Processing ODS steels and microstructure characterization

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Outline

- Background and objectives
- Study of Y_2O_3 evolution during processing by using powder diffraction techniques
- Manufacturing and microstructure characterisation of ODS
    - Primary route: powder metallurgy
        - ✓ Conventional: HIP
        - ✓ Novel: Spark plasma sintering (SPS)
    - Secondary approach: liquid metal processing – ongoing
- Conclusions & future work

Background and objectives

- Incomplete processing knowledge
  - How to identify the acceptable powder?
  - Can nanocluster behaviour be correlated with processing variables?
  - Limited commercial products for comparison

- Objectives:
  - Create own bespoke ODS alloys to support microstructural analysis, testing and model validation

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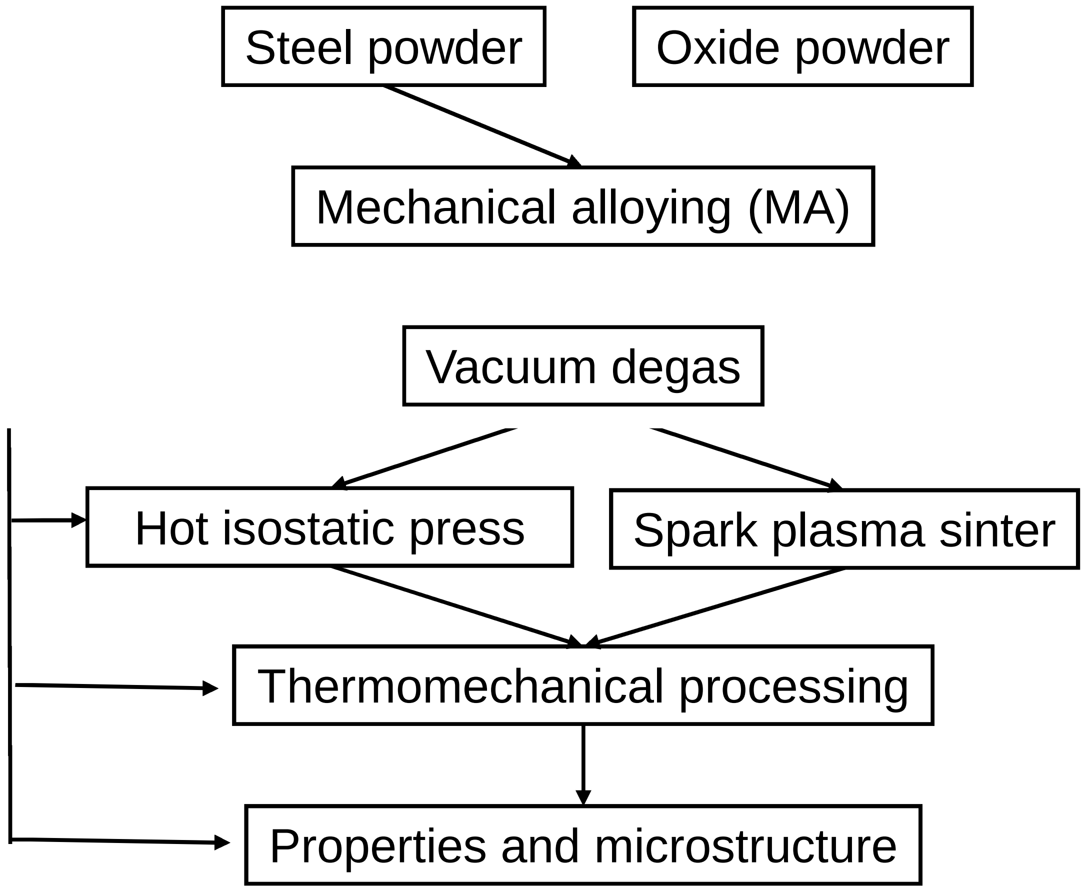
Powder metallurgy processing routes

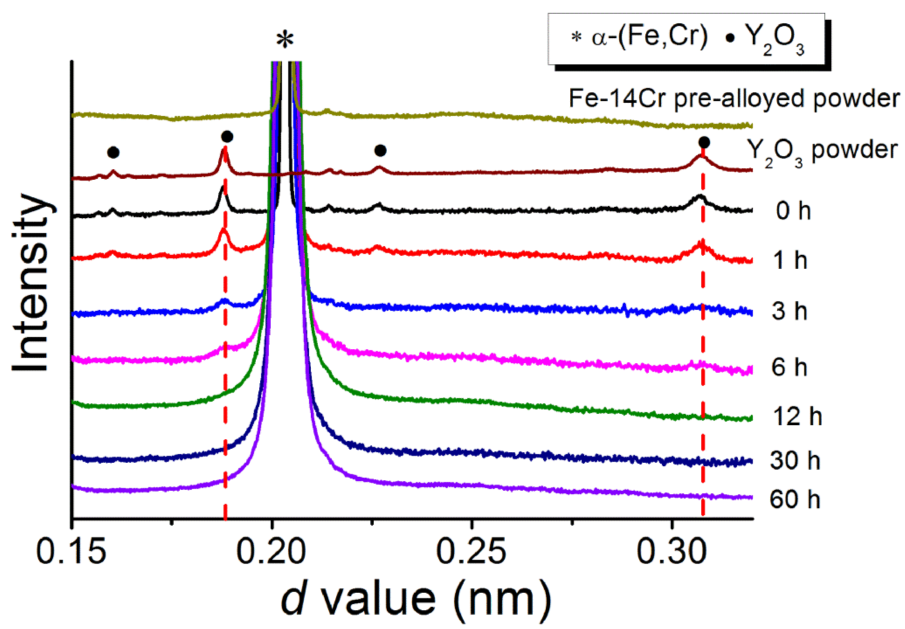


Study of Y_2O_3 evolution during processing

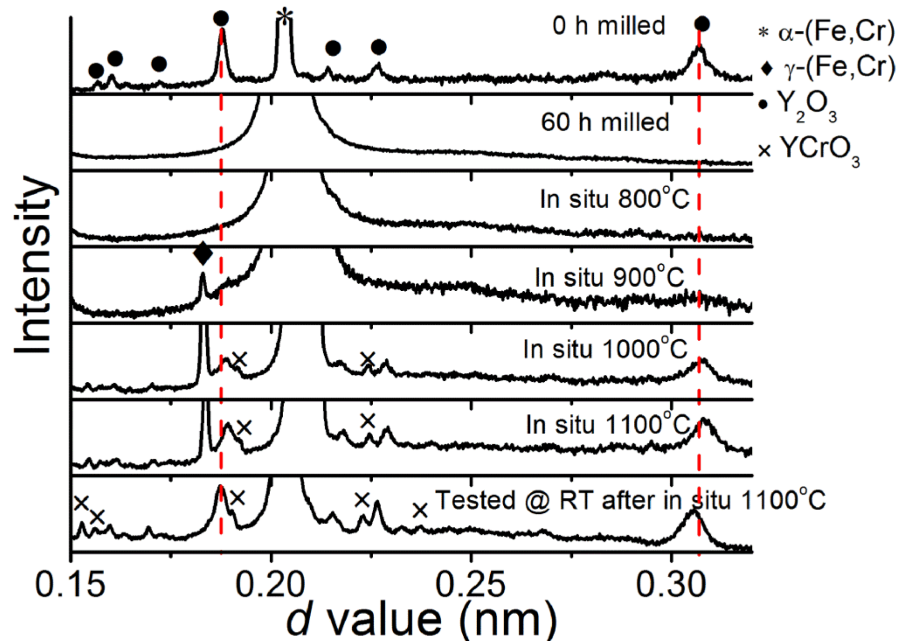
- Core process aspects to be understood
  - o Dissolution of Y_2O_3 during milling
  - o Re-precipitation as nanoclusters during hot working

- Techniques employed: high resolution powder diffraction (HRPD)
  - o In-house processing to know detailed process history
  - o Easy to prepare powder samples
  - o Penetration depth of neutrons (~25 mm) or synchrotron X-rays much greater than laboratory XRD (~5 μm)
  - o In-situ high temperature monitoring

Powder Neutron diffraction of Fe-14Cr-10Y_2O_3



Microstructure evolution as a function of MA time



Microstructure evolution during high temperature in situ annealing.

- 10% Y_2O_3 for confident detection
- Dissolution of Y_2O_3 during milling
- Re-precipitation of Y_2O_3 peaks during *in-situ* annealing > 900°C
- First real-time observation

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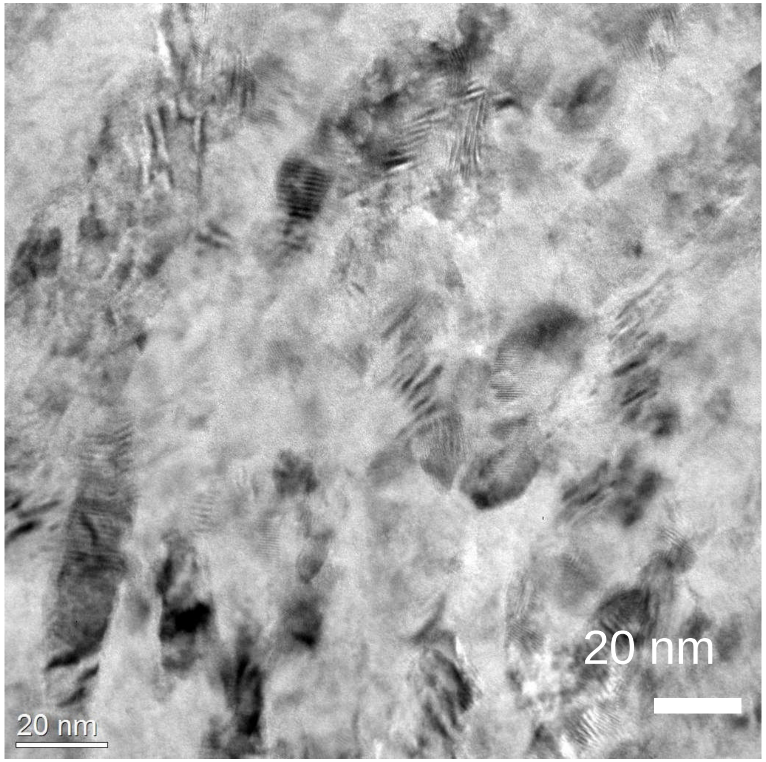
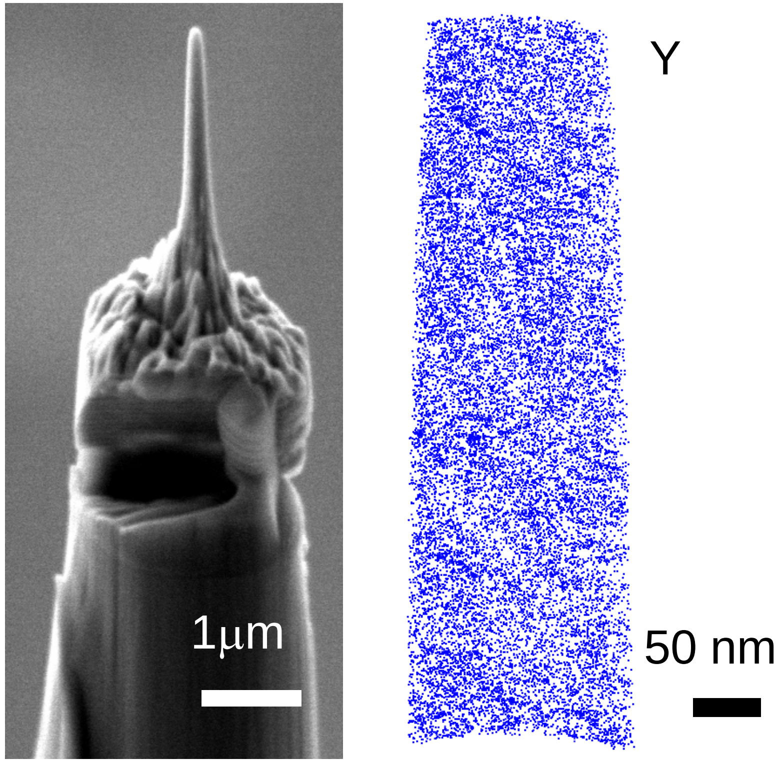
Nanoscale analysis of as-milled ODS powder

TEM of $Fe\text{-}14Cr\text{-}10Y_2O_3$

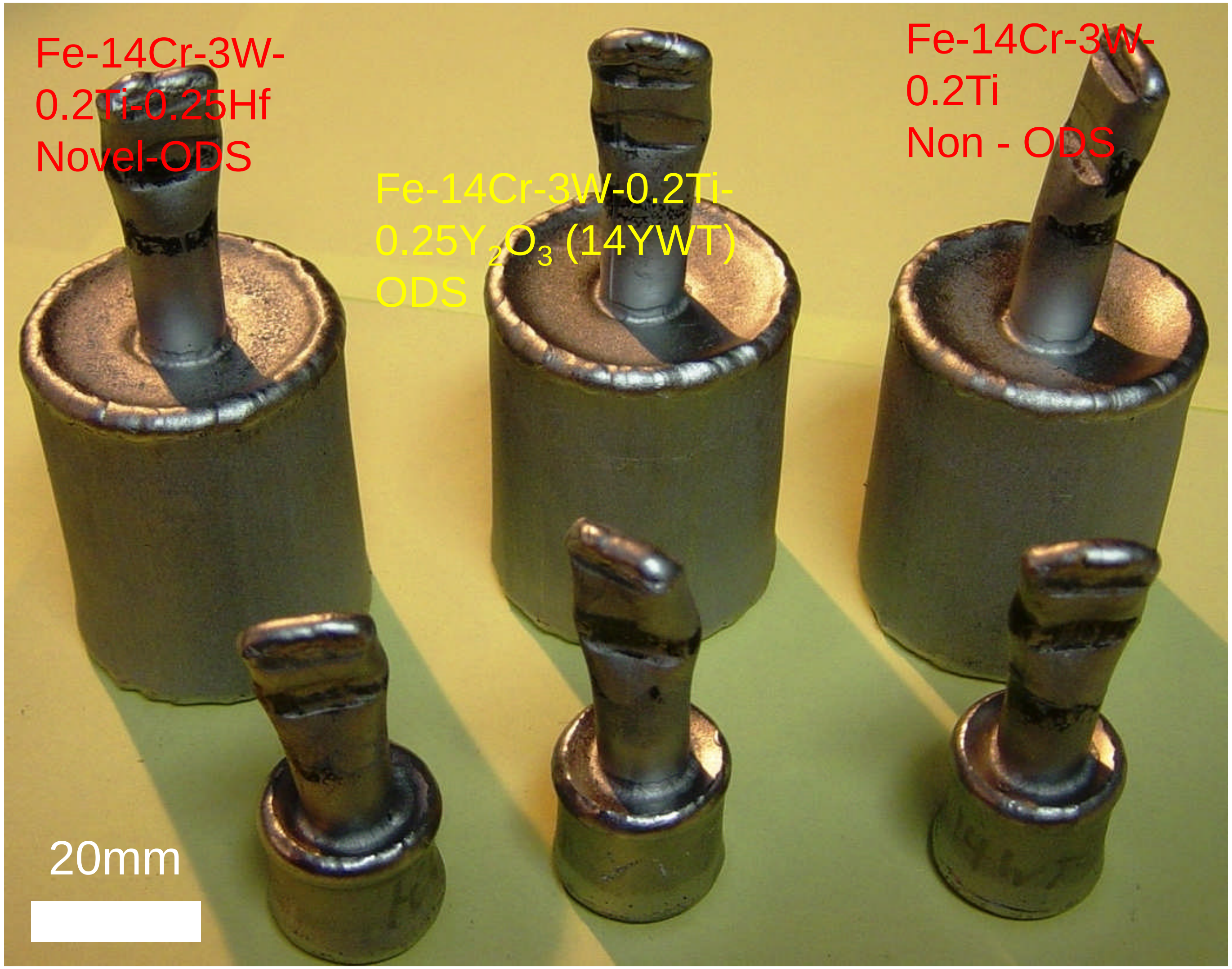


APT of $Fe\text{-}14Cr\text{-}0.25Y_2O_3$



- Y_2O_3 particles not convincingly resolved - dissolved
- High defect density due to work hardening during milling
- Homogeneous distribution of Y from APT, TEM & neutron diffraction

Consolidation of ODS by HIPing

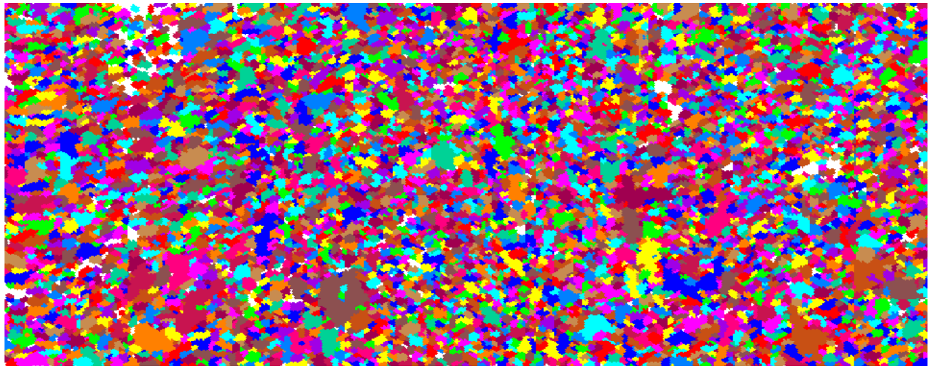


- HIP conditions: 1150°C x 150 MPa x 4 h

Microstructures of HIPed 14YWT

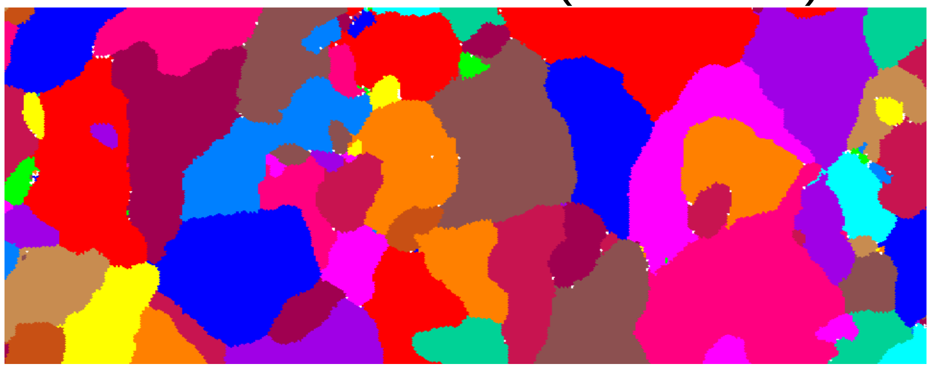
EBSD

Fe-14Cr-3W-0.2Ti-0.25Y_2O_3 (ODS)



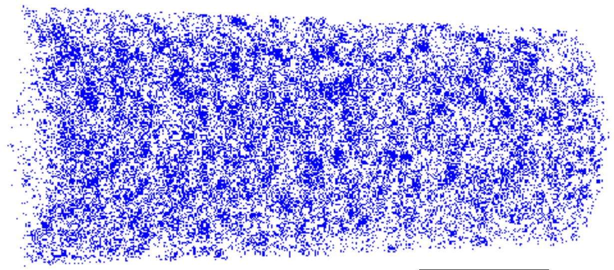
15 μm

Fe-14Cr-3W-0.2Ti (Non-ODS)



15 μm

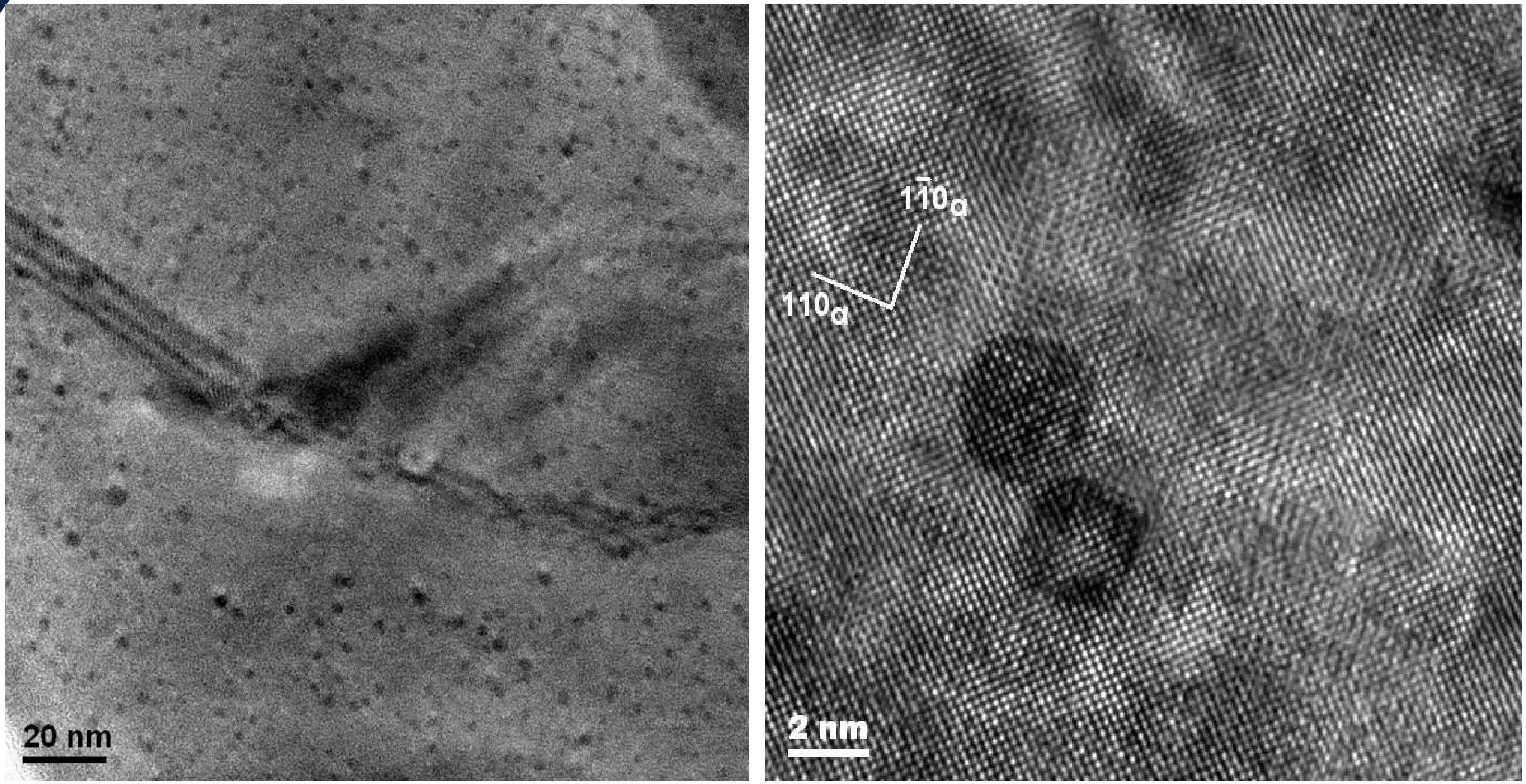
Atom probe tomography (APT)



50 nm

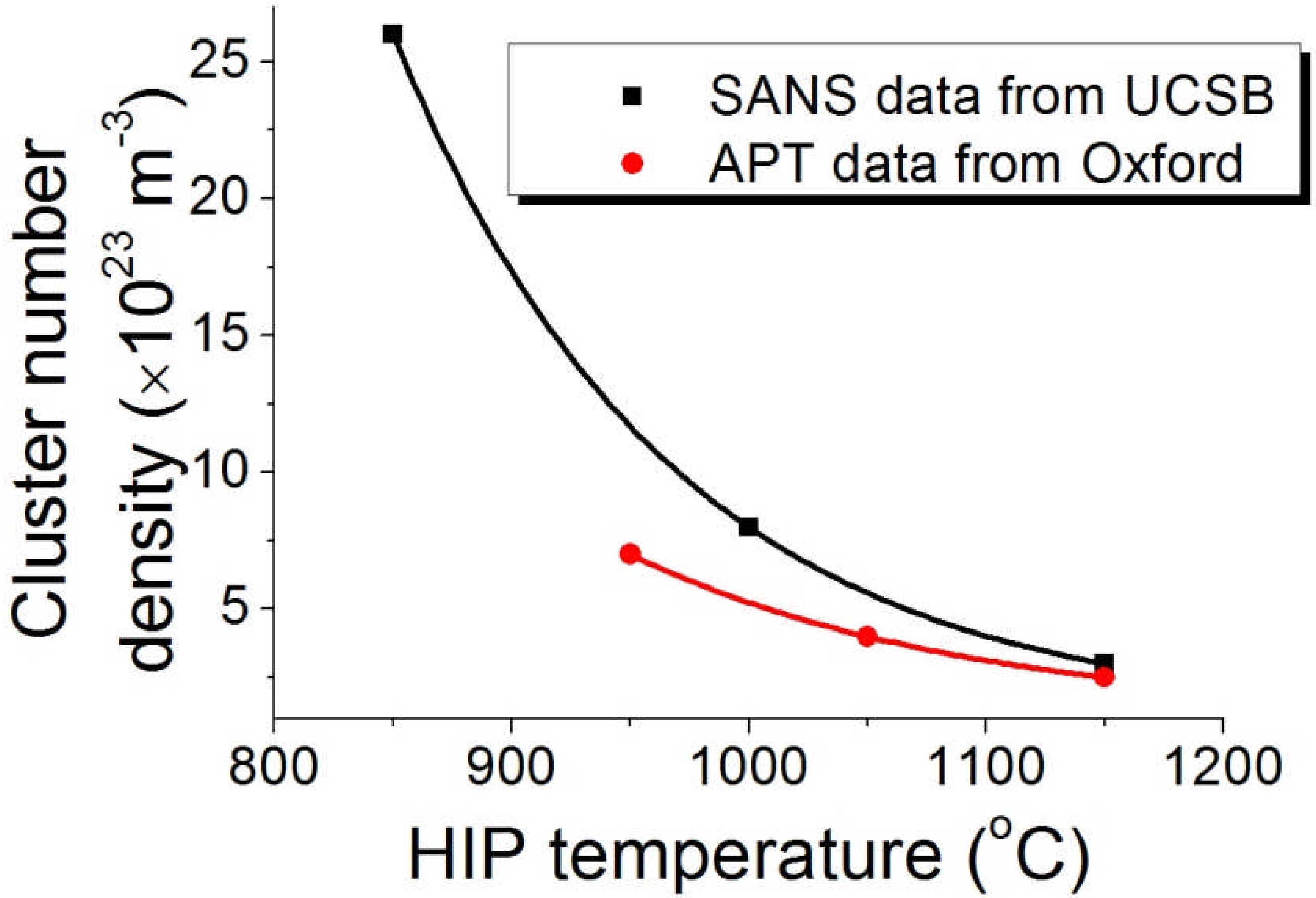
- Refined grain sizes due to addition of Y_2O_3
- Average nano-cluster size (1.9 nm) and density (2.4×10^{23} m^{-3}) are comparable to reported values from other leading groups in ODS processing.

Microstructures of HIPed 14YWT



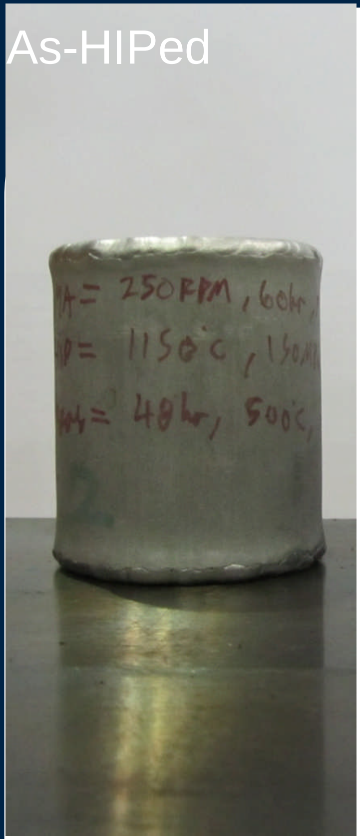
- Nano-clusters with uniform distribution and high number density

Effect of HIP temperature on nano-clusters



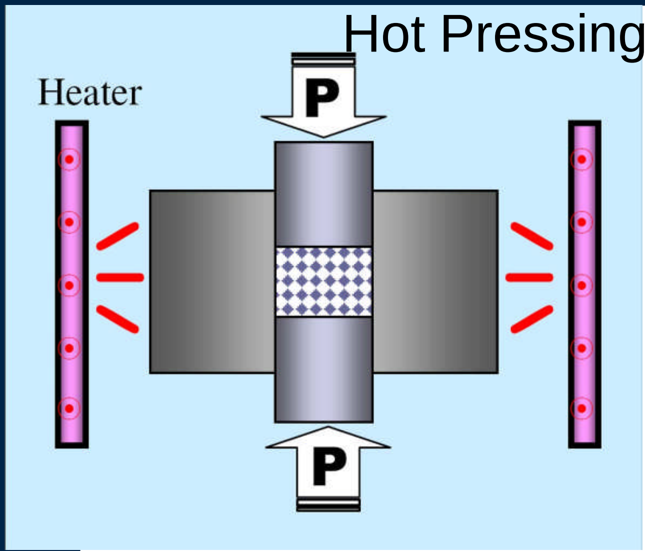
- With increasing HIP temperature, size of nano-clusters increases and their number density decreases.

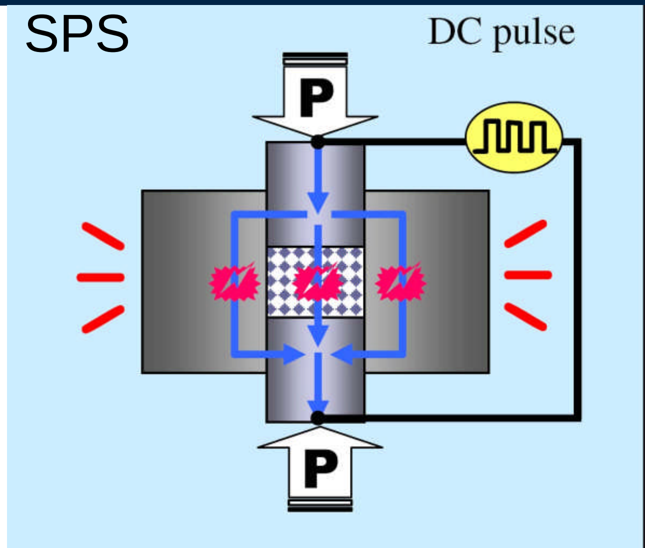
Thermomechanical treatment of ODS by Forging

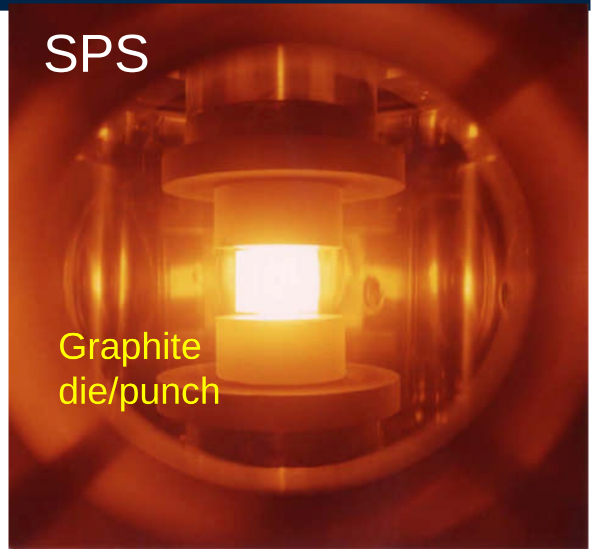


- Forging @ Adv. Forming Res. Cent. (AFRC), Strathclyde.
- Forging conditions: sample temp = 1150ºC, die temp = 300ºC, 35% reduction, unidirectional forging.
- Mechanical test being performed by KIT.

Spark Plasma Sintering (SPS)





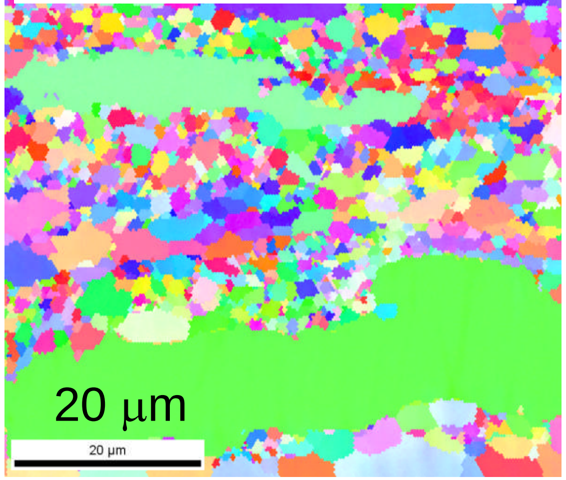


Advantages:

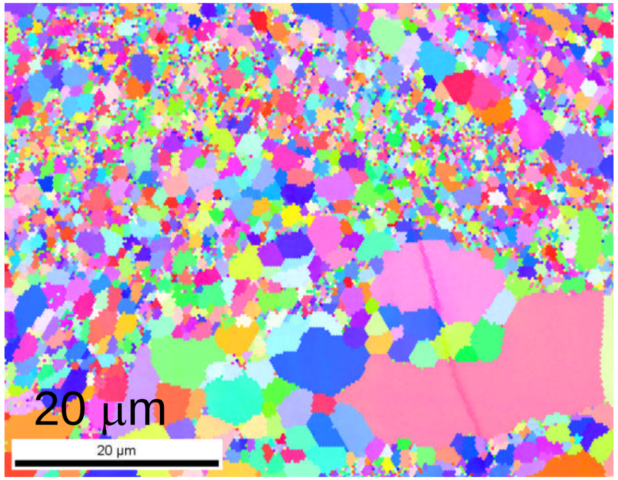
- Rapid heating rates (up to 1000K/min) – sintering in minutes
- Homogenous temperature distribution
- Preservation of nano- and metastable structures in bulk materials
- High throughput and low energy consumption
- Little work on ODS

Consolidation of ODS by SPS

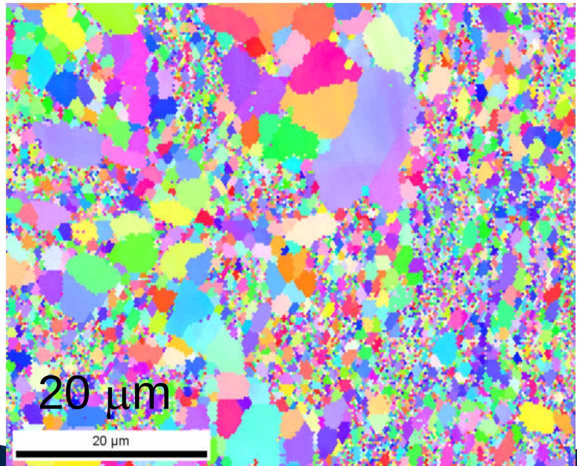
Fe-14Cr-0.25Y_2O_3



Fe-14Cr-0.4Ti-0.25Y_2O_3



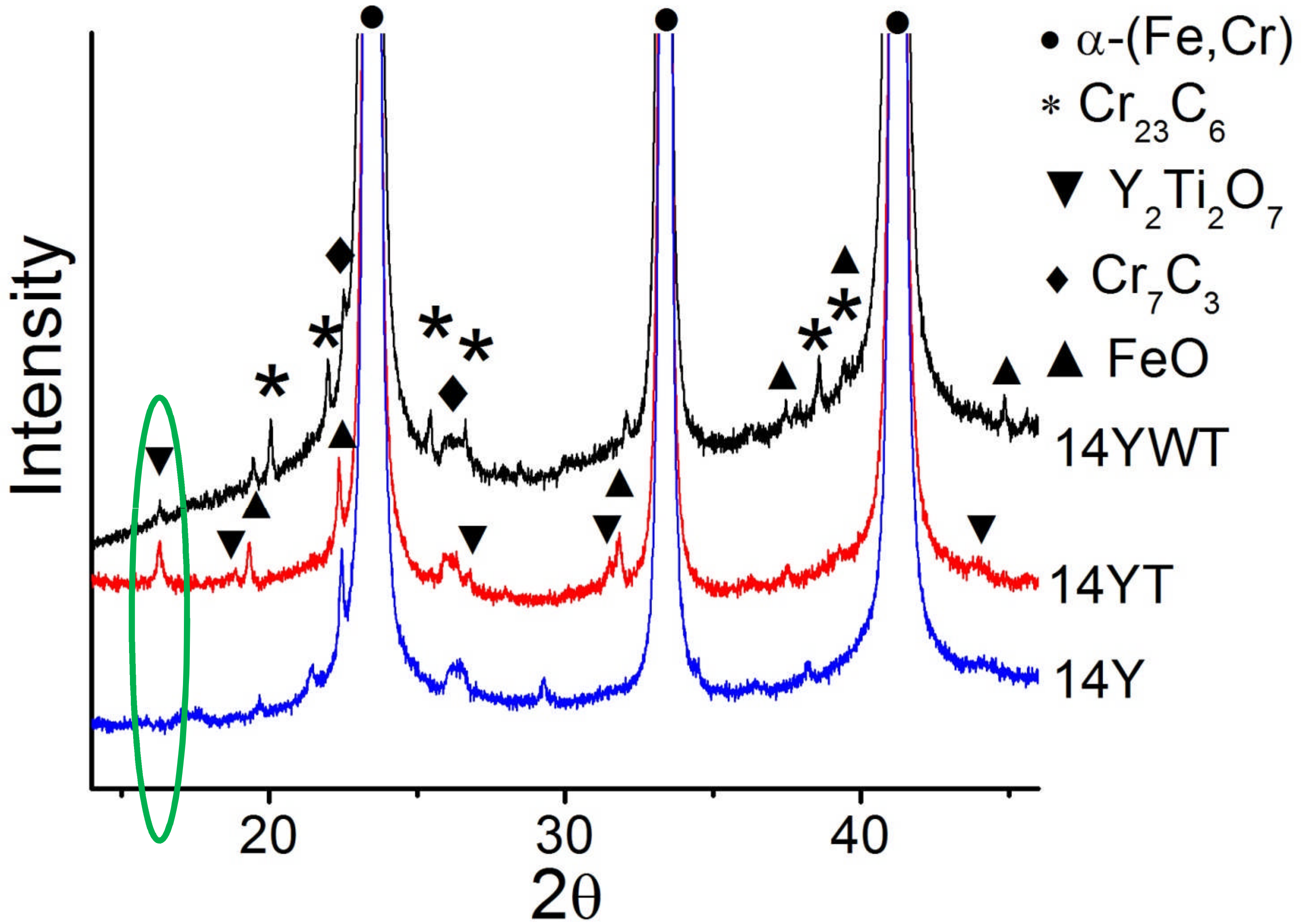
Fe-14Cr-3W-0.2Ti-0.25Y_2O_3



SPS vs. HIP

Force: 50MPa vs. 200MPa
Sintering duration: 5min vs. 3-4 hr
Density: 99.6%

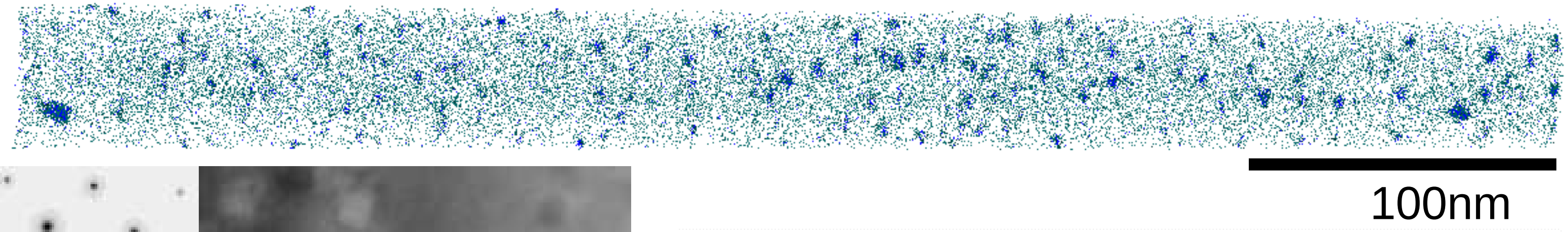
High resolution synchrotron XRD of SPSed ODS

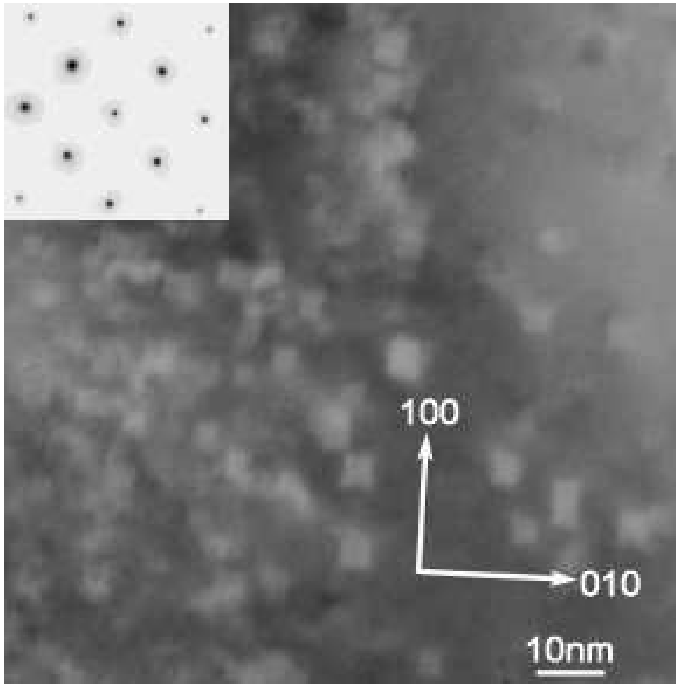


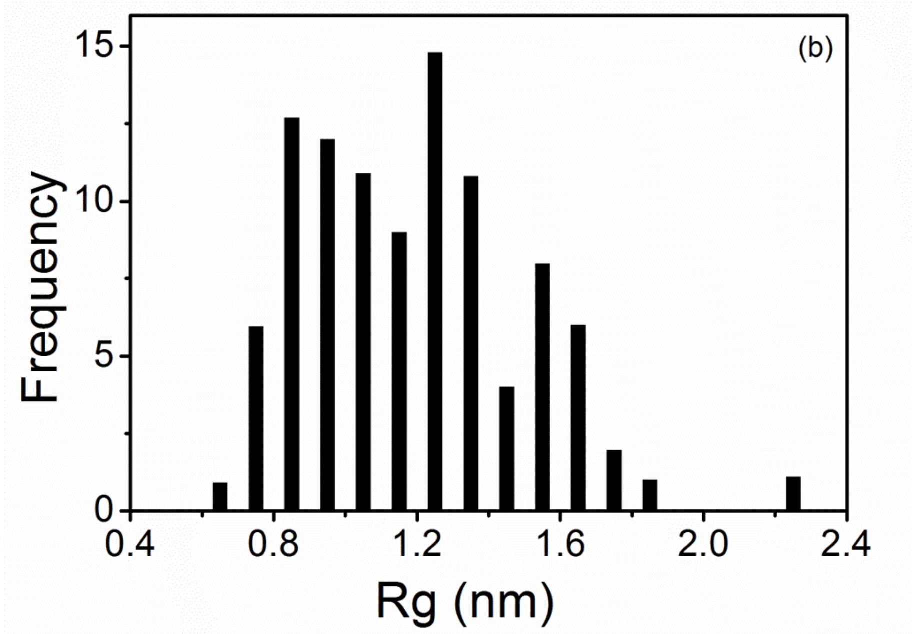
- Some evidence for $Y_2Ti_2O_7$ in Ti-contained ODS

Microstructures of SPSed ODS

Fe-14Cr-0.4Ti-0.25Y_2O_3

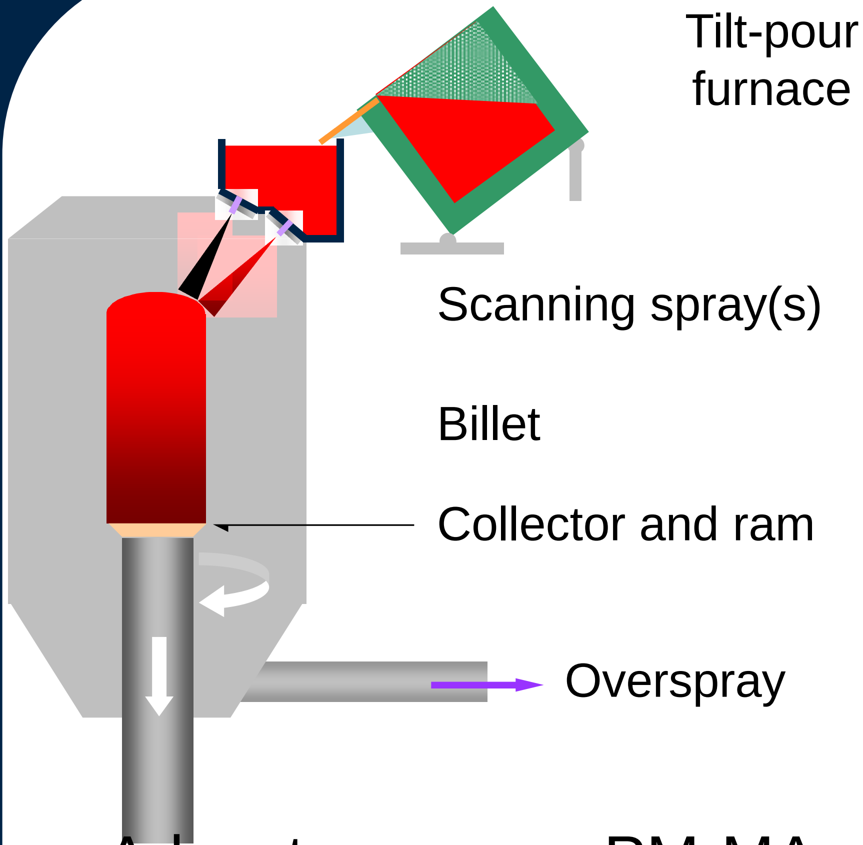


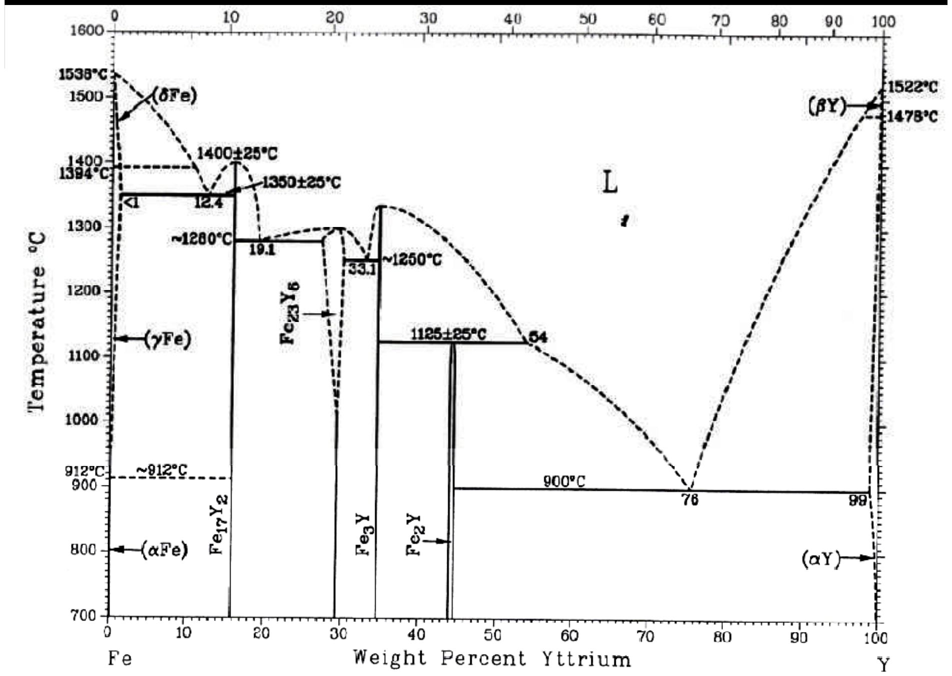




- Y-Ti-O nano-clusters with mean R_g=1.2nm and number density =2.7x10^{23} m^{-3}

Liquid metal processing of ODS - 1st route: spray forming





Advantages over PM-MA

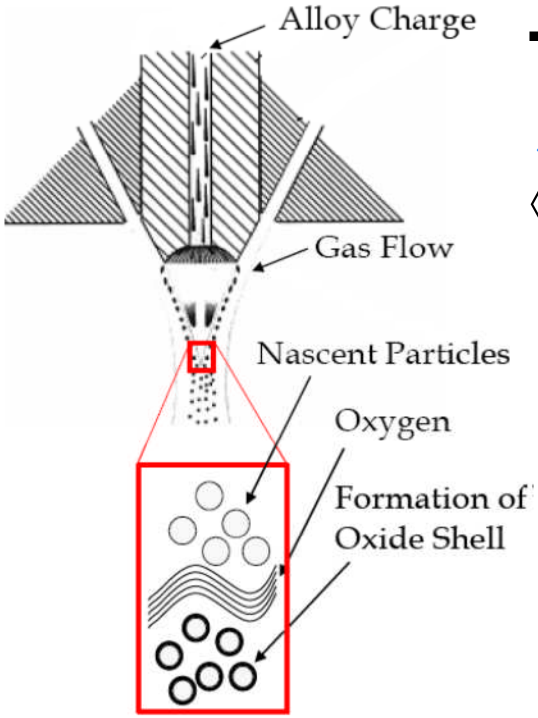
- Rapid solidification
- Very low oxide (<80 ppm)
- Production rate = 15 kg/min

Disadvantages over PM-MA

- Low yield (but same as PM?)
- Loss of Y
- How to introduce oxide?

Liquid metal processing of ODS - 1st route: spray forming

- Rationale
  - o Gas atomization reaction synthesis (Ar-O_2)
  - o In situ surface oxidation of the most kinetically favoured oxide phase (metastable Cr-enriched oxide)
  - o O exchange reaction during annealing to form Y-enriched oxide dispersoids

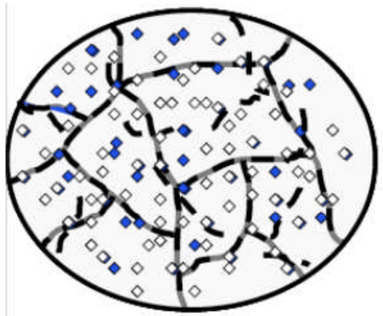


— Less stable oxides along the prior particle boundaries

♦ Oxygen

◊ Y-enriched oxides

Annealing



Ref: *J. Nucl. Mater*. 396 (2010) 86

ODS produced by spray forming

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ODS produced by spray forming

Challenges:

- Highly reactive of Y
- High temperature of 1700ºC
- High vacuum for big chamber
- Reaction between wash coat (Al_2O_3 and ZrO_2) and Y

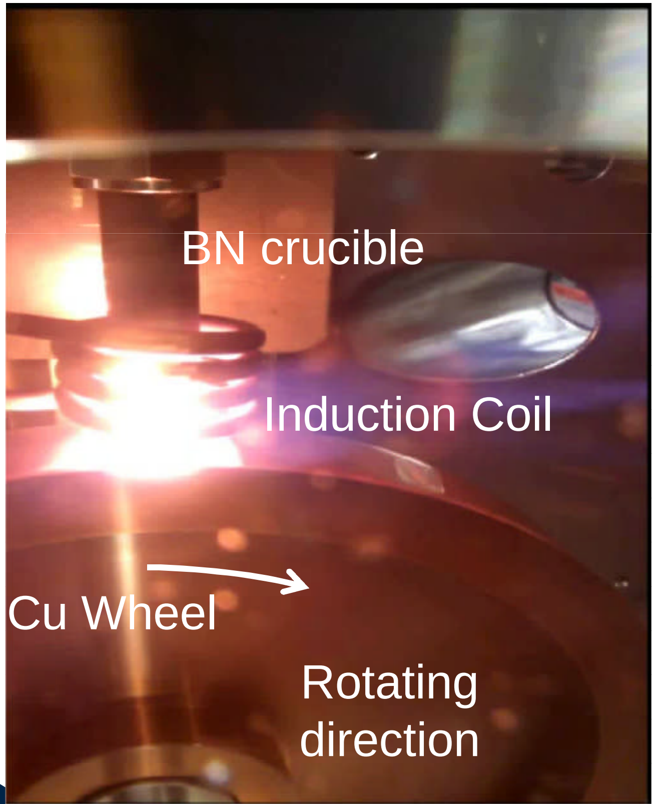
Processing condition:

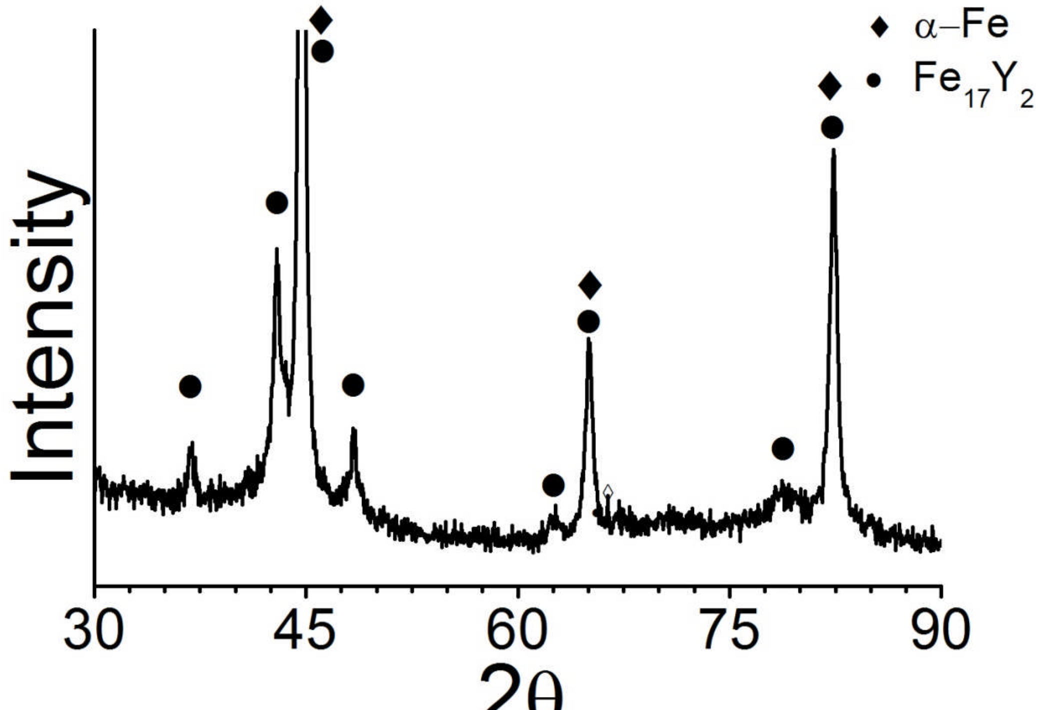
- 22kg Fe-17Cr + 300g Fe-75Y for target composition: Fe-17Cr-1Y
- Final composition: Fe-17Cr-0.1Y

Liquid metal processing of ODS - 2nd route: melt spinning + internal oxidation

Methodology

- Produce Fe-Y ribbons by melt spinning
- Seal Fe-Y ribbons with Fe-Fe_2O_3 powder mixture in evacuated quartz tube
- Ball milling of mixture of Fe-Y ribbons and Fe_2O_3





- No Y_2O_3 in as-spun ribbons

Summary

- Fundamental study on nano-precipitate formation mechanism
  - First time to observe re-precipitation behaviours of Y_2O_3 in real time.
- Establish in-house manufacture capability for high quality ODS
  - Better understanding of the effect of processing variables & compositions
  - Similar microstructures to reports from other leading groups.
  - Supply of bespoke alloys to whole group
  - SPS is a cost-effective technique for ODS consolidation.
  - Full processing route from in-house powder to forged and tested bespoke materials with reasonable large scale.
- Liquid metal processing routes are progressing.

Future work

- Mechanical testing at KIT
  - Dedicated vacuum tensile testing rig (RT-1200^{o}C)
  - Charpy testing (Liquid nitrogen to 1000^{o}C)
- Alternative ODS processing routes
  - Reactive ball mill
  - Spray forming
  - Melt spinning + internal oxidation
- More neutron and synchrotron X-ray beam time

Thank you for your attention