# «Effect of Heavy-Ion Irradiation on the Nanoscale State of Advanced Reactor Ferritic-Martensitic Steels»

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## Introduction

Oxide Dispersion Strengthened (ODS) ferritic–martensitic steels are considered for nuclear applications as structural components for fusion or fission reactors. To ensure good performance in service, the microstructure of such ODS steels have to remain stable under irradiation at high temperature. Oxide dispersion strengthened F/M steels have been developed using the addition of nanoscale oxide particles to increase the high-temperature strength. These nano-oxides act as pinning points for dislocations, improving creep strength. Additionally, the nano-precipitates are expected to promote recombination of irradiation-produced point defects. Since the nanoparticles are believed critical to the high temperature strength and potential radiation resistance, the long-term stability of the nanoparticles under irradiation is an important issue.

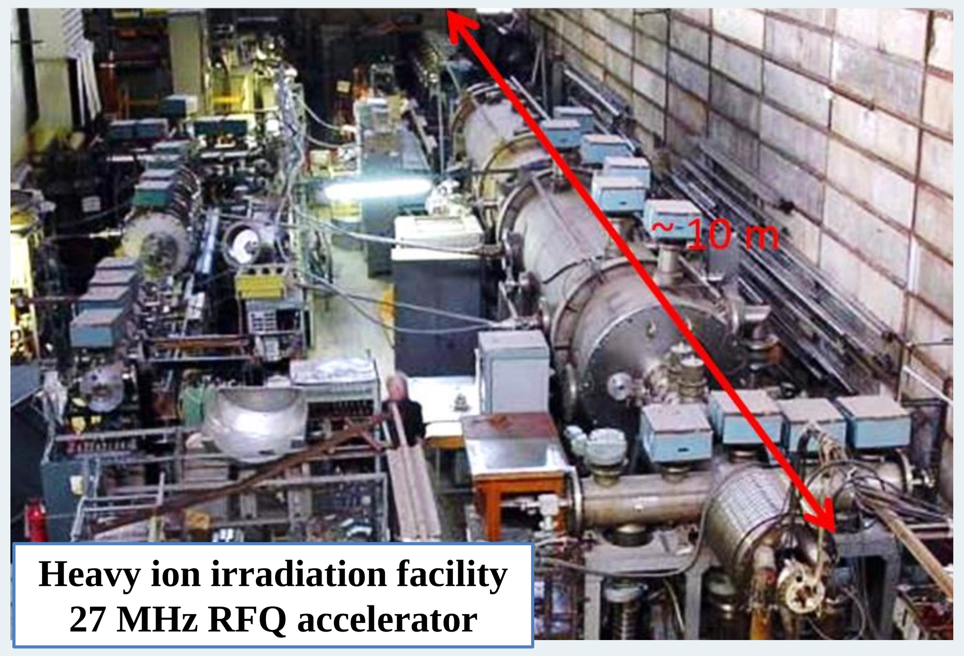
The main purpose of this work was to investigate the processes of evolution of oxide particles in ODS perspective steels under heavy ions irradiation to simulate neutron damage.

## Investigated materials

| Composition, at.% | Cr | Ti | Y | O | V | Oxides: Mean size, nm | Oxides: Number density, m$^{-3}$ | Clusters: Size, nm | Clusters: Number density, m$^{-3}$ |
|---|---|---|---|---|---|---|---|---|---|
| ODS Eurofer | 9.65 | - | 0.25 | 0.37 | 0.38 | 6 | $2\cdot10^{21}$ | ~ 2-4 | ~ $10^{24}$ |
| ODS 13.5%Cr | 14.60 | - | 0.15 | 0.22 | - | 14 | $10^{20}$ | ~ 2-4 | ~ $10^{23}$ |
| ODS 13.5%Cr-0.3%Ti | 14.60 | 0.35 | 0.15 | 0.22 | - | 8 | $2\cdot10^{21}$ | ~ 2-4 | ~ $10^{24}$ |

| Composition, at.% | Cr | V | Mn | Si | C | N | Clusters: Size, nm | Clusters: Number density, m$^{-3}$ |
|---|---|---|---|---|---|---|---|---|
| EK-181 | 11,9 | 0,31 | 0,95 | 0,73 | 0,64 | 0,16 | ~ 2-4 | ~ $10^{24}$ |

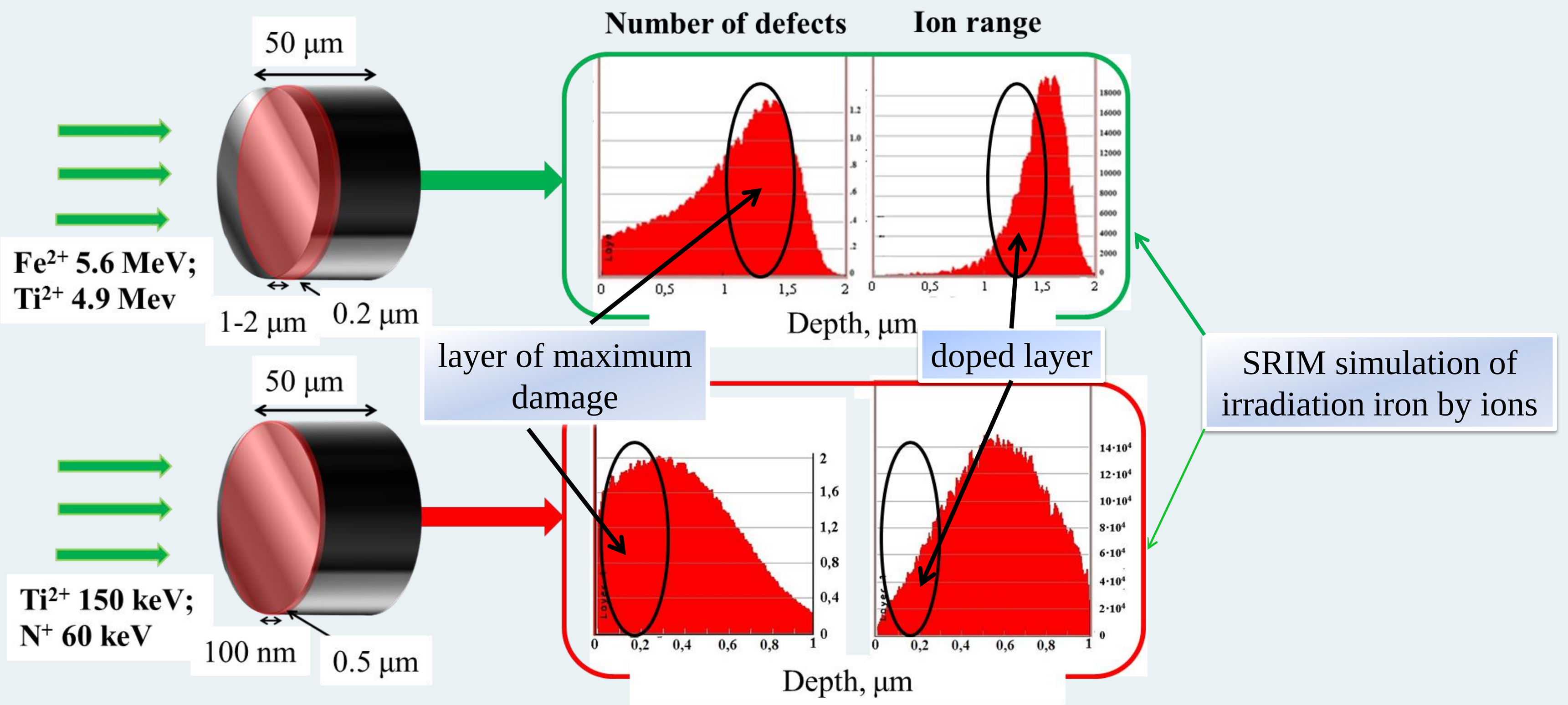
## Irradiation experiment



Heavy ion irradiation facility
27 MHz RFQ accelerator

**Parameters of irradiaton:**

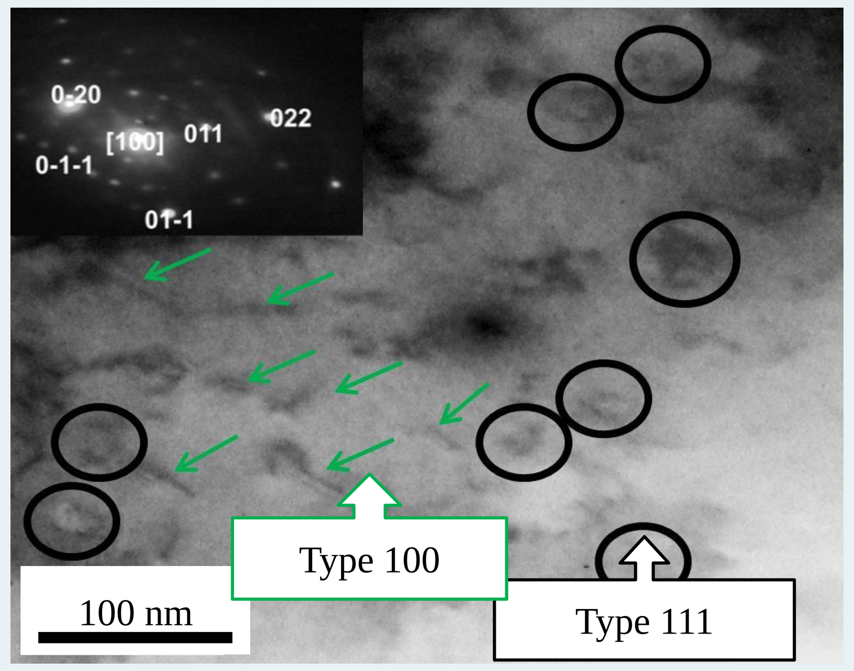
Fluence: $10^{15}$ ions/cm$^2$;
Temperature: 300 K;
Ions: $Ti^{2+}$ - 4.9 MeV; $Fe^{2+}$ - 5.6 Mev;
$Ti^{2+}$ - 150 keV; $N^+$ - 60 keV



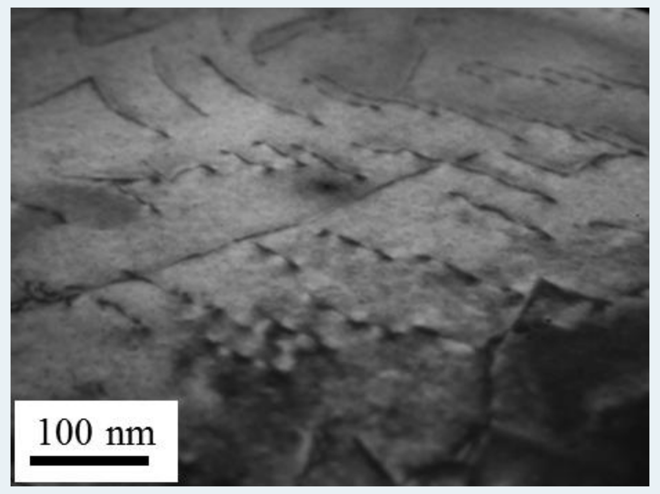

**The TEM samples preparation technique that forms a thin foil at a predetermined depth in ion irradiated material has been worked out.**

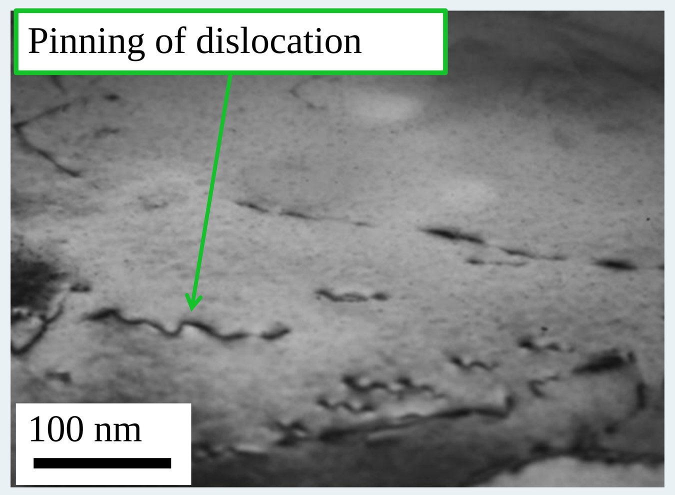
## Microstructure of materials after ion irradiation

### ➢High density of dislocation defects

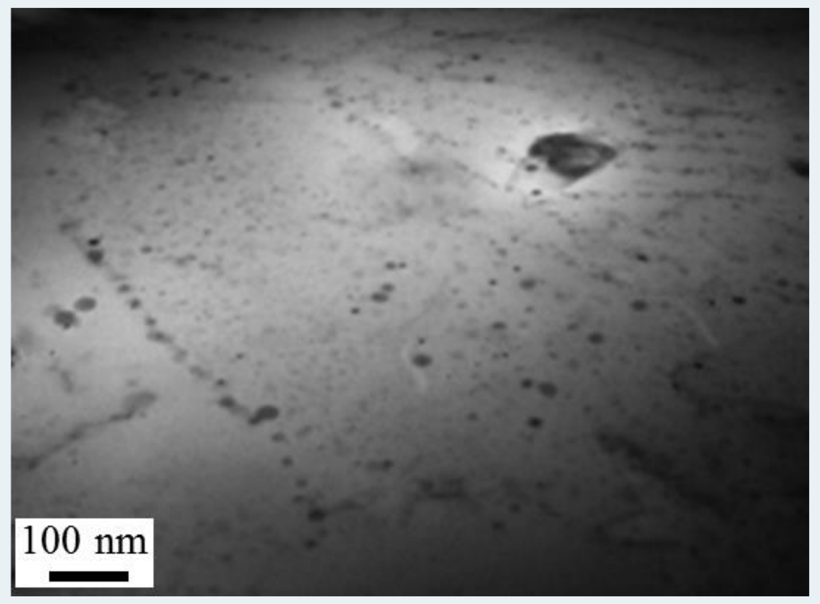


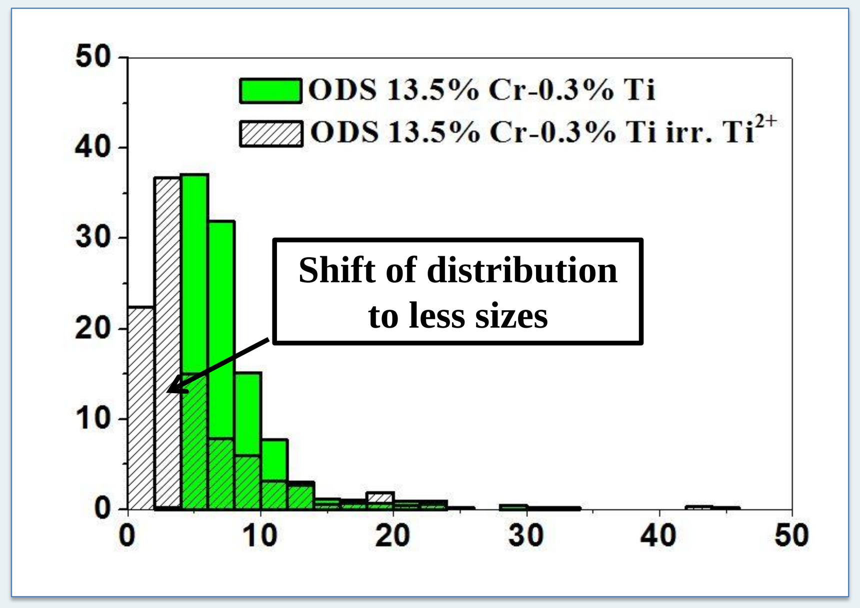

| Number density of dislocation loops (cm$^{-2}$) | | | | |
|---|---|---|---|---|
| Material | Initial | Fe | Ti | N |
| EK-181 (low energy) | $10^8$ | - | $10^{10}$ | $<10^{10}$ |
| ODS Eurofer (low energy) | | - | $7\cdot10^9$ | $3\cdot10^9$ |
| ODS 13.5%Cr | | $10^{12}$ | $10^{12}$ | - |
| ODS 13.5%Cr-0.3%Ti | | $10^{12}$ | $10^{12}$ | |



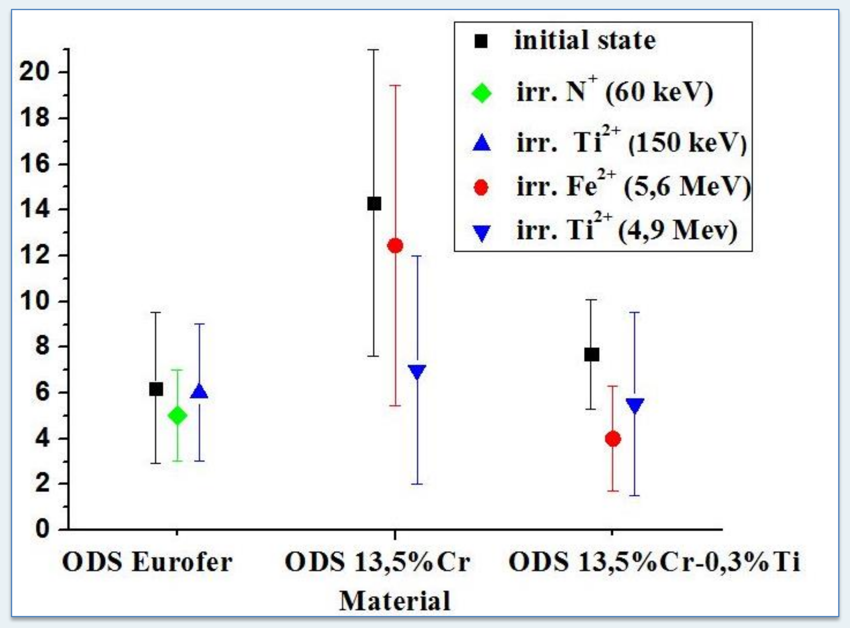

### ➢Changes in size distribution of oxide particles



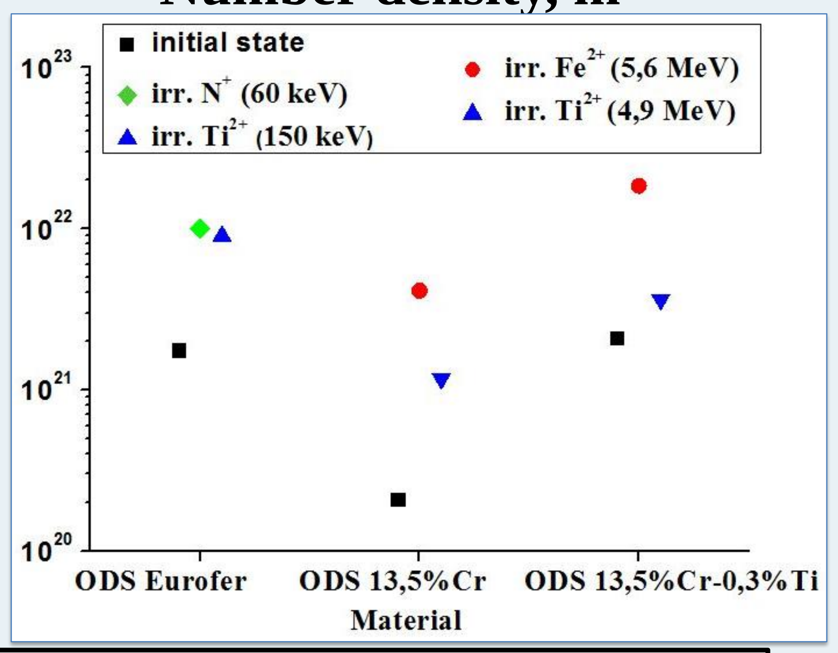




## Statistical analysis of the oxide particles in the materials before and after irradiation

**Mean size, nm**



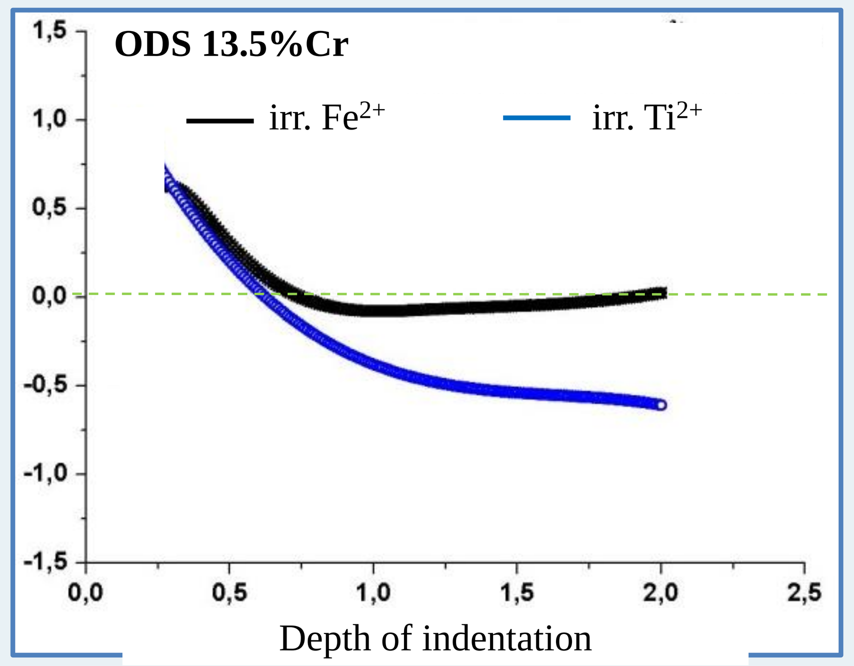

**Number density, m$^{-3}$**

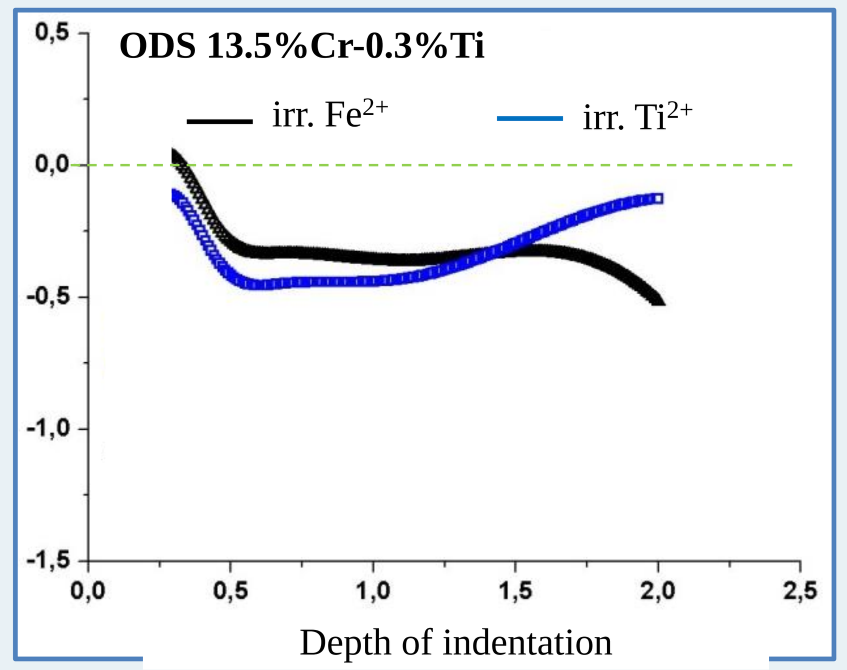



**Ion irradiation leads to decrease of the mean size and to increase of the number density of oxide particles due to the formation of small (<5 nm) oxide**

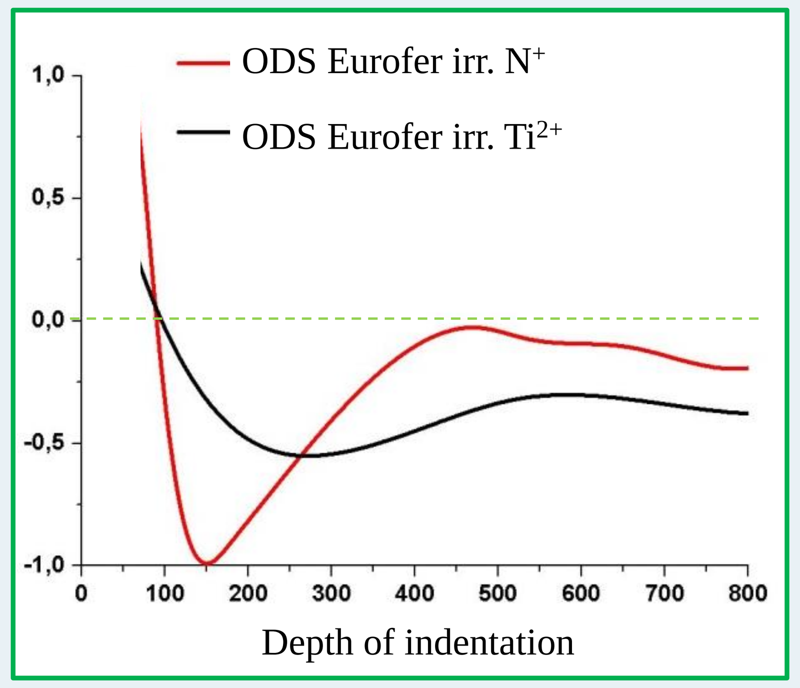
## Change of nanohardness between irradiated and initial states

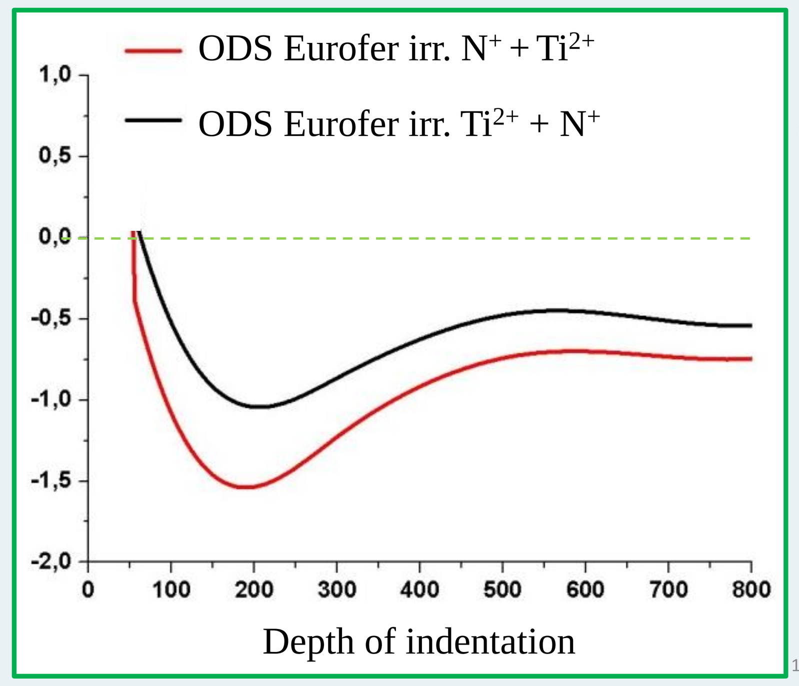
**Ions: $Ti^{2+}$ 4,9 MeV; $Fe^{2+}$ 5,6 MeV**

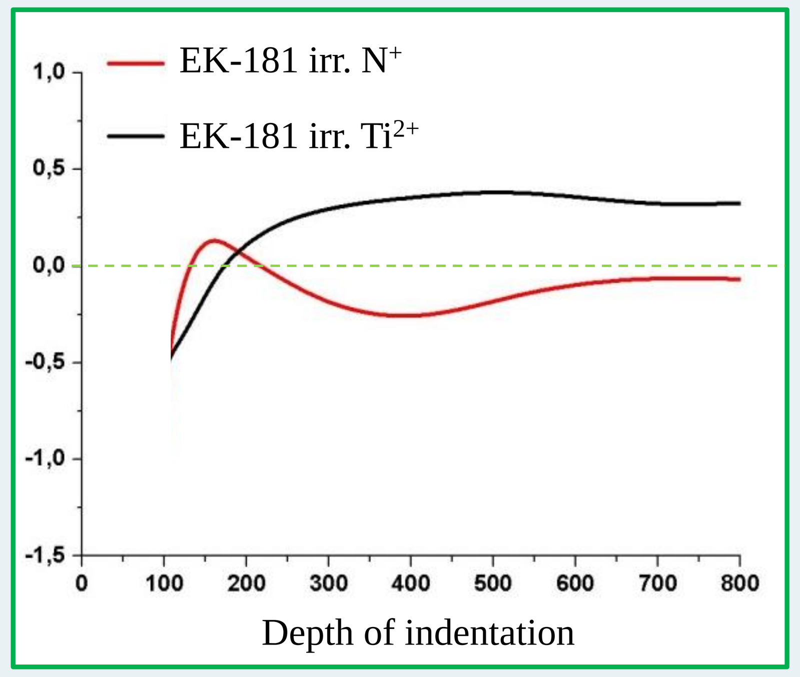


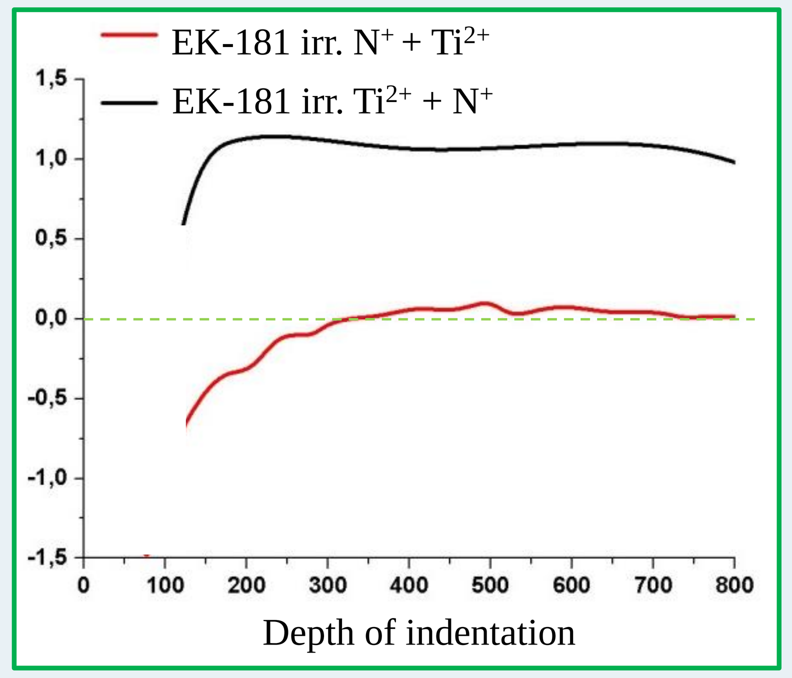




**Ions: $Ti^{2+}$ 150 keV; $N^+$ 60 keV**




## Summary


- The study of the microstructure of the initial and ion irradiated samples of EK-181, ODS Eurofer, ODS 13.5%Cr and ODS 13.5%Cr-0.3%Ti steels have been carried out. Samples have been irradiated by ions $Fe^{2+}$, $Ti^{2+}$ with energy 5.6 MeV, 4.9 MeV respectively and by ions $Ti^{2+}$ and $N^+$ with energy 150 keV, 60 keV respectively.
- In all irradiated materials observed increased number density of dislocation defects in the region of maximum damage. Highest value has been found in materials irradiated by high-energy ions ~ $10^{12}$ def./cm$^2$.
- Impact of ions on oxide inclusions reduces the proportion of large (> 20 nm) particles and increasing the total number density of oxides.
- The highest value of hardening (~ 10%) was found out for EK-181 steel irradiated by $Ti^{2+}$, and sequential irradiated by $Ti^{2+}$, and then $N^+$ (~20%).