KIT Karlsruhe Institute of Technology

IAM Institute for Applied Materials

Formation and development of nanoclusters in ODS steels and their influence on mechanical properties

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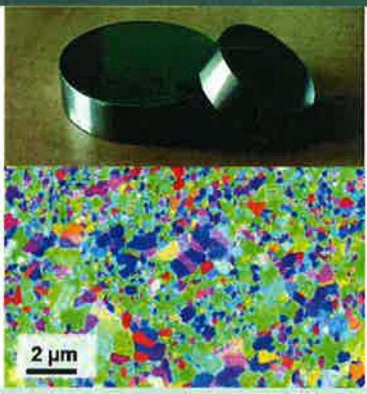
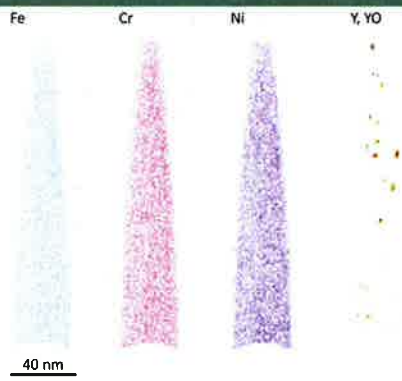
Institute for Applied Materials IAM–WK





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Motivation

ODS steels

Nanoclusters · **Ultrafine-grained**

Radiation resistance · **Corrosion resistance** · **Creep resistance**

➢ How are the mechanical properties influenced by the microstructure?

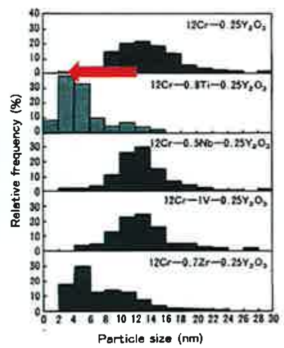
- Expections
  - High thermal stability of Y-Ti-O particles
  - Pinning of grain boundaries by particles
  - Little grain growth

Materials

- Analysis of ferritic and austenitic ODS steels
- Improved high temperature properties of austenitic (fcc) ODS steel expected

In wt. %	Fe	Cr	Ni	Ti	Y_2O_3
FNC 14	Bal.	**14**		0.4	0.25
ANC 25/20	Bal.	**25**	**20**	0.4	0.25

- Decreasing Y_2O_3 particle size by titanium additions
- Formation of Y-Ti-O-containing nanoclusters (~ 4 nm)



S. Ukai et al., J. Nucl. Mater. 307 (2002)

Processing

Zoz Simoloyer CM01
www.zoz-gmbh.de

Retsch PM400
www.retsch.de

- Cylinders with very low porosity
- Diameter between 20 and 40 mm

Mechanical Alloying

- Attritor or planetary ball mill
- Elemental powders + Y_2O_3
- Argon atmosphere

FAST

- Field Assisted Sintering Technique
- Pressure of 50 MPa
- 5 min at 1000 °C
- Fast heating and cooling rate (100 K/min)

- APT
- SEM / EBSD
- Annealing
- Compression tests

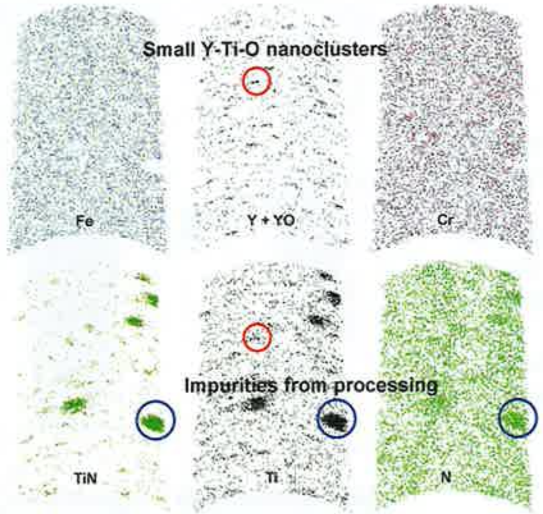
Atom probe measurements

- Tips produced by SEM/FIB
- LEAP 4000X HR by Cameca®

FNC 14: mainly voltage mode
- detection rate: 0.3 %
- pulse rate: 200 kHz
- pulse fraction: 19 to 20 %
- temperature: 50 to 60 K

ANC 25/20: laser mode
- detection rate: 0.3 %
- pulse rate: 200 kHz
- pulse energy: 100 pJ
- temperature: 50 K

Reconstruction of a FNC 14 (FAST) tip



- 5 nm thick, 2-dimensional longitudinal section of the reconstruction

- ➢ Proof of cluster formation in FNC 14
- ➢ Identification of clusters with maximum separation method

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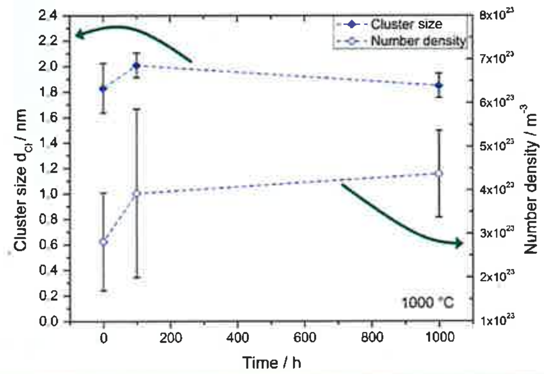
Comparison of ANC 25/20 and FNC 14

ANC 25/20 (FAST)
- Cluster size: 4.9 nm
- Number density: $1.2 \cdot 10^{23}\ m^{-3}$

FNC 14
- ➢ Similar volume fraction
- ➢ Smaller clusters
- ➢ Higher number density



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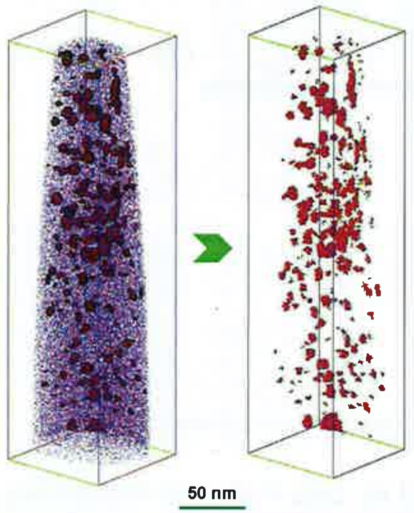
Composition of nanoclusters

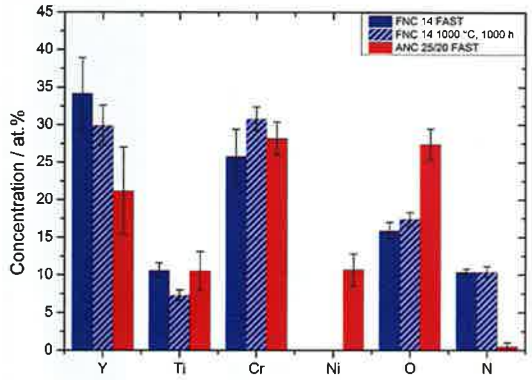


- Removal of matrix
- Modification of mass spectrum
- Decomposition of peaks

- Fe content artificially set to 0
- Cr (and Ni) content reduced proportionally

➤ Average composition of all identified nanoclusters

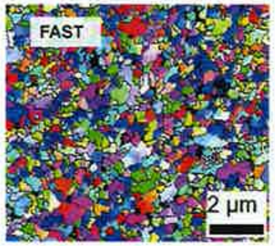
Composition of nanoclusters



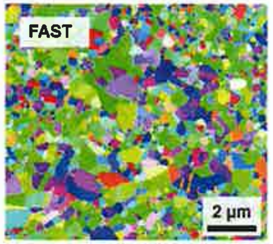
- No significant changes in **FNC 14** during annealing
- N seems to substitute O in **FNC 14**
- Ni seems to substitute Y in **ANC 25/20**

➤ Ratio of (Y,Ti,Cr,Ni) to (O,N) is around 3:1 in both alloys

Grain size



FNC 14



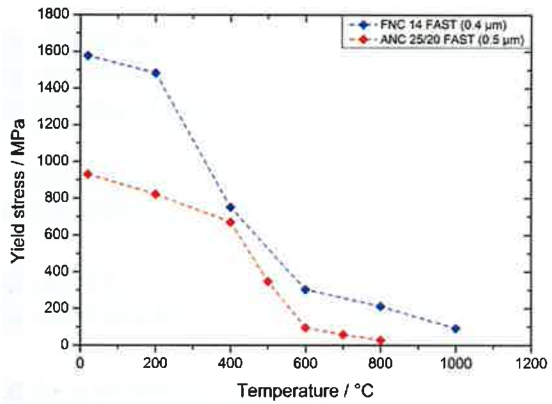
ANC 25/20

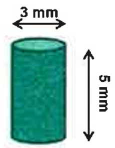
- Determination of grain size
- Orientation mappings from EBSD measurements
- Area weighted averaging

➢ Comparable grain size of all ODS steels after consolidation (between 0.4 and 0.5 µm)
➢ No grain growth even after 1000 h at 1000 °C
➢ Nanoclusters effectively pin grain boundaries and prevent grain growth

➢ No thermally induced changes in microstructure up to 1000 °C!

Mechanical properties – Compression tests





- Compression tests between room temp. and 1000 °C
- Constant strain rate: 10^{-4} s^{-1}

- Strength of **FNC 14** > **ANC 25/20** at all temperatures

➢ Influence of smaller clusters and higher amount of clusters
➢ Drop of strength for **ANC 25/20** at higher temperature

Modeling of strength

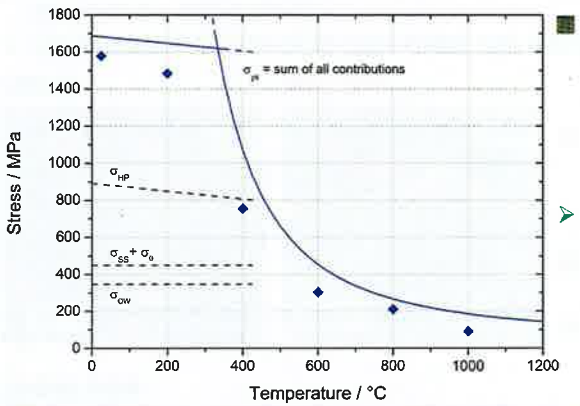
$$\sigma_{ys} = \sigma_0 + \sigma_{HP} + \sigma_{OW} + \sigma_{ss}$$

$$\sigma_{HP} = \frac{k_{HP}}{\sqrt{d}}$$

Hall-Petch strengthening

$$\sigma_{OW} = \frac{MGb}{d_p}\sqrt{\frac{6f}{\pi}}$$

Orowan strengthening



- Calculation of strengthening contributions in the low temperature range

➢ Calculated strength can depict results from compression tests very well.

Summary

- Production and analysis of ferritic and austenitic ODS steels
- APT for characterization of nanoclusters

- Large number of nanoclusters (< 5 nm) in both steels
- Ratio of Y, Ti, Cr(, Ni) to O (and N) was 3:1

- Nanoclusters and grain size (around 0.5 µm) stable during long-term annealing at 1000 °C

- Strength of **FNC 14** higher at all temperatures
- Drop of strength at higher temperature for **ANC 25/20**
- Calculation of strengthening contributions possible

Thank you very much for your attention!

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