measurements improvements
- Limited density variation, below the uncertainty limit
- TEM characterizations of pre-irradiated materials: cavities identification, limited associated swelling

### Prospects

- To improve density measurements uncertainty (new measurements of the 'before GONDOLE irradiation state' - virgin specimens and some pre-irradiated archive specimens)
- To perform TEM characterizations of specimens available after phase 8

# Thank you for your attention

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