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## Evaluation of neutron irradiation induced embrittlement of T91 steel

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- Motivation
- SPT experiments
- Annealing effect
  - Hardness tests
  - Positron Annihilation Analysis
  - Microstructure observation
- Summary

## ➤ Motivation

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## ➤ Summary

Ferritic/Martensitic steels are considered as candidate materials for the containers of the liquid target of spallation neutron sources and accelerator driven systems (ADS) in the future.

A major concern for ferritic/martensitic steels such as T91 is the irradiation induced embrittlement.

In the present work, the embrittlement of T91 irradiated in fission reactor has been examined by using the small punch technique.

In addition, annealing temperature effect on hardness has been studied by TEM observation and positron annihilation analysis.

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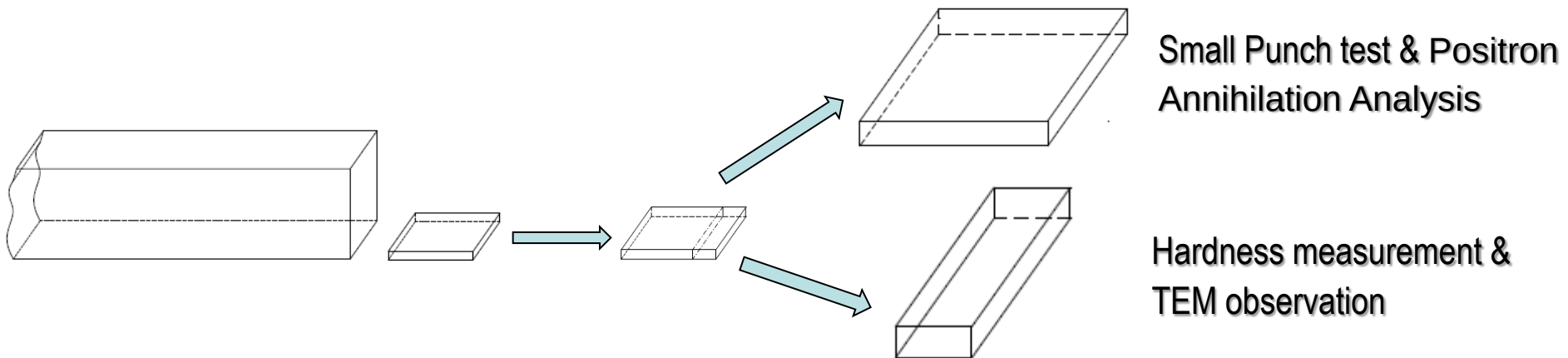
## Material & Irradiation conditions:

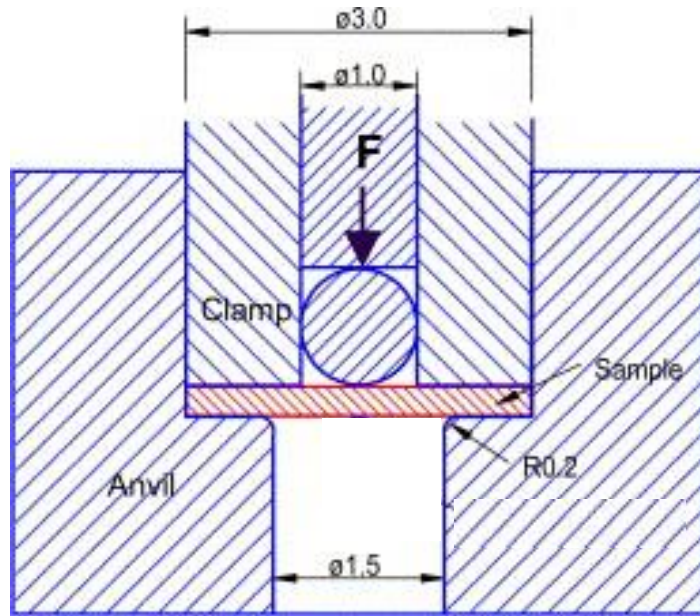
The commercial ferritic/martensitic steel T91(9Cr-1MoVNb) irradiated in BR2 during the SPIRE irradiation campaign, up to 3.58 dpa at 200 °C.

| Element | C    | Cr   | Mo   | W       | Nb   | V    |
|---------|------|------|------|---------|------|------|
| wt%     | 0.1  | 8.32 | 0.96 | <0.01   | 0.06 | 0.24 |
| Element | P    | Mn   | Ni   | B       | N    | Si   |
| wt%     | 0.02 | 0.43 | 0.24 | <0.0005 | 0.03 | 0.32 |

## Samples:

- ✓ 3\*3 mm<sup>2</sup> sheets have been cut from KLST sample by diamond wire saw, the thickness are ~ 400 μm;
- ✓ Polished with sand paper down to 243 ~ 254 μm, the final polishing is made with 1200 Grid sand paper;
- ✓ Cleaned in acetone and ethanol with ultrasonic bath.





*A schematic overview of the set-up of small punch test (from X. Jia, Y. Dai / JNM 323 (2003) 360–367).*

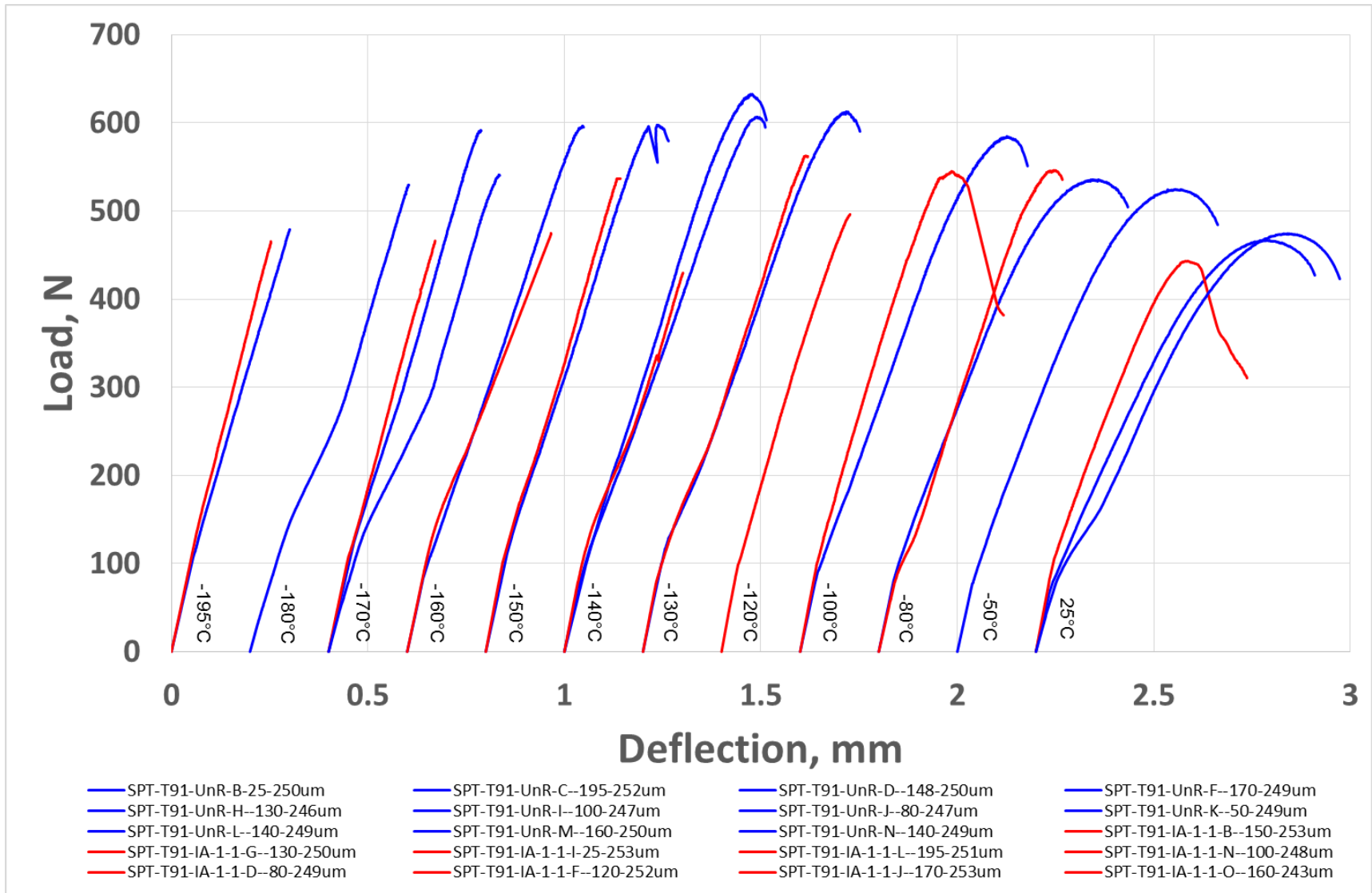
Puncher is installed into MTS mechanical testing machine (max. load capacity of 2 kN);

Balls have  $\varnothing = 1 \text{ mm}$ ;

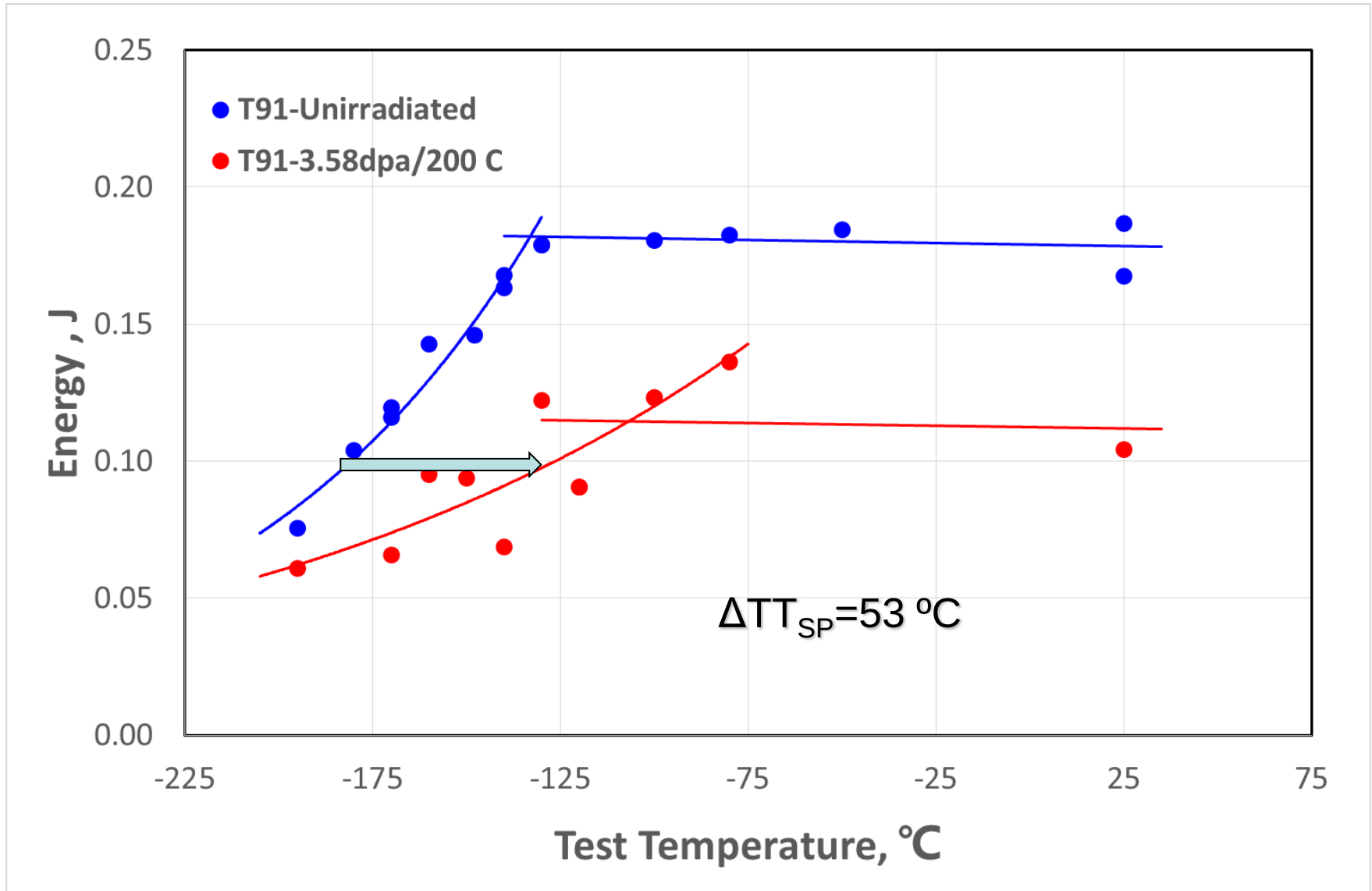
Constant crosshead speed of  $0.3 \text{ mm} \cdot \text{min}^{-1}$ ;

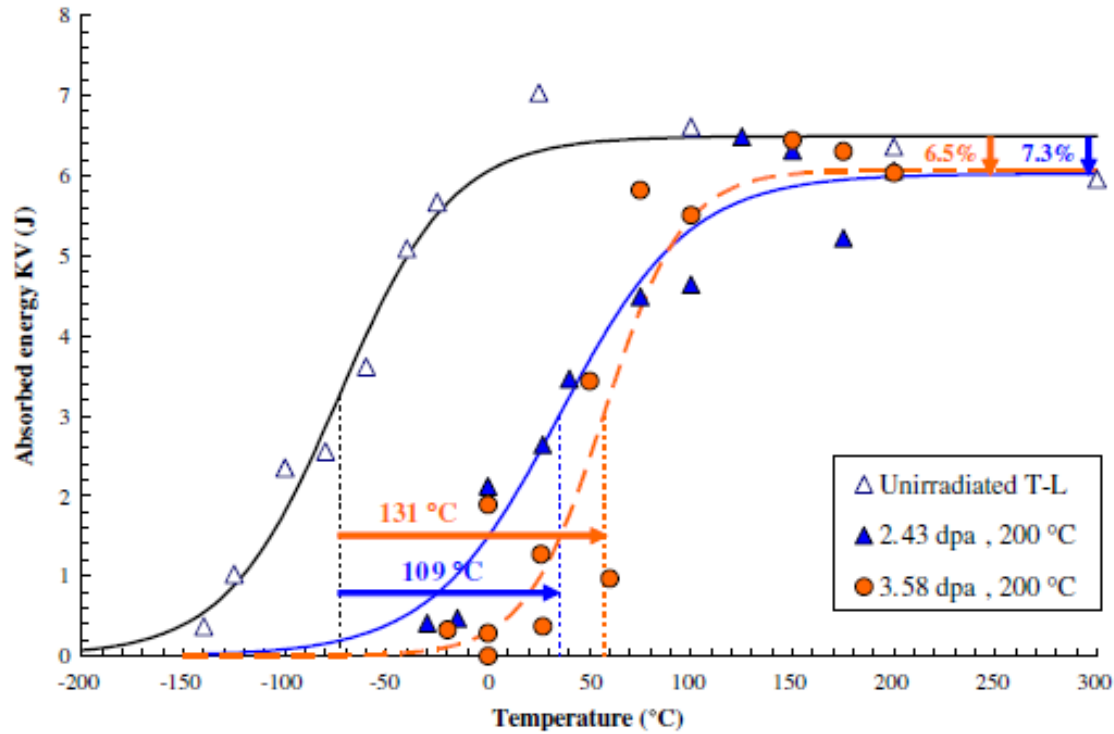
Temperature is measured in 0.5 mm away from the sample;

The displacement is measured as displacement of the testing machine crosshead.



*The curves are shifted in deflection on  $N \cdot 0.2$  (mm); N – the number of curve*





$$\Delta DBTT_{KLST-Charpy} = 131 \text{ }^{\circ}\text{C}$$

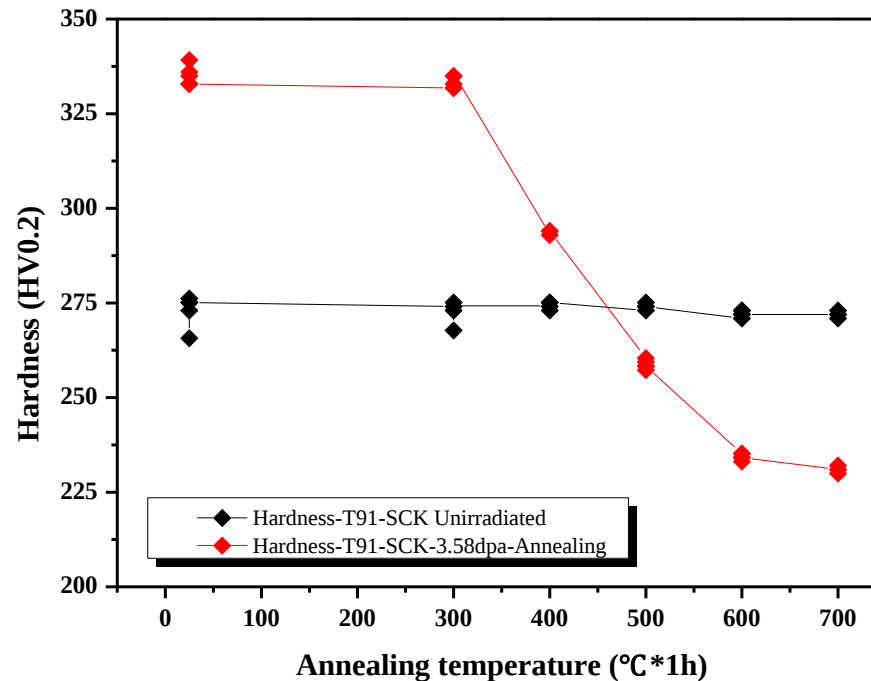
$$\alpha = \frac{\Delta TT_{SP}}{\Delta DBTT_{Charpy}} \approx 0.40$$

*DBTT shift from KLST Charpy tests (M. Matijasevic, E. Lucon, A. Almazouzi/ JNM 377 (2008) 101–108)*

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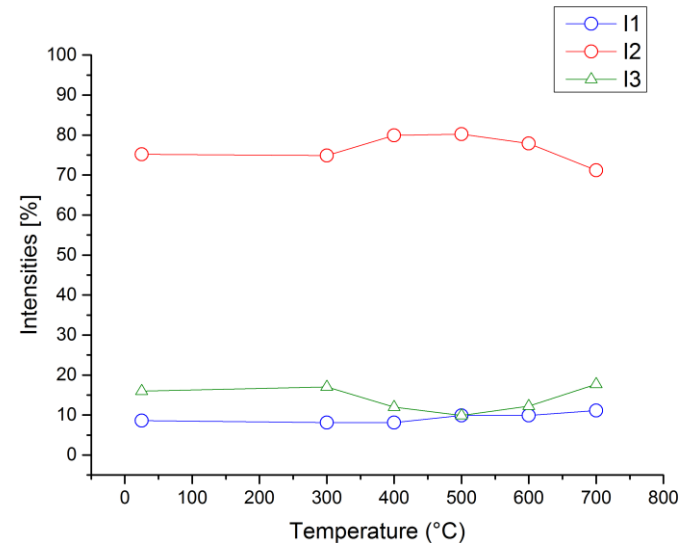
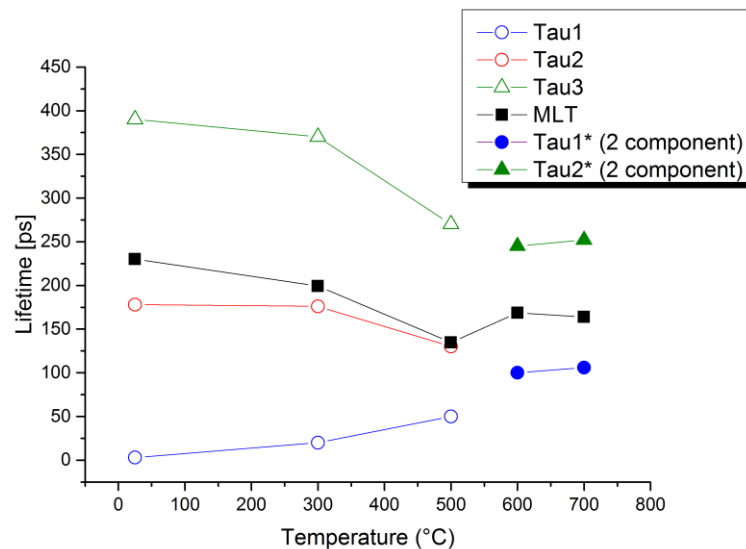
Annealing on the samples of hardness tests, Positron Annihilation Analysis and TEM were conducted step by step at a series temperature of 300~700°C;

Micro-hardness have been measured before and after annealing.



# Positron Annihilation Analysis

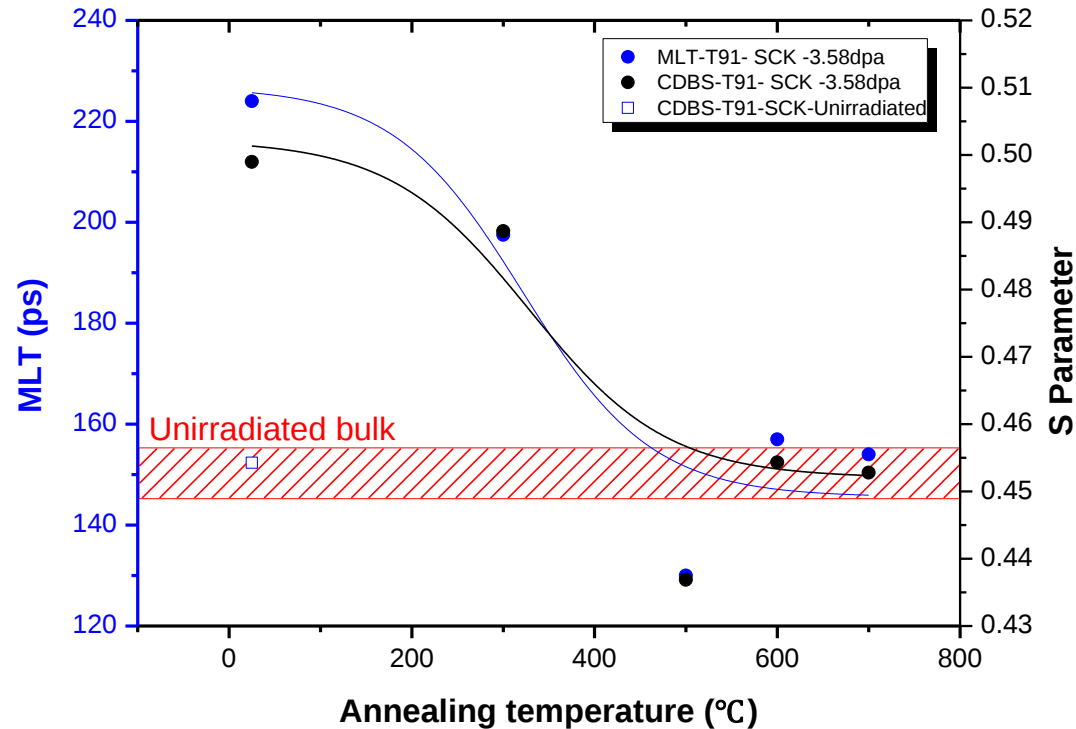
Positron annihilation techniques were used to investigate small defects induced by irradiation;



## Characterization of the major trapping centers in the as-irradiated materials

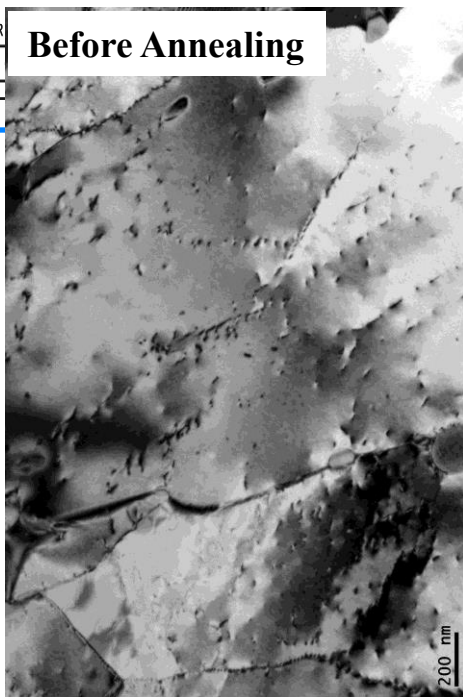
| n – irradiated T91 (2.95dpa) |                                   |
|------------------------------|-----------------------------------|
| Defect 1 (tau2)              | Dislocations + some point defects |
| Defect 2 (tau3)              | Void (~20 vacancies)              |
| $N_1$ [m <sup>-3</sup> ]     | $5.2 \times 10^{24}$              |
| $N_2$ [m <sup>-3</sup> ]     | $5.0 \times 10^{22}$              |

Number density of defect 1 was calculated using specific trapping rate for mono-vacancy ( $t_{1V} \cong t_{12V-12He}$ ). For defect 2, data on irradiated Cu were used [Eldrup, Singh, *J. Nucl. Mater.* 251 (1997) ]

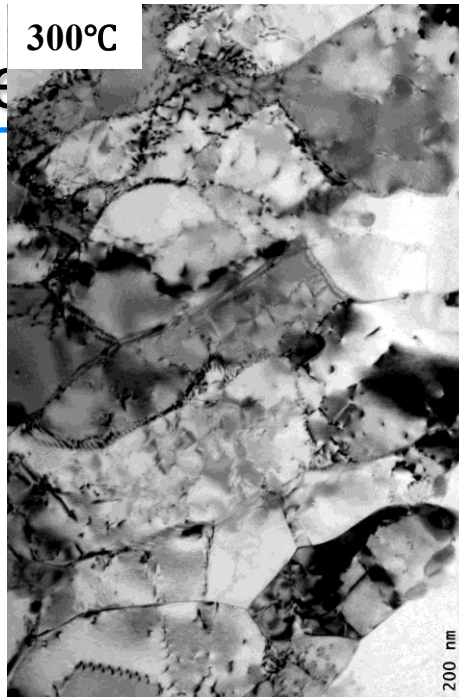


T91 steel irradiated in a fission reactor shows a complete recovery of the microstructure after annealing at  $> 500^{\circ}\text{C}$ .

**Before Annealing**



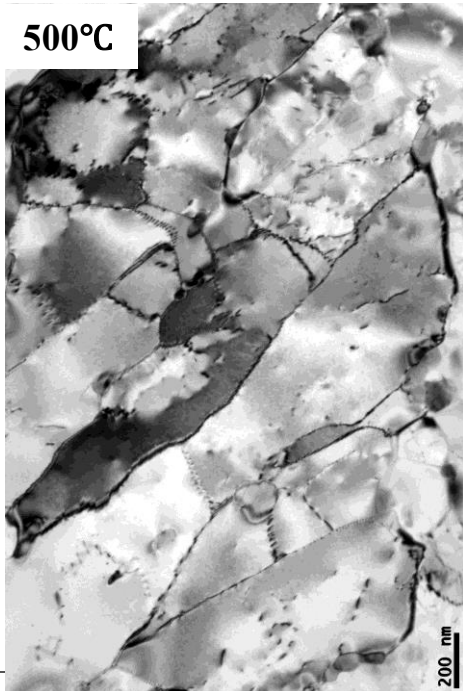
**300°C**



**400°C**



**500°C**



**600°C**

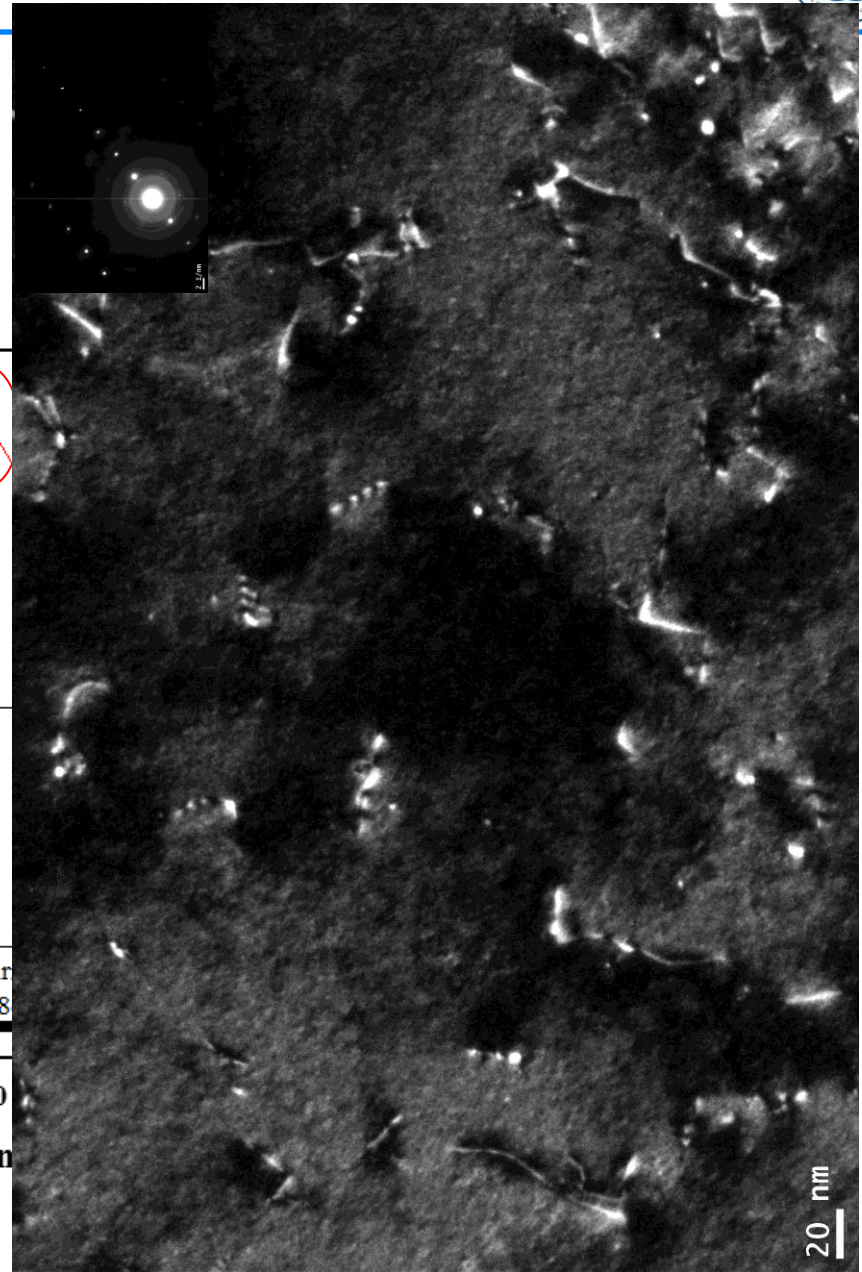
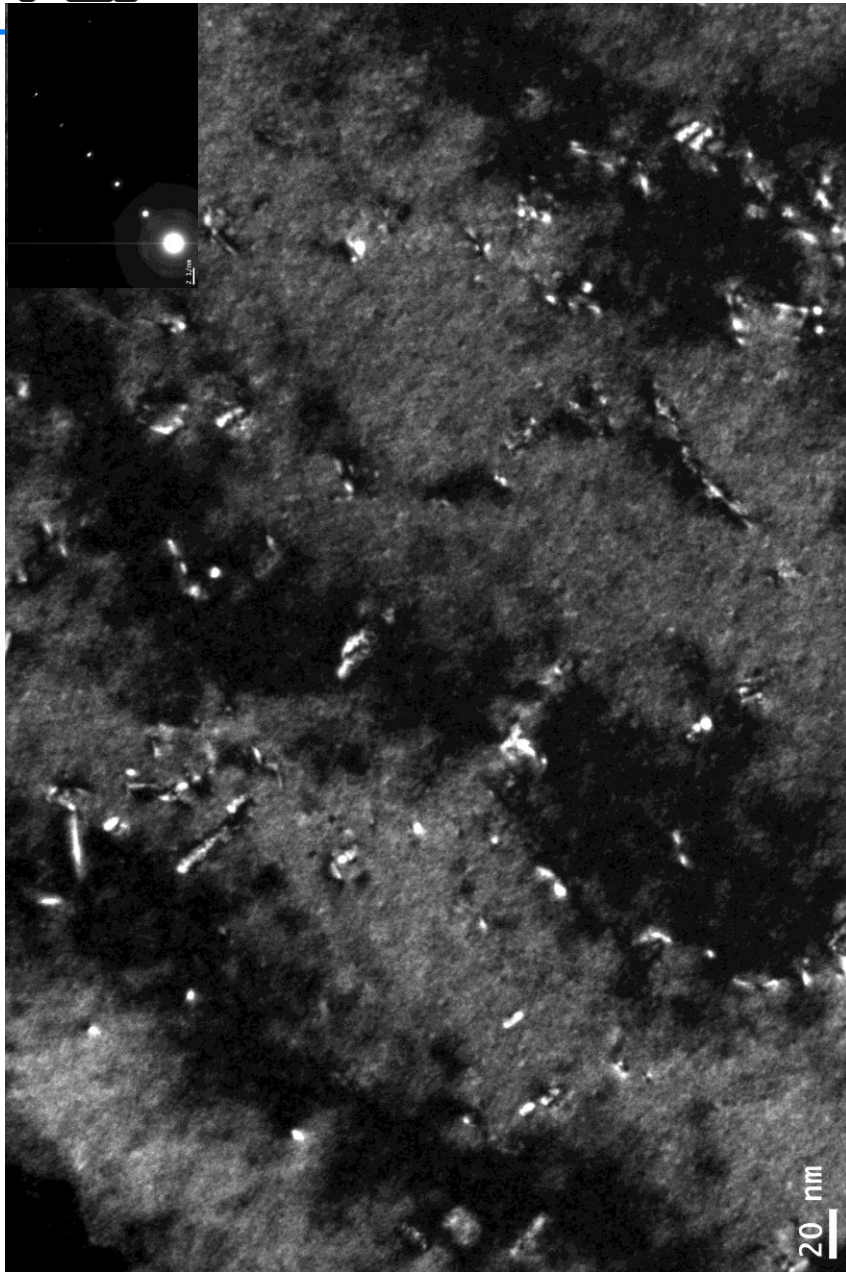


**700°C**

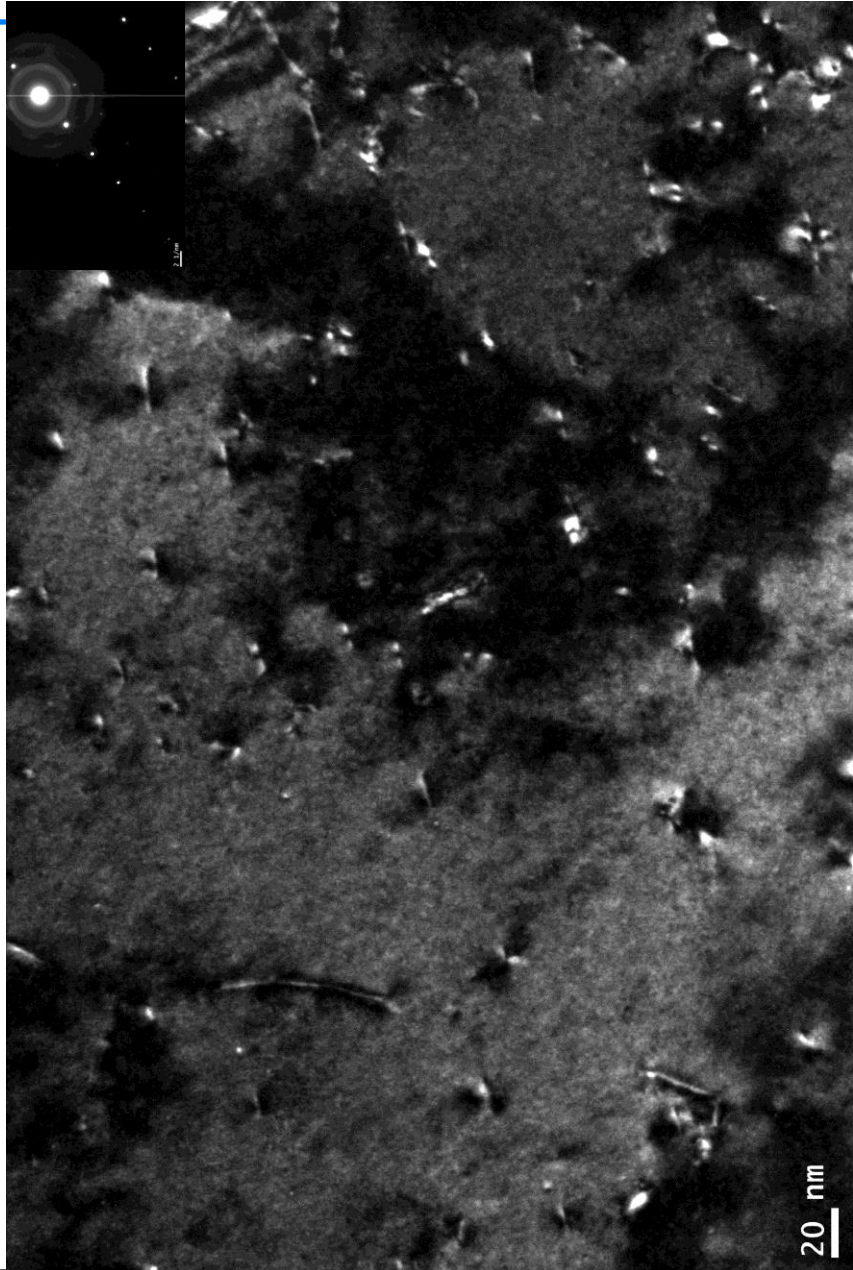


Before annealing

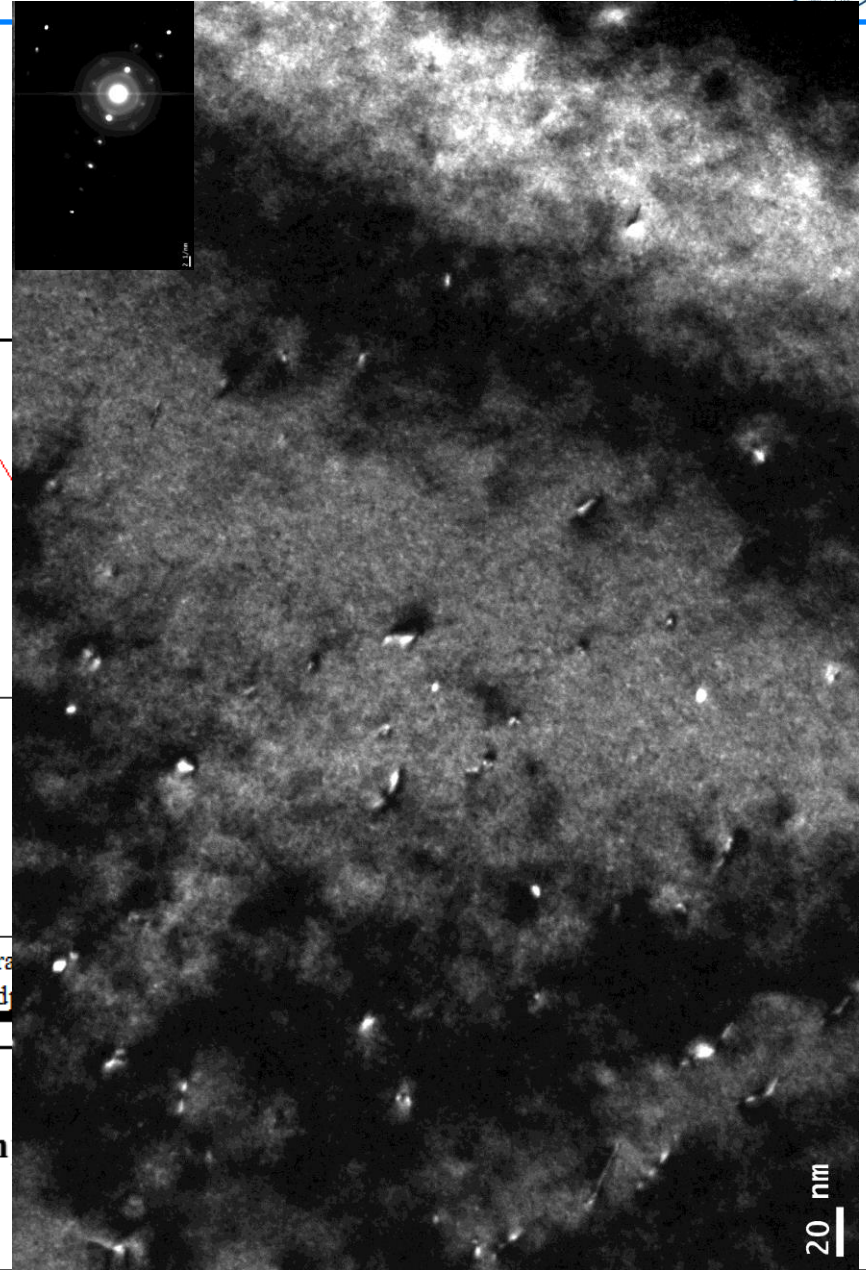
After annealing (300°C)



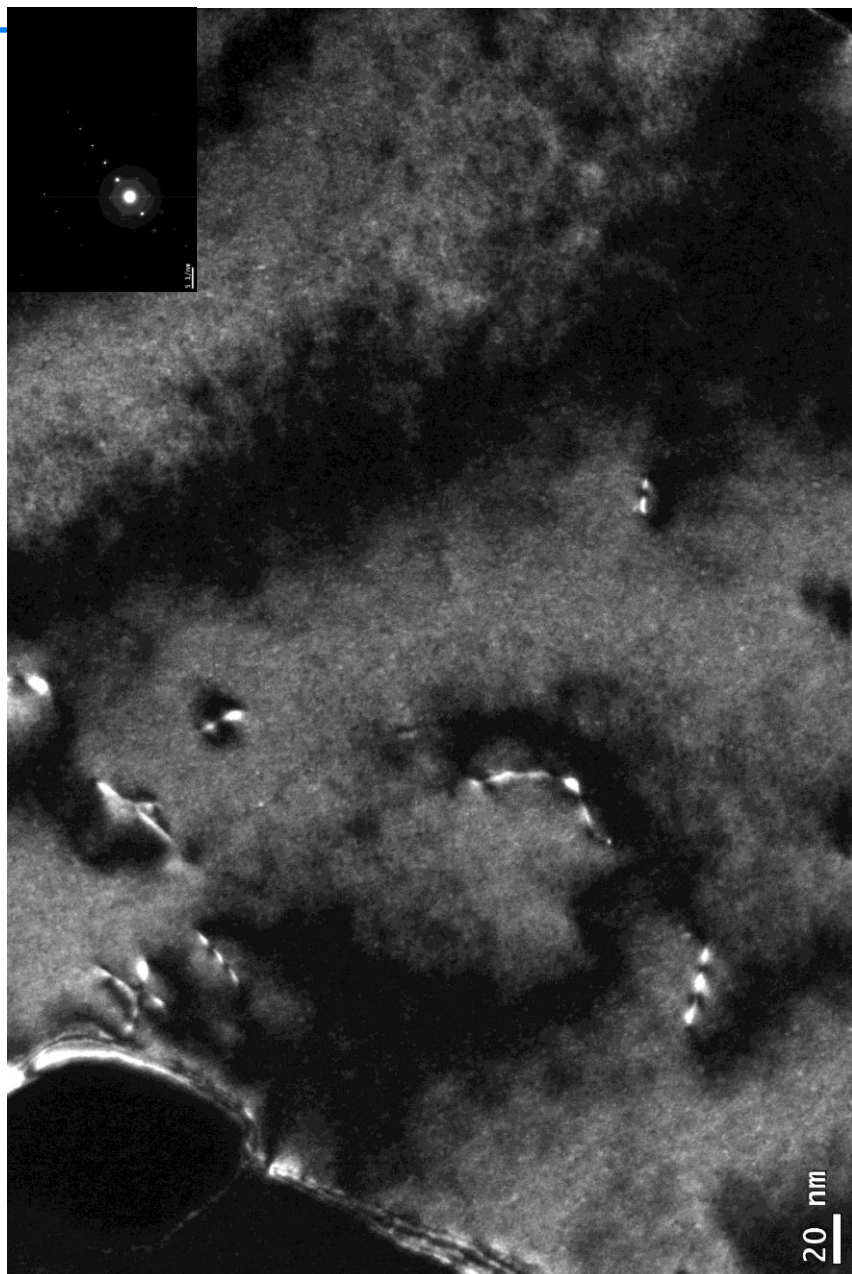
After annealing (400°C)



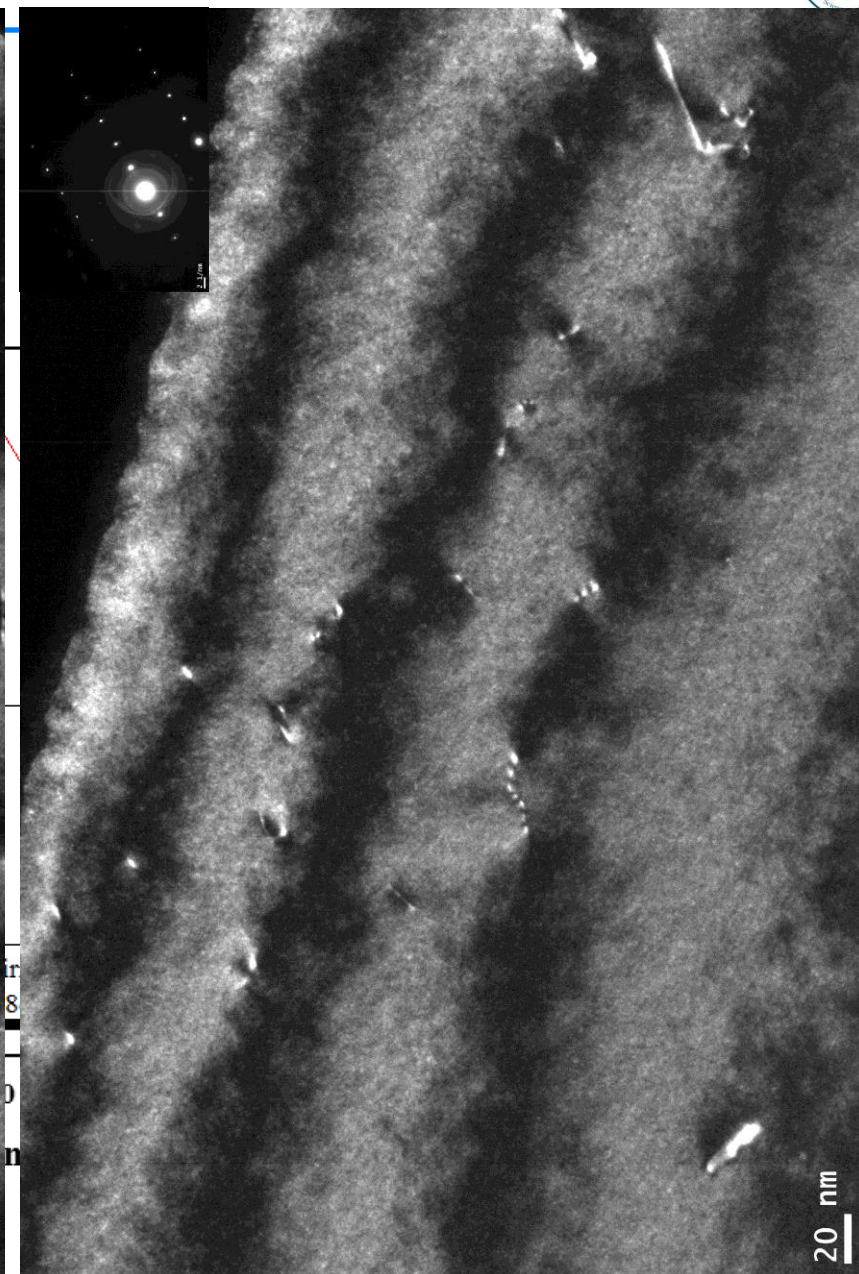
After annealing (500°C)

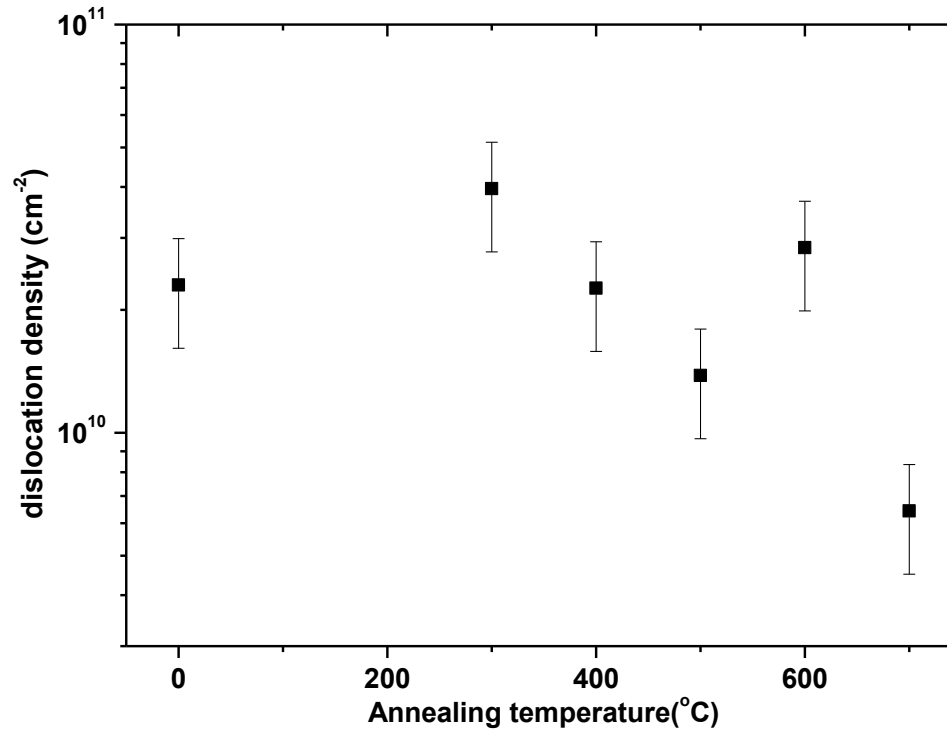


After annealing (600°C)



After annealing (700°C)





### Dislocation line density

TEM observation showed that the density of dislocation line decreased with the increasing of annealing temperature.

Before annealing, average loop size is 2.49 nm and its density is  $1.78 \times 10^{11} \text{ cm}^{-2}$ .

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- Set of SP experiments for  $TT_{SP}$  evaluation has been performed, and the ratio of  $\Delta TT_{SP}$  to  $\Delta DBTT_{KLST}$  is 0.40;
- Hardness decreased with the increasing of annealing temperature, and it become stable at the annealing temperature  $>500\text{ }^{\circ}\text{C}$ ;
- Positron Annihilation analysis results showed that a complete recovery of the microstructure after annealing at  $> 500\text{ }^{\circ}\text{C}$ ;
- TEM observation showed that the density of dislocation line after annealing have the similar tendency with hardness.

# Thanks for SCK-CEN to provide the samples !

# Thank you !