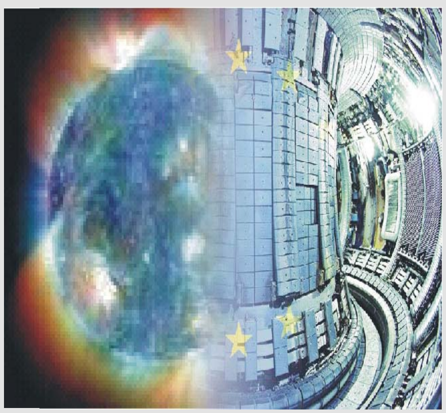
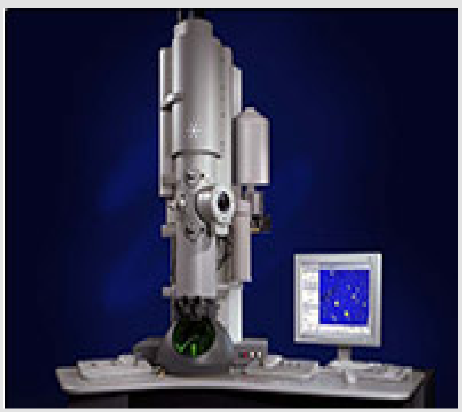
Karlsruhe Institute of Technology

Microstructure- property correlation of 13.5%Cr nanostructured ODS steels for fusion application

P.He, **M. Klimenkov, R. Lindau, A. Möslang**



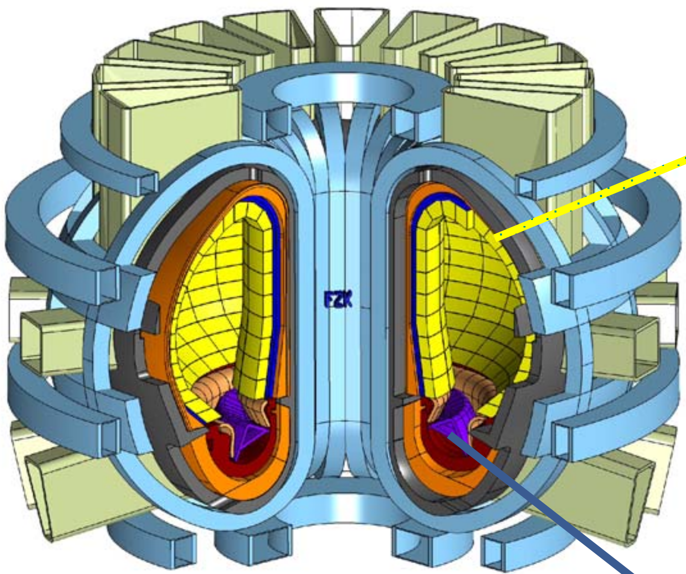


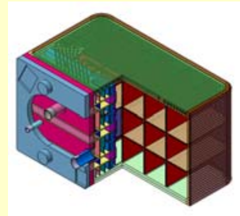
Karlsruhe Institute of Technology
Institute for Applied Materials (IAM)
Karlsruhe, Germany

Outline

- Introduction
  - Requirements for structural material in fusion reactor
  - Reduced-activation F/M steels and ODS RAFM steels
  - Development of ODS RAF steels
- Experimental
  - Fabrication route
  - Analytical TEM (EDX and EELS)
- Microstructure
  - Bimodal grain size distribution
  - Chemical composition of coarse precipitates and ODS particles
  - Influence of Ti content on the mean particle size
- Mechanical properties
  - Hardness, tensile property, Charpy impact property
- Summary

Material challenges in Fusion Reactor





Blanket: ≤30 dpa/year, 2.5MW/m²

Reduced-activation ferritic-martensitic steels:

- EUROFER (9Cr-WVTa) 250-550 °C
- EUROFER-ODS 250-650 °C

Divertor: ≤10 dpa/a, 10-15 MW/m²

Nanostructured RAF(M)-ODS-steels

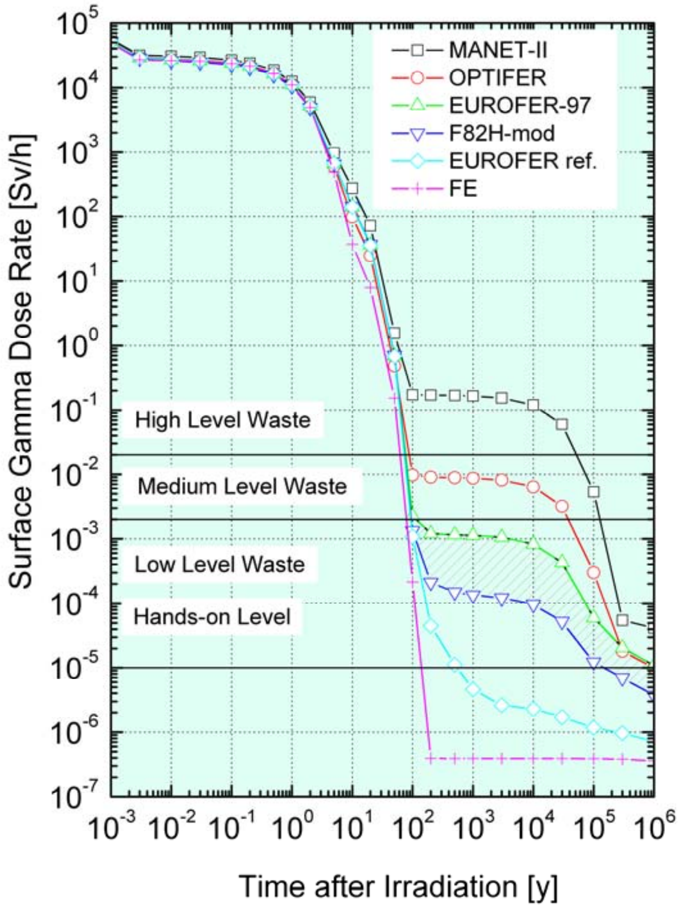
→ ~300 – 800 °C

RAFM-ODS (EUROFER-ODS)

Alloy	Heat	C	Si	Mn	Cr	V	W	Ta		Y_2O_3
EUROFER	E 83699	0.12	0.06	0.42	8.87	0.19	1.10	0.14	+	0.3 or 0.5

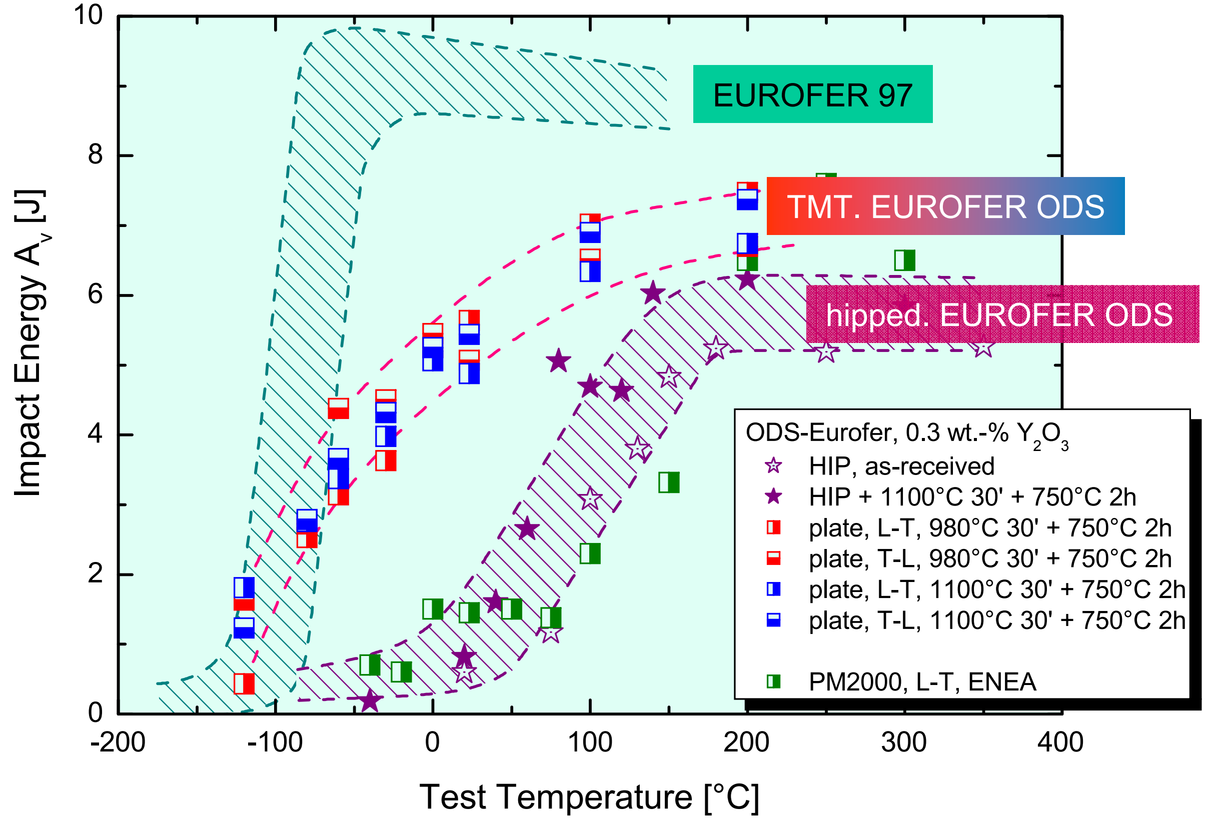
Reduced Activation Structural Materials:

RAFM Steel



Priority:
Low activation capability

ODS-RAFM Steel



Priority:
- Superior high temperature ductility
- Promising impact property

Experimental Steels for Higher Operating Temperatures

- ODS steels are candidate materials for several Gen.IV reactor concepts
  - Sodium-Cooled Fast Reactor (SFR) : cladding, in-core, out-of-core
  - Super-Critical Water-Cooled Reactor (SCWR): cladding, in-core, out-of core
  - Very –High-Temperature Reactor (VHTR): in-core, out-of-core
- ODS steels are candidate structural materials for future DEMO reactor
- Trends: 12-20 Cr ODS ferritic steels for higher operating temperature (higher thermal efficiency)

☺ Superior tensile and creep strength compared to 9Cr ODS

☺ Better HT corrosion resistance

☺ Good radiation resistance

☹ Inferior impact property and ductility

Basic composition of 13.5% Cr ODS ferritic steels

RAF Steels:

Replace critical alloying elements e.g. Mo ⇒ W

Capsel-No.	Heat	Cr	W	Ti	Y_2O_3	Density
K12	159/0	13.5	2.0	**0.0**	0.0	99.1%
K2	159/1	13.5	2.0	**0.0**	0.3	99.5%
K4	159/3	13.5	2.0	**0.2**	0.3	99.9%
K5	159/4	13.5	2.0	**0.3**	0.3	99.1%
K6	159/5	13.5	2.0	**0.4**	0.3	99.5%

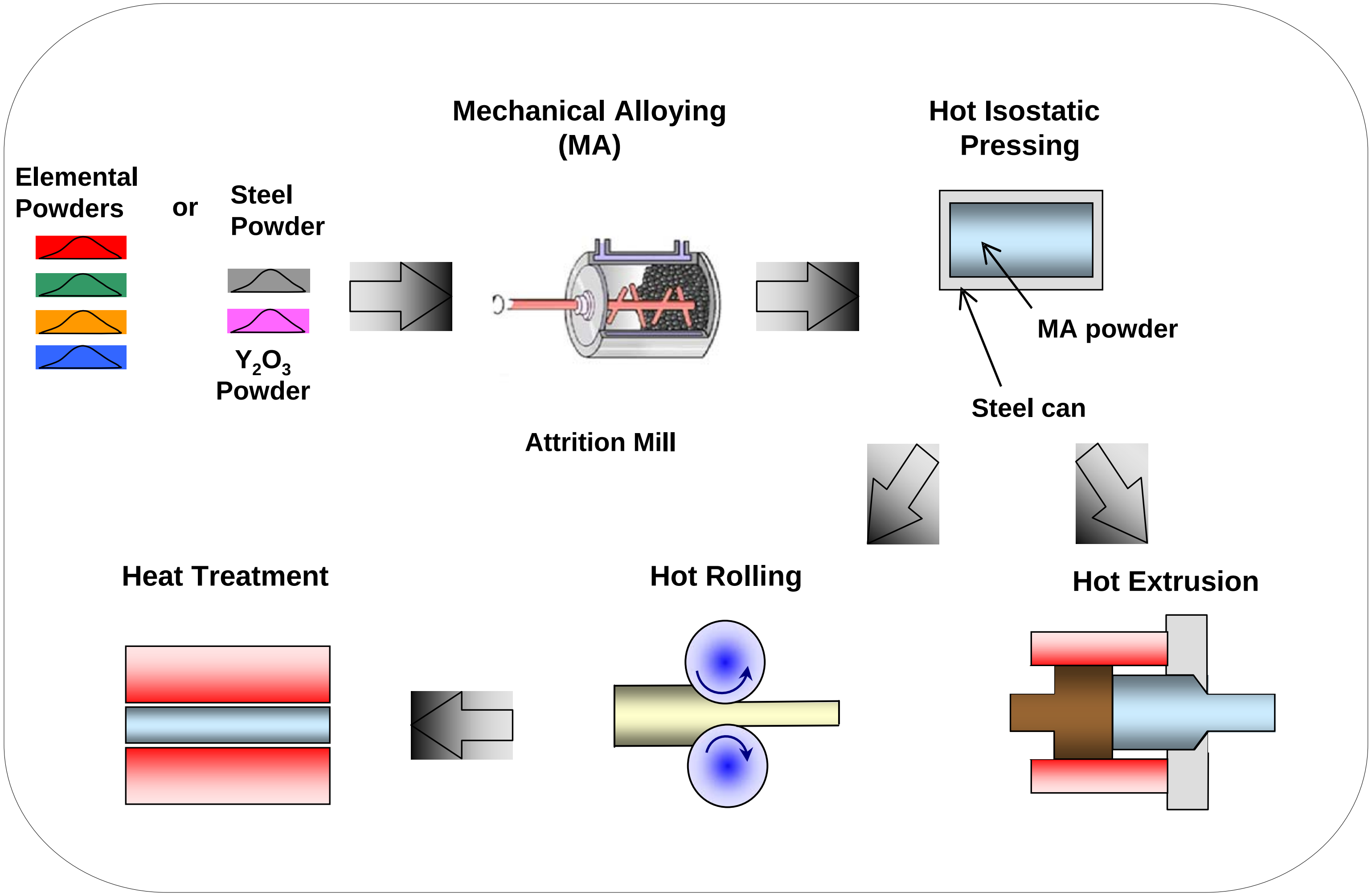
Theoretical density:

for pure metal: $\rho = m/v$

for alloys: $1/\rho = \omega_1 \%/\rho_1 + \omega_2 \%/\rho_2 + \omega_3 \%/\rho_3$

This work aims to investigate Ti dependency of microstructure and mechanical properties for ferritic ODS steels

Fabrication route



Mechanical alloying process

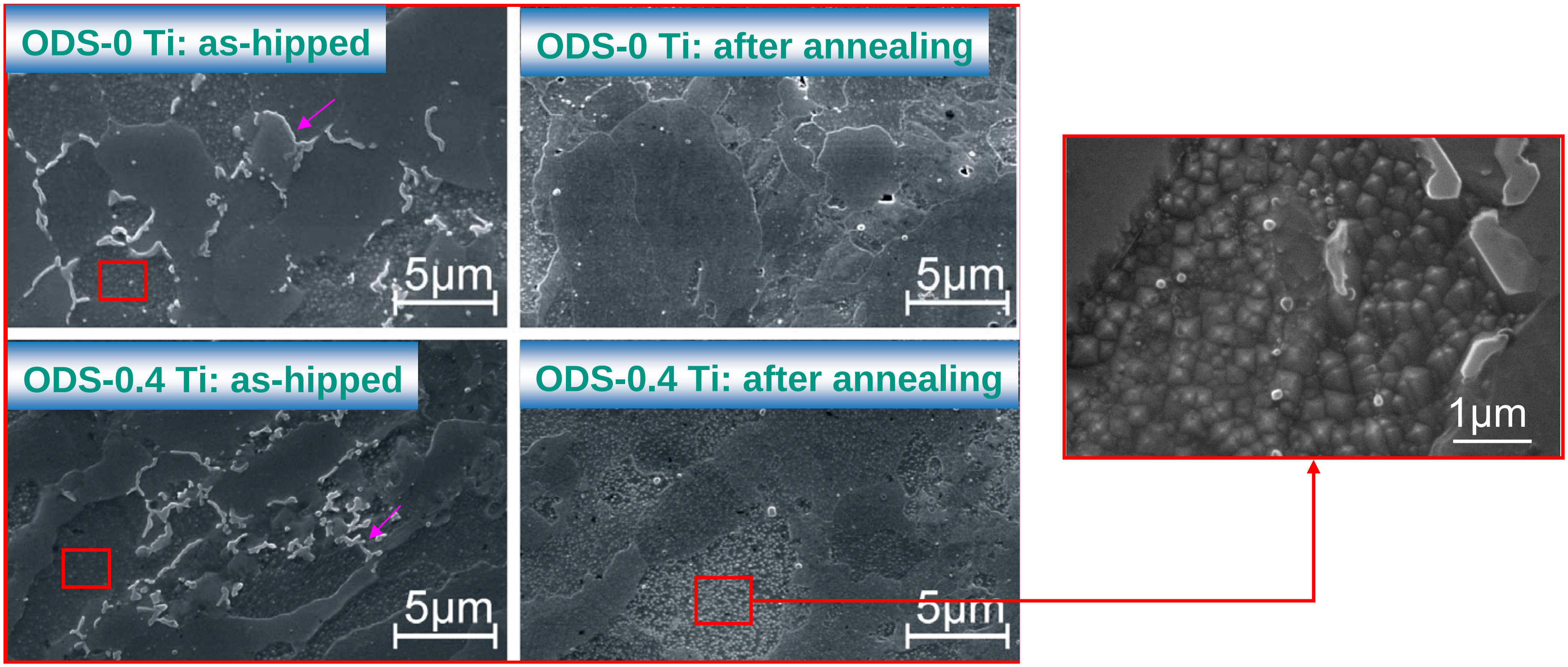
ZOZ Simoloyer CM01

View into milling container

Capsules after HIP

- MA: under H2, 1000/4'/700/1'/24h, Glove-Box
- HIP: 1150°C, 100 MPa, 2.5h
- Anealing: 1050°C, 2h, Vacuum

Microstructure by SEM



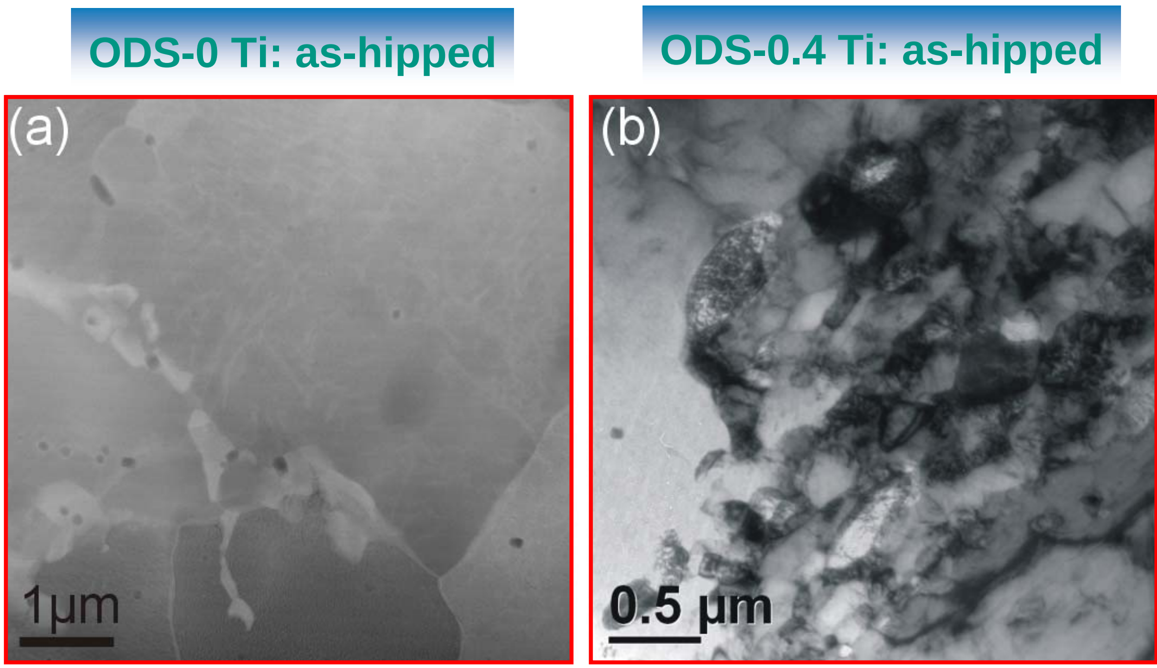
☺ No visible porosity after HIP in all alloys ☞ high density

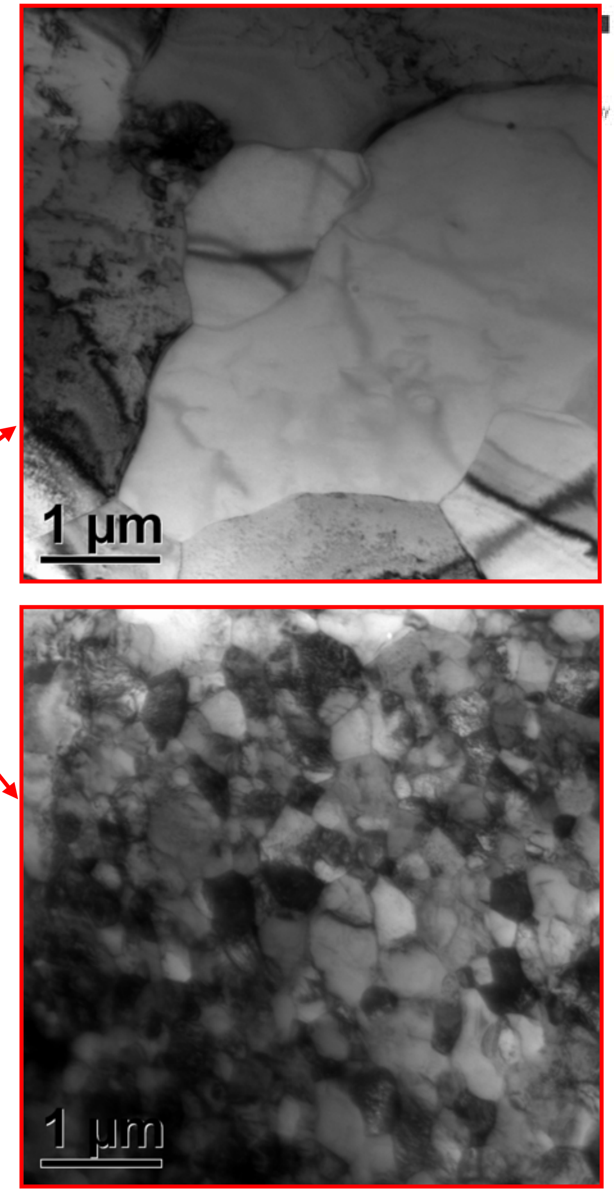
☺ Elongated coarse precipitates align preferentially along GB in all ODS steels after HIP, eliminated by annealing

☺ Spherical precipitates (100-300nm in diameter) in all ODS steels in both conditions

☹ Grain structure (especially nano grains) is not clearly visible due to etching problem

Bimodal grain size by TEM





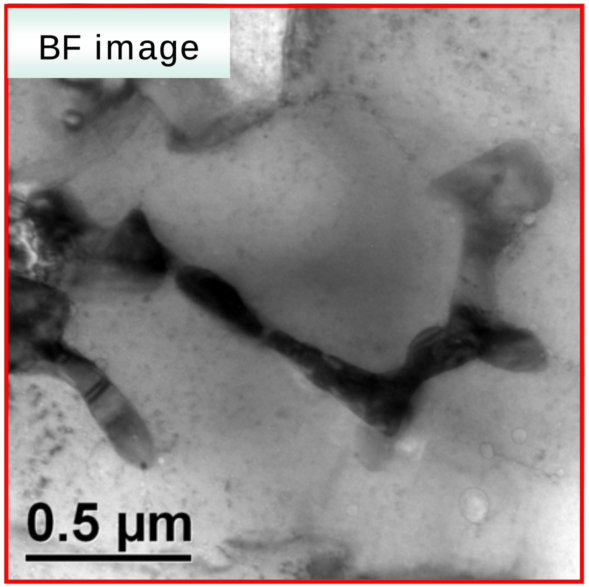
- Bimodal grain size distribution in all Ti-containing ODS steels
- Coarse grain ~1-8µm free of dislocations; nano grains ~200-800 nm with much higher dislocation density (darker contrast)
- It is very stable and resistant to annealing
- It is assumed to caused by recrystallization and abnormal grain growth during HIP.

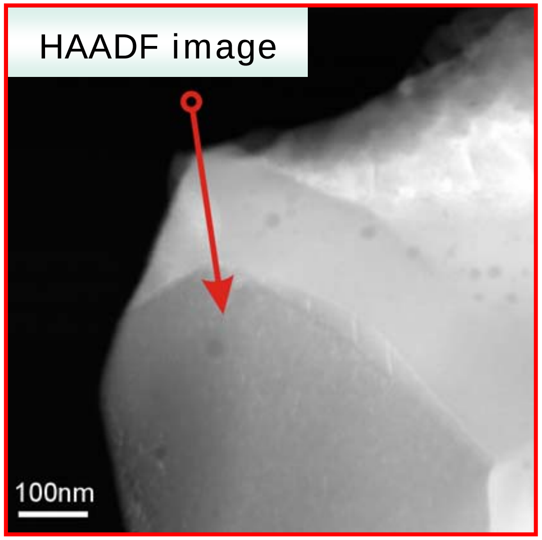
Chemical composition of precipitates

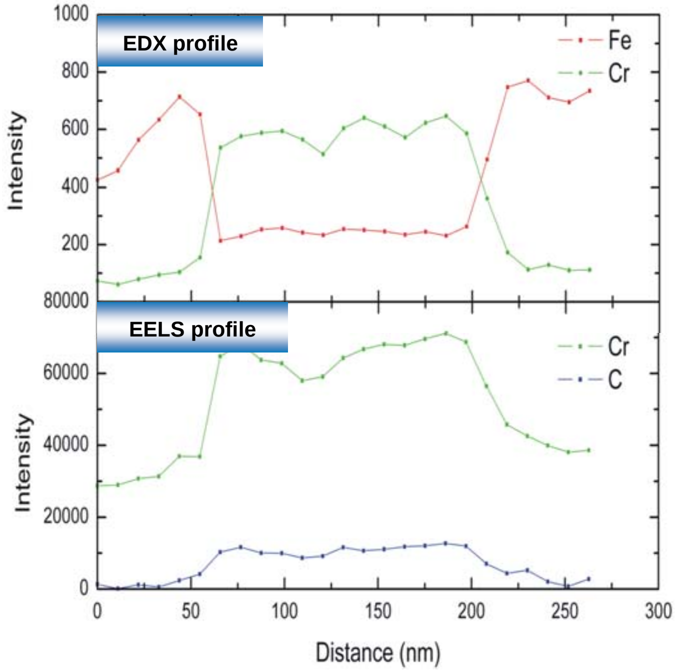
- TEM: Tecnai F20 S (200keV) - GIF 200
- STEM-HAADF technique was used for imaging and analyical investigations
- EDX and EELS experiments were performed in the STEM mode
- EELS investigations were performed on coarse precipitates and ODS particles as an excellent complement for the detection of light elements.

Chemical composition of precipitates

ODS-0 Ti: as-hipped



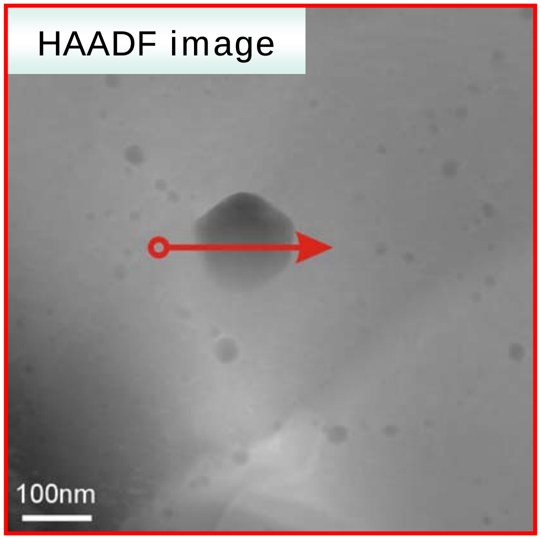


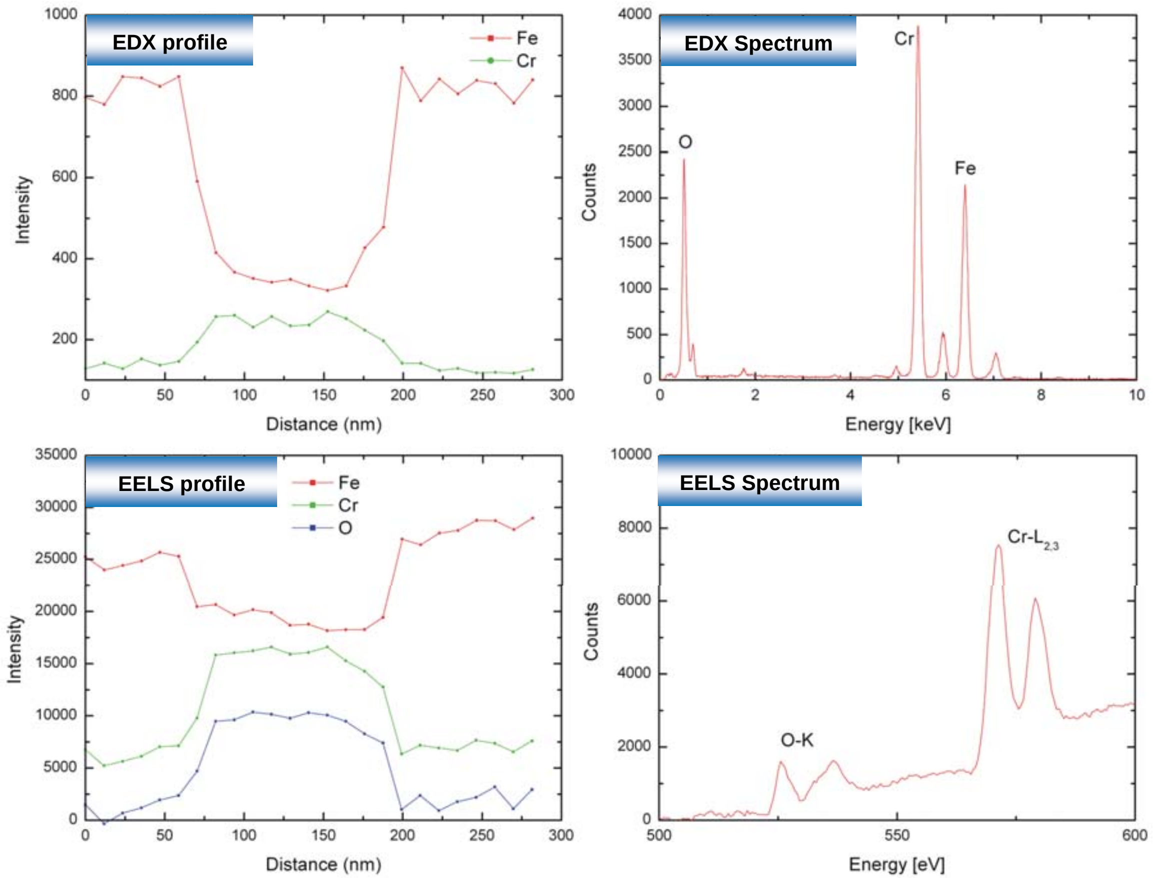


- Darker contrast in BF while brighter in HAADF
- Size:1-3 µm in length, 0.1-0.2 µm in thickness
- EDX and EELS experiments were performed at the position located near to the edge
- Cr carbide

Chemical composition of precipitates

ODS-0 Ti: as-hipped

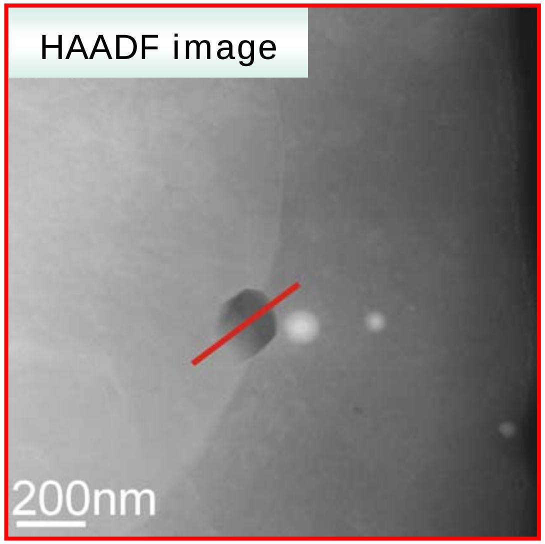


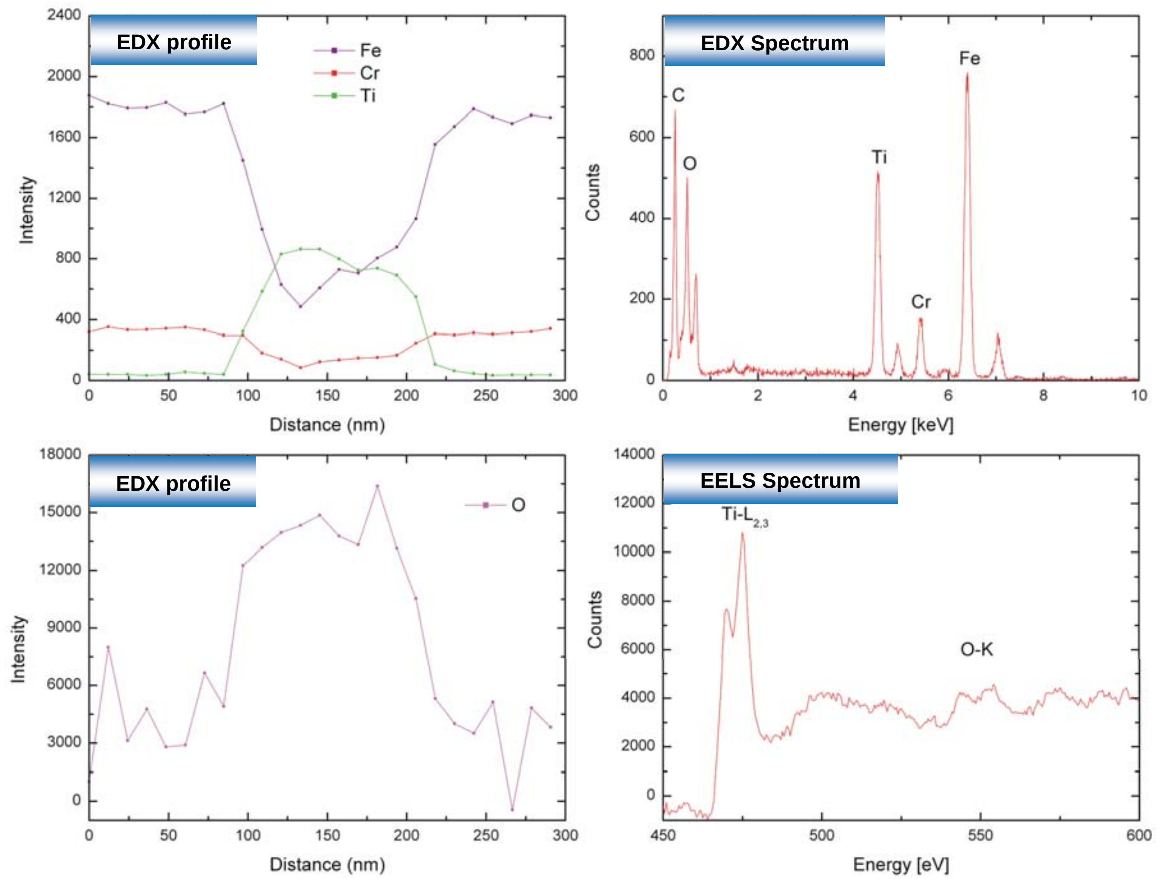


- Spherical precipitate (50-300 nm in diameter) in the non-Ti ODS steel are proven to be Cr oxide.

Chemical composition of precipitates

ODS-0.4 Ti: as-hipped

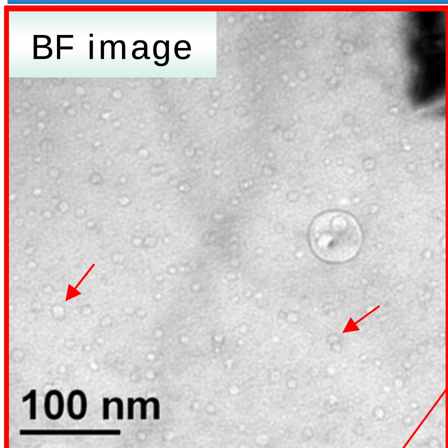


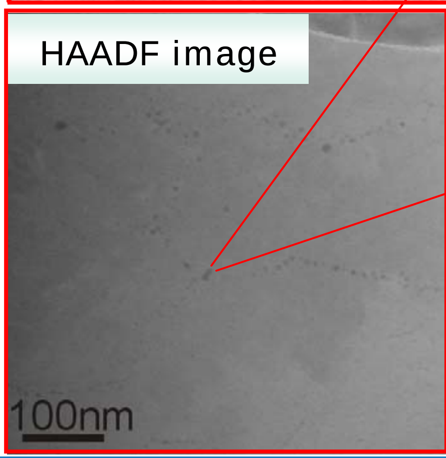


- Spherical precipitate (100-300 nm in diameter) in the 0.4%Ti ODS steel are proven to be Ti oxide.
- Only a few Al oxides were found with a similar size and contrast.

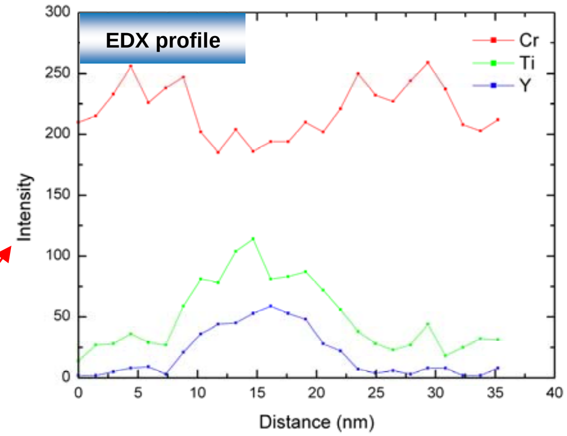
Chemical composition of ODS particles

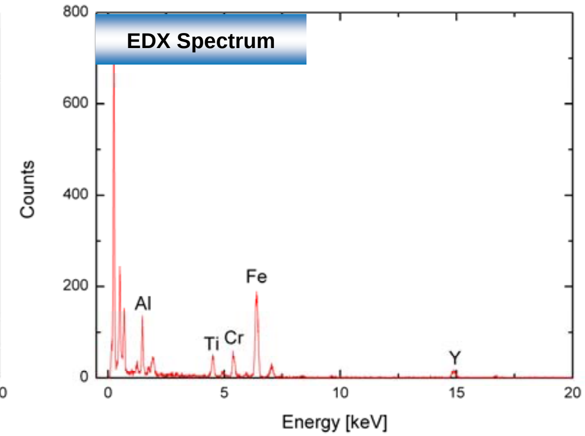
ODS-0 Ti: as-hipped

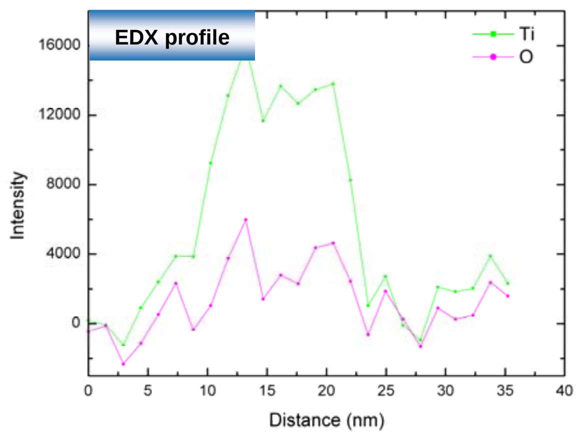


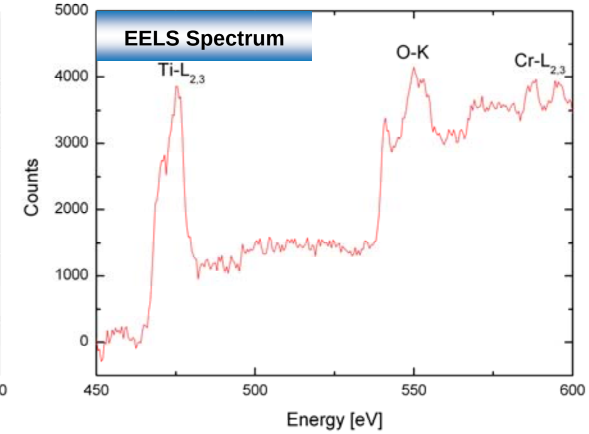


ODS-0.4 Ti: as-hipped



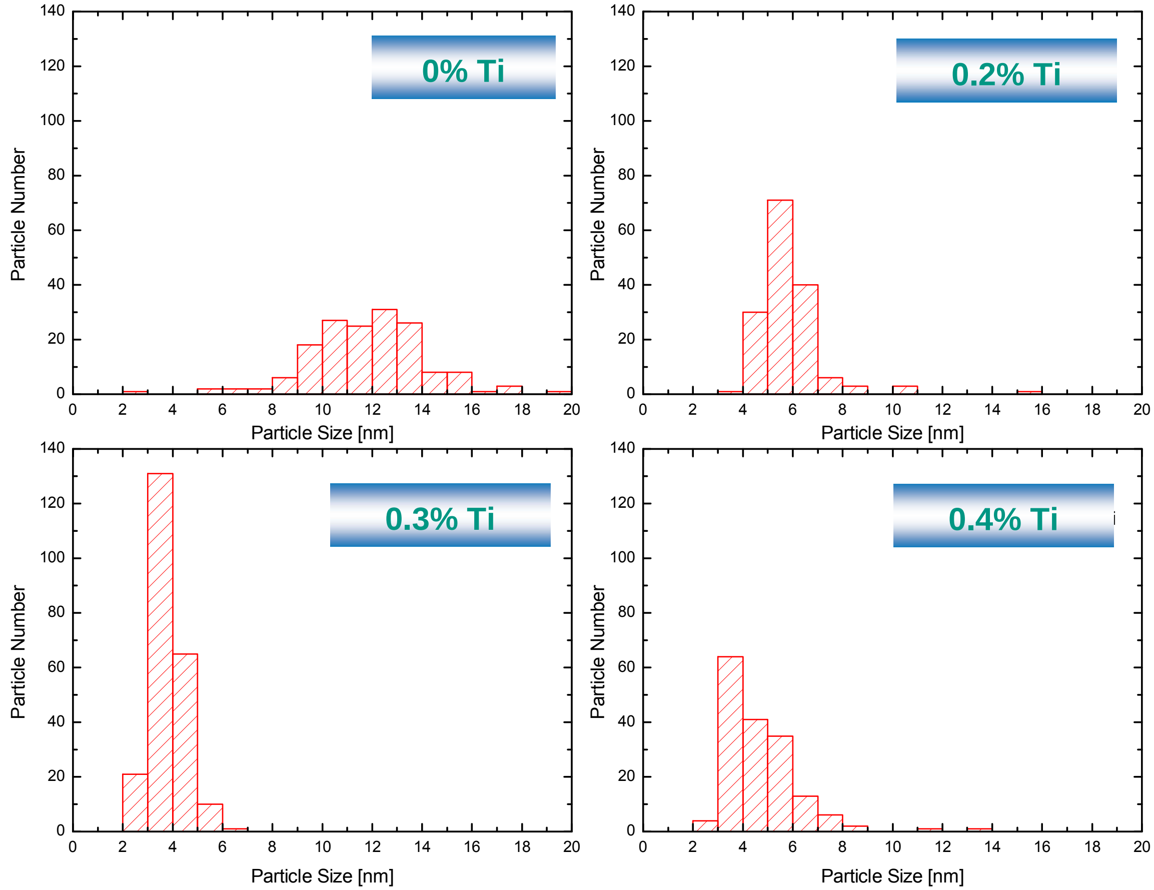


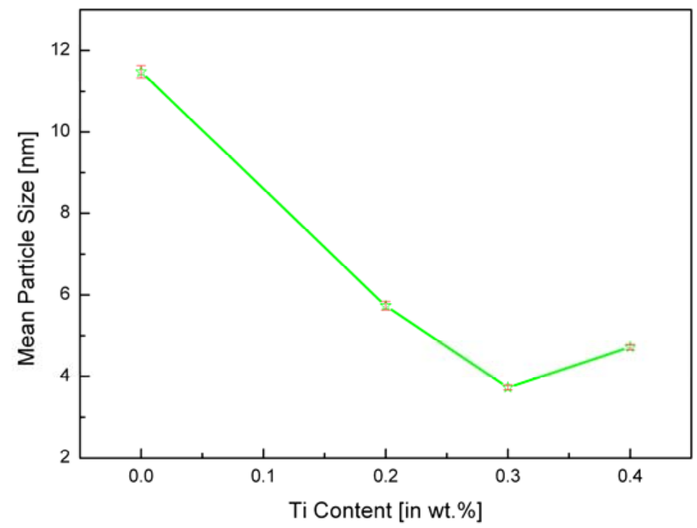




- In non-Ti ODS steel, ODS particles visible as spherical dots with similar contrast to that of the matrix in BF image mainly consist of Y and O.
- In 0.4Ti ODS steel, ODS particles are visible as dark dots consist of Y-Ti-O. A non negligible Al is also detected in some particles.
- Al contamination may result from MA process.

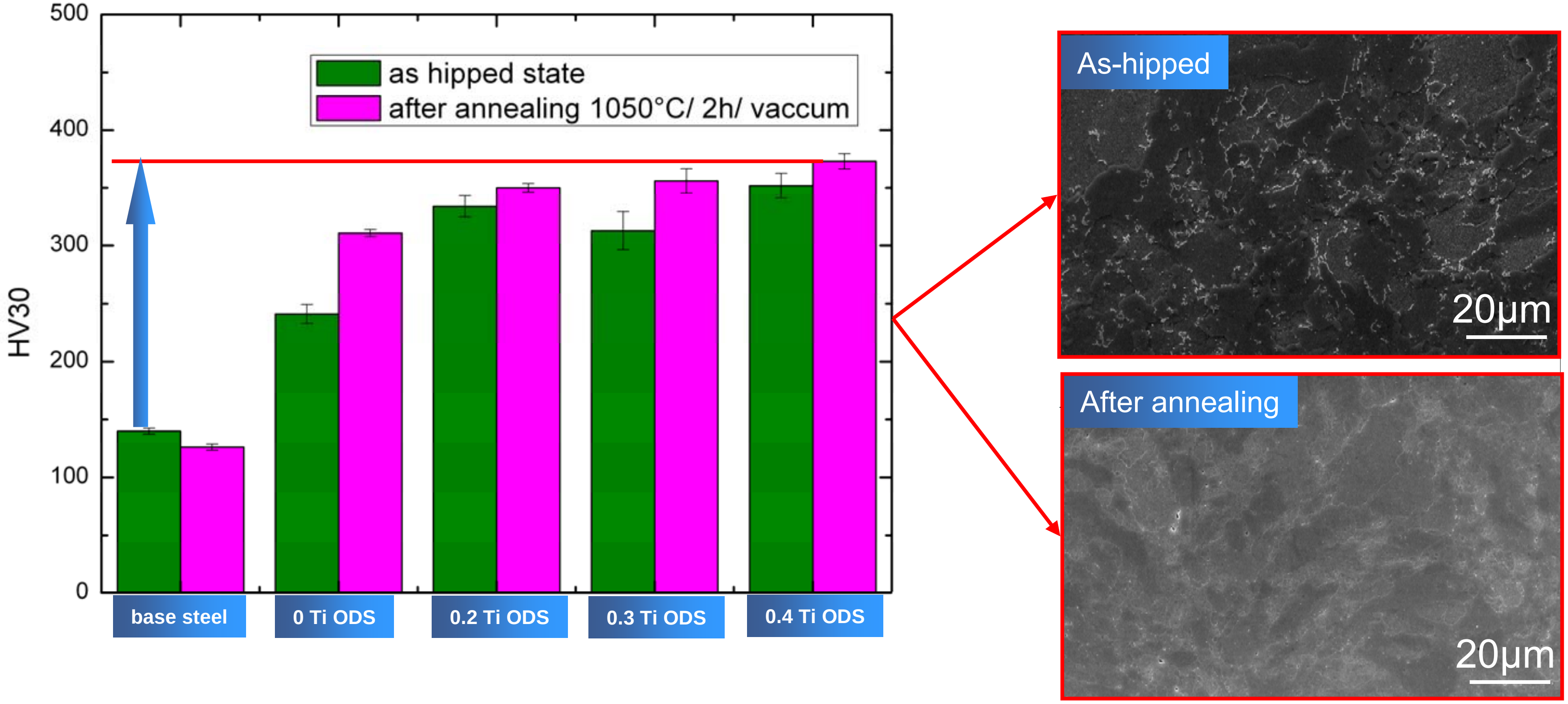
Ti dependence of ODS particle size





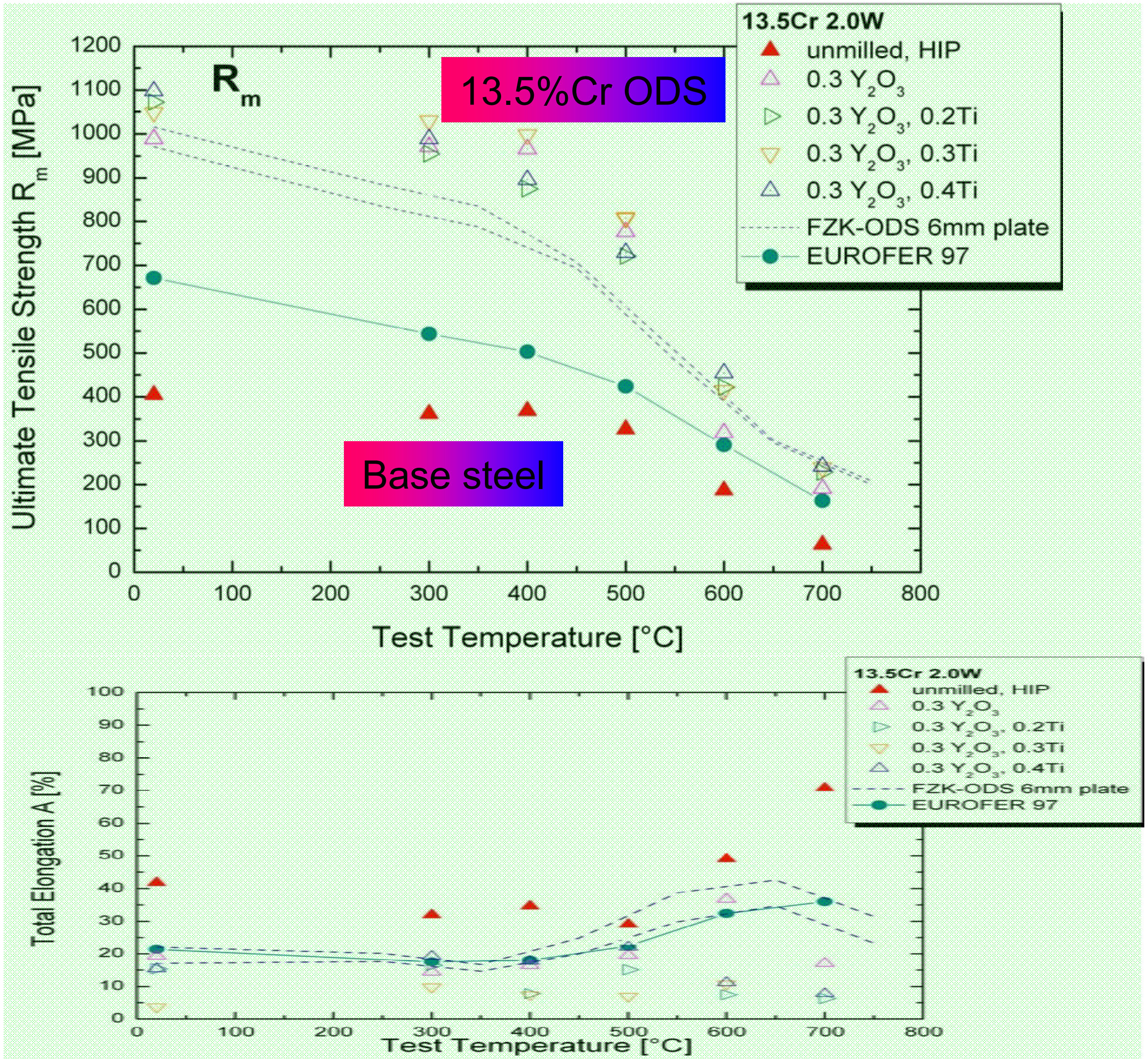
- Ti addition is effective in particle size refinement
- The optimum effect is obtained with 0.3% Ti in terms of narrow range and the smallest mean particle size
- Further increase in Ti may increase the amount of coarse Ti oxides.

Mechanical property- Vickers Hardness



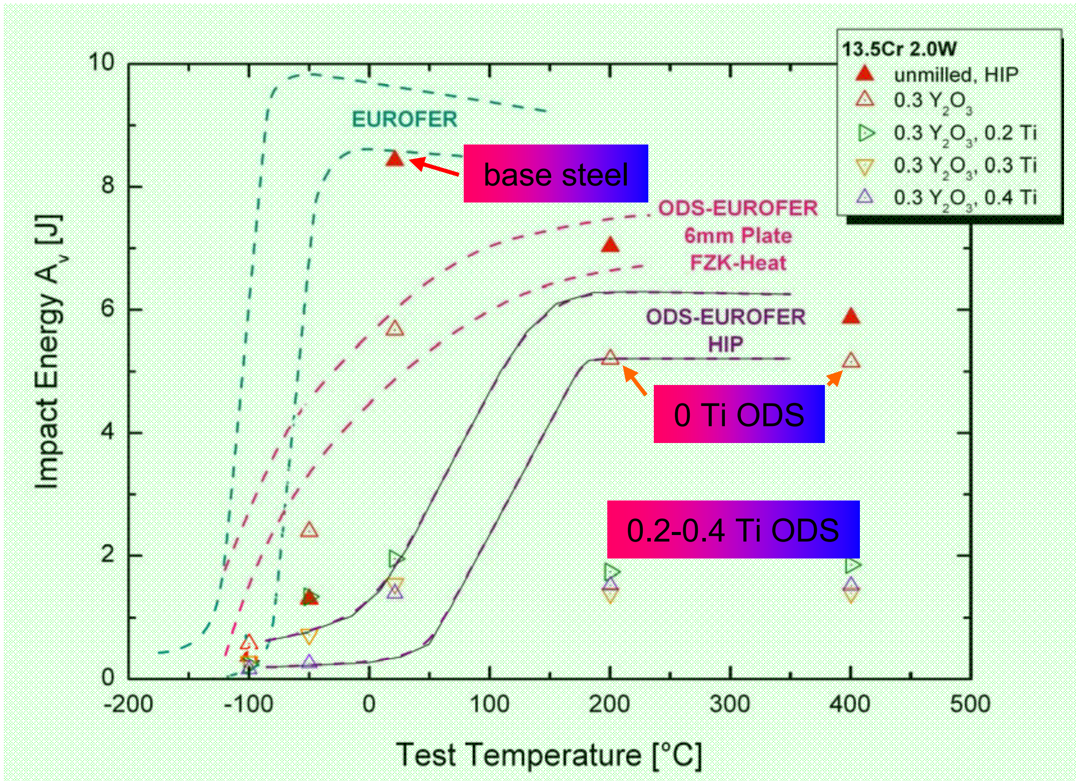
- ODS steels significantly increase the hardness by 160%.
- Generally hardness increases with increasing Ti content.
- Annealing leads to a decrease for base steel ☞ recovery and recrystallization☞ dislocation density↓ grain size ↑
- All ODS alloys exhibit hardness increment after annealing☞ dissolution of Cr carbides during annealing and enhanced Cr solid solution

Mechanical property- Tensile Property



- ODS ferritic steels show more profound strengthening effect compared to 9%Cr ODS
- 0.3Ti ODS steel shows best tensile strength over the test temperature range with lowest high temperature ductility
- 0-Ti ODS steel exhibits satisfactory strength up to 500°C and show a steep drop in strength at higher temperature; comparable ductility to ODS Eurofer

Mechanical properties - Impact Properties



- Base steel exhibits similar USE with 2nd Gen.ODS Eurofer
- 0-Ti ODS steel shows comparable USE of 5.8 J and lower DBTT with the hipped ODS Eurofer
- Ti-containing ODS steels show inferior impact property (USE < 2J)

☹ **There is always trade-off between strength and fracture property!**

☞ **Appropriate Thermal Mechanical Treatment ☺**

Summary

- Fully dense materials were received after HIP. All Ti-containing ODS steels exhibit a bimodal grain size distribution even after annealing.
- Precipitates: coarse precipitates and fine ODS particles
  - elongated Cr carbides in all as-hipped ODS samples; eliminated by annealing
  - Spherical Cr oxide for the 0-Ti ODS steel; spherical Ti oxides (a few Al oxides) for 0.4Ti ODS in as-hipped state as well as in after-annealing state
  - ODS particles consist of Y and O for 0Ti ODS and Y-Ti-O in 0.4Ti ODS (Y-Ti-Al-O).
- 0.3Ti ODS exhibits the optimum effect in particle size refinement ☞ highest ultimate tensile strength
- Mechanical properties
  - Superior hardness and tensile strength. Increase with increasing Ti content.
  - weaker impact properties for all Ti-containing ODS steels (USE<2 J); 0 Ti ODS steel shows comparable USE (5.8J) with 1st Gen. ODS Eurofer and satisfactory tensile strength up to 500° C.
- Future work aims to improve high temperature ductility and impact properties.

Thank you very much for your attentation!