# Modelling oriented experiments: Behaviour of Fe-Cr-C model alloys under neutron irradiation

Work supported by EFDA/TTMS-007

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## Content

- Introduction and Objectives
- Experimental techniques
- Results and discussion
- Conclusion

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# Introduction

In Fusion technologies:

| Operating conditions | Properties needed |
|---|---|
| Temperature (200 to 550°C) | High thermal conductivity and heat resistance; low thermal expansion |
| High flux and high dose (100 to 200 dpa); high He/dpa production rate (3 to 15 appm/dpa) | Low DBTT shift; sufficient strength with limited loss of ductility and toughness; low swelling rate |
| High stress level (100 MPa) | High creep resistance |



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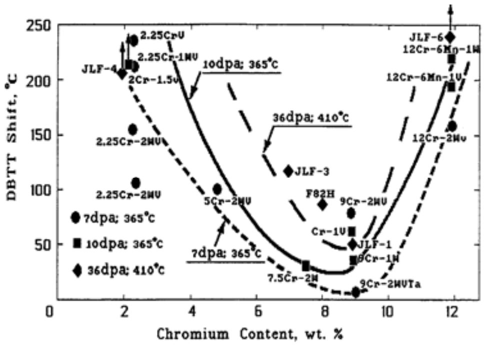
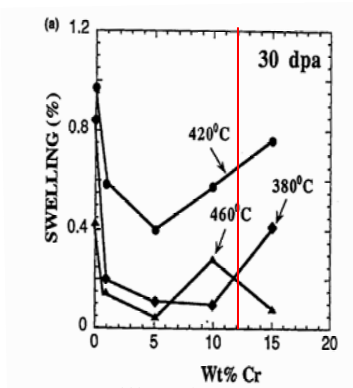
# Why 9% Cr ?




Lowest DBTT shift was found for 9% Cr steels → material is less brittle




Low swelling for Cr-content between 5 and 10%

Fe-9%Cr seems to be the best candidate

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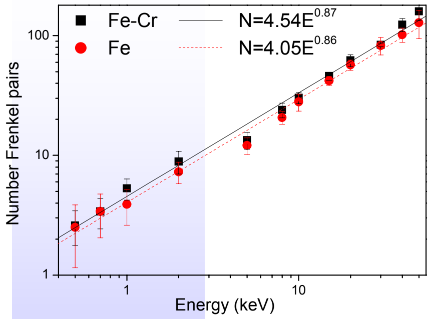
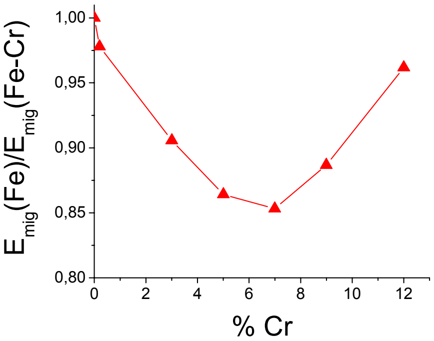
# Effect of Cr on defect production and migration

Molecular Dynamic Simulation by D. Terentyev et al.




Cr has almost no effect on defect production rate




However: the presence of Cr affects strongly the diffusitivities of irradiation induced defects

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# Objectives

- To assess experimentally the effect of Cr – concentration on defect production and accumulation in model alloys: how do they compare with steels under irradiation?
- To investigate the mechanisms of irradiation induced changes of the mechanical properties of high Cr F/M steels
- **Ultimate aim**: Provide reliable experimental database for model validation

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# Approach

- Experimental investigation of Fe, model alloys of different Cr content and industrial steels after neutron irradiation (same neutron flux, doses & temperature):

We chose as matrix:

- I- Pure Fe and ultra-pure Fe-9Cr
- II- Industrial pure Fe and Fe-2,5,9,12 Cr
- III- Conventional and LA Ferritic martensitic steels



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# Investigated Materials with chemical composition and heat treatment

| Alloy | Mn | Si | P | Si | Al | Ti | Cr | Ni | Cu | Nb | B | Mo | C | N | V | W |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 251 | 0.009 | 0.02 | 0.013 | 0.0020 | 0.003 | 0.004 | **2.5** | 0.044 | 0.005 | / | / | / | 0.008 | 0.0173 | 0.001 | / |
| 259 | 0.02 | 0.04 | 0.011 | 0.006 | 0.0033 | 0.0028 | **5.04** | 0.06 | 0.01 | / | / | / | 0.02 | 0.0344 | 0.001 | / |
| 252 | 0.03 | 0.09 | 0.012 | 0.00066 | 0.0069 | 0.0034 | **9** | 0.07 | 0.01 | / | / | / | 0.02 | 0.0353 | 0.002 | / |
| 253 | 0.03 | 0.11 | 0.05 | 0.006 | 0.003 | 0.0037 | **11** | 0.09 | 0.01 | / | / | / | 0.03 | 0.0397 | 0.002 | / |

| steel | C | Cr | Mo | W | Nb | Ta | V | P | Mn | Ni | B | N | Si |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| T91 | 0.1 | **8.32** | **0.96** | **<0.01** | **0.06** | **--** | **0.24** | 0.02 | 0.43 | 0.24 | <0.0005 | 0.03 | 0.32 |
| E97 | 0.12 | **8.96** | **<0.001** | **1.1** | **<0.001** | **0.13** | **0.19** | <0.005 | 0.43 | 0.007 | <0.001 | 0.016 | 0.07 |

**Normalisation**:

**for alloys :1050 °C for 3h, after air cooling**

**for steels: 1040-1070°C for 1h**

**Tempering**:

**for alloys : 730°C for 4 h 10min, after air cooling**

**for steels: 730-780°C for 1h**

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## Experimental characterisation techniques (before and after irradiation)

### Microstructure:

- Metallography / Optical Microscopy
- SEM (Scanning Electron Microscopy)
- TEM (Transmission Electron Microscopy) JOEL 3010:
  - 1 mm disk specimen technique (magnetism)
  - Bright field (BF), Dark field (DF)
  - Weak beam dark field (WBDF), g(4g-6g)
- PAS

### Mechanical properties:

- Hardness (HV5)
- Tensile testing
- Charpy
- Small punch
- Fracture thougness
- Compression (EPFL/CH)



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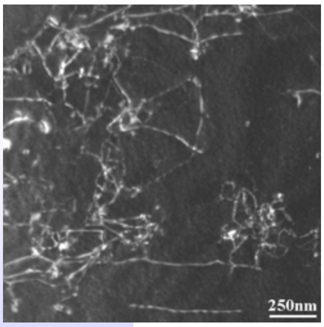
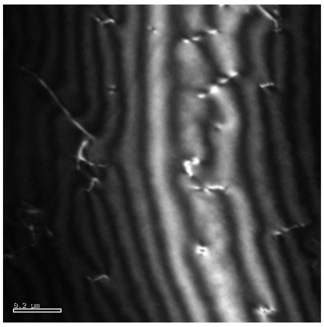
## Microstructure of pure Fe and ultra high pure Fe-9Cr before irradiation




**Pure Fe**



**Pure Fe-9 Cr**

- Ferrite , with grains size from 15-25 µm
- Dislocations are of type b= a/2 <111>, and their density is ($1.7x10^9$ $cm^2$ for FeCr, and $7x10^8$ $cm^2$ for Fe)

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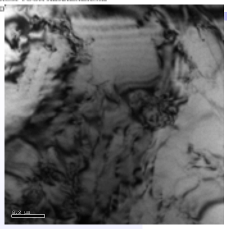
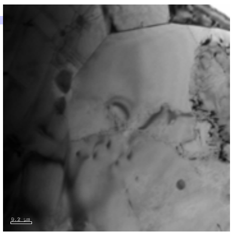
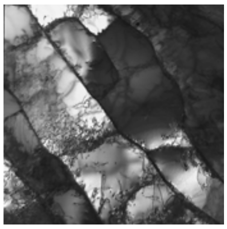
## TEM micrographs with microstructure of Fe-Cr alloys and steels before irradiation



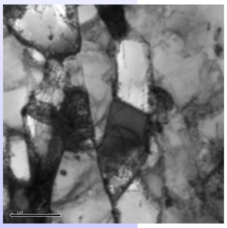
**Fe-2.56 %Cr**



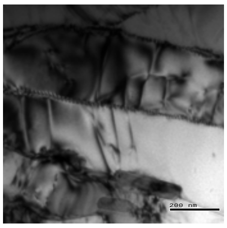
**Fe-4.62 %Cr**



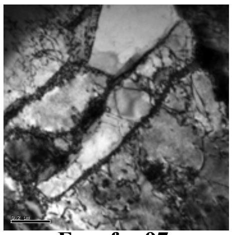
**Fe-8.39 %Cr**



**Fe-11.62%Cr**



**T91**



**Eurofer 97**

the structure changes from fully ferritic to ferrite + bainite: grain size decreases

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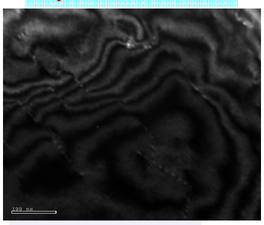
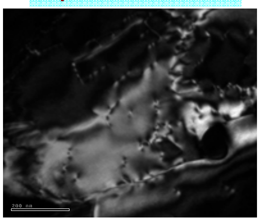
## Dislocation structures in unirradiated material with their density

$\rho = 1.2 \times 10^9\ cm^2$



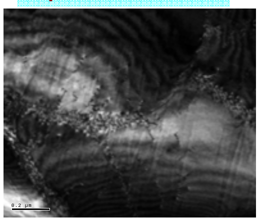
**Fe-2.56%Cr**

$\rho = 5.8 \times 10^9\ cm^2$

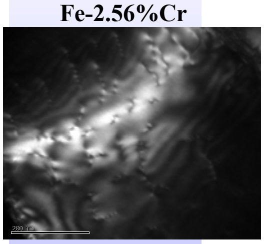


**Fe-4.62%Cr**

$\rho = 6.3 \times 10^9\ cm^2$

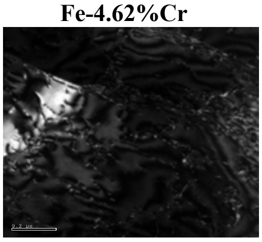


**Fe-8.39%Cr**



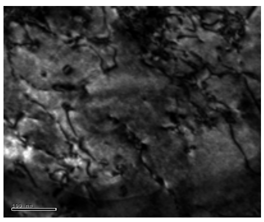
**Fe-11.62%Cr**

$\rho = 5.5 \times 10^9\ cm^2$



**T91**

$\rho = 4.7 \times 10^9\ cm^2$



**Eurofer 97**

$\rho = 7.8 \times 10^9\ cm^2$

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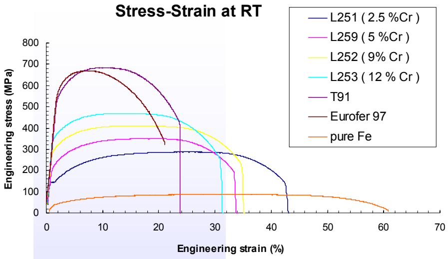
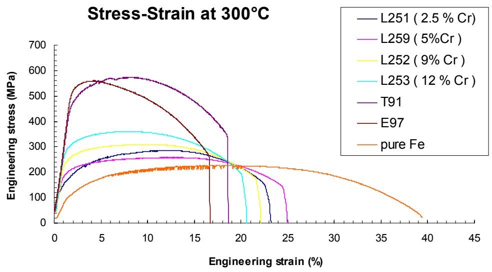
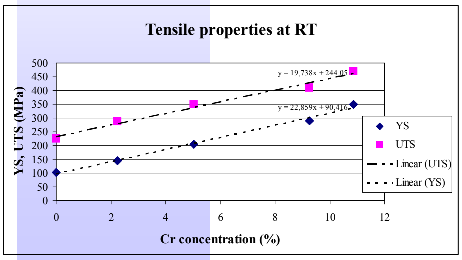
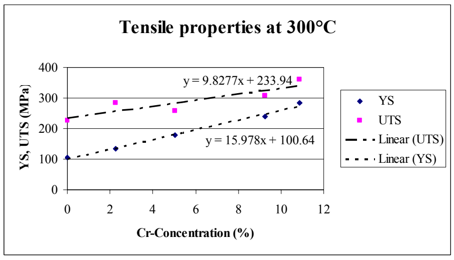
# Tensile tests

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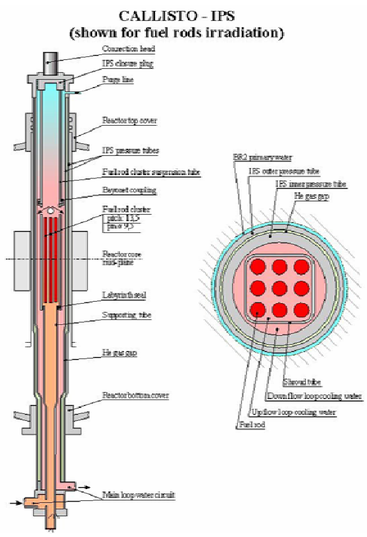
# Irradiation in BR2 (MIRE-Cr)

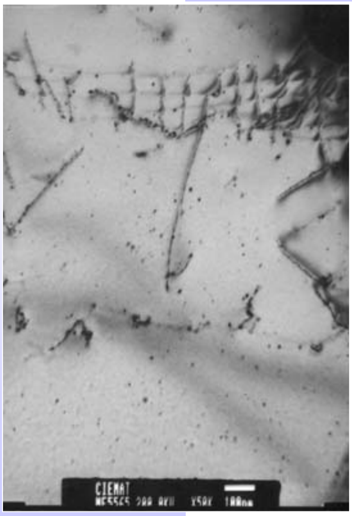
## Irradiation conditions:

- **Temperature: 300°C**
- **Pressure: 1.0 MPa ≤ P ≤ 15.7 MPa**
- **neutron flux ( > 1Mev) : $10^{13}$ n/(cm$^2$s)**
- **3 groups of specimens for 1, 3 & 5 cycles**
- **3 doses of 0.06, 0.6 and 1 dpa**

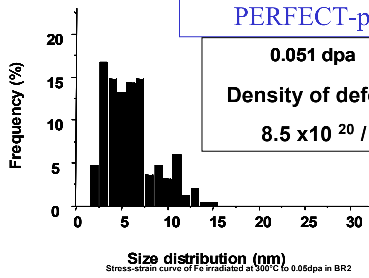


# Microstructure of pure Fe after irradiation at BR2

CIEMAT ( Spain ) within PERFECT-project

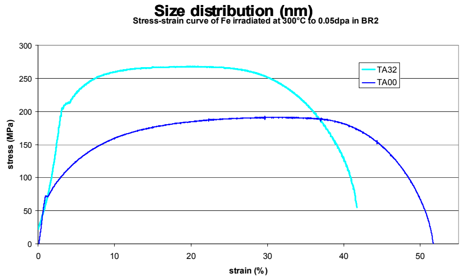



**Pure Fe**




**0.051 dpa**

**Density of defects = 8.5 x10 $^{20}$ / m$^3$**

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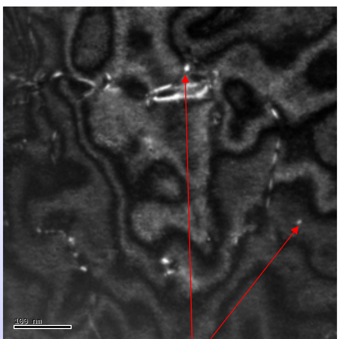
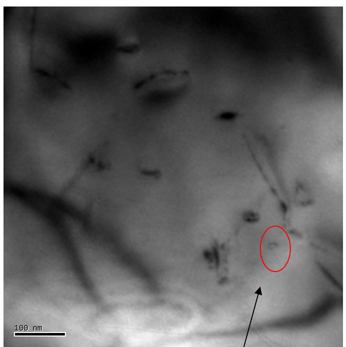
# Defects induced by irradiation

- Need of WBDF (g, 4-6g) imaging technique:

  Example for 2.5 % Cr alloy with <110> image condition:






Defects

d= 6-8 nm , **ρ= 0.7 x 10 $^{21}$/ m$^3$**

small loop

d=12 nm

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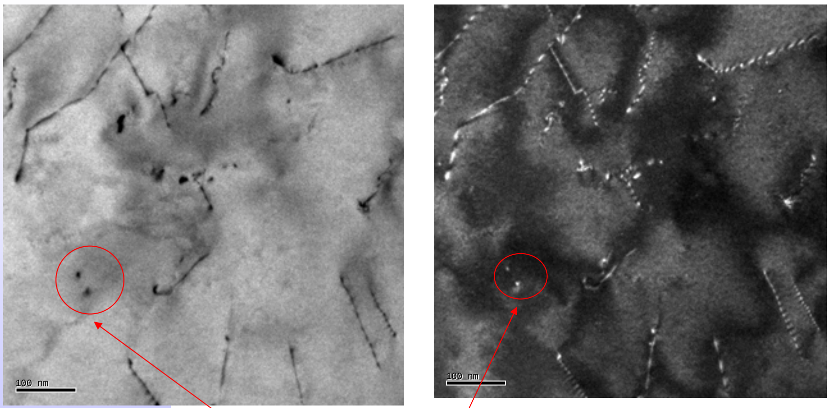
## Fe-9% Cr alloy after irradiation




Defects , d = 4 -7 nm and ρ = 1.325 x10 [21] / m[3]

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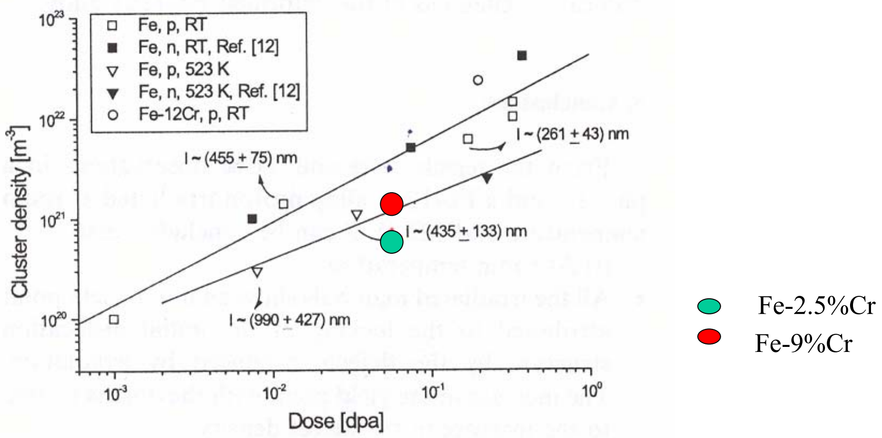
## Dose dependence of the defect density in Fe and Fe-Cr alloys




M.I.Luppo et all.JNM(2000)

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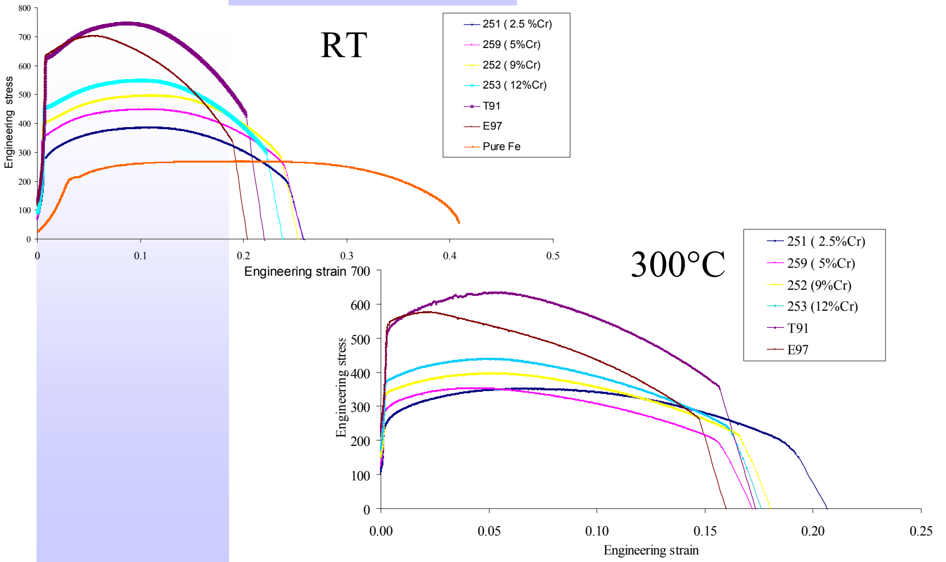
## Tensile test at RT and 300°C for 0.06 dpa

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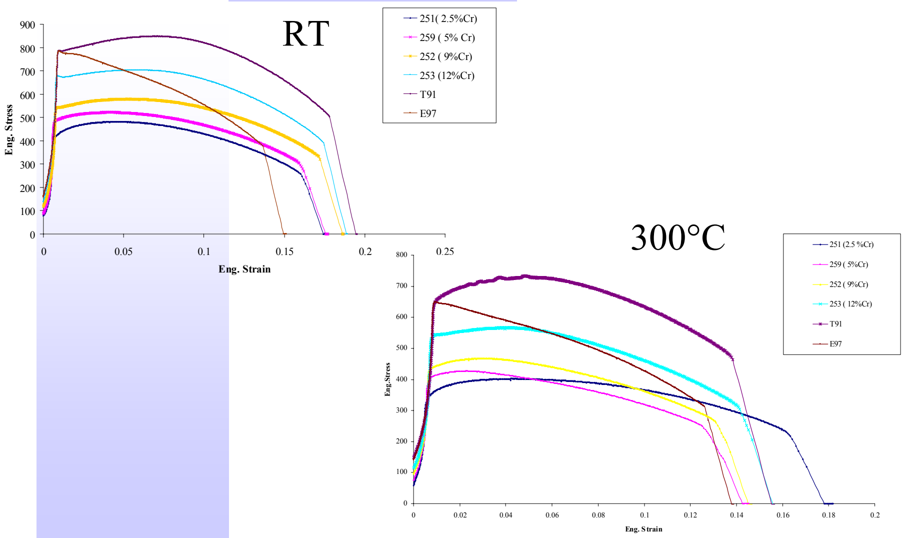
## Tensile test at RT and 300°C for 0.6 dpa



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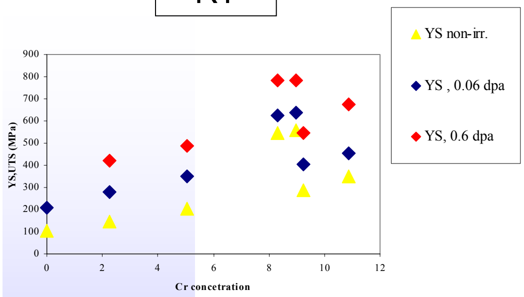
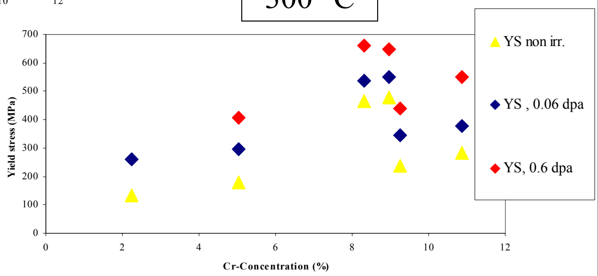
## Tensile properties and their comparison

RT




Neutron irradiation at 300°C results in hardening of Fe-Cr alloys and it increases with doses

300 °C




Increase of YS can be observed, as well as loss of ductility , compared with non-irradiated material

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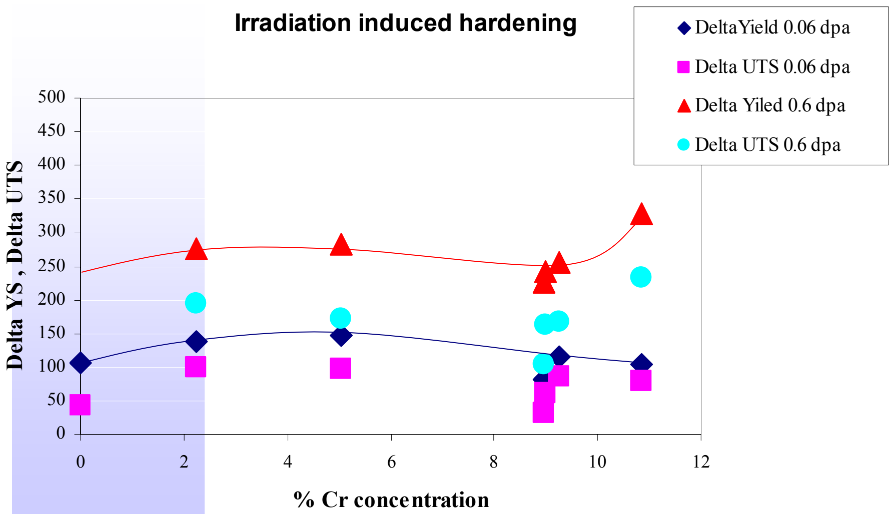
## Hardening



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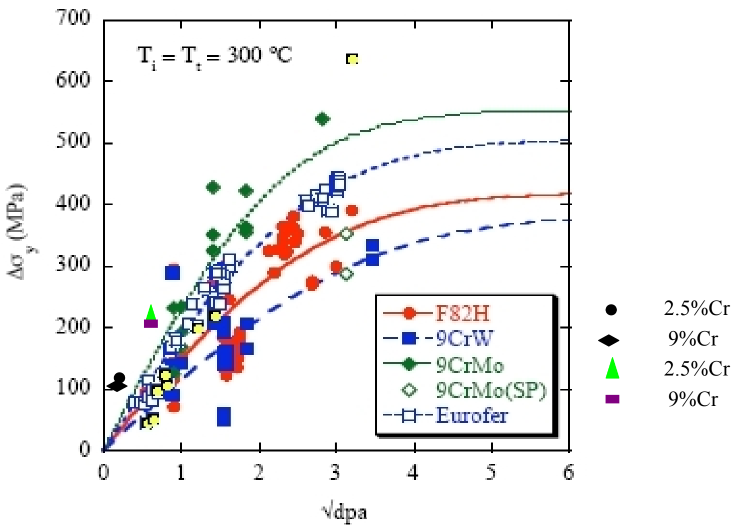
## Comparison with other FM steels

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## Summary after irradiation at 300°C for doses of 0.06 dpa and 0.6 dpa

- Microstructure showed small defects induced by irradiation (work is still in progress)
- Neutron irradiation results in hardening of Fe-Cr alloys followed by reduction of their ductility
- The hardening depends on Cr concentration and it increases with doses of irradiation

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# Outlook

- The irradiation campaign should finish by end 2005
- 2005-2006 will hence be needed for PIE, using all mentioned techniques
- The combination of characterisation techniques on model materials will provide data on both microstructure and mechanical properties for systems close to steels
- These data will both be useful for model validation and to allow a wide correlation (model alloys, steels, ...) between microstructure, composition and mechanical behaviour under irradiation