

# Forschungszentrum Karlsruhe in der Helmholtz-Gemeinschaft

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## NUCLEAR RESPONSES IN IFMIF CREEP-FATIGUE TESTING MACHINE

### How ODS particles are formed?

Oxide dispersion strengthened (ODS) steels are produced by mechanical alloying followed by powder consolidation (e.g., hot isostatic pressing=hipping) at temperature around 1000-1200°C and pressure ~100 MPa.

**Contemporary view:** Oxide particles found after hipping are the remnants of the initial oxide powder crushed by **mechanical alloying (MA)** incorporated into steel matrix. This view is supported by the fact that the hipping temperature is much lower than the melting temperature of yttria ( $T_m \sim 2410^\circ\text{C}$ ) and therefore any thermal processes of oxide particle transformation should be excluded.

#### New insight:

Experiments performed by other groups on 12-14%Cr ferritic steels have shown:

- SAXS, SANS and TEM analysis of MA powder show no traces of yttria particles
- SANS shows formation of **nanoclusters (NCs)** after annealing of MA powder at temperatures higher than 850°C. The NC sizes increase and their volume fractions and number densities decrease with increasing the consolidation temperature in the manner consistent with Ostwald ripening process.
- ODS particles are stable during annealing at hipping temperature for a long time (e.g., 243 h @ 1150°C)
- Addition of Ti could hardly affect MA process, however, resulting in formation of smaller ODS particles
- Addition of Ti in the form of oxides results in formation of mixed Y-Ti oxides
- 3D atomic probe performed on hipped ODS ferritic steels reveals:
  - presence of Y and O in solid solution
  - significant (x16 times) increase of Y concentration near dislocations
  - formation of nm-sized spherical zones with increased concentration of Y and O.

The experiments performed on 9%Cr ferritic-martensitic ODS steels (Eurofer) presented in this work are consistent with the results obtained by other groups for 14%Cr ferritic steels.

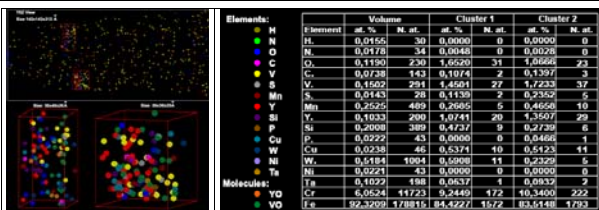
### Results: 3D Atomic Probe Analyses of Eurofer ODS

**Materials:** Eurofer and Eurofer ODS (0.5%Y<sub>2</sub>O<sub>3</sub>)

**Samples preparation:** cutting performed by electro-chemical erosion method

**Method:** Energy Compensated Optical Tomography Atomic Probe (ECOTAP).

Material	ECOTAP results
Eurofer	No impurity segregations
Eurofer ODS	Two types of zones with increased impurity concentrations were found: <ul style="list-style-type: none"> <li>• Spherical zones of ~3x3x3 nm<sup>3</sup></li> <li>• Plate-like zones with thickness of ~2 nm, spacing between layers varies from 2 to 30 nm within the same sample</li> </ul>



### Results: ODS-Ferrite Matrix Orientation Relationship

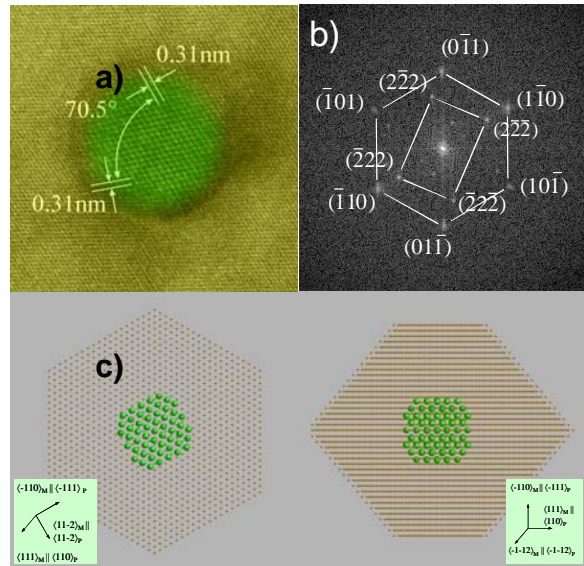
**Material:** Eurofer ODS (0.5% Y<sub>2</sub>O<sub>3</sub>) hipped and tempered

Kurdjumov-Sachs type orientation relationship was observed:

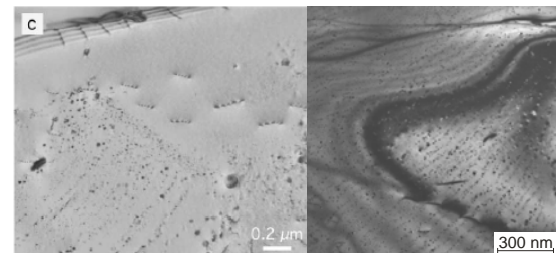
$$\langle 111 \rangle_M \parallel \langle 110 \rangle_F \text{ and } \langle \bar{110} \rangle_M \parallel \langle \bar{111} \rangle_F$$

ODS particles are partly coherent with ferrite matrix.

HRTEM micrograph of Y<sub>2</sub>O<sub>3</sub> particle embedded into ferrite matrix (a) and its Fourier transformation as well as crystallographic model showing yttrium and iron atom positions corresponding to the observed orientation relationship (c).

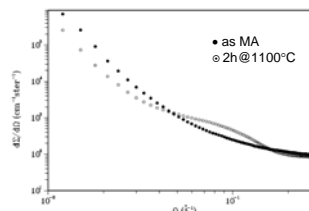


### Results: TEM of Eurofer Samples



R. Schaublin et al., JNM 307-311 (2002) 778-782

### Results: SANS on MA Eurofer ODS Powder



**Material:** 9%Cr EUROFER ODS (0.5% Y<sub>2</sub>O<sub>3</sub>) powder

Production step	SANS results
powder as MA	no NCs observed
MA powder annealed at 1100°C for 2 h	NCs of ~10 nm

### Conclusions

based on the results for ferritic and ferritic-martensitic ODS steels:

- Yttrium oxide particles added to steel powder are destroyed and, at least, partly dissolved in the matrix during MA.
- ODS particles form during hipping (or during cooling after hipping) by precipitation of dissolved atoms
- Once formed ODS particles are stable and coarsen only at temperatures higher than the hipping one.
- Small spherical zones found by 3D atomic probe can be considered as precursor of precipitates.
- Addition of Ti, which compete with Y for O, changes the kinetics of precipitation resulting in formation of smaller particles.