# Stability of Structures in Nanocomposited Ferritics (NCF)

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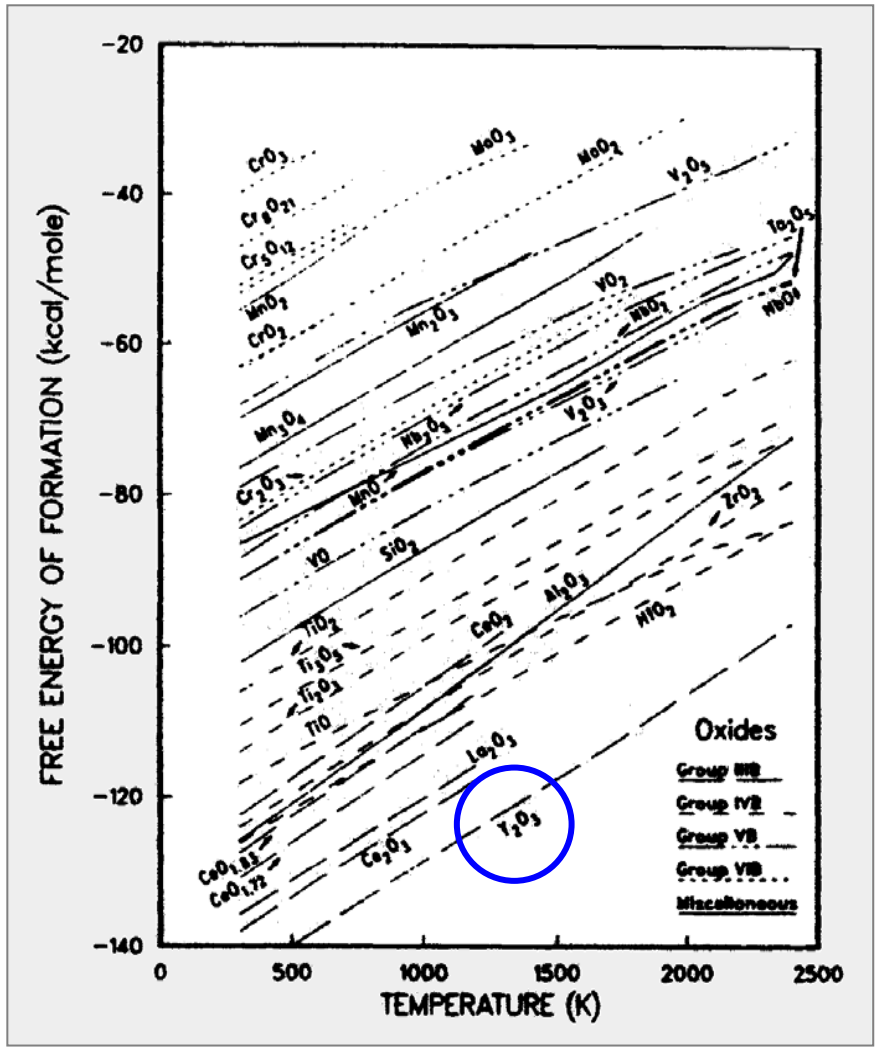
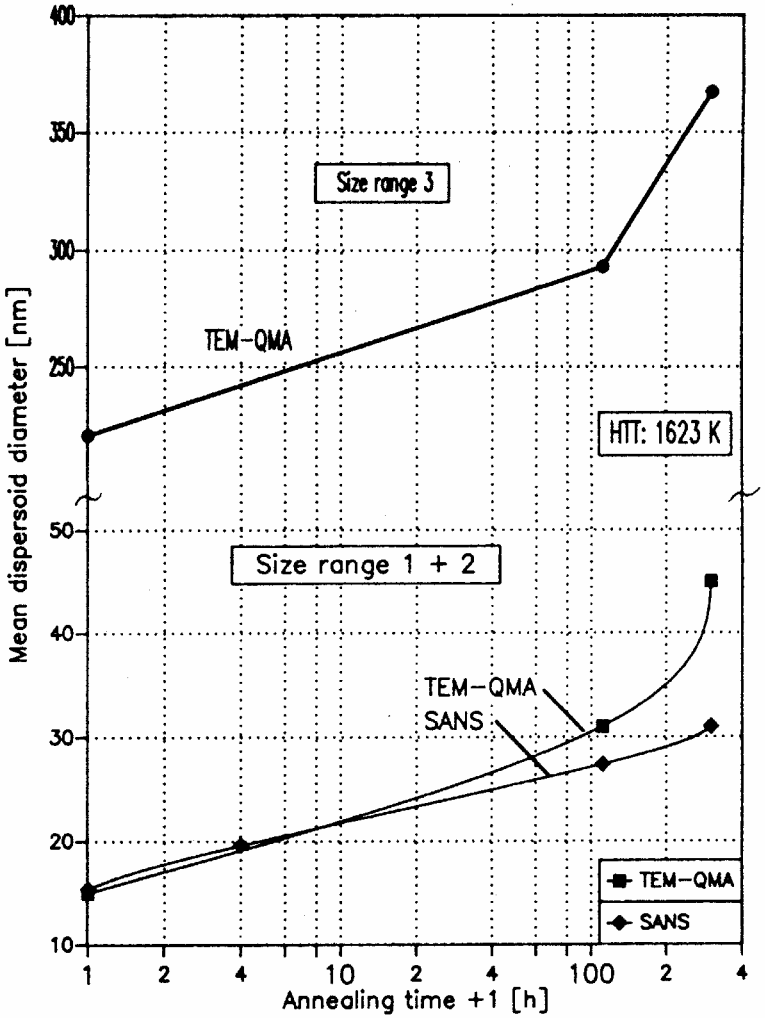
# High Temperature Stability of Oxide Phases

- The free energy of formation of oxides is typically larger than that of most carbides, nitrides, and sulfides
- Yttrium-oxide ($Y_2O_3$) is one of the most thermodynamically stable phases
- A dispersion of oxide particles improves the high temperature strength of an alloy (ODS) by acting as stable, hard obstacles to mobile dislocations and grain boundaries



# Dispersed Oxides in an Alloy Undergo Ostwald Ripening at Elevated Temperatures

P. Krautwasser et. al (1994)

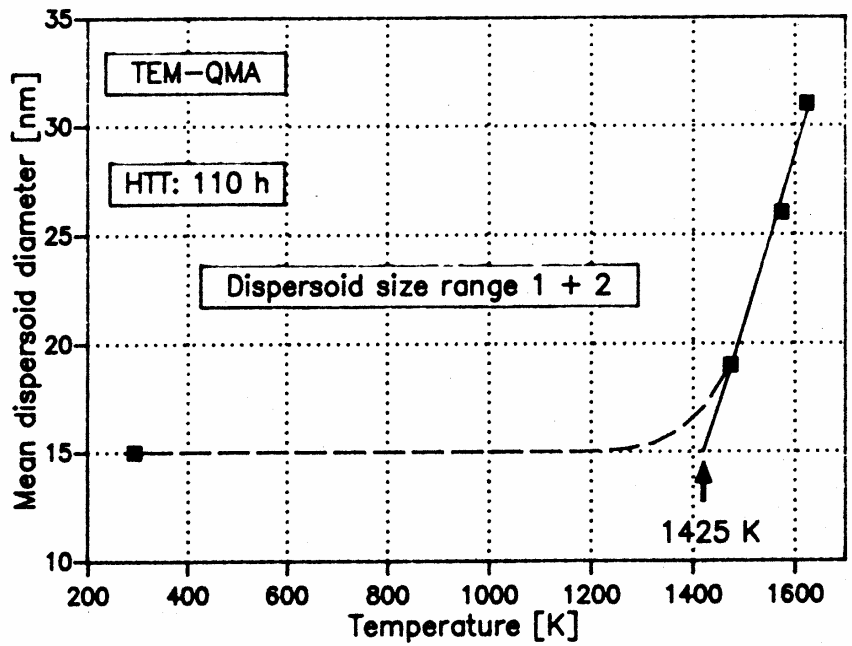



- ODS Ferritic Alloy (PM2010)
  - MA Fe-20Cr-5.5Al-0.5Ti + 1.0$Y_2O_3$
  - dispersion of Y-Al oxides
- Coarseing did not obey Lifshitz-Slyozov-Wagner (LSW) theory
  - could not discern between interface or volume diffusion controlled coarsening
  - structural transitions at high temperatures

    Perovskite (YAP) to Garnet (YAG)

# Coarsening Behavior of Y-Al Oxides in ODS Ferritic Alloy (PM2010)

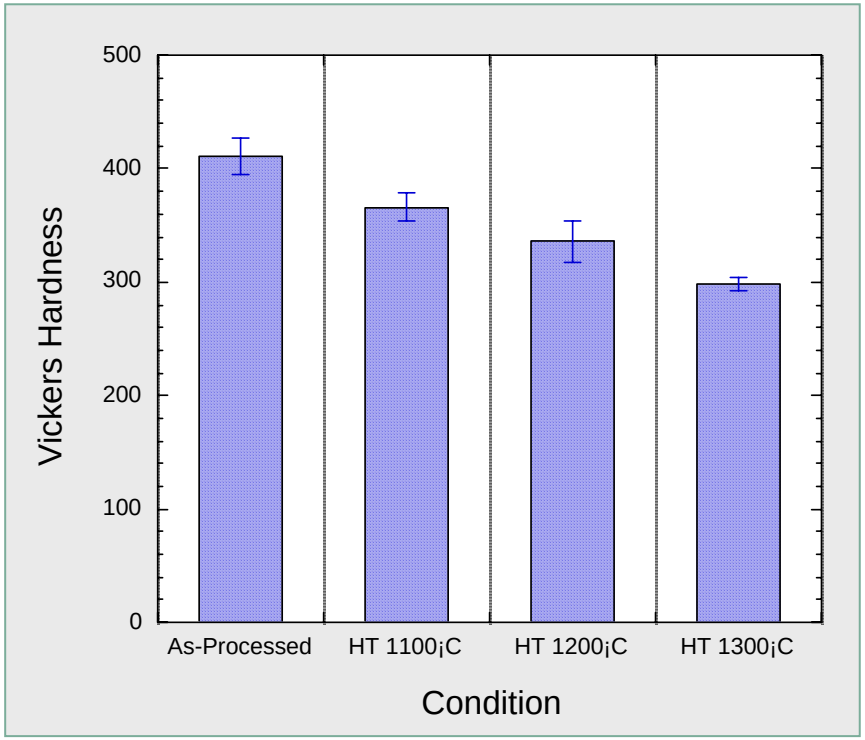
MA Fe-20Cr-5.5Al-0.5Ti + 1.0$Y_2O_3$




- The coarsening rate of the YAG/YAP particles does not change below a temperature of ~1150°C in the PM2010 alloy
  - low solubility and diffusion rate of Y in Fe lattice

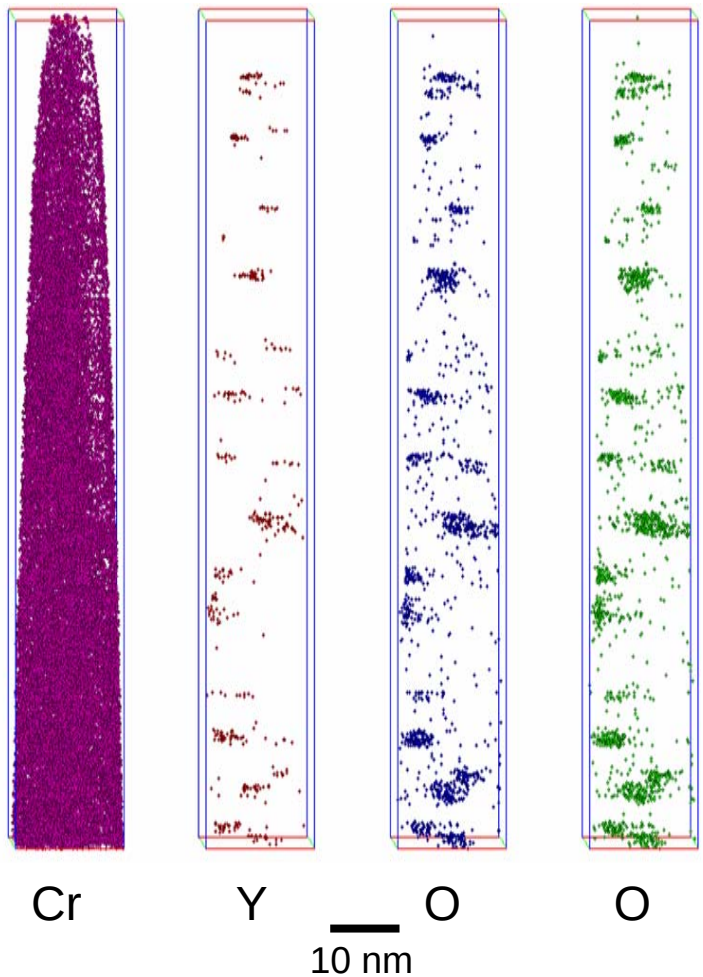
P. Krautwasser et. al (1994)

# Coarsening and Structural Stability of the Nanoclusters was Investigated at Very High Temperatures

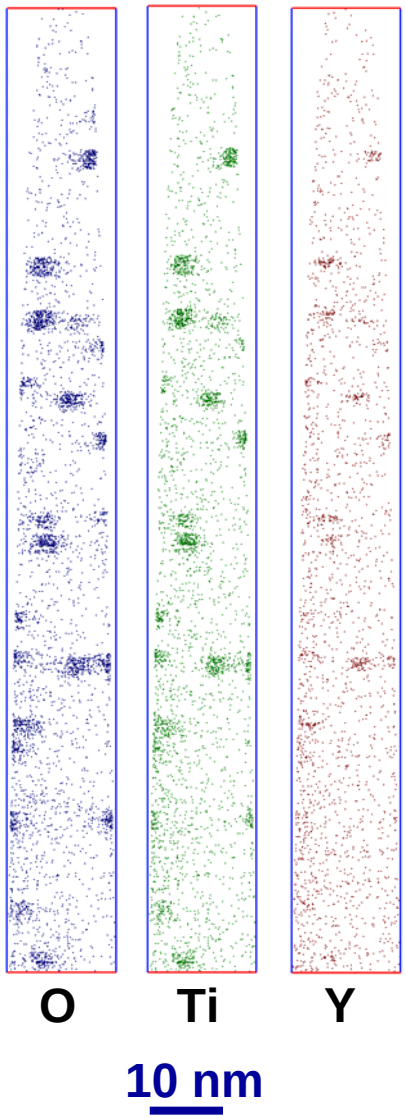



- Softening is not significant
- ~50% of the microstructure has recrystallized after annealing for 1h at ~86% $T_{MP}$(~1520°C)

MA 12YWT Alloy
As-Processed

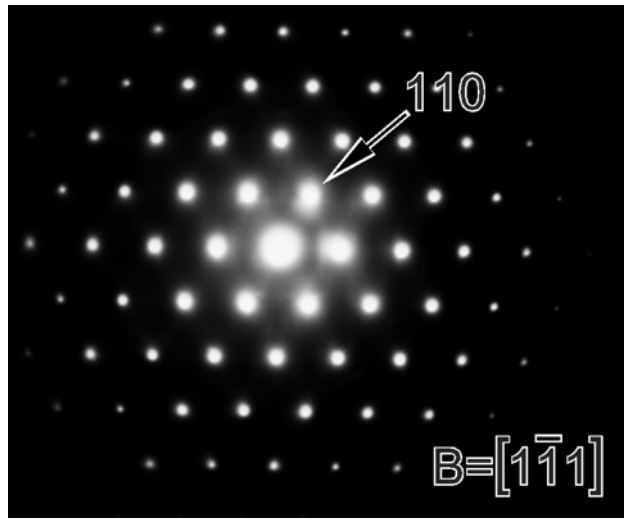


# 3-D APT indicates nanoclusters are still present after 1 h at 1300°C




- No significant changes in size, composition, and number density
- Similar results have also been obtained after 10 h at 1300°C

- SAD patterns in TEM only show reflections from bcc $\alpha$-Fe matrix
- Some coarser oxides particles observed ($TiO_2$ and $Y_2Ti_2O_7$)



# Composition of Nanoclusters Analyzed by APT for Several Conditions

MA Fe-12Cr-3W-0.4Ti + $0.25Y_2O_3$

| Condition | Cr | W | Y | Ti | O | M:O |
|---|---|---|---|---|---|---|
| 12YW AP | 14.5 ± 5.5 | 0.19 ± 0.19 | 21.2 ± 9.4 | 1.41 ± 1.5 | 11.4 ± 10.7 | 2.98 ± 1.4 |
| 12YWT AP | 7.0 ± 4.3 | 0.21 ± 0.21 | 9.2 ± 7.8 | 19.9 ± 8.7 | 23.6 ± 10.6 | 1.27 ± 0.3 |
| 1 h 1300°C | 7.9 ± 3.8 | 0.57 ± 0.53 | 5.6 ± 7.8 | 14.5 ± 8.7 | 20.5 ± 14.0 | 1.00 ± 0.3 |
| 10 h 1300°C | 6.5 ± 4.5 | 0.73 ± 1.5 | 9.9 ± 9.2 | 13.5 ± 7.1 | 21.2 ± 10.5 | 1.09 ± 0.3 |
| Crept 650°C | 7.8 ± 4.5 | 0.12 ± 0.12 | 7.6 ± 7.3 | 21.4 ± 8.9 | 23.8 ± 8.1 | 1.24 ± 0.4 |
| Crept 800°C | 5.6 ± 3.9 | 0.33 ± 0.33 | 7.9 ± 4.9 | 24.6 ± 8.5 | 28.1 ± 9.8 | 1.19 ± 0.2 |
| Crept 850°C | 7.2 ± 4.7 | 0.45 ± 0.98 | 10.9 ± 9.4 | 17.2 ±8.2 | 21.7 ± 10.8 | 1.42 ± 0.6 |
| Crept 850°C | 7.8 ± 4.1 | 0.33 ± 0.29 | 5.7 ± 3.0 | 23.6 ± 7.5 | 24.6 ± 8.1 | 1.21 ± 0.1 |

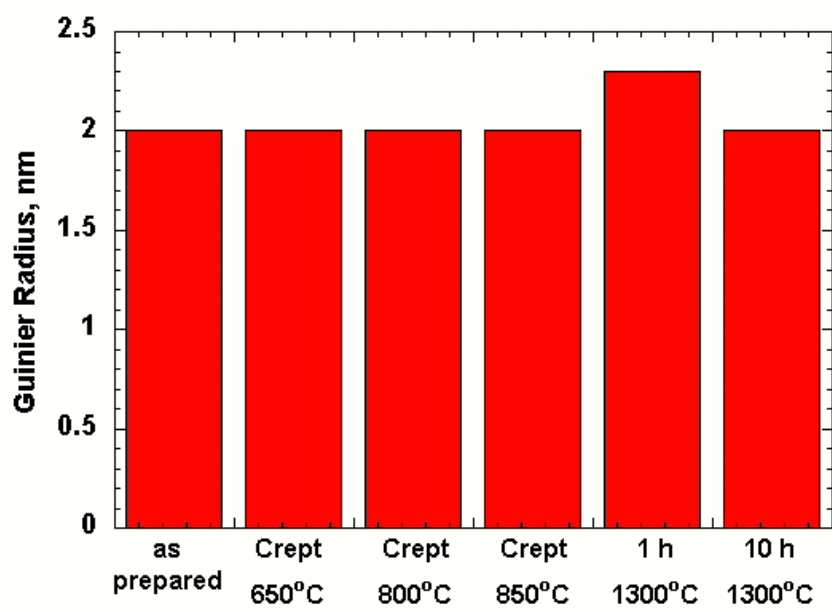
**Error is standard deviation of measurements**

Balance Fe

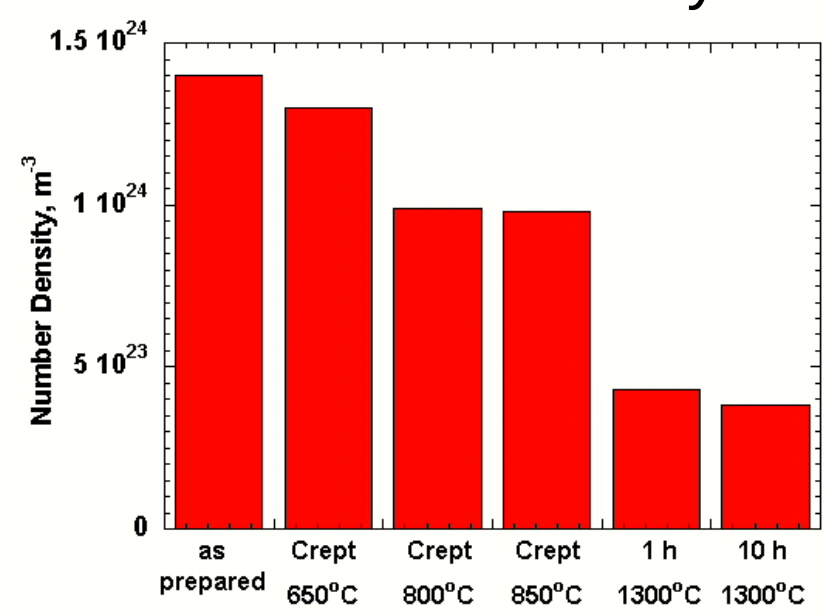
- The composition of each nanoscale particle was estimated with the envelope method

# Changes in Size and Number Density of Nanoclusters During High Temperature Exposures

## Size




## Number Density




14,230h at 138MPa

***Determined from $N_v = N_p / v$***

# Key Research Issue

- During neutron irradiation, several processes occur that can lead to instabilities of the oxide or nc dispersion
  - thermally driven ostwald coarsening process
  - ballistic effects that disrupt structural lattices and redistribute solutes
  - transport processes such as segregation caused by solute-point defect interactions and sink interactions
- Only a limited amount of information is currently available concerning the stability of oxide particles in ODS ferritic steels during neutron irradiation?
  - FFTF/MOTA experiment using INCO MA957 and MA956
  - JOYO experiment on two experimental ODS ferritic alloys
- Neutron irradiation experiments have not been performed on the nanocluster dispersion strengthened (NCDS) 12YWT alloy. Only one study using 3.2 MeV fe+ ion irradiation with simultaneous He injection has been performed (Kim et al., JNM, 2000)

# Neutron Irradiation Studies on ODS Steels

- FFTF/MOTA irradiation test …….(D.S. Gelles, JNM, 1996)
  - INCO MA957 and MA956 alloys
  - ~420°C to a dose of 200 dpa (peak swelling range in ferritic alloy class)

| Alloy | Composition (wt.%) | Swelling (% density change) | Highest Swelling (%) | Mean Void Size (nm) | Void Density ($^{\#}/cm^3$) |
|---|---|---|---|---|---|
| T91 | Fe-8.43Cr-0.89Mo-0.24V-0.37Mn | 1.76 | 5.0 | 21.4 | $4.8 \times 10^{15}$ |
| MA956 | Fe-19.1Cr-4.20Al-0.34Ti + 0.48$Y_2O_3$ | 1.17 | 0.21 | 26.7 | $1.9 \times 10^{14}$ |
| MA957 | Fe-13.8Cr-0.26Mo-0.90Ti + 0.26$Y_2O_3$ | 1.75 | 3.73 | 6.1 | $9.2 \times 10^{14}$ |
| MA957 | Fe-13.1Cr-0.27Mo-0.95Ti + 0.25$Y_2O_3$ | 0.25 | 3.5 | 35.4 | $8.6 \times 10^{14}$ |

- MOTA irradiation test….(M.L. Hamilton, D.S. Gelles, et al., PNNL, 2000)
  - INCO MA957
  - 370 to 750°C to doses in the range from 8.5 to 38.5 dpa
  - *The $Y_2O_3$ dispersoid was unaffected by neutron irradiation up to 750°C and 38.5 dpa*

    **(2 nm size** $Y_2O_3$ dispersoid was observed in the unirradiated MA957)

# Neutron Irradiation Study on ODS Steels in Japan

- JOYO irradiation test…….(S. Yamashita, K. Oda, S.Ohnuki, N. Akasaka, and S. Ukai, ICFRM-10, 2001)
  - Fe-11Cr-2.7W-0.4Ti-0.63$Y_2O_3$ & Fe-12.8Cr-2.8W-0.6Ti-0.34$Y_2O_3$
  - Temperatures: 450°C and 561°C
  - Doses: 10.5 and 21.0 dpa
- Reported the possibility of oxide ($Y_2O_3$ and complex Ti-Y oxide) dissolution induced by recoil resolution
  - stability of the oxide particles was more sensitive to dose that to temperature

# Key Issues

- Limited data exists, but studies based on neutron, ion, and electron irradiation indicate that oxide particles are stable over large ranges of temperatures and doses
  - ODS ferritic steels
  - ~7 nm size $Al_2O_3$ oxides very stable in ODS GlidCop at 450°C to ~200dpa
  - need further investigations of oxide dispersion stability at higher irradiation temperatures

- We do not understand the mechanism for the nanocluster stability in a thermal environment, but conducting neutron and ion irradiation studies would be beneficial
  - cascade damage could disrupt the mechanism for their stability, with subsequent transofmri