Thermal stability of ferritic and austenitic nanocluster containing ODS steels

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Motivation

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Steels for high temperature applications

- Advantages: low cost, low density, well characterized
- Disadvantages: use limited up to 700 °C

Benefits of oxides addition

- Formation of nanoclusters → limit mean free path of dislocations
- Pinning of grain boundaries hinder grain growth

Characteristics of typically used ferritic oxide dispersion strengthened (ODS) steels

- Stable long-term microstructure at high temperatures
- Resistance against radiation-induced swelling
- Superb corrosion and oxidation resistance
- Extraordinary good creep properties

➢ Do austenitic ODS steels have even better properties?

Overview

- Materials
  - Ferritic
  - Austenitic
- Thermal stabiltiy
- Cluster analysis by APT
- Mechanical properties

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Materials

Two **ferritic** alloys were prepared

- Nominal composition:

in wt.%	Fe	Cr	Ti	Y_2O_3
NC 0.4Ti-0.25Y_2O_3	balance	14	**0.4**	**0.25**
NC 1Ti-0.5Y_2O_3	balance	14	**1**	**0.5**

- Mechanical alloying from elemental powders in attritor mill
  - 1000 rpm
  - Steel balls (BPR = 10:1)
  - 4800 cycles of 45 s milling and 15 s cooling
  - Ar atmosphere and -10 °C
- Compaction by field assisted sintering technique (FAST)
  - Heating/cooling rate: 100 K/min
  - Temperature: 1000 °C
  - Pressure: 50 MPa
  - Time: 5 min

Materials

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Additionally one **austenitic** alloy was prepared

in wt.%	Fe	Cr	Ni	Ti	Y_2O_3
NC 0.4Ti-0.25Y_2O_3	balance	14	--	0.4	0.25
NC 1Ti-0.5Y_2O_3	balance	14	--	1	0.5
ANC 0.4Ti-0.25Y_2O_3	balance	16	16	0.4	0.25

- Mechanical alloying from elemental powders
- Low gains and changes in composition in attritor mill

- Use of planetary ball mill
  - 200 rpm
  - WC balls (BPR = 10:1)
  - 300 cycles of 1 min milling and 2 min cooling
  - Ar atmosphere
- Compaction by field assisted sintering technique (FAST)

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XRD measurements on ANC 0.4Ti-0.25Y_2O_3

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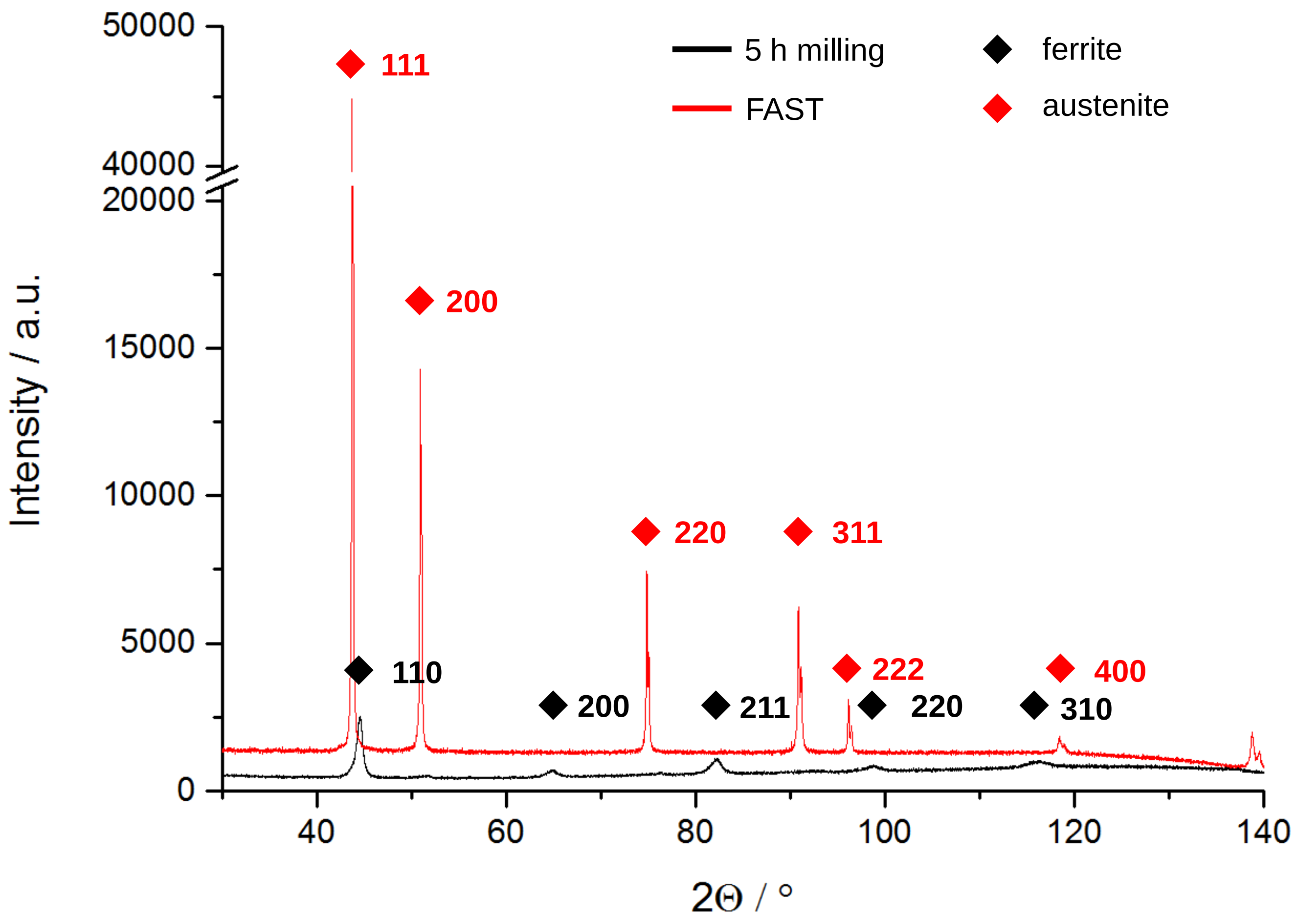

Milling ➔ **pure ferrite** ➔ *compaction* ➔ pure austenite

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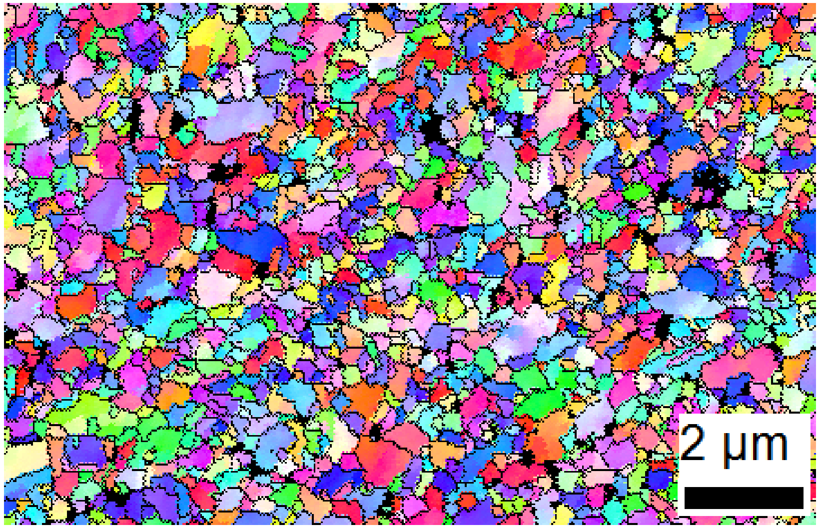
Microstructure after compaction

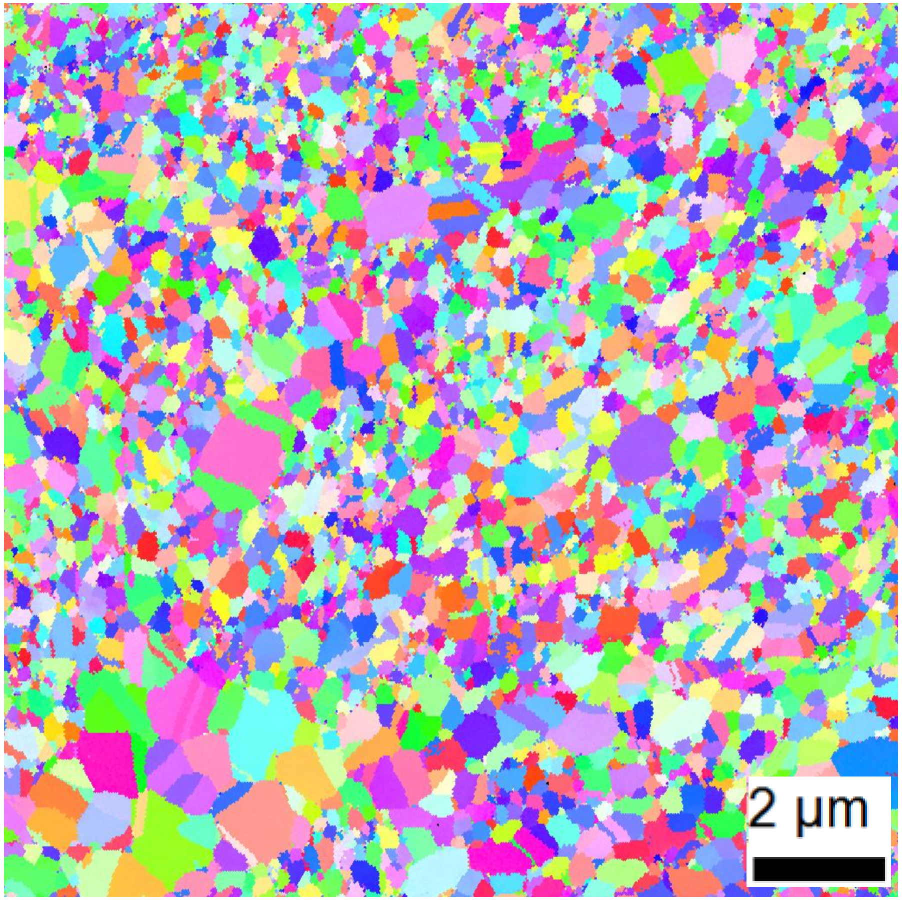


NC 0.4Ti-0.25Y_2O_3

- Initial grain size in all alloys similar

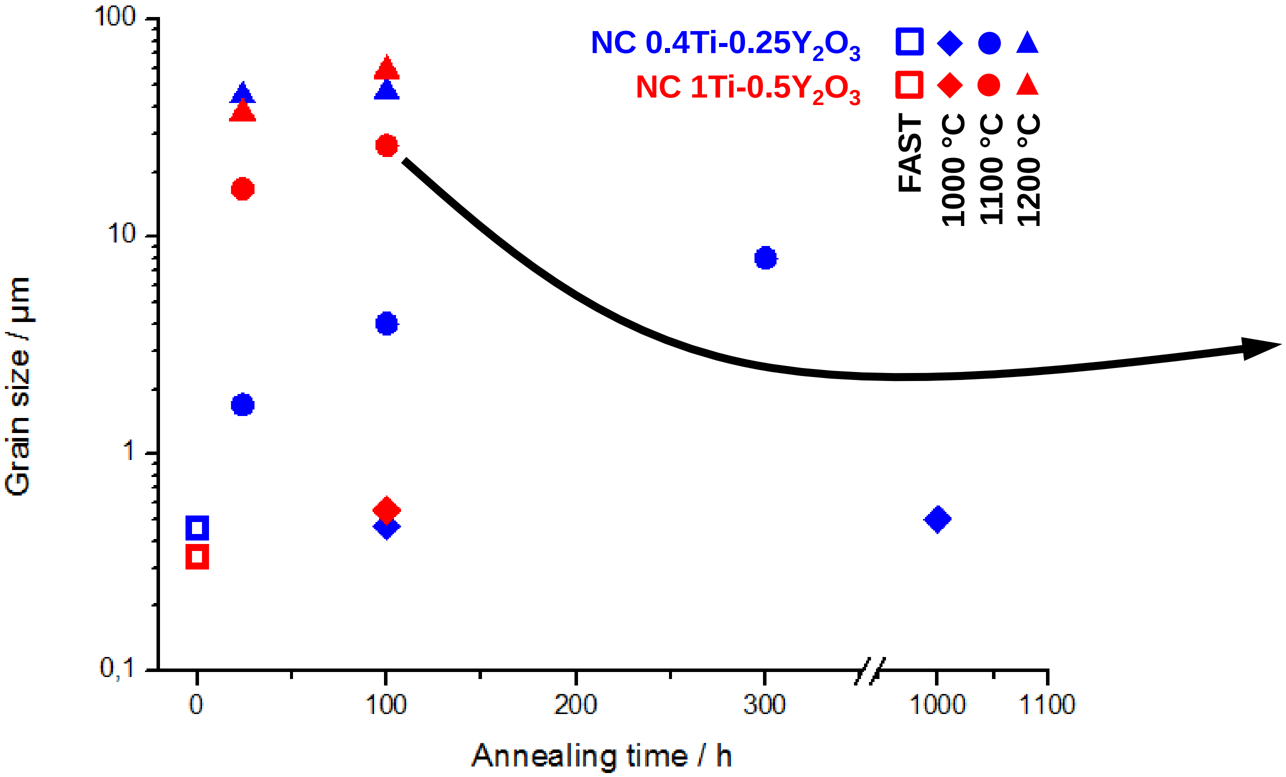
NC 0.4Ti-0.25Y_2O_3	0,46 µm
NC 1Ti-0.5Y_2O_3	0,34 µm
ANC 0.4Ti-0.25Y_2O_3	0,38 µm

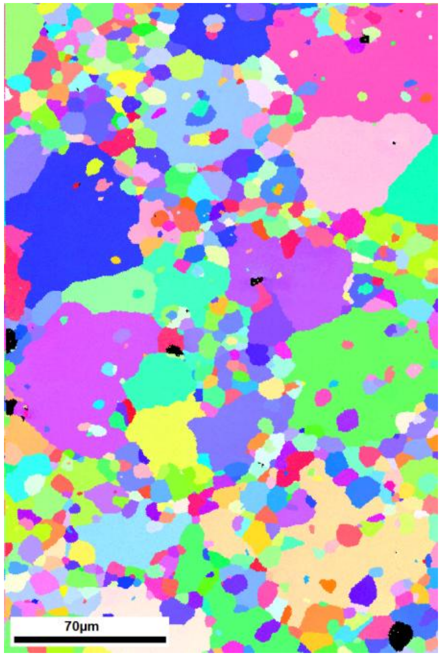
- No texture caused by FAST



ANC 0.4Ti-0.25Y_2O_3

Heat treatment of ferritic ODS steels

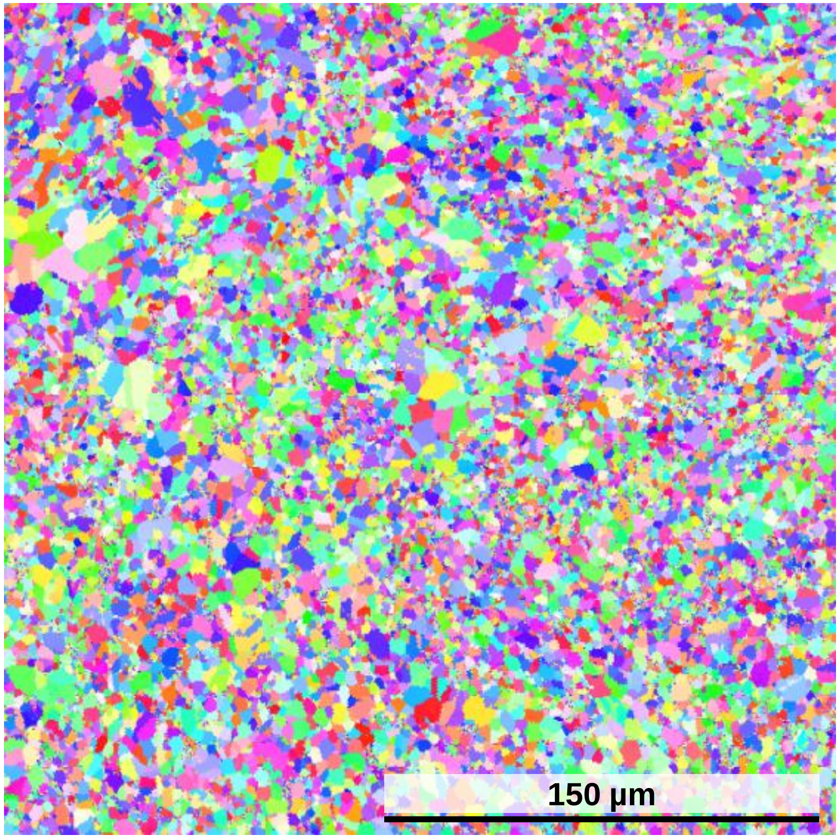




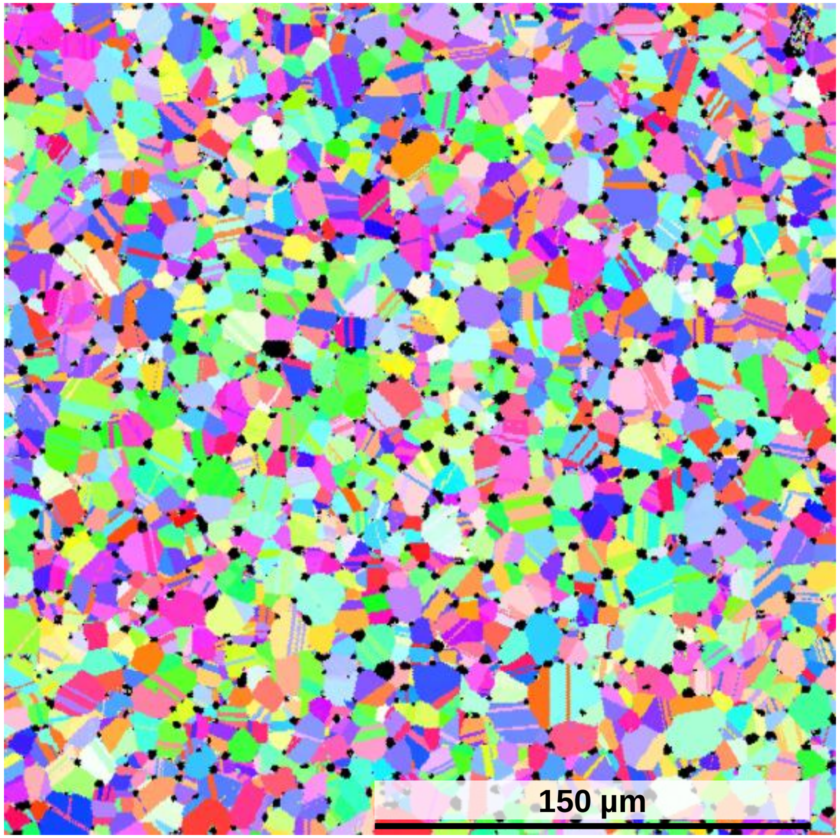
NC 1Ti-0.5Y_2O_3 after 100 h at 1100 °C

- Stable grain size at 1000 °C
- Growth of selected grains at 1100 °C for NC 1Ti-0.5Y_2O_3
- Fast grain growth at 1200 °C

Heat treatment of ANC 0.4Ti-0.25Y_2O_3

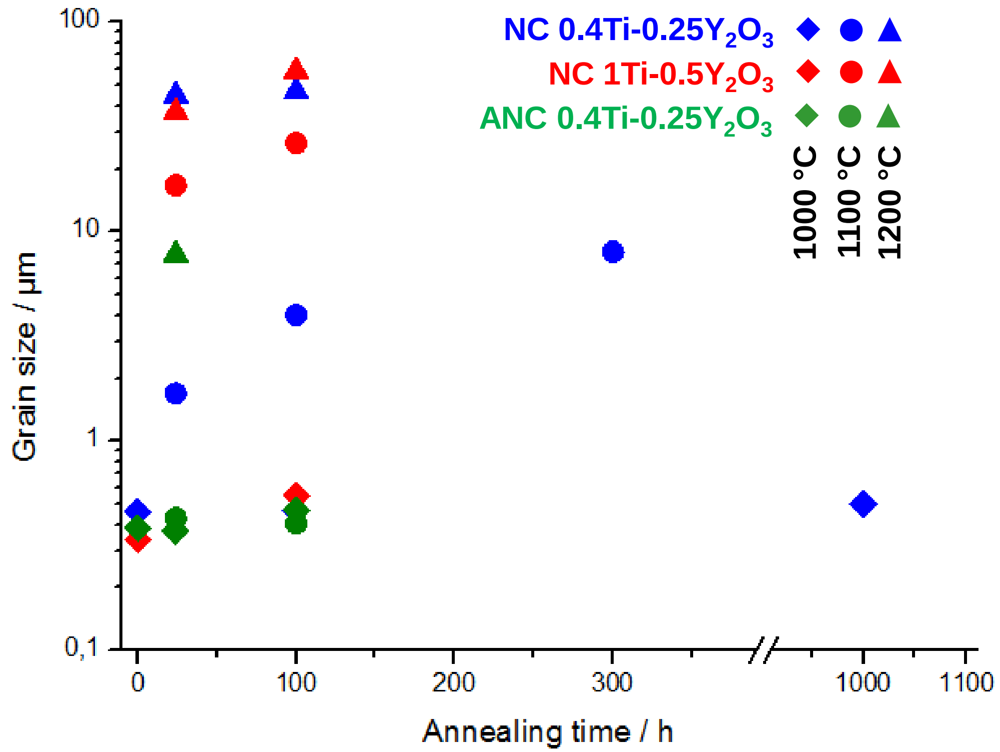


100 h at 1100 °C



24 h at 1200 °C

Heat treatment of austenitic ODS steels



ANC 0.4Ti-0.25Y_2O_3

No grain growth at 1000 °C and even 1100 °C

Smaller grains after 24 h at 1200 °C compared to ferritic ODS steels

- Grain growth starts possibly at higher temperatures for austenitic ODS steel

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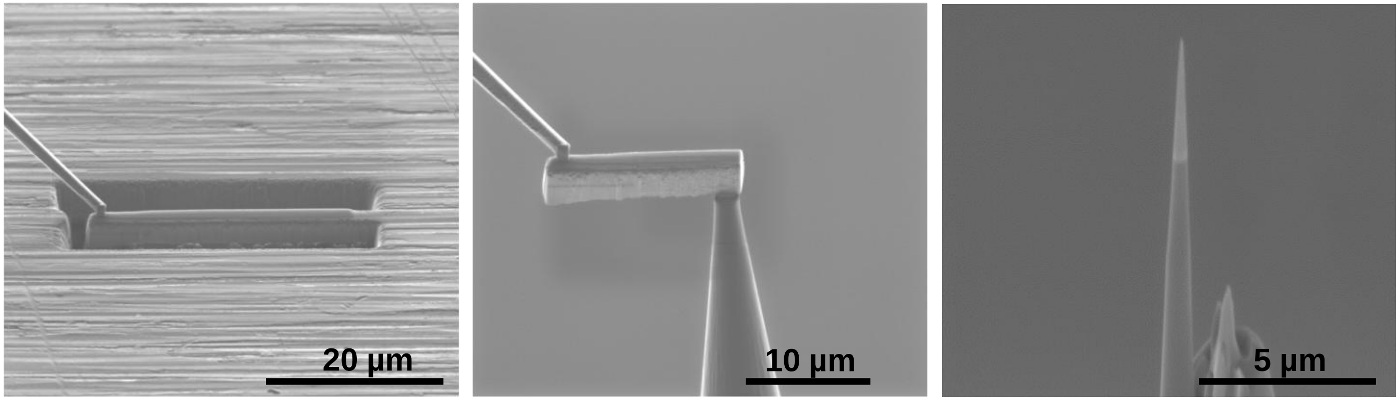
Analysis of nanoclusters

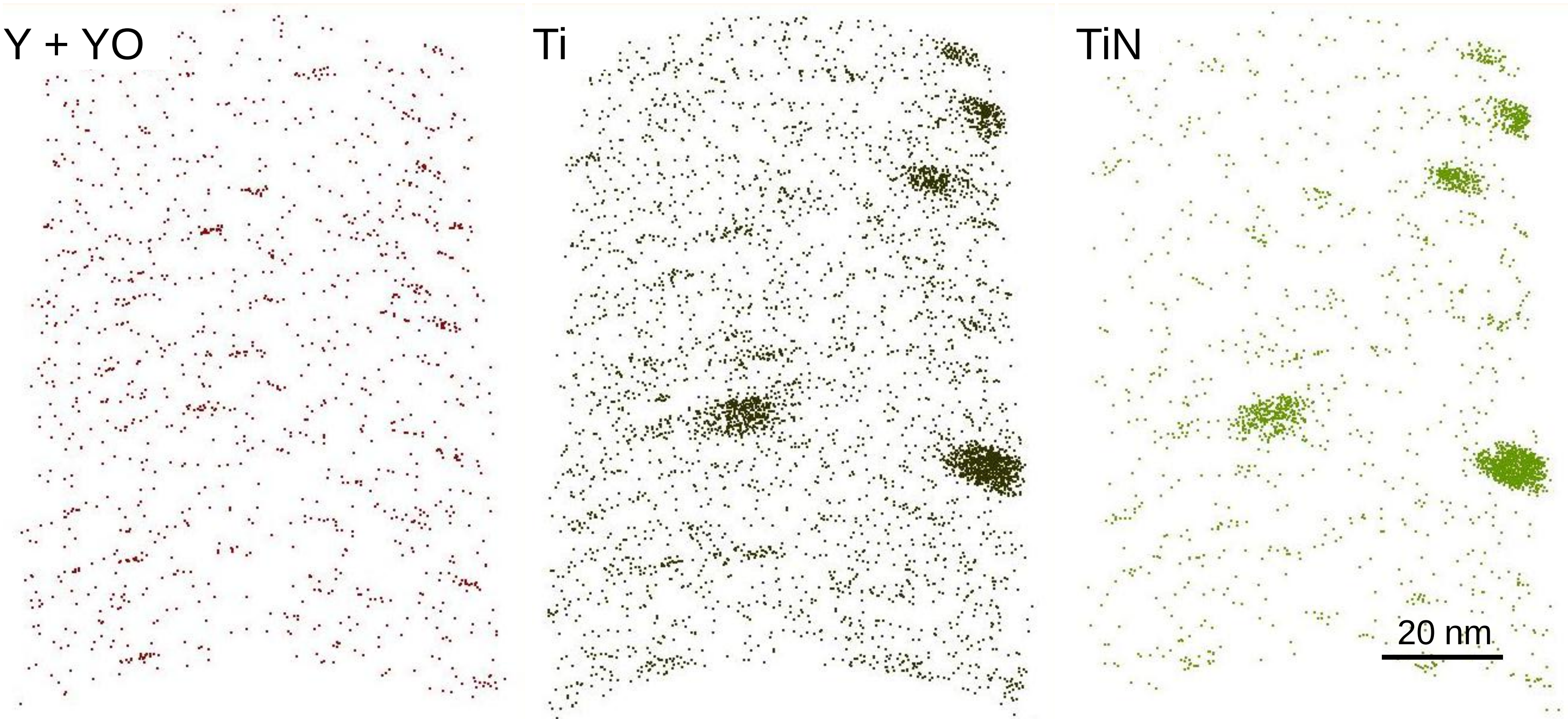
Nanoclusters most likely responsible for high thermal stability of ODS steels

- Analysis of nanoclusters by Atom Probe Tomography
- Three states of NC 0.4Ti-0.25Y_2O_3 were investigated
  - As FAST
  - After 100 h and 1000 h at 1000 °C
- FIB lift out specimens

<ins>Parameters</ins>
Voltage atom probe
T_{set} = 50 to 60 K
Detection rate = 0.3 to 0.5 %
Pulse rate = 200 kHz
Pulse fraction = 19 to 20 %

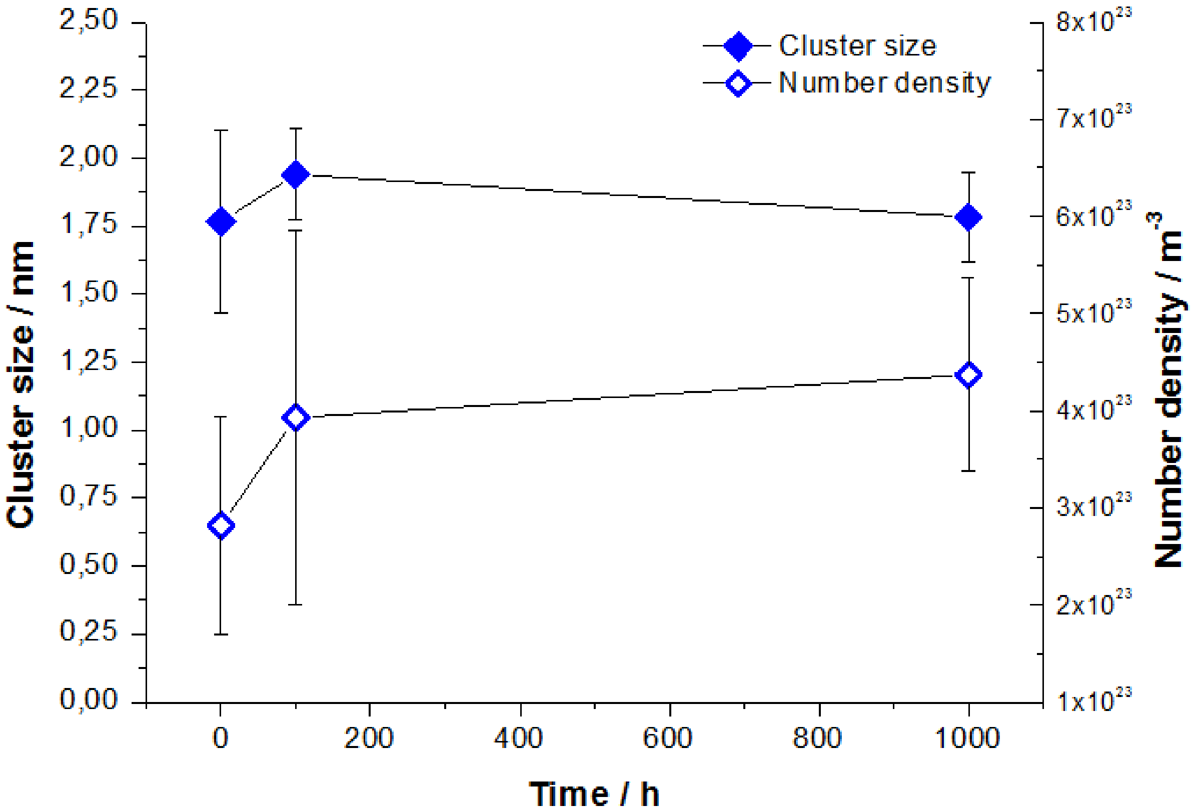


APT of NC 0.4Ti-0.25Y_2O_3 initial state



- Nanoclusters are detected
- Small clusters coincide with Y and Ti
- Impurities from processing

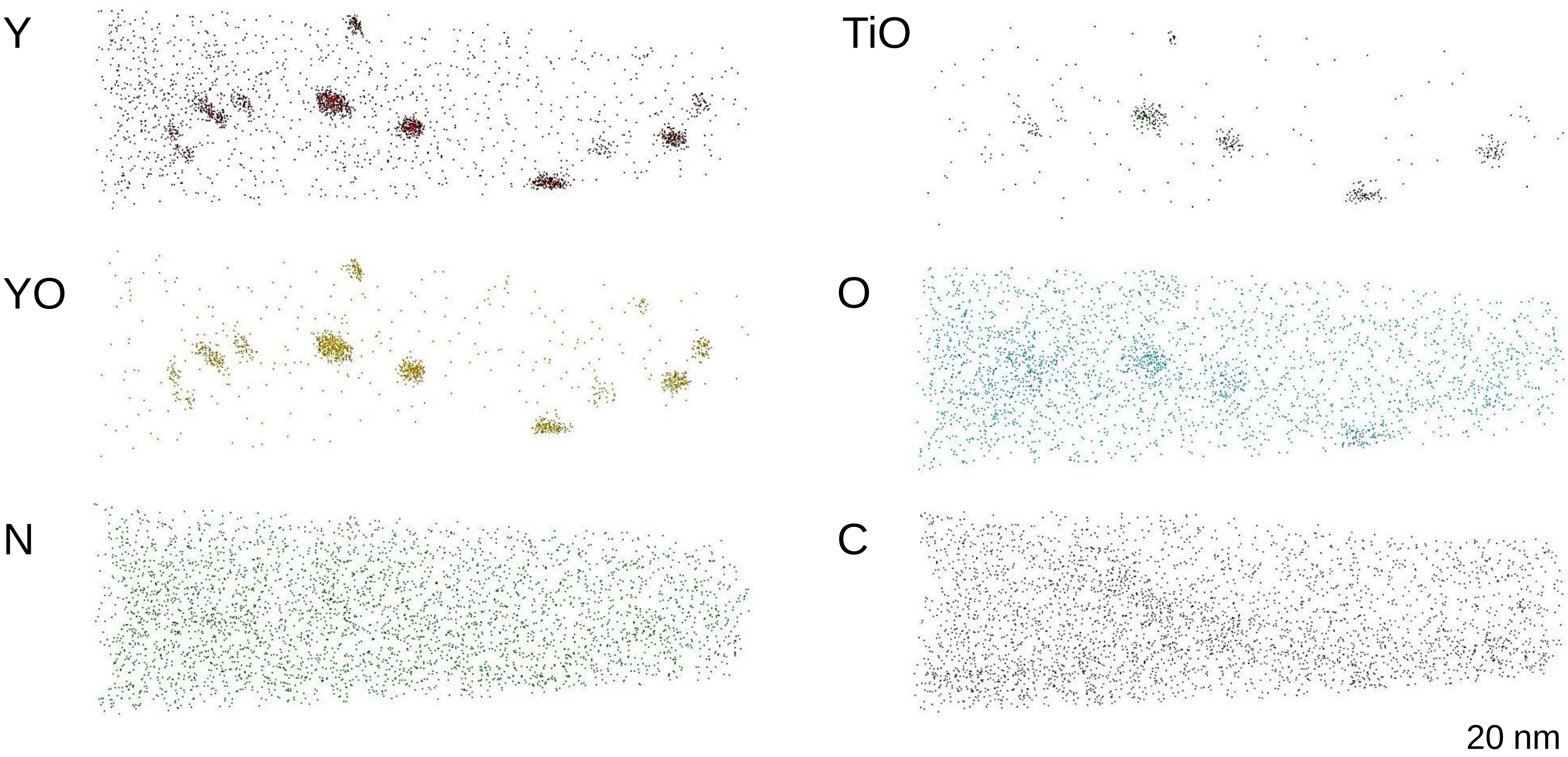
Development of cluster size during annealing



- $d = 2\sqrt{5}\, r_g$
- Initial state:
  3 to 5 nm large Ti-N-rich particles
- After 1000 h at 1000 °C: no Ti-N-rich particles found

- Cluster size is stable up to 1000 h at 1000 °C
- Number density is also stable

Evidence of nanoclusters in ANC 0.4Ti-0.25Y_2O_3



- Y-Ti-O rich clusters are found in this sample
- Less and may be larger clusters compared to ferritic ODS steels
- Nitrogen is homogenously distributed
- Clusters were only found in few tips > inhomogenously distributed

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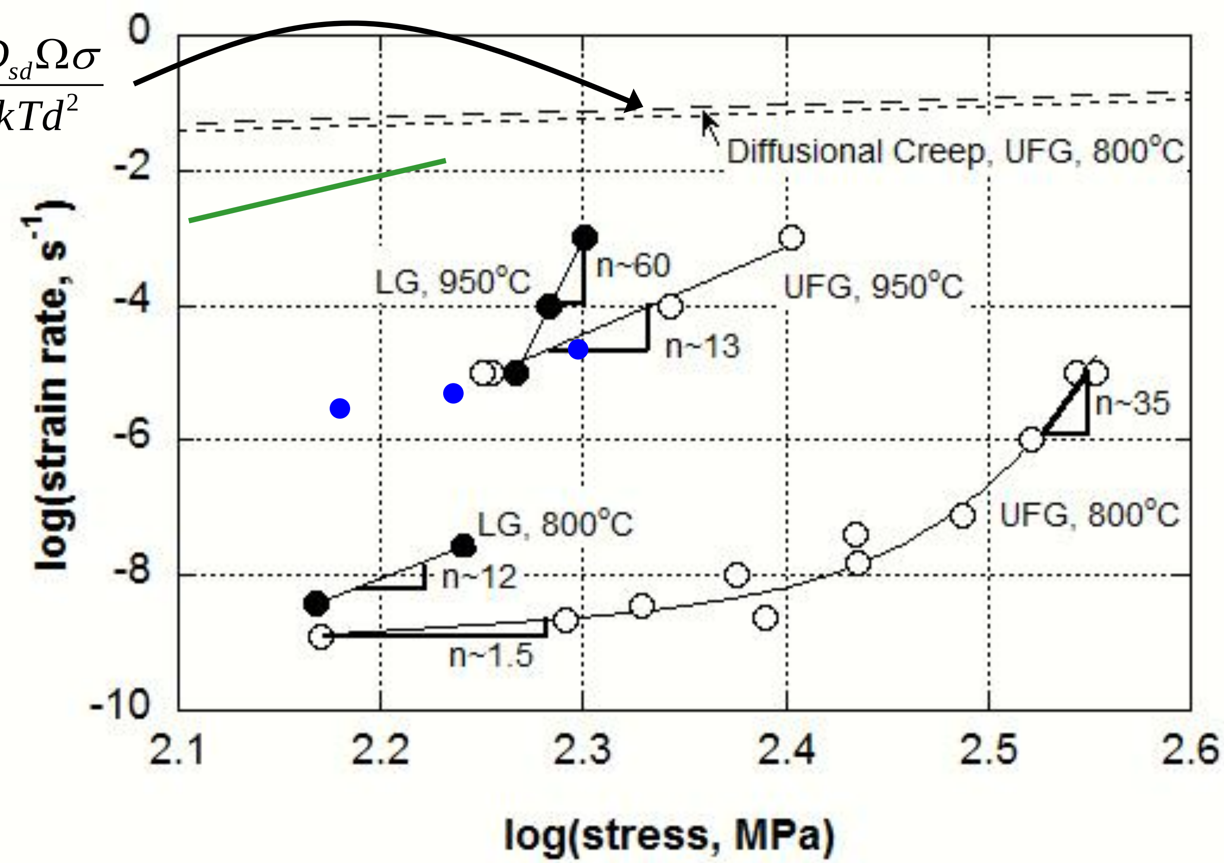
Consequences on mechanical properties – Creep behaviour

$$Nabarro-Herring\ Creep: \dot{\varepsilon} \sim 40 \frac{D_{sd}\Omega\sigma}{kTd^2}$$

$$Coble\ Creep: \dot{\varepsilon} \sim 47 \frac{\delta D_{gb}\Omega\sigma}{kTd^3}$$

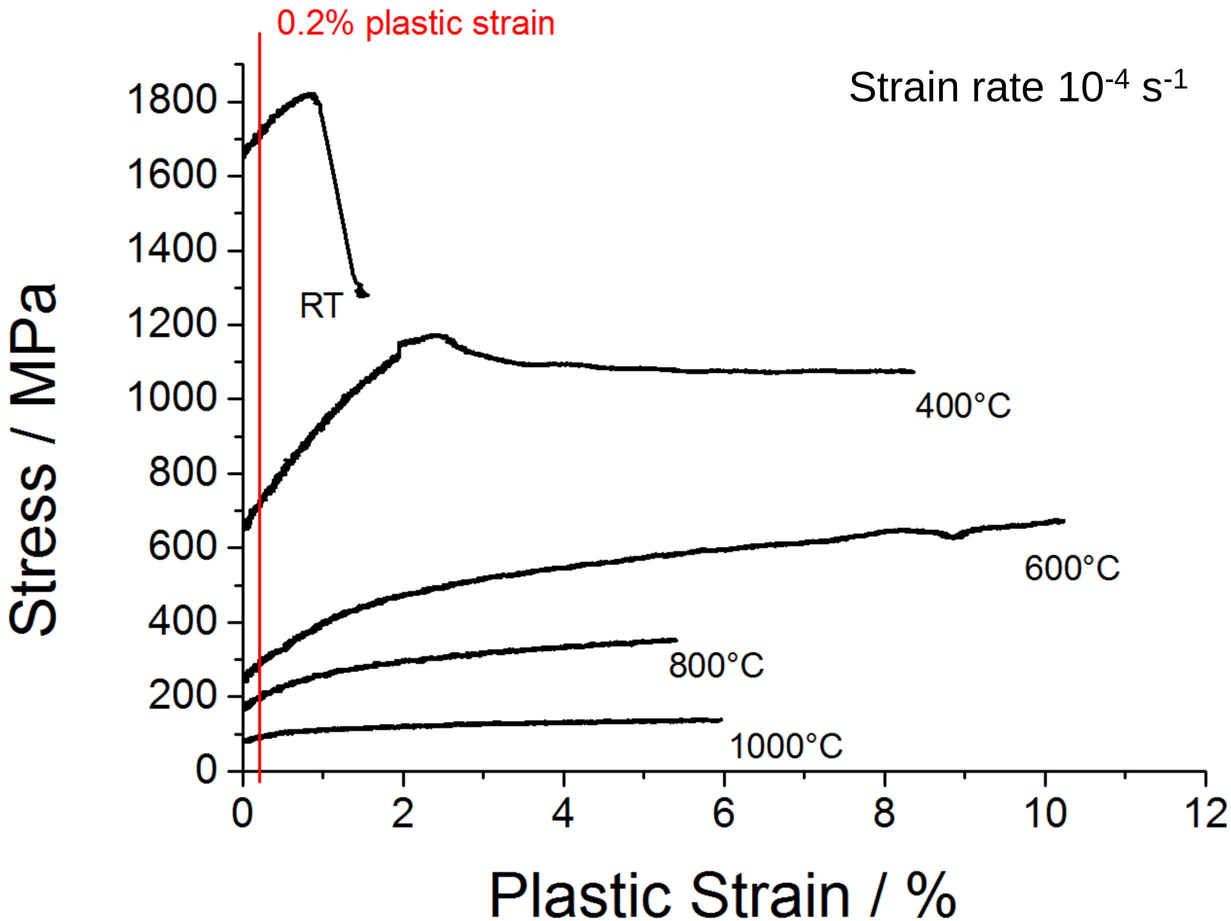


- ○ 14YWT UFG
- ● 14YWT LG
- ● NC 0.4Ti-0.25Y_2O_3 (this work)
- — ODS steel without Ti at 950°C (Susila et al., MSE A528, 2011)

➢ Unusual high creep resistance of NC 0.4Ti-0.25Y_2O_3

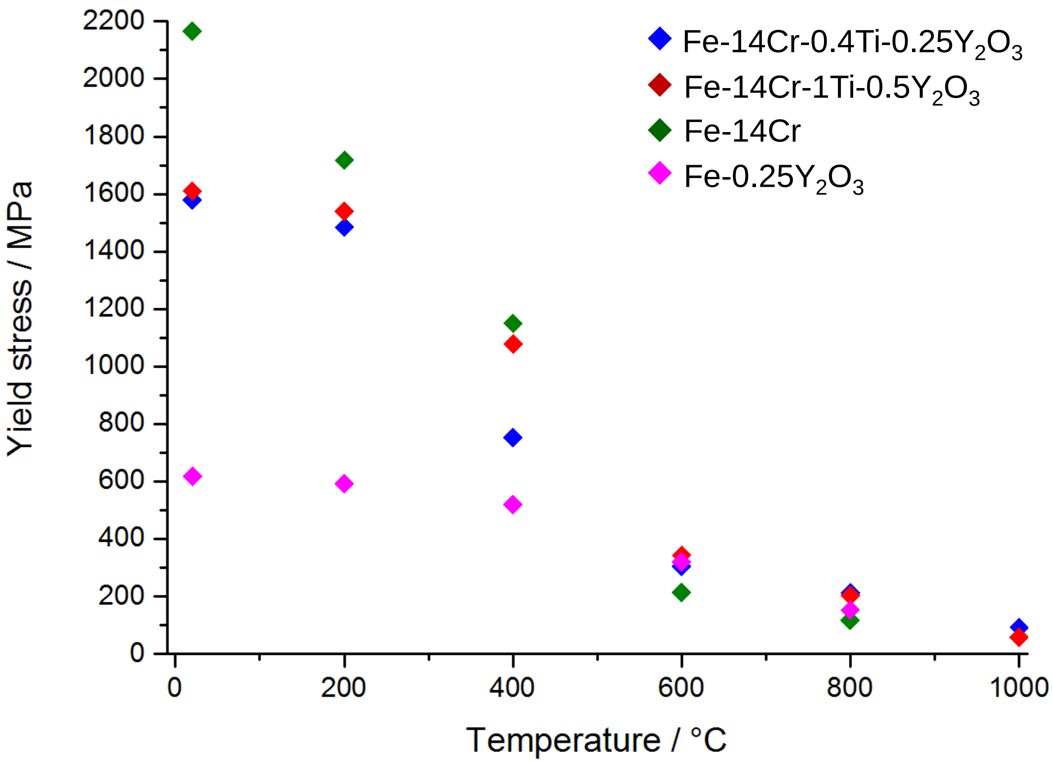
Schneibel et al., Script. Mater. (2009)

Consequences on mechanical properties – Stress-strain curves for NC 0.4Ti-0.25Y_2O_3



- Decreasing yield stress with increasing temperature
- Largest difference between 400 and 600 °C

Consequences on mechanical properties – Temperature dependence of yield stress



- ODS steels compared to base alloys
- Strong decrease of yield stress in steel without dispersion strengthening
- Low yield stress without solid solution hardening

Summary

Ferritic and austenitic ODS steels

- Same initial fine grain size (around 0.5 µm)
- Grain size of austenite stable up to higher temperatures
- Clusters supposed to be responsible for that

Cluster analysis

- Ferritic ODS steels
  - High number density of clusters in ferritic ODS steels
  - Stable cluster size of Y-Ti-O clusters at 1000 °C up to 1000 h
- Austenic ODS steels
  - Less but larger clusters than in ferritic counterpart in the initial state

Resulting mechanical properties of ferritic ODS steels

- Extraordinary creep resistance
- High yield stress up to 600 °C

Outlook

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Ferritic ODS steels

- Searching for reasons of yield stress breakdown and creep behaviour
- Cluster evolution at higher temperatures during annealing

Austenitic ODS steels

- Detailed tests of mechanical properties at high temperatures
- Cluster evolution at higher temperatures during annealing

Thank you for your attention!