# NANOSTRUCTURE EVOLUTION IN STRUCTURAL MATERIALS UNDER HEAVY ION IRRADIATION

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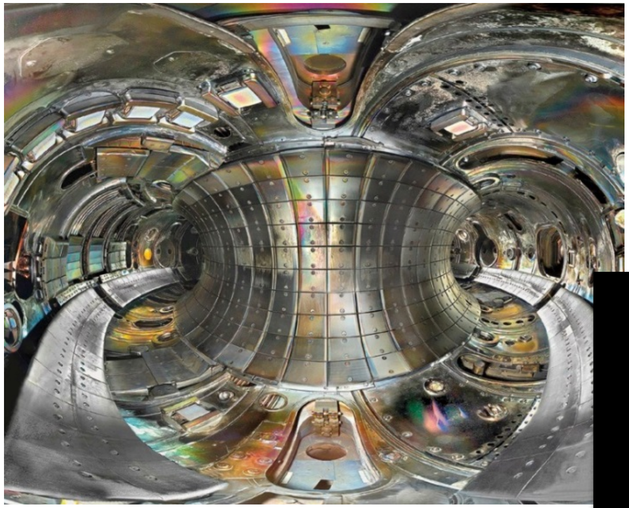
# Outline

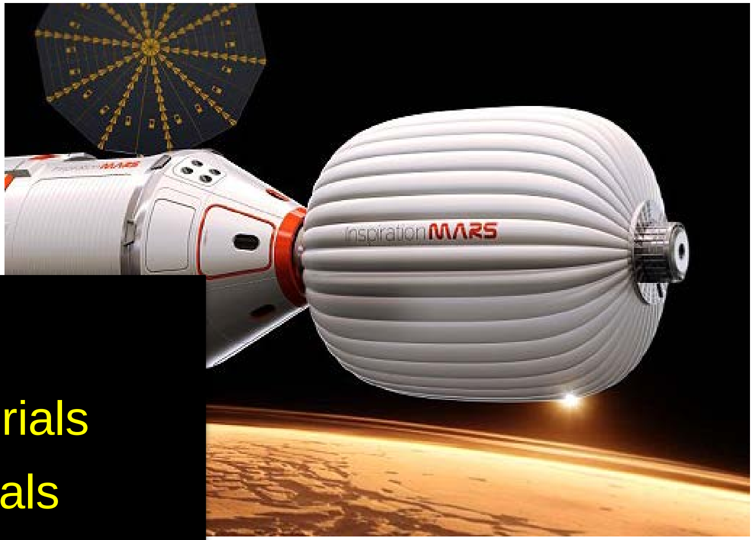
1. Introduction. Materials for extreme conditions. Interaction of heavy ions with matter
2. Heavy ion irradiation experiments
3. Microscopic techniques for study of materials reconstruction under irradiation
4. Behavior nanostructured steels under heavy ion irradiation (TEM&APT)
5. Mater science complex for NICA
6. Conclusions

# Materials for extreme environments

Advanced fusion and fission reactors



Components for space devices



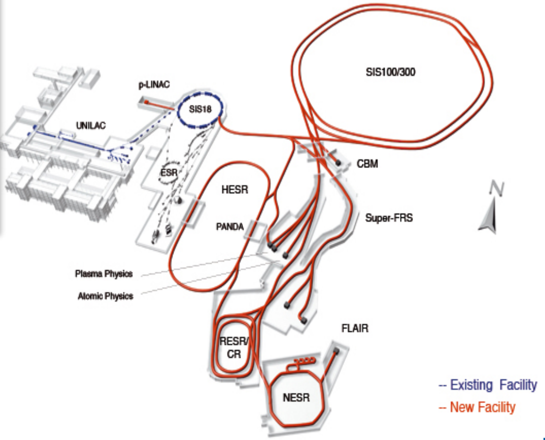

Reliability of:

- functional materials
- isolating materials
- electronic devices

Risks:
embrittlement, cracking, swelling, activation...

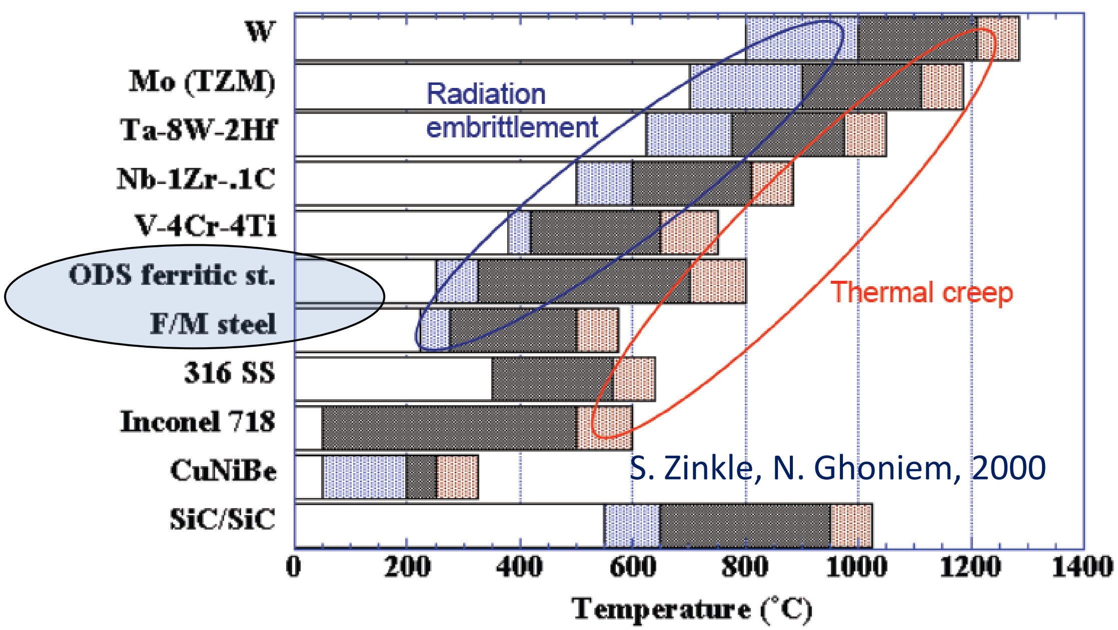
LHC

FAIR

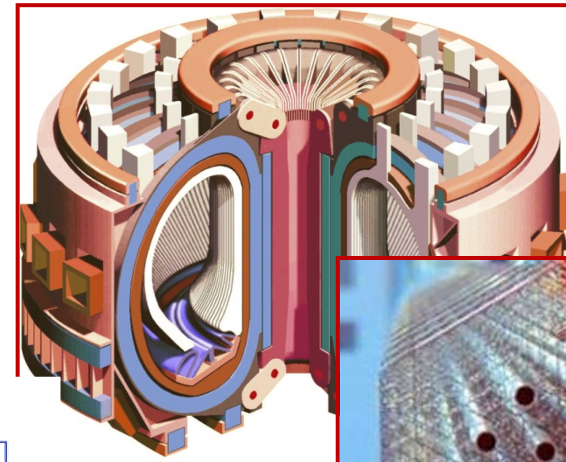


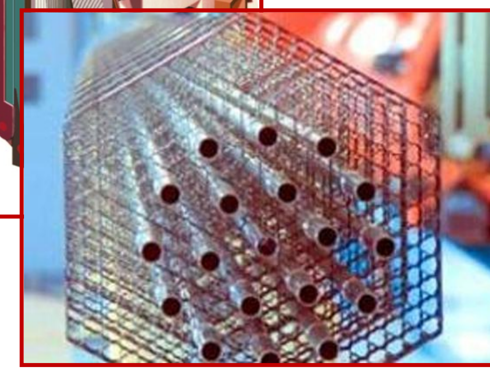
# New Generation Structural Materials (Steels)

✓ **Requirements:**
- Higher damage dose >150 dpa;
- Higher operating temperatures >700ºC;
- Should have reduced activation;
- Corrosion resistance;



Structural Material Operating Temperature Windows: 10-50 dpa

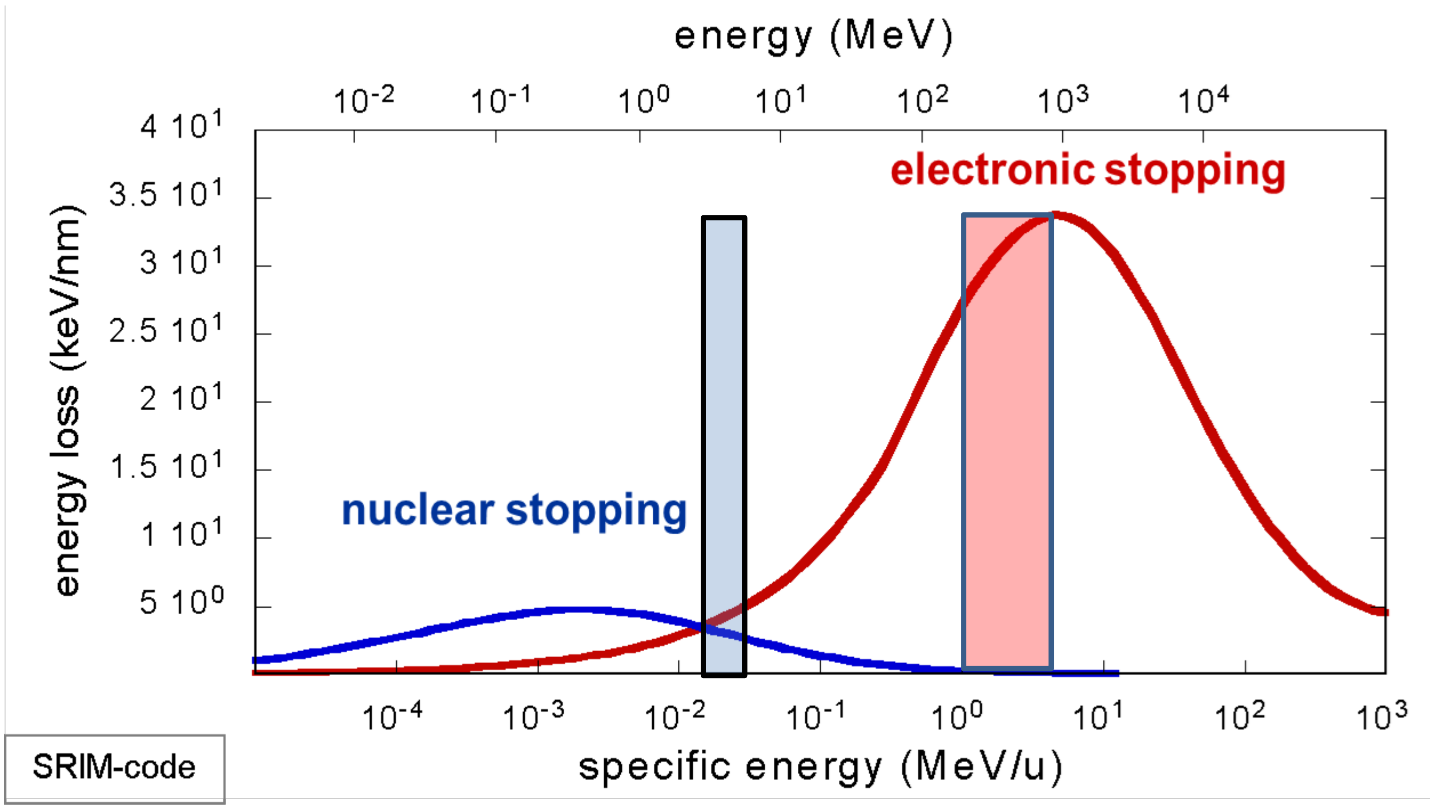




✓ **General way: nanostructured materials**

Oxide and carbonitride hardening: formation of nanosized $Y_2O_3$, $Y_2Ti_2O_7$, VC, M(C,N), $M_{23}C_6$ etc. particles

# Ion energy loss for low and high energy heavy ion irradiation

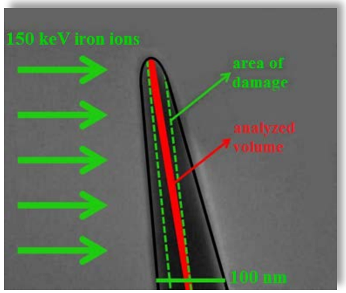


# Irradiation experiments

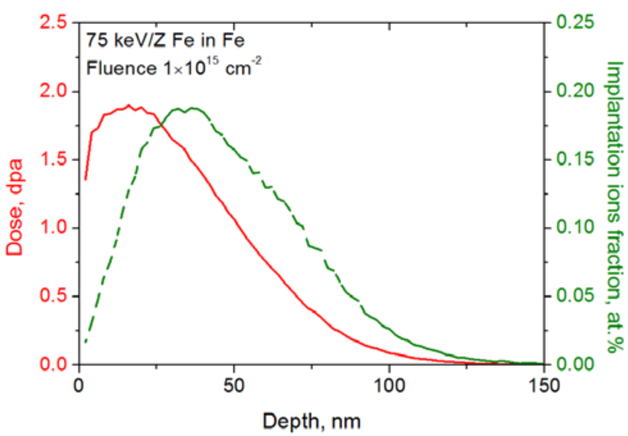
## Low energy heavy ion irradiation

### 75 keV/Z Fe ions

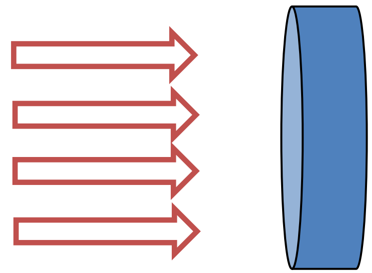
**$Fe^{+}$ (25%), $Fe^{+2}$ (68%), $Fe^{+3}$ (7%)**



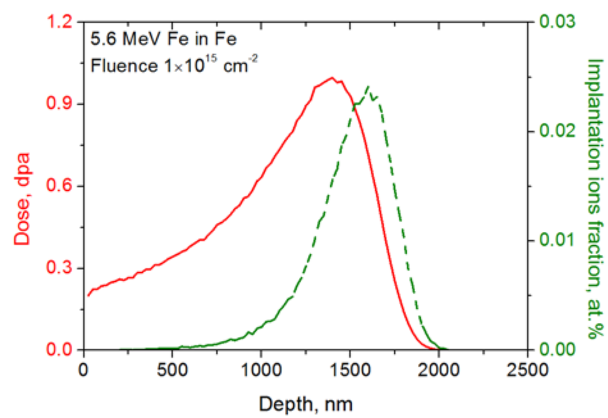

**APT specimens**




### 5.6 MeV Fe ions



**TEM specimens**




Temperature: RT - 500 °C

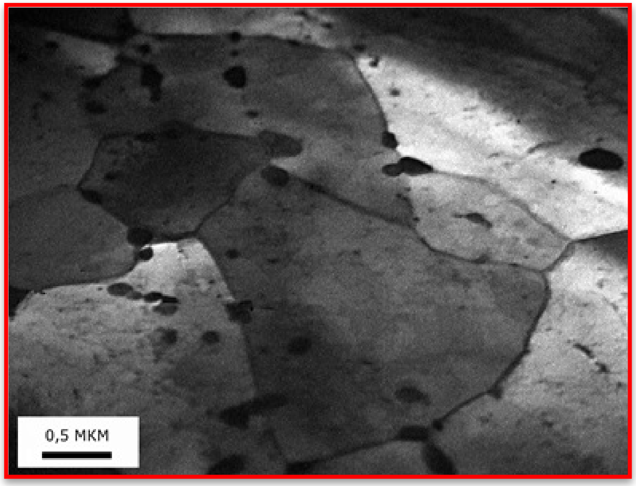
## Swift heavy ion irradiation

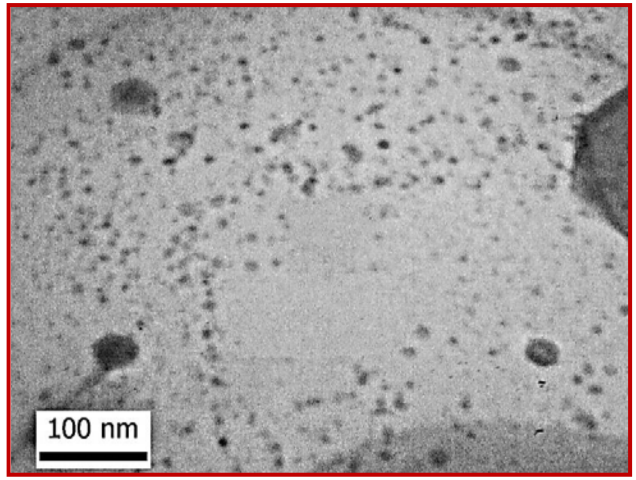
| |
|---|
| **UNILAC (GSI)**<br>**Au, 5.6 MeV/n** |
| IC100 (FLNR JINR)<br>Xe, 3.1 MeV/n |

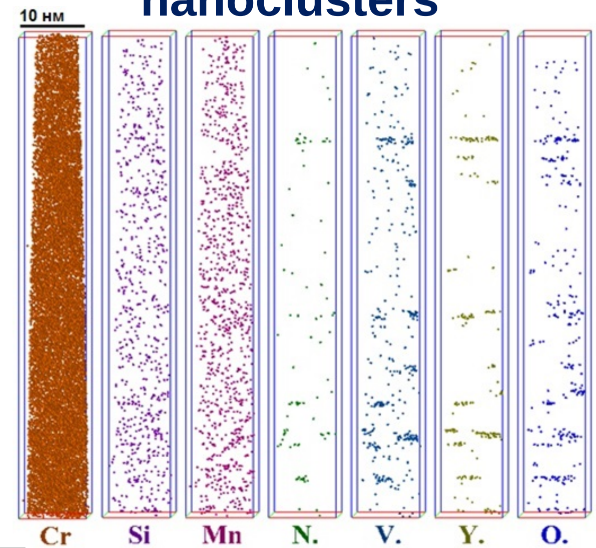
Temperature: RT

# Multilevel characterization nanostructured materials

**Grains** → **Secondary phases (oxides, carbides, dislocations)** → **Precipitates, nanoclusters**




**Different scales of research**

TEM

Microstructure
Nanostructure
Chemical analysis

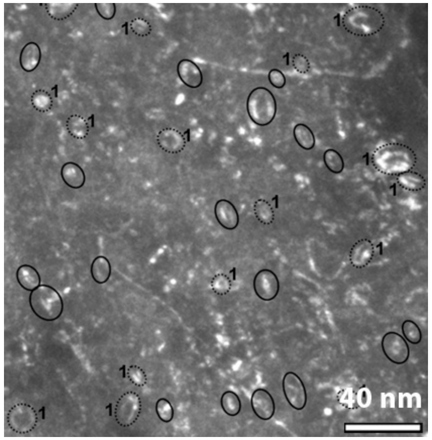
APT

Local analysis
Atomic structure
3D atom map

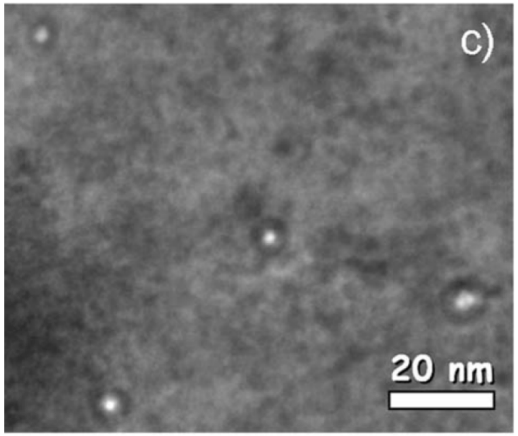
# TEM-visible changes after irradiation

## European steel Eurofer 97 (32 dpa, 330 °C)

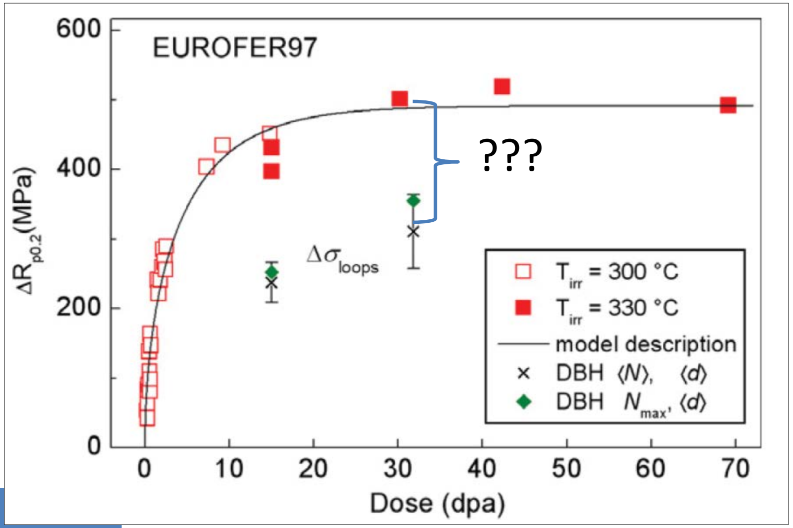
Dislocation loops




Voids




Hardening




| 32 dpa (BOR 60) | Loops&clusters | Voids |
|---|---|---|
| Number density, $m^{-3}$ | $1.7 \times 10^{22}$ | $2.3 \times 10^{21}$ |
| Size, nm | 4.8 | 1.6 |

O.J. Weiß et al., *J. Nucl. Mater.* 2012

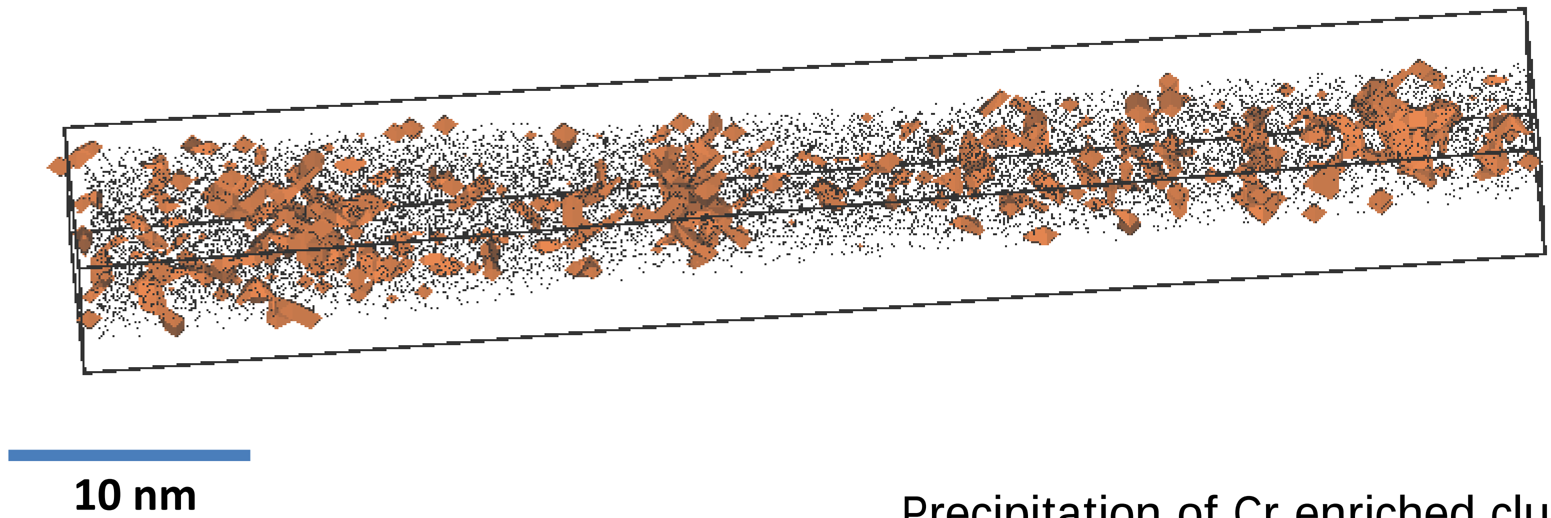
Hardening due to irradiation induced precipitate growth of MX and $M_{23}C_6$ is negligible (< 0,2%)

C. Dethloff et al., *J. Nucl. Mater.* 2014

# APT-visible changes under neutron irradiation

## European steel Eurofer 97 (32 dpa, 330 °C)

Isosurface of 20 at.% Cr showing Cr precipitates (•) in Fe matrix (•).




Precipitation of Cr enriched clusters

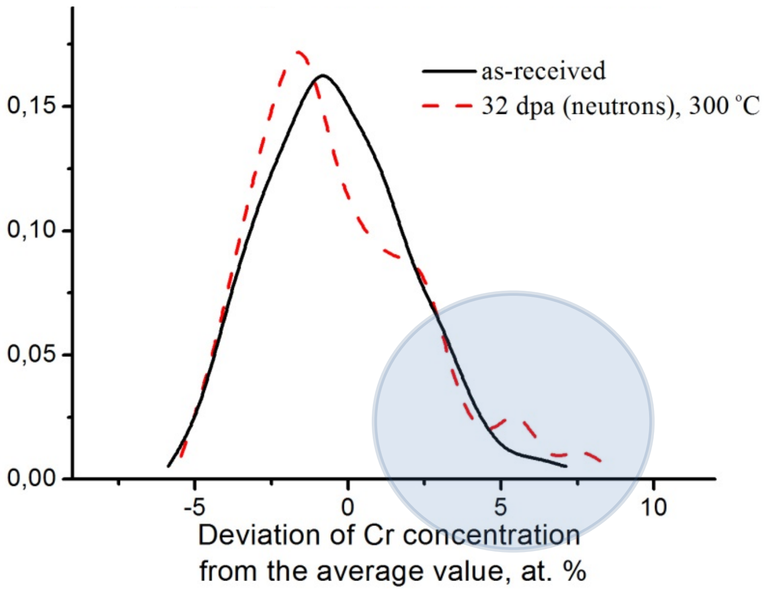
| 32 dpa | Cr-Mn clusters |
|---|---|
| Number density, $m^{-3}$ | ~ $5\times10^{24}$ |
| Size, nm | 2,6/3,7 |

S.V. Rogozhkin et al., *Inorg. Mater. Appl. Res.* 2013

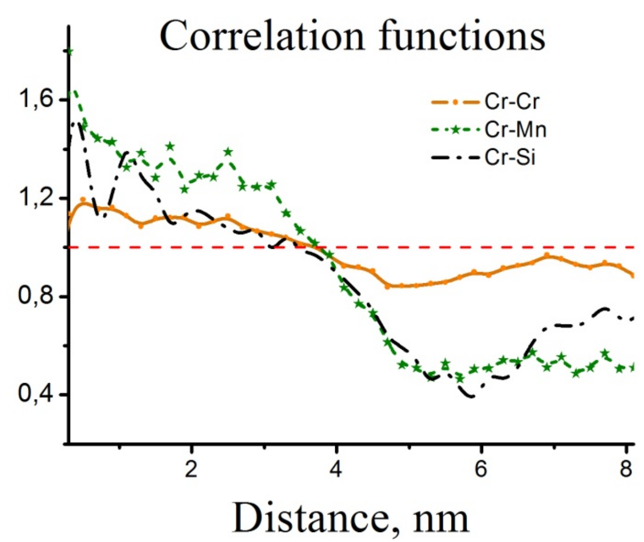
# APT-visible changes after irradiation. Sophisticated analysis

## European steel Eurofer 97 (32 dpa, 330 °C)

Frequency distribution of Cr



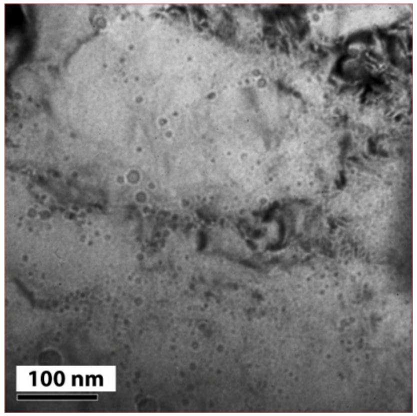

Cr-Cr and Cr-Mn correlation

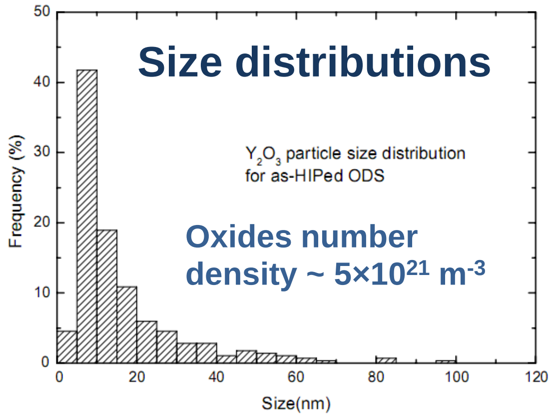



S.V. Rogozhkin et al., *Inorg. Mater. Appl. Res.* 2013

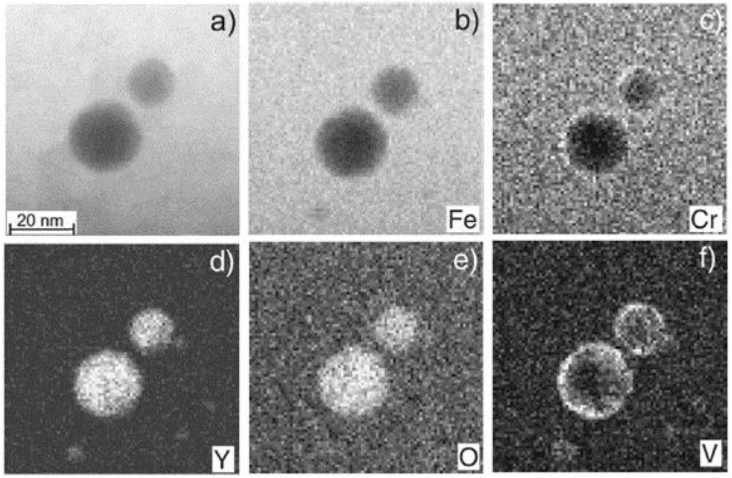
# TEM-visible features

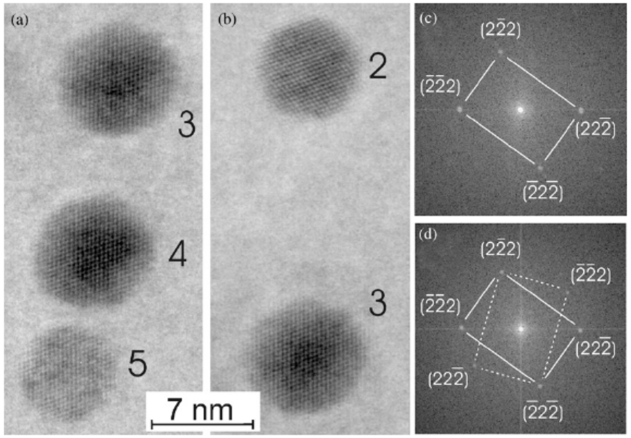
## European nanostructured steel  ODS Eurofer




**Oxide particles $Y_2O_3$**



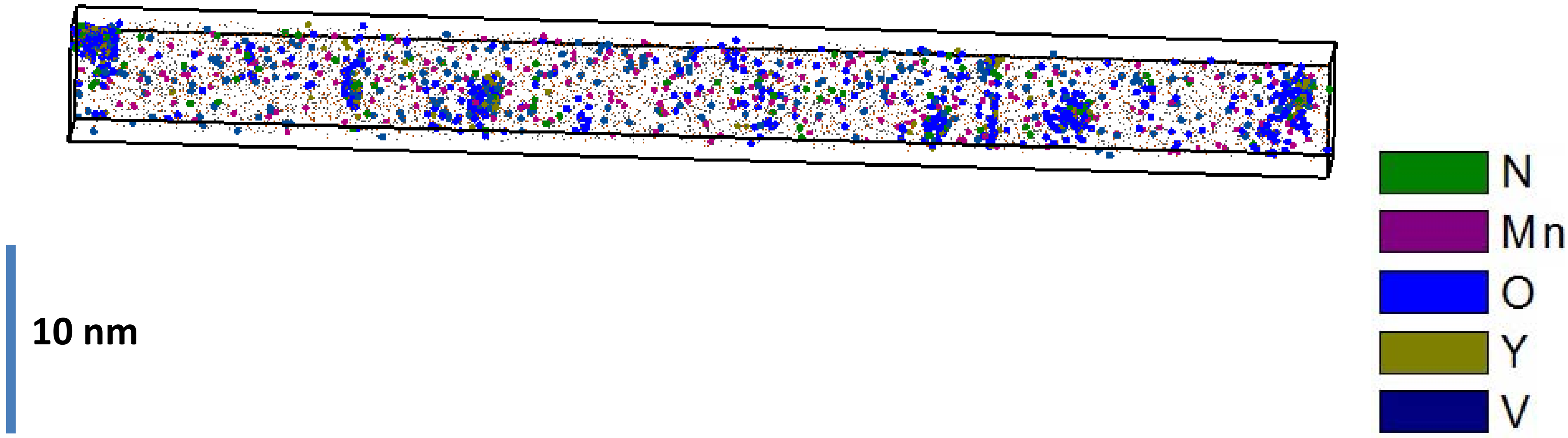




V. de Castro et al., *J. Nucl. Mater*. 2007
M. Klimenkov et al., *J. Nucl. Mater.* 2009
M. Klimiankou et al., *J. Cryst. Growth* 2003

# APT-visible features

## European nanostructured steel ODS Eurofer

### Clusters enriched in Y, O, V, N and Cr



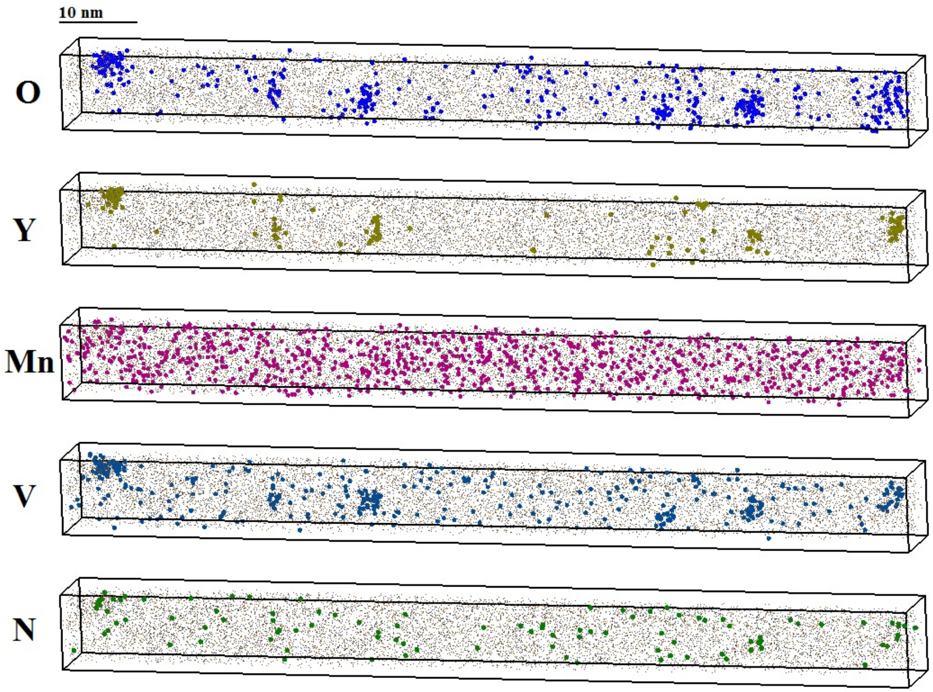

S.V. Rogozhkin, et al., *J. Nucl. Mater. 2011*

# APT-visible features

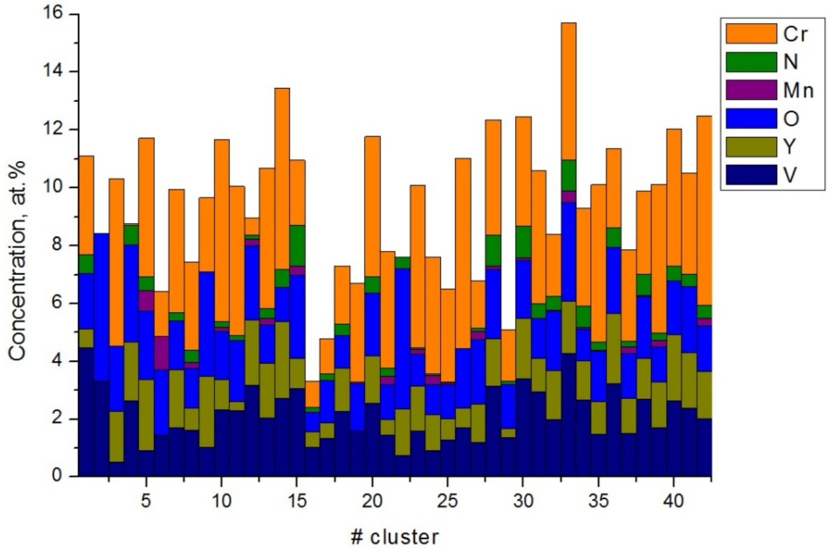
## European nanostructured steel ODS Eurofer

### Clusters enriched in Y, O, V, N and Cr

10 nm




### Clusters chemical composition



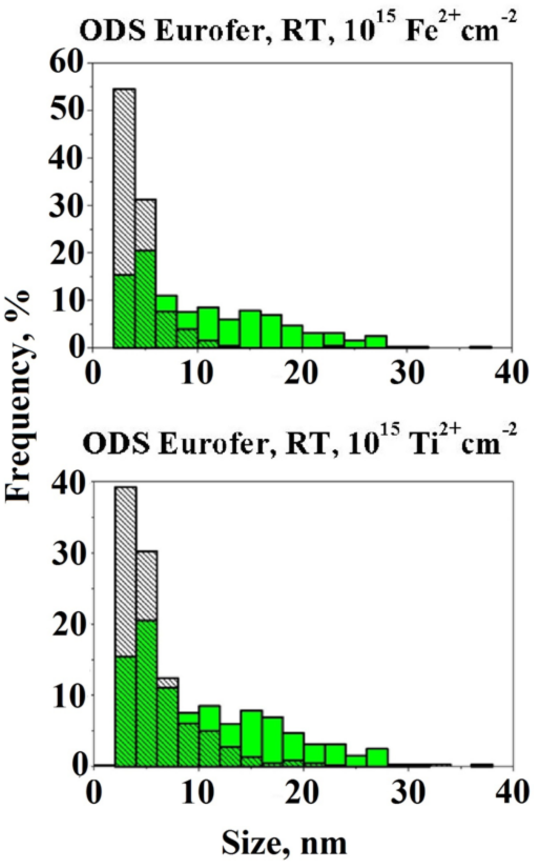

**Cluster number density:**
**$2 \times 10^{24}$ $m^{-3}$**
**Size: 1-4 nm**

S.V. Rogozhkin, et al., *J. Nucl. Mater. 2011*

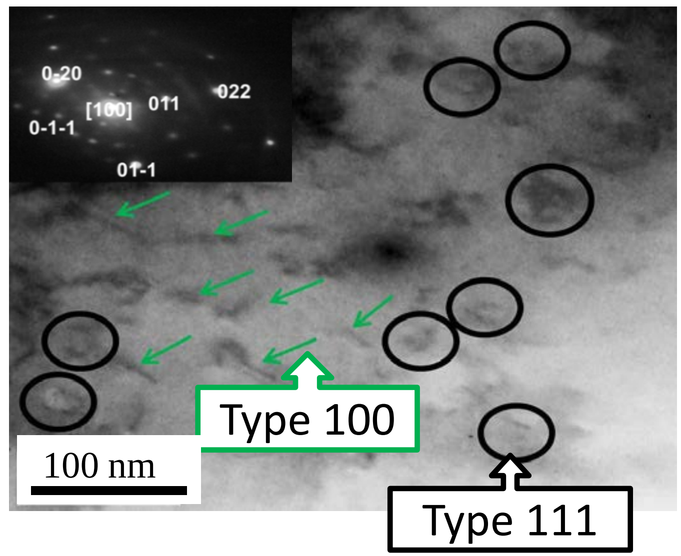
# TEM-visible changes under Fe ion irradiation

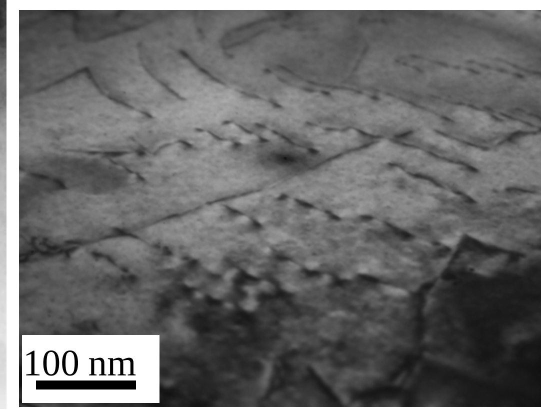
## European nanostructured steel ODS Eurofer

### Size distribution of oxide particles after irradiation




### Dislocation loops



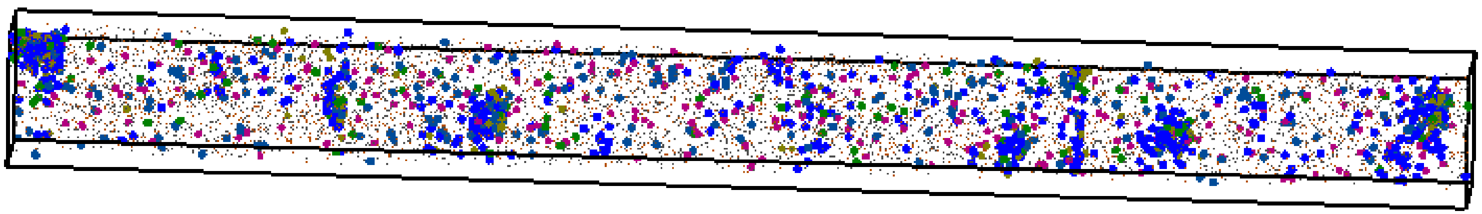

Number density of dislocation defects grew from $10^8$ to $10^{12}$ $cm^{-2}$

Oxides number density increased from $5\times10^{21}$ to $19\times10^{21}$ $m^{-3}$

S.V. Rogozhkin et al., *J. Nucl. Mater. Energy 2016*

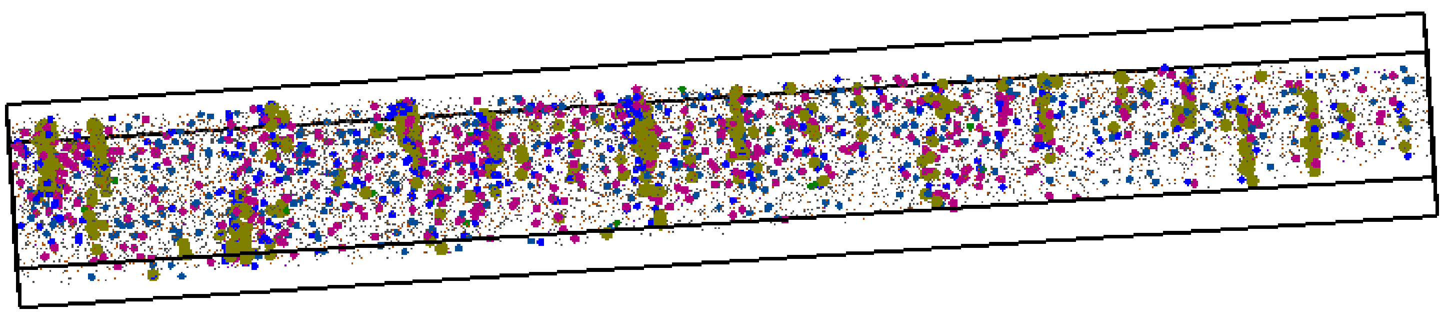
# APT-visible changes under irradiation

## Unirradiated ODS Eurofer




10 nm

## Irradiated ODS Eurofer (32 dpa)

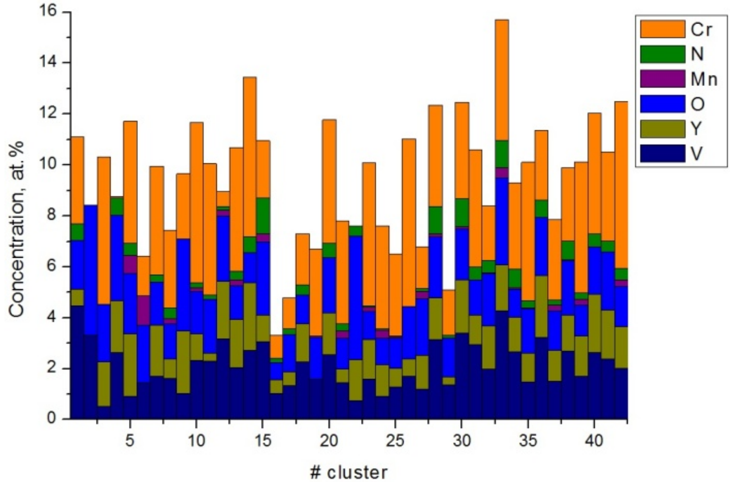


S.V. Rogozhkin et al., *J. Nucl. Mater. 2011*

# APT-visible changes under irradiation

## Unirradiated ODS Eurofer

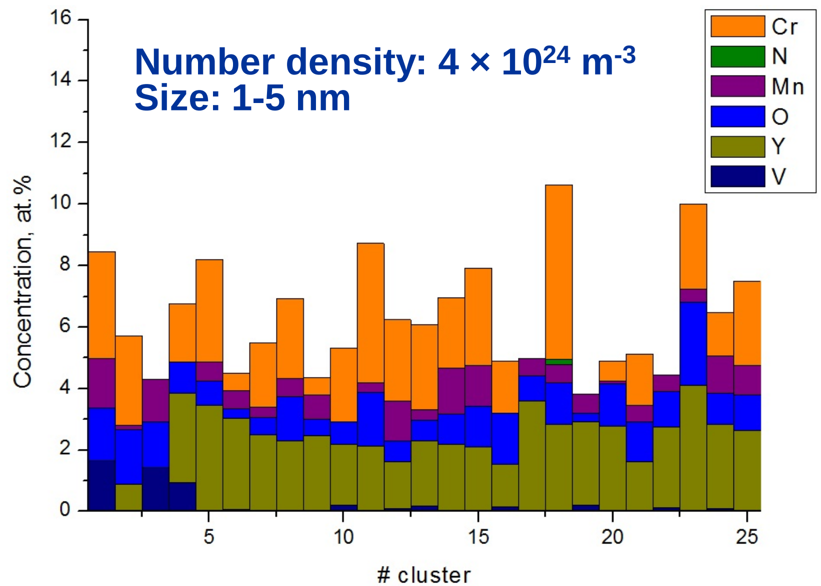
Clusters chemical composition




**APT study found a significant change in the nanocluster composition and an increase in cluster number density**

## Irradiated ODS Eurofer (32 dpa)

Clusters chemical composition

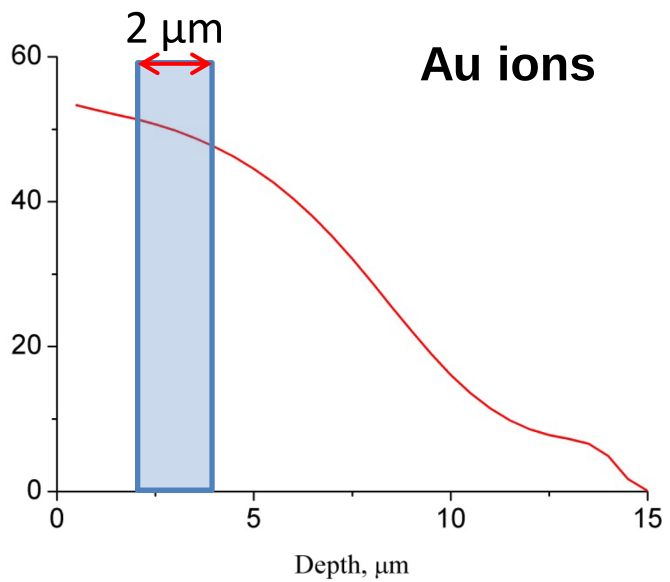


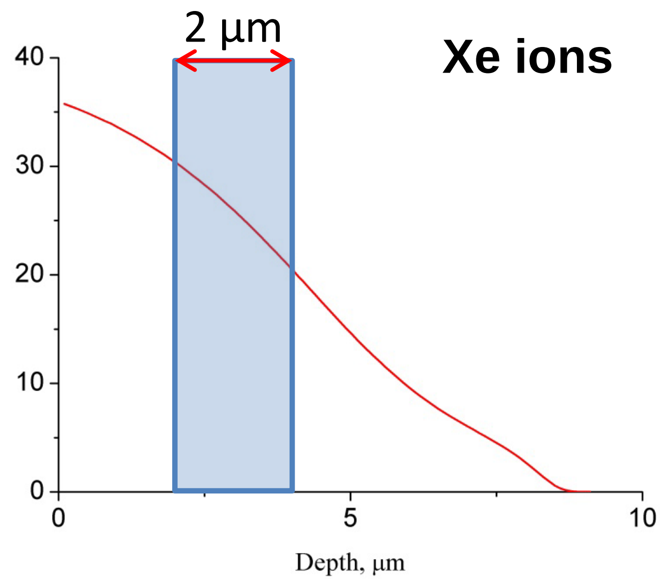

S.V. Rogozhkin et al., *J. Nucl. Mater. 2011*

# Swift heavy-ion irradiation of ODS Eurofer

| Accelerator | Type of ions | Energy, MeV/n | Fluence, ions/cm$^2$ |
|---|---|---|---|
| UNILAC (GSI) | Au | 5.6 | $1\times10^{11}$; $5\times10^{12}$ |
| IC100 (FLNR JINR) | Xe | 3.1 | $1\times10^{13}$; $1\times10^{14}$ |

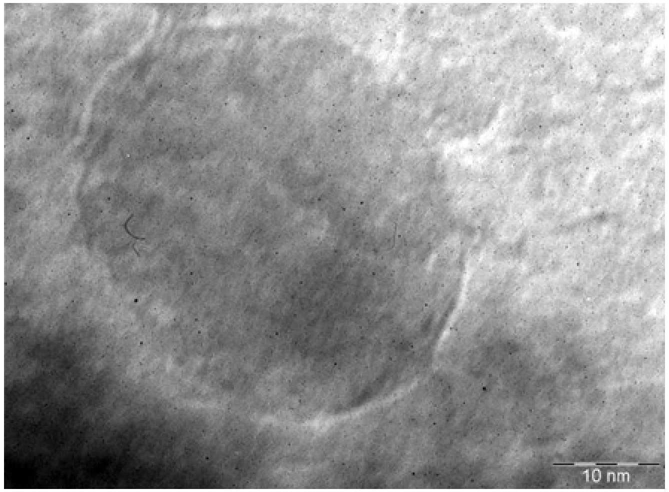
Results of SRIM calculation of ionization energy loss in pure Fe:



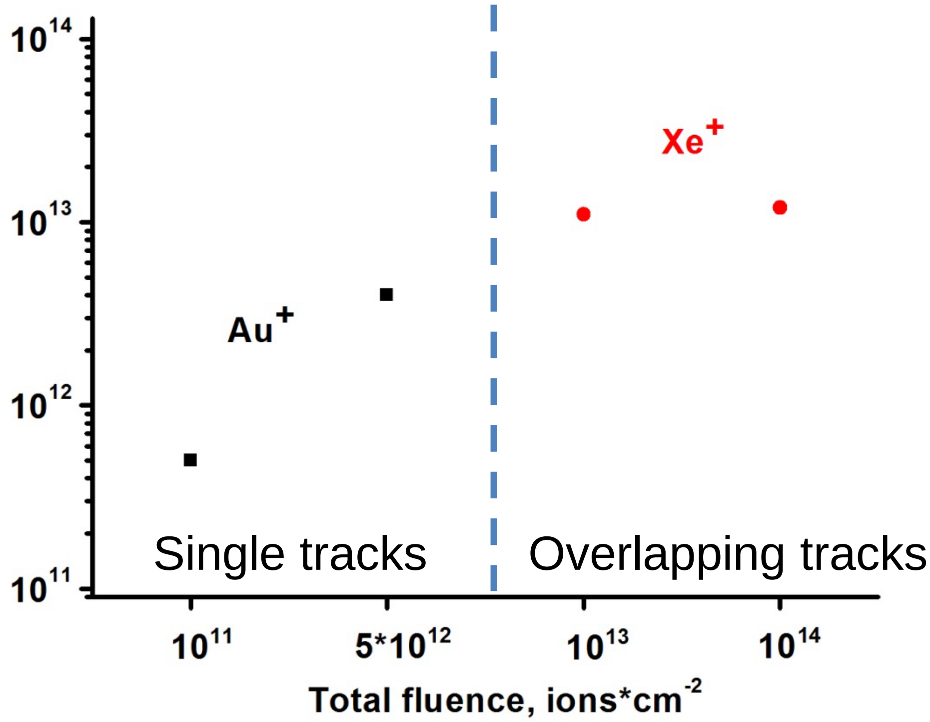
# TEM: Track formation within oxide inclusions

Before irradiation:




After (up to $10^{14}$ $Xe^{+}\times cm^{-2}$):

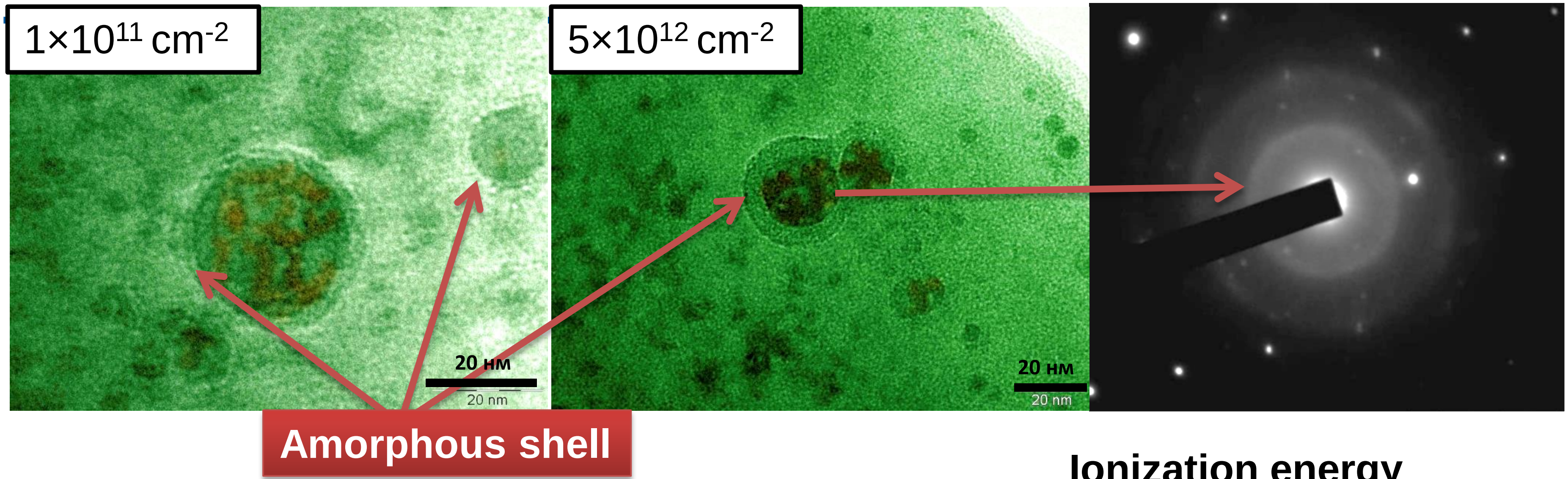

**Track density, $cm^{-2}$**



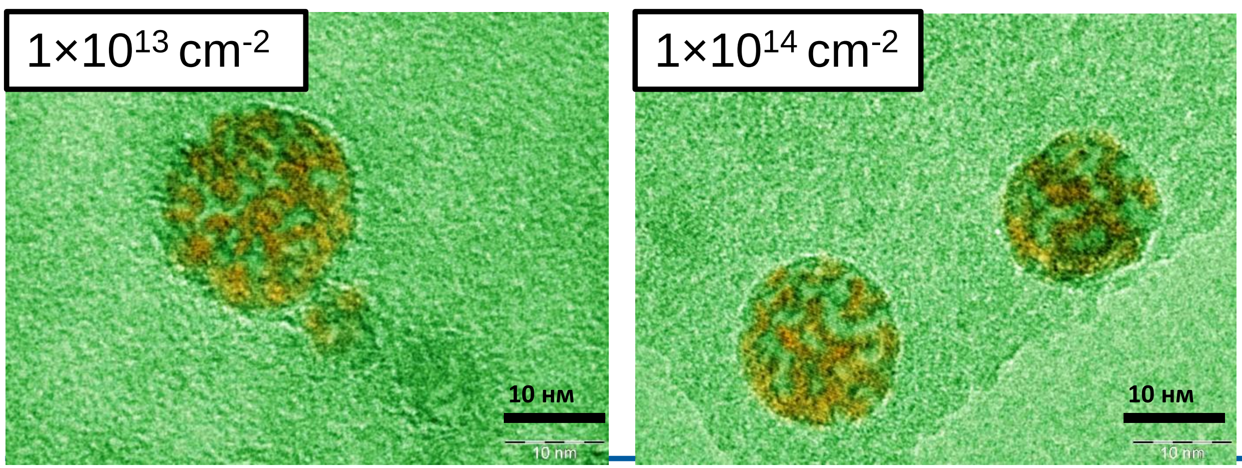

Average size of tracks:
2±1 nm for Xe ions
4±2 nm for Au ions

# TEM: Amorphization of oxides

**Au ions:**




**Xe ions:**




**Ionization energy losses:**

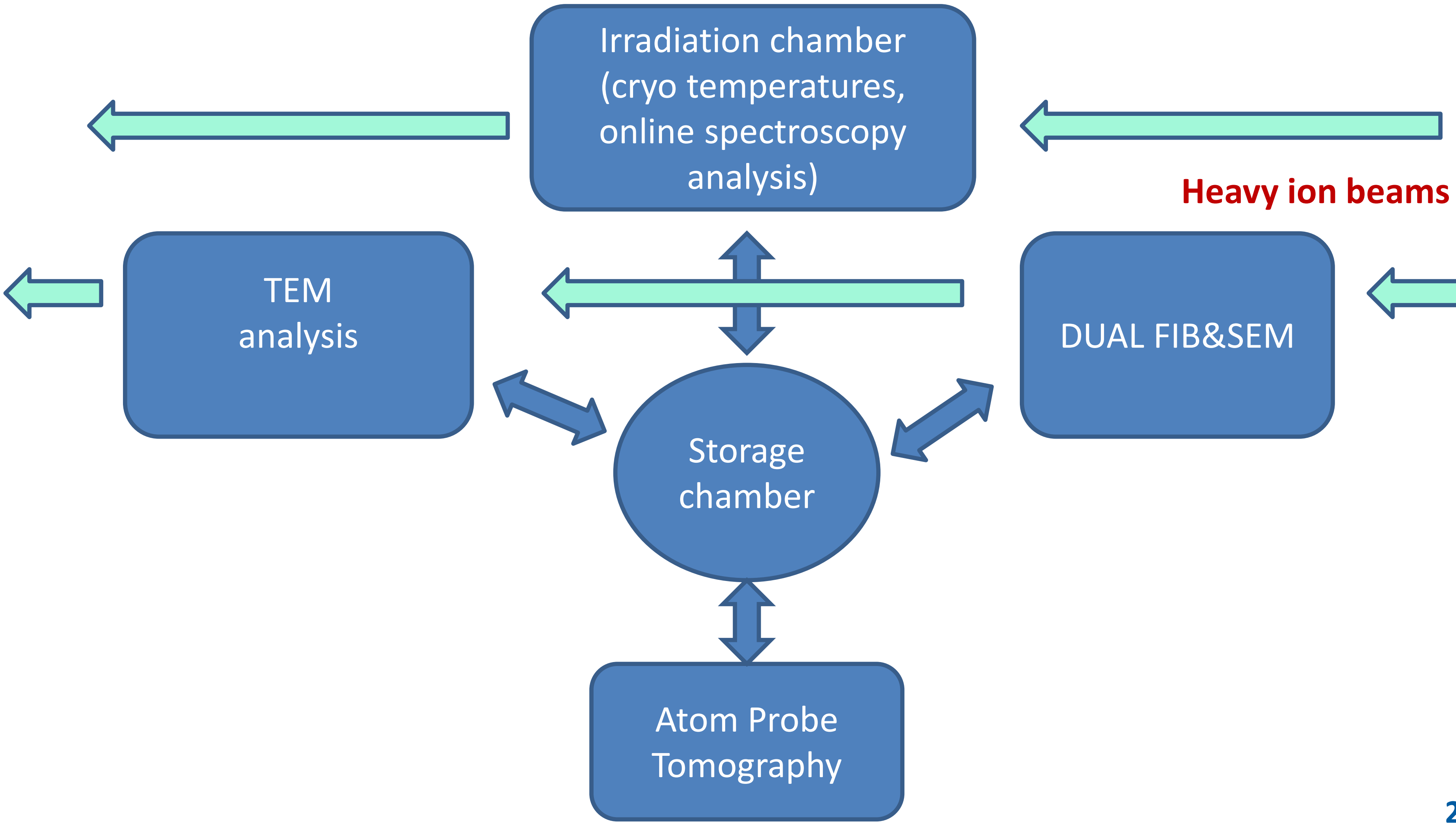
Au ions: 55 keV/nm

Xe ions: 25 keV/nm

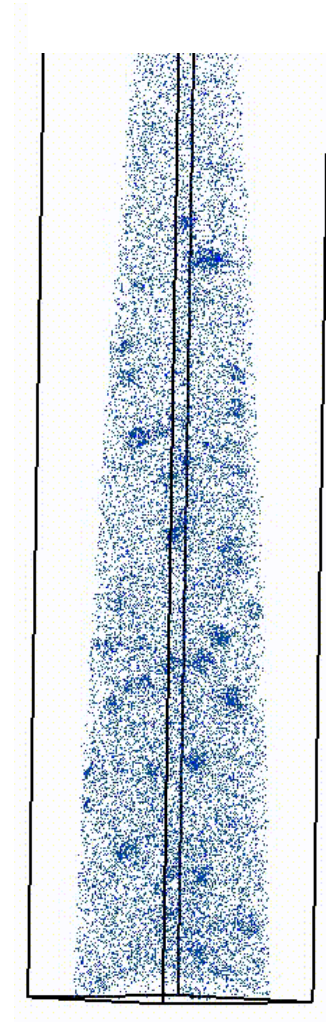
Tracks formation leads to amorphization of oxide particles and peripheral layer (for Au ions).

# Advanced Experiments in Material Science on heavy ion beans at NICA

# Atom scale analysis complex for heavy ion irradiated samples



# Atom probe tomography with femtosecond laser evaporation (ITEP NRC "Kurchatov institute")



- ✓ Investigated volume 50 × 50 × 1000 $nm^3$
- ✓ Mass resolution M/ΔM more than 800
- ✓ Automatic control , software for APT data analysis
- ✓ Flexible design

# Conclusion

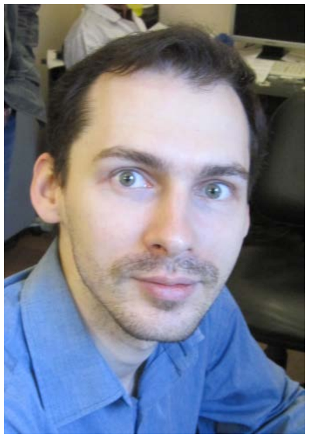
Material science complex with high resolution techniques (TEM, SEM&FIB and APT) will provide wide range studies in the field of radiation degradation and in advanced nanotechnology

**Center of**

**Material Science&Nanotechnology**

# SSC RF Institute for Theoretical and Experimental Physics

## Department of atomic-scale and nuclear physic techniques for investigation of nuclear materials



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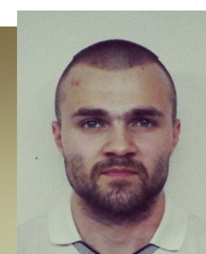

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# THANKS FOR YOUR ATTENTION!