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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 ~2410°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:

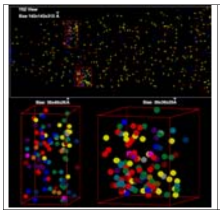
- SAXS, SANS and TEM analysis of MA powder show no traces of yttria particles
- SANS shows formation of nanoclusters (**NCs**) after annealing of MA powder at temperatures higher than 850°C. The NC sizes increase and their volume fractions and number densities decrease with increasing the consolidation 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

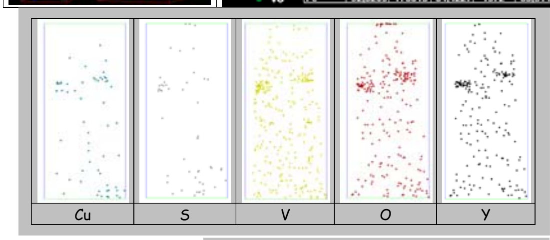
Materials: Eurofer and Eurofer ODS (0.5%Y_2O_3)
Samples preparation: cutting performed by electro-chemical erosion method
Method: Energy Compensated Optical Tomography Atomic Probe (ECOTAP).

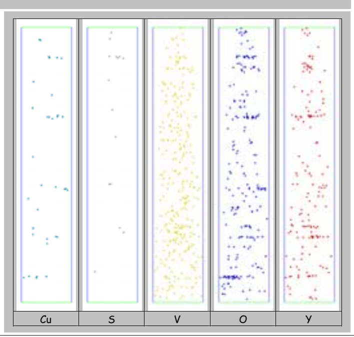
Material	ECOTAP results
Eurofer	No impurity segregations
Eurofer ODS	Two types of zones with increased impurity concentrations were found: • Spherical zones of ~3x3x3 nm3 • Plate-like zones with thickness of ~2 nm, spacing between layers varies from 2 to 30 nm within the same sample



Elements: H, N, O, C, V, S, Mn, Y, Si, P, Cu, W, Ni, Ta
Molecules: YO, VO

	Volume		Cluster 1		Cluster 2	
Element	at. %	N. at.	at. %	N. at.	at. %	N. at.
H.	0,0155	30	0,0000	0	0,0000	0
N.	0,0178	34	0,0048	0	0,0028	0
O.	0,1190	230	1,6520	31	1,0666	23
C.	0,0738	143	0,1074	2	0,1397	3
V.	0,1502	291	1,4501	27	1,7233	37
S.	0,0143	28	0,1139	2	0,2352	5
Mn	0,2525	489	0,2685	5	0,4658	10
Y.	0,1033	200	1,0741	20	1,3507	29
Si	0,2008	389	0,4737	9	0,2739	6
P.	0,0222	43	0,0000	0	0,0466	1
Cu	0,0238	46	0,5371	10	0,5123	11
W.	0,5184	1004	0,5908	11	0,2329	5
Ni	0,0221	43	0,0000	0	0,0000	0
Ta	0,1022	198	0,0637	1	0,0932	2
Cr	6,0524	11723	9,2449	172	10,3400	222
Fe	92,3209	178815	84,4227	1572	83,5148	1793





Results: ODS-Ferrite Matrix Orientation Relationship

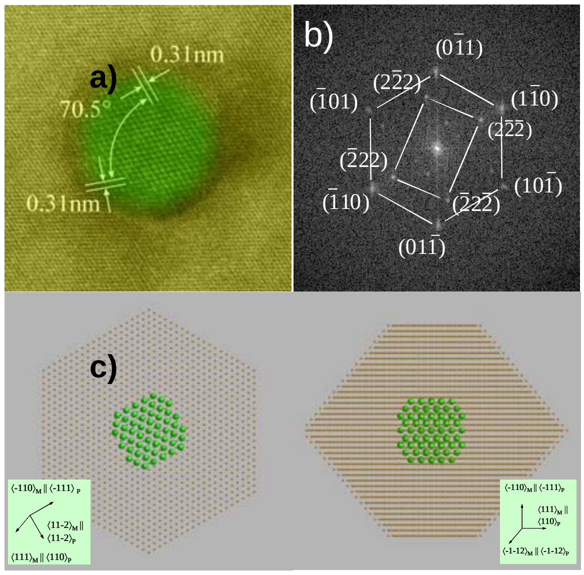
Material: Eurofer ODS (0.5% Y_2O_3) hipped and tempered

Kurdjumov-Sachs type orientation relationship was observed:

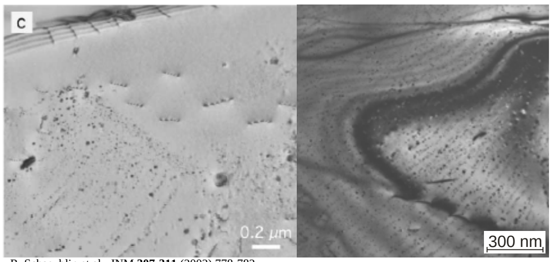
$\langle 111\rangle_M \parallel \langle 110\rangle_P$ and $\langle -110\rangle_M \parallel \langle -111\rangle_P$

ODS particles are partly coherent with ferrite matrix.

HRTEM micrograph of Y_2O_3 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).

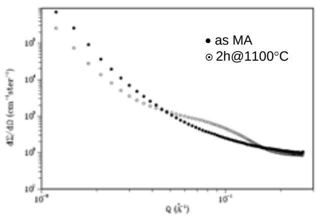


Results: TEM of Eurofer Samples



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

Results: SANS on MA Eurofer ODS Powder



Material: 9%Cr EUROFER ODS (0.5% Y_2O_3) 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 that 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.