Investigation on different oxides as candidates for nano-sized ODS particles in reduced-activation ferritic (RAF) steels

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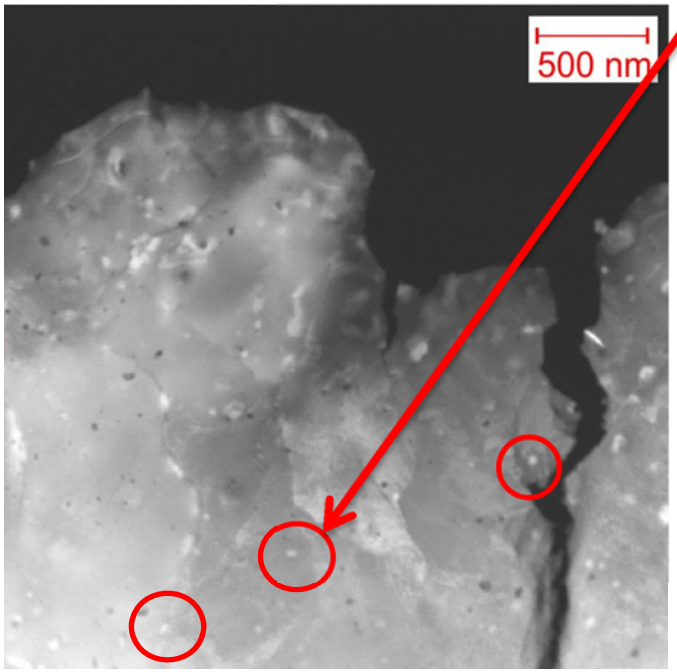
Contents

- Introduction
- Production of ODS alloys
- Mechanical tests
- XAFS study
- Outlook

Introduction

- What are ODS alloys?

Oxide **D**isperions-**S**trengthened alloys



(TEM High-Angle-Annular-Dark-Field Image)

Nano-sized oxide-particles 10-20nm

+ good corrosion-resistance

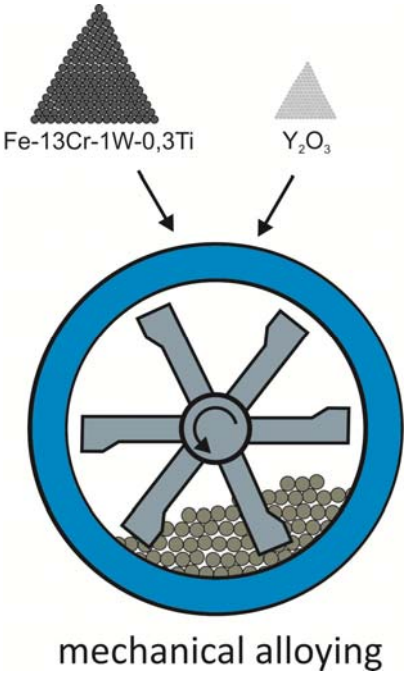
+ excellent high-temperature properties

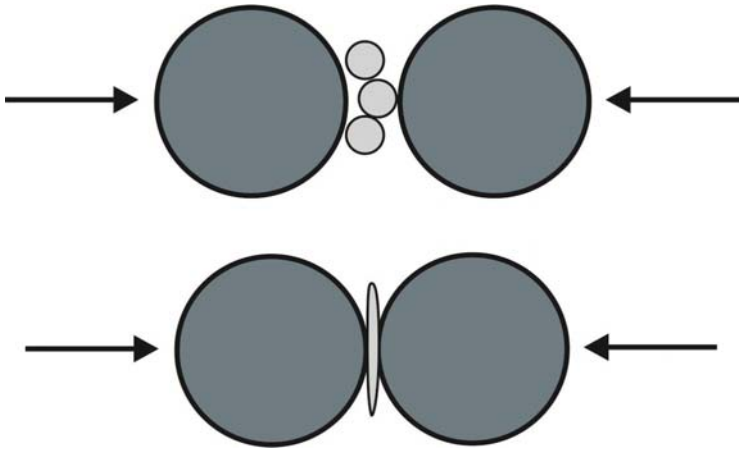
++ improved creep-strength

- material tends to be brittle

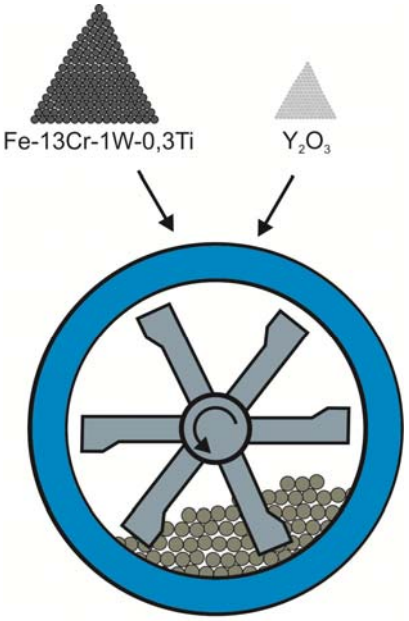
- High production costs

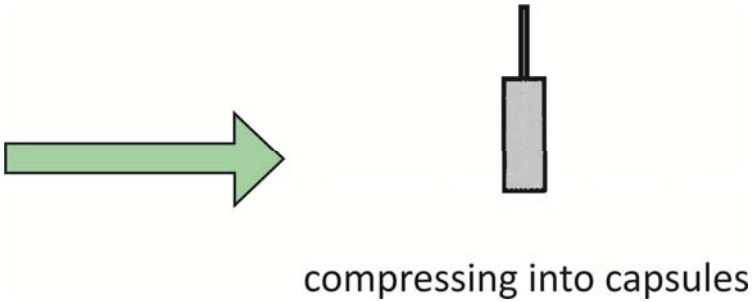
Production of ODS alloys



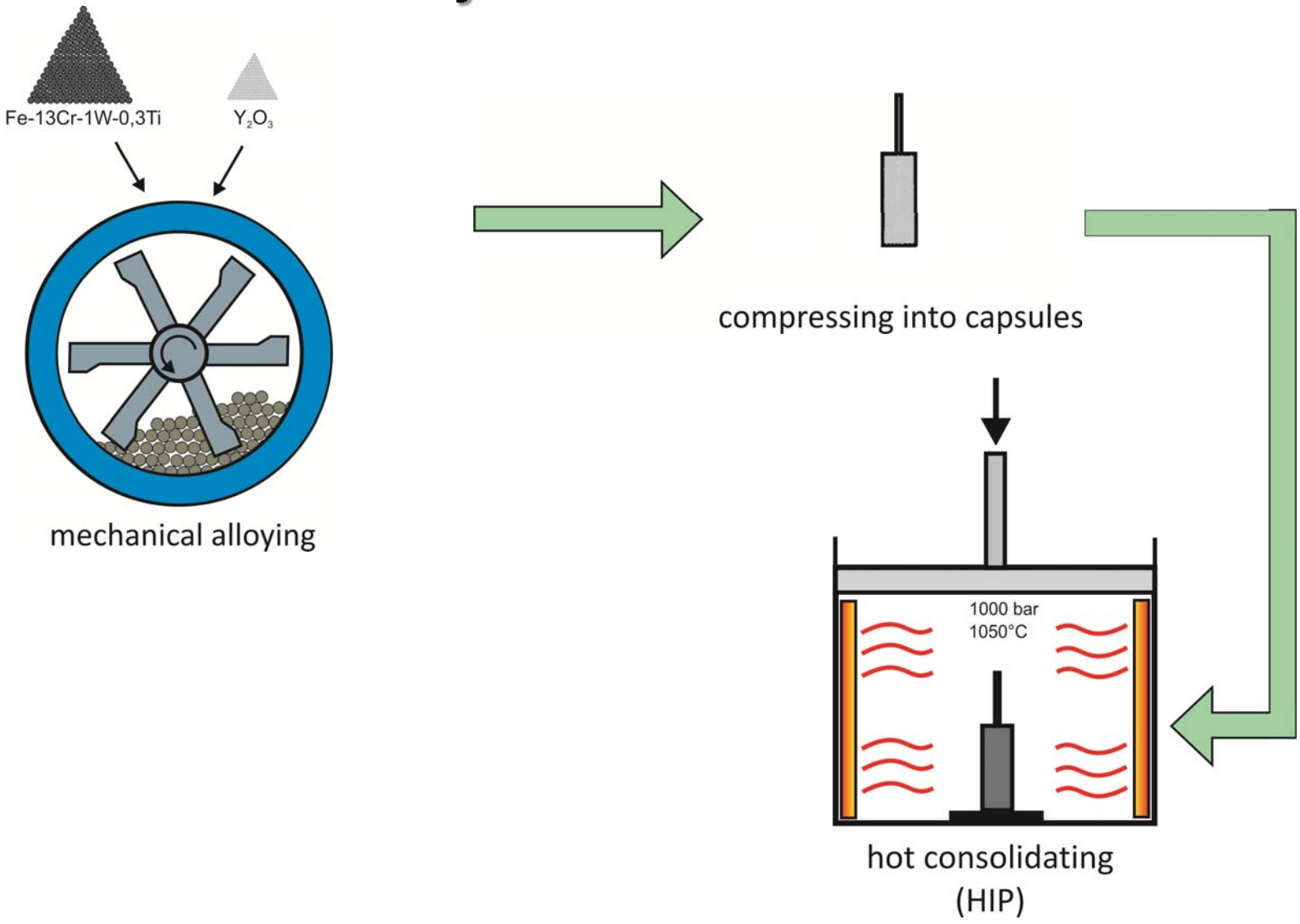


Production of ODS alloys

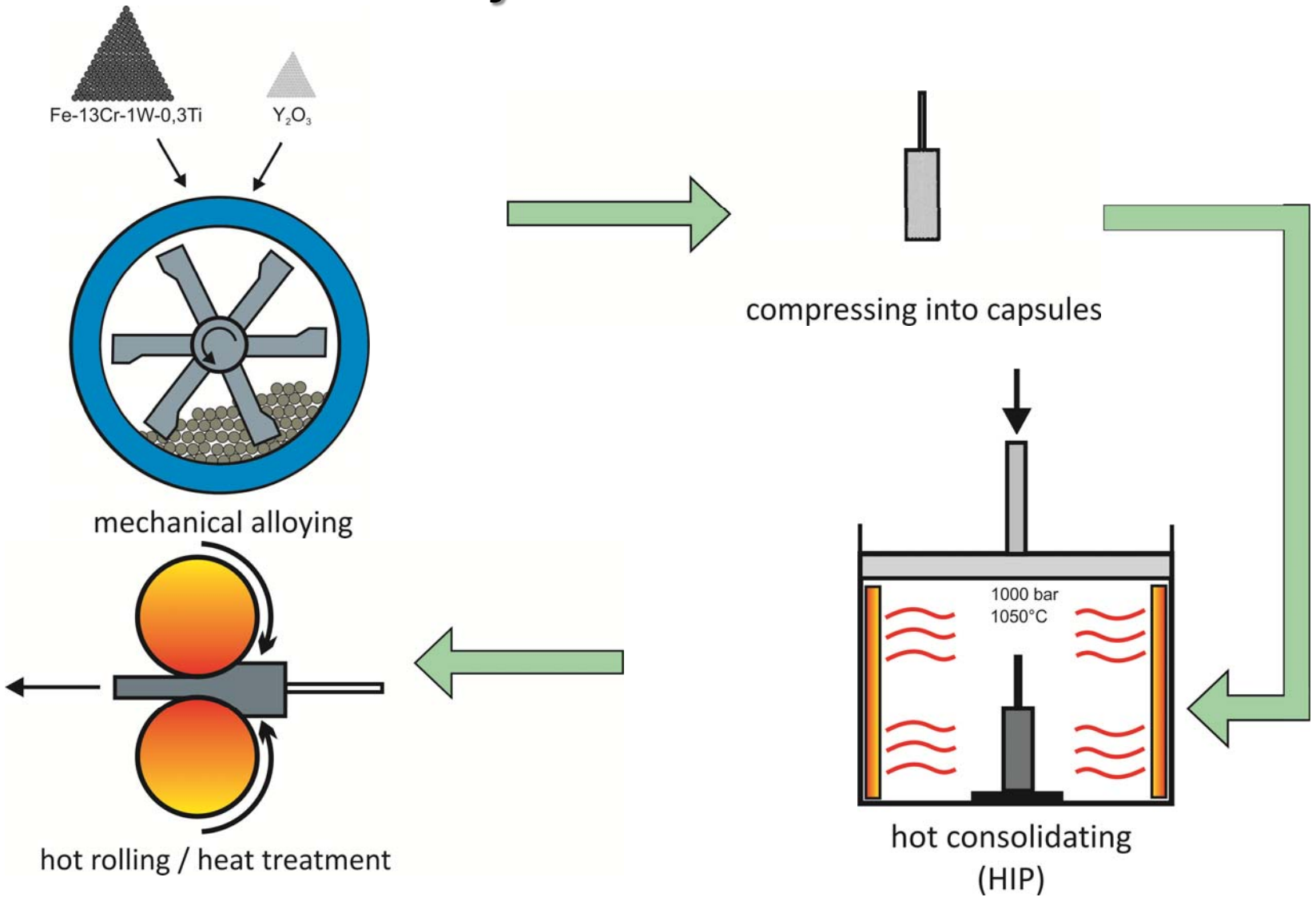




Production of ODS alloys

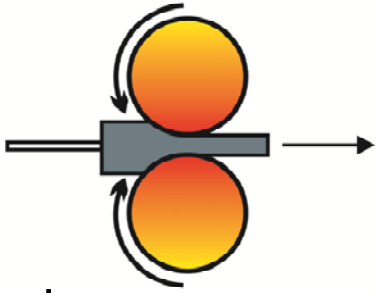


Production of ODS alloys



Production of ODS alloys

Compacting of the powders containing the different oxides:



- HIP at 1100°C / 100 MPa for 2 hours
- Hot-rolling at 1100°C
- Reduction from 45 mm diameter to 6 mm thickness
- 5 passes needed for final shape, with reheating after each pass

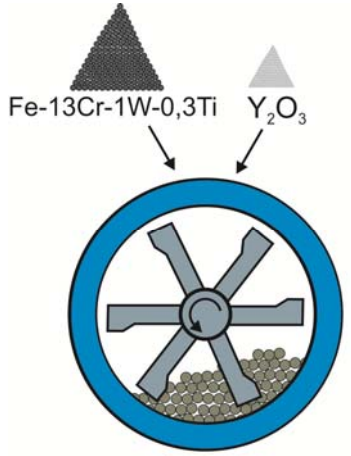
TU Clausthal

Production of ODS alloys

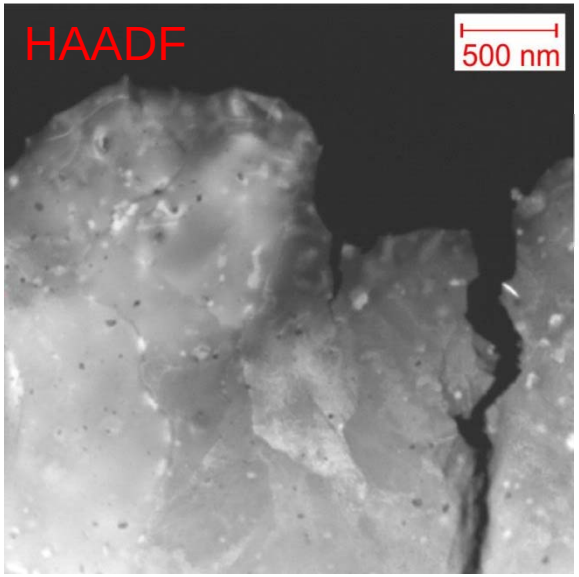
Production-parameters:

No.	composition	milling-speed	milling-time
1	Fe13Cr1W0.3Ti + La_2O_3	1200 / 800	80h
2	Fe13Cr1W0.3Ti + Ce_2O_3	1200 / 800	80h
3	Fe13Cr1W0.3Ti + MgO	1200 / 800	80h
4	Fe13Cr1W0.3Ti + ZrO_2	1200 / 800	80h
Ref.	Fe13Cr1W0.3Ti + Y_2O_3	1200 / 800	80h

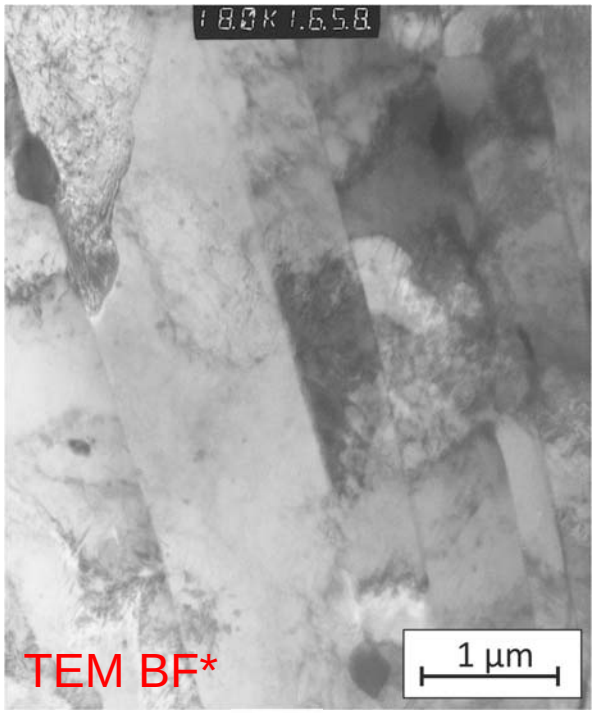
- milling in argon-atmosphere
- ball to powder ration 10:1 (2000g : 200g)
- complete produktion in argon (glove-box)

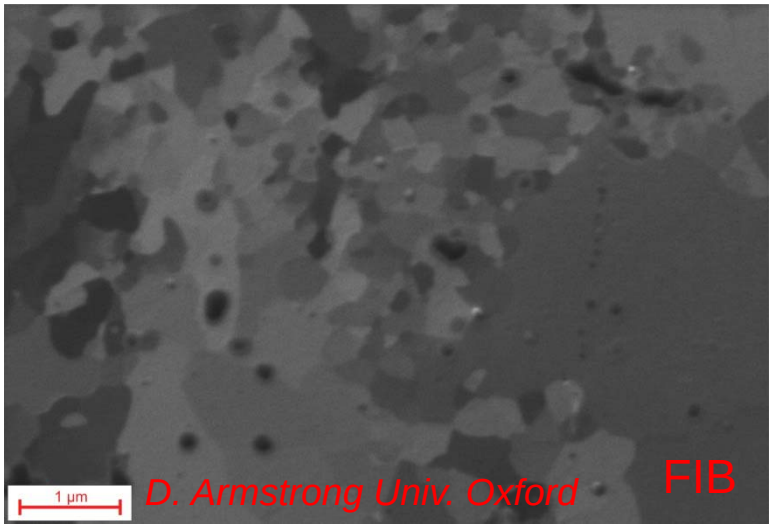


Characterization (FIB/TEM)



Fe13Cr1W0.3Ti +
Y_2O_3

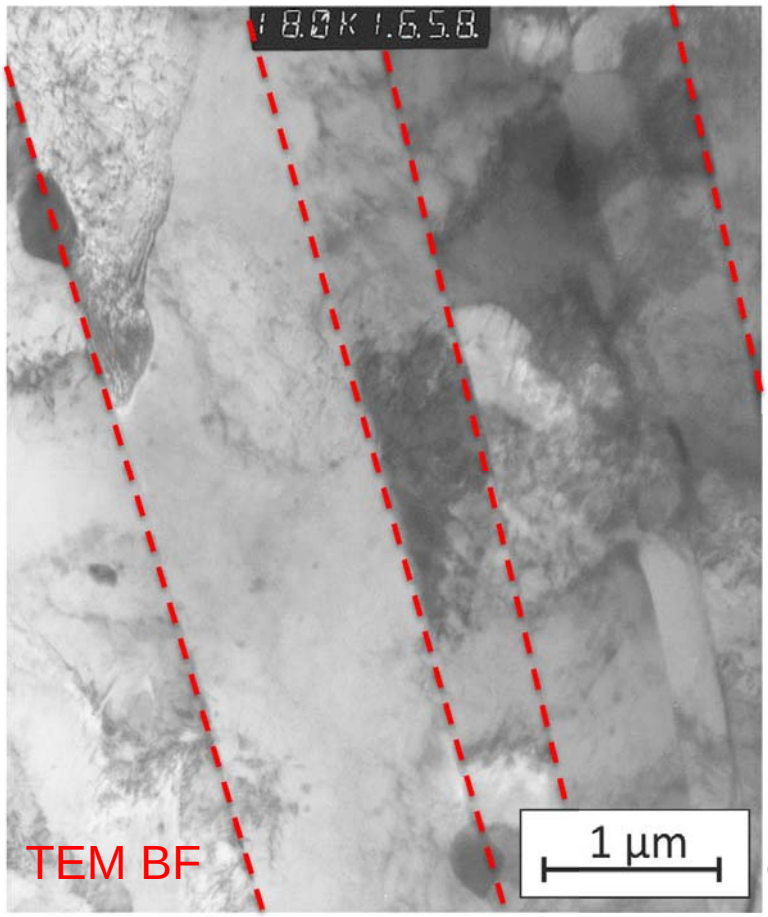




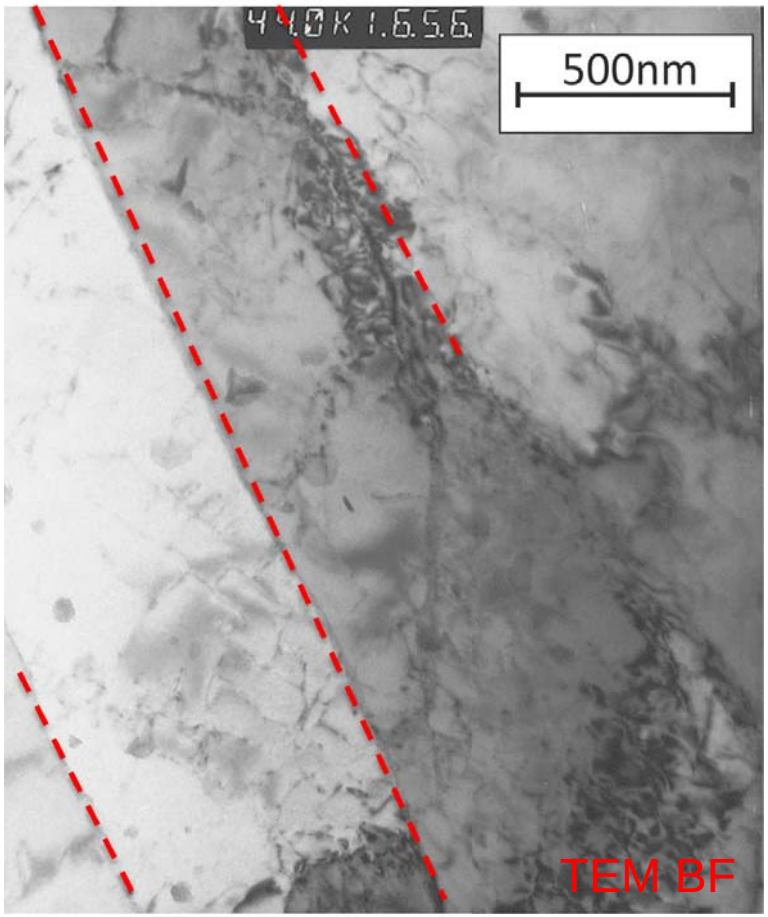
Fe13Cr1W0.3Ti + La_2O_3 rolled

*GRC - Physical Metallurgy 2011, August 3., Boston, USA

Characterization (microstructure)

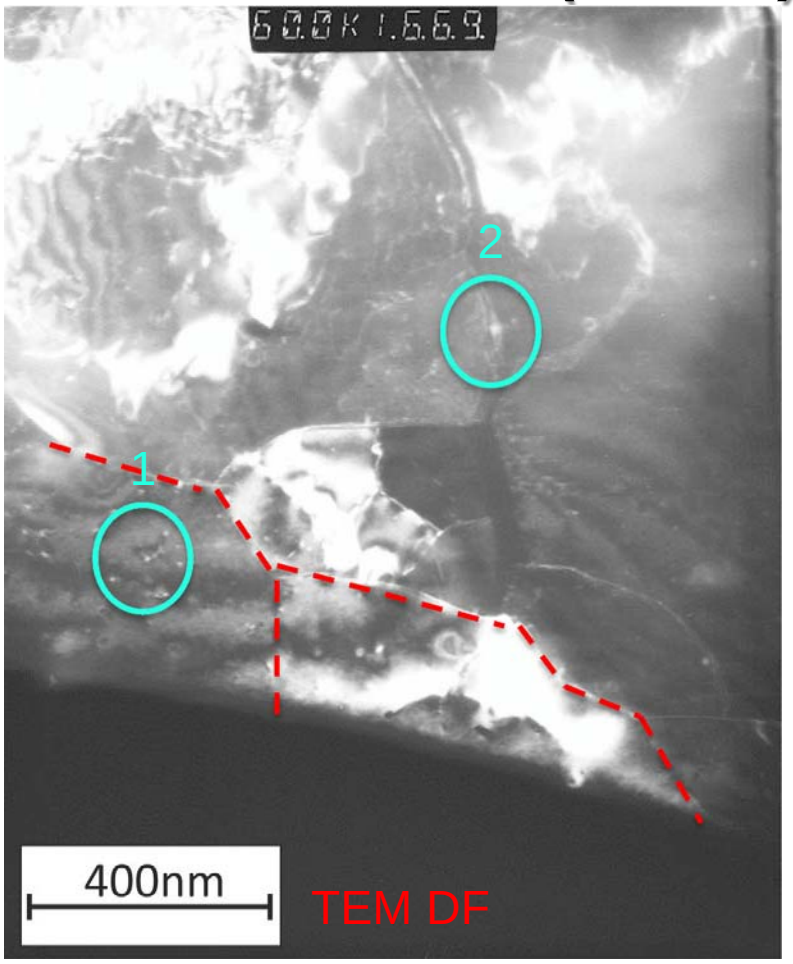


90°RD

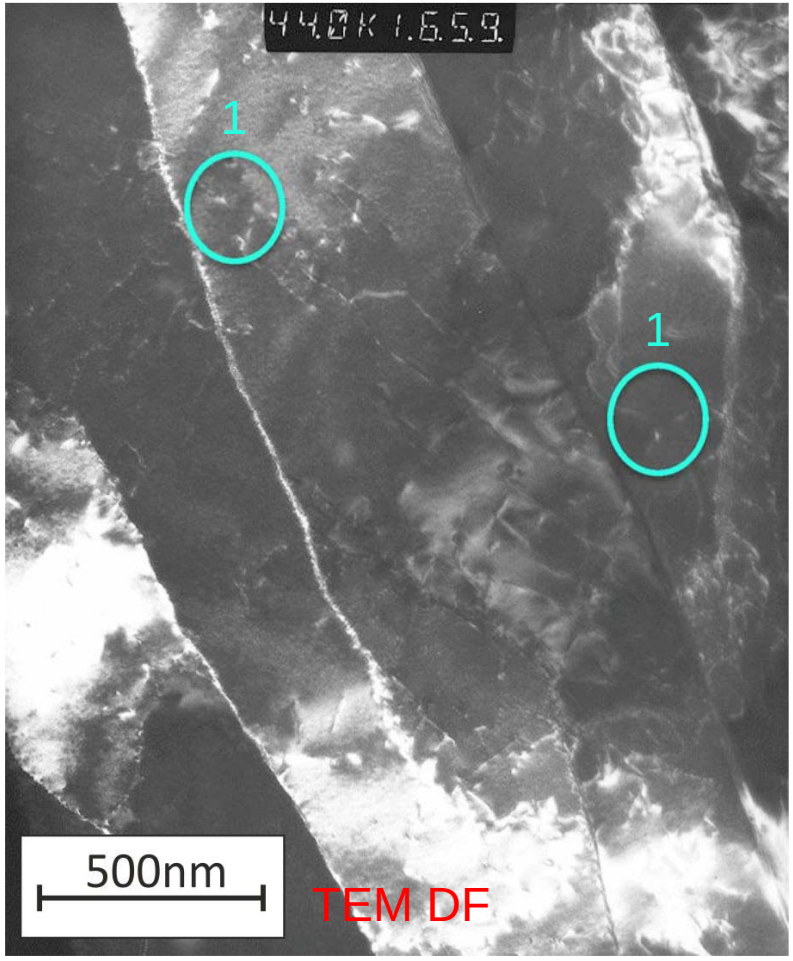


- clearly visible rolling texture
- grain size approx. 400 to 800 nm width, but micrometer-sized length

Charakterization (oxides)

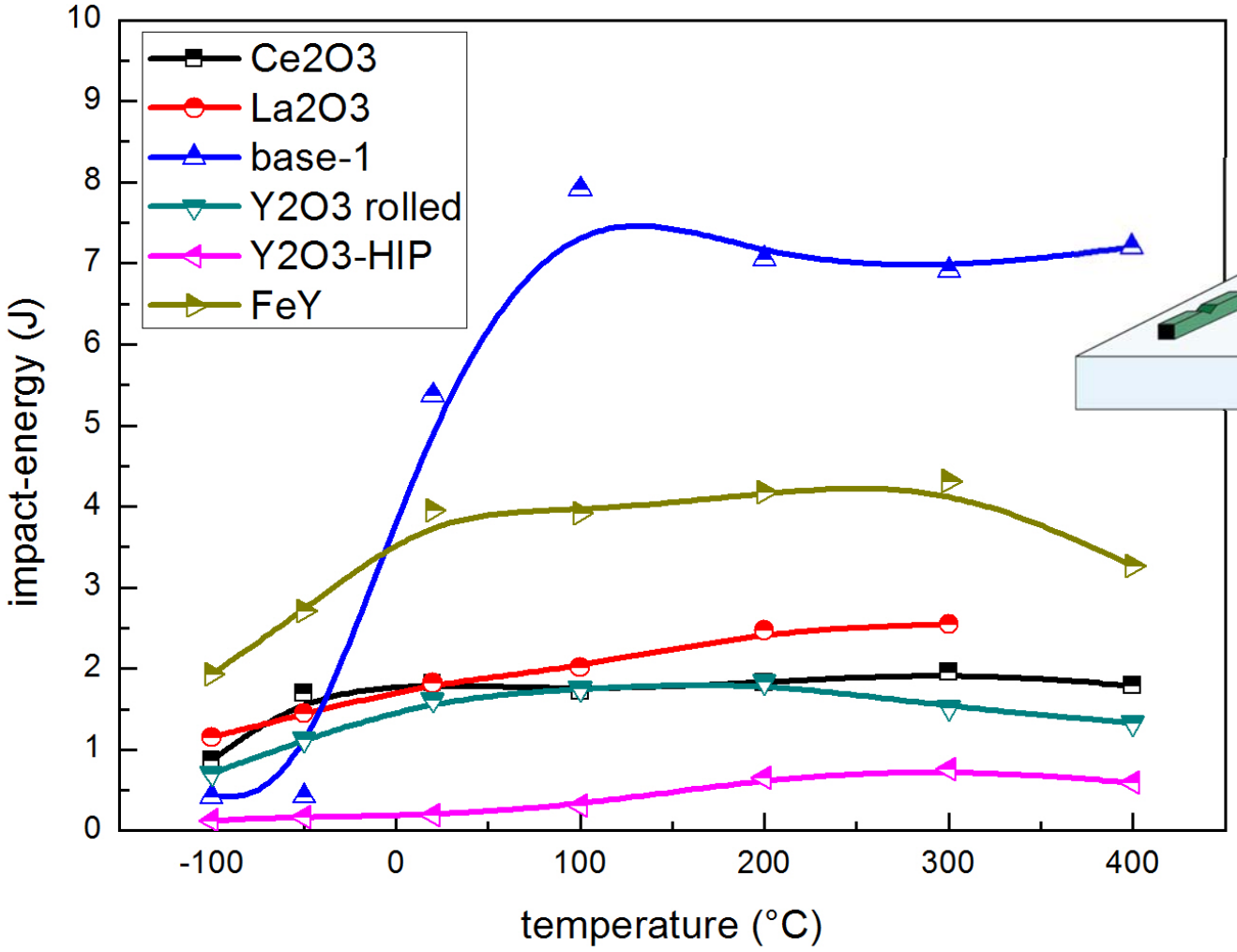


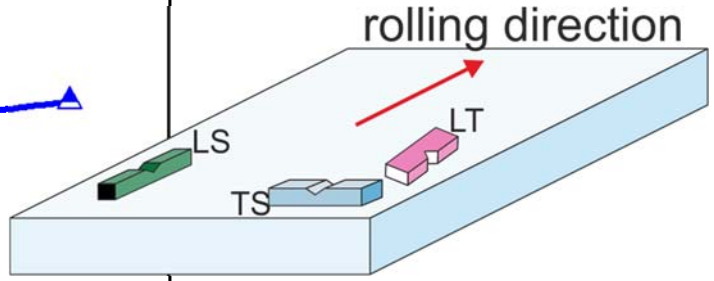
90°RD



- formatation of ODS particles with La_2O_3
- fine distribution of the oxides (inside grains[1] and on GB[2])

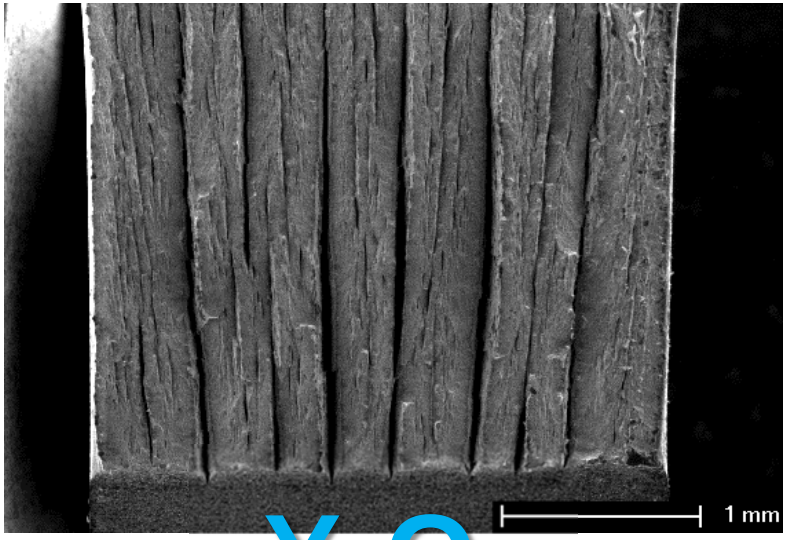
mechanical tests (charpy-impact test)





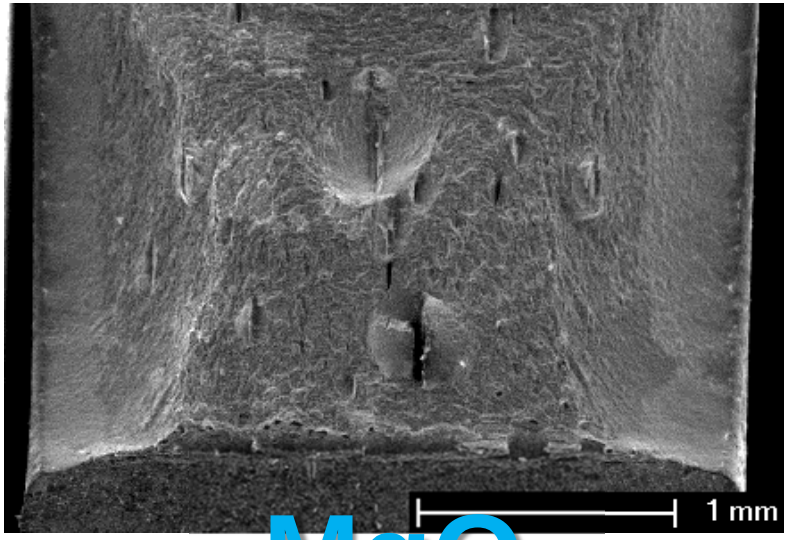
All specimens
in LT- orientation

mechanical tests (charpy-impact test)



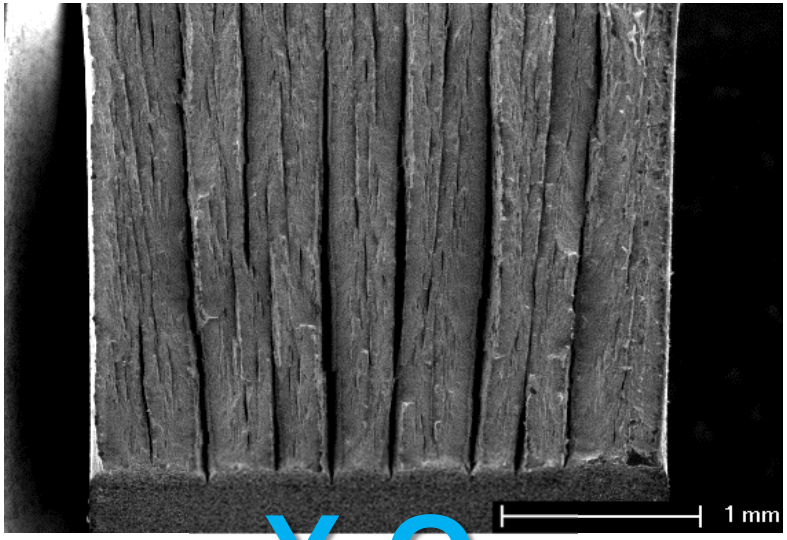
Y_2O_3

RT

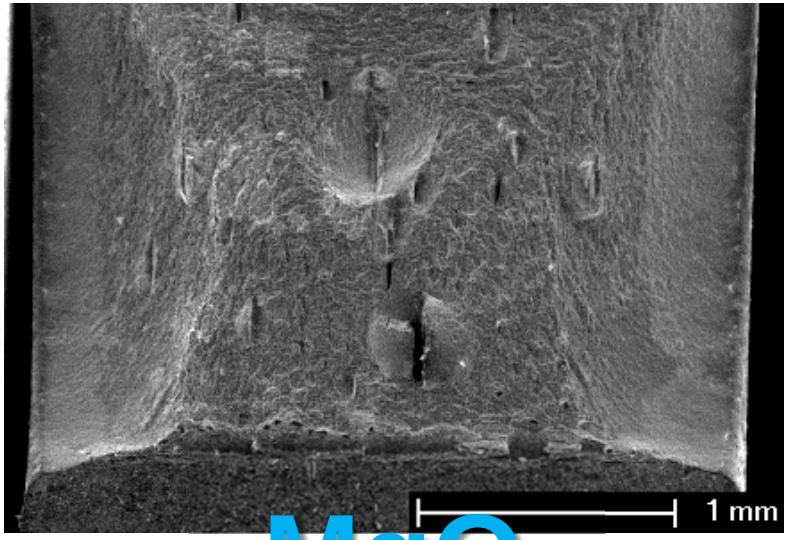


MgO

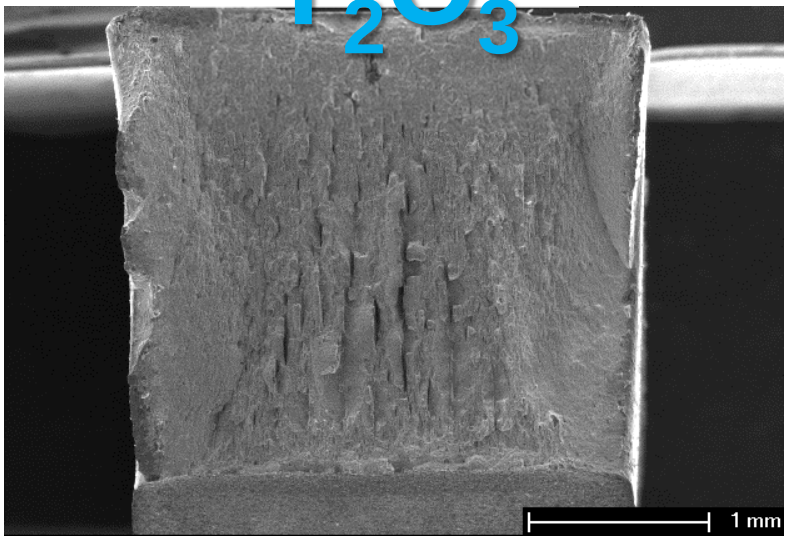
mechanical tests (charpy-impact test)



RT

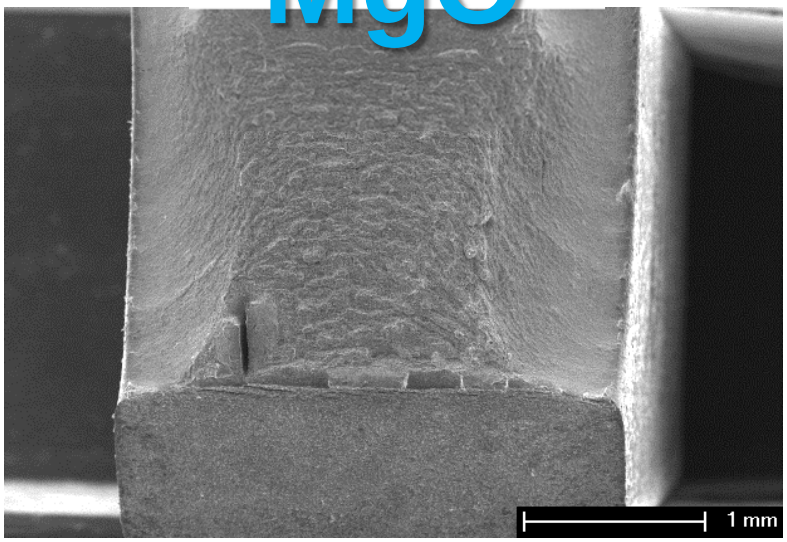


Y_2O_3

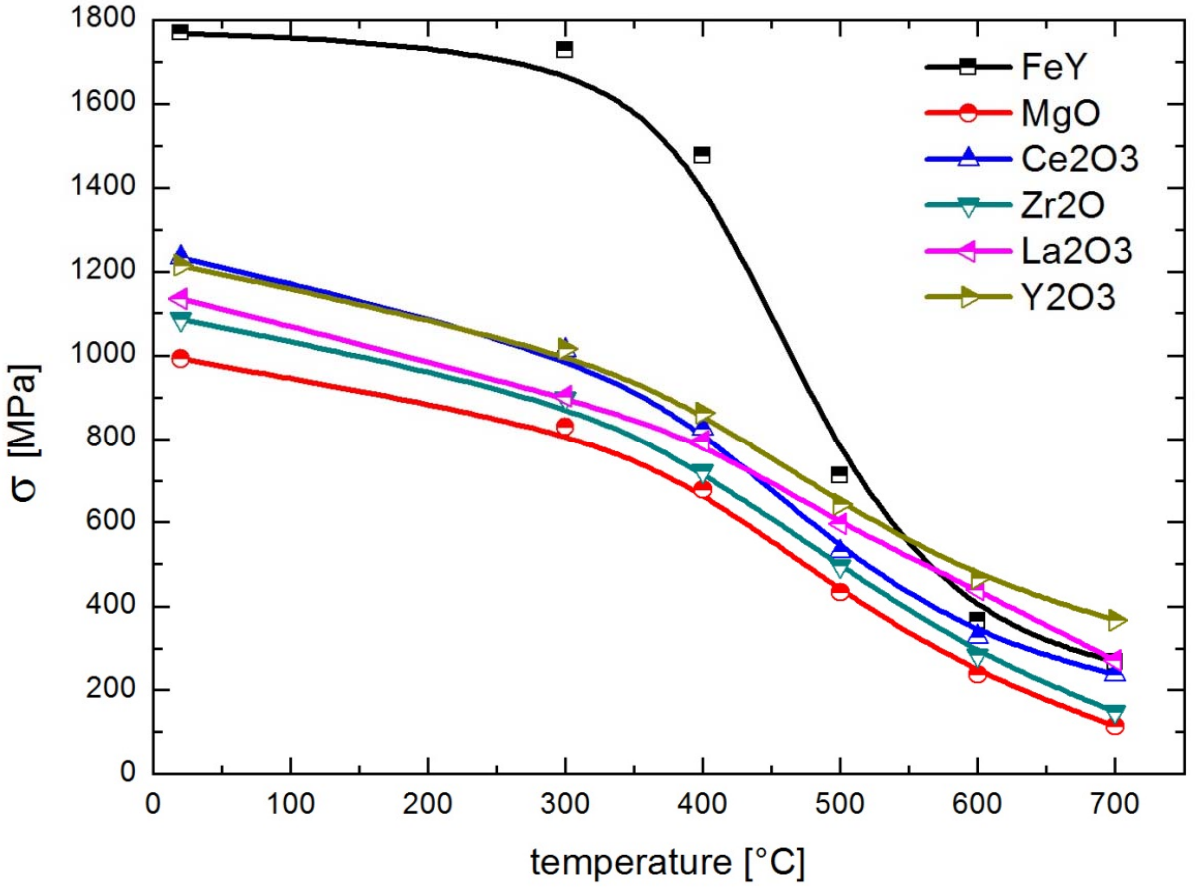


300°C

MgO

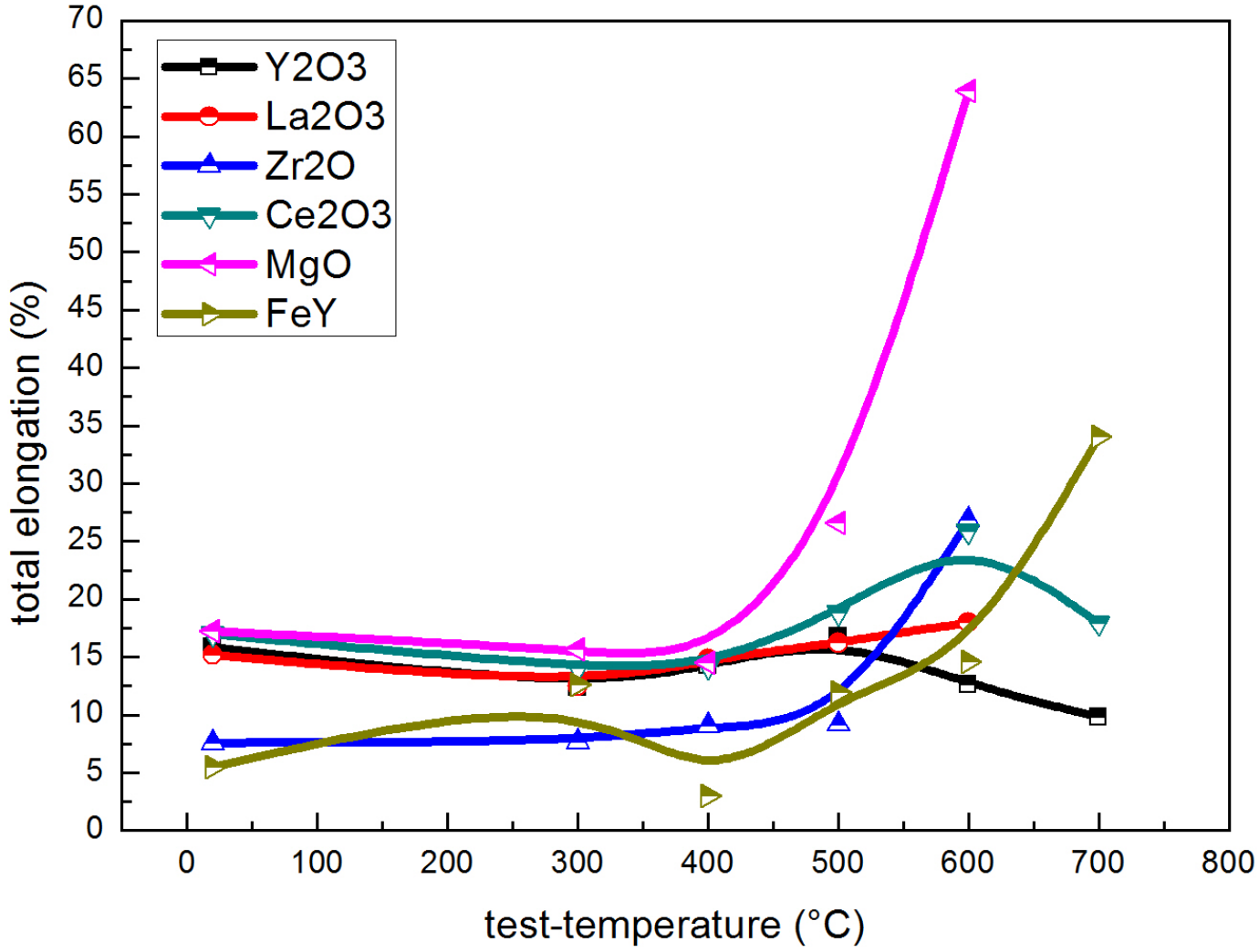


mechanical tests (tensile tests – yield strength)

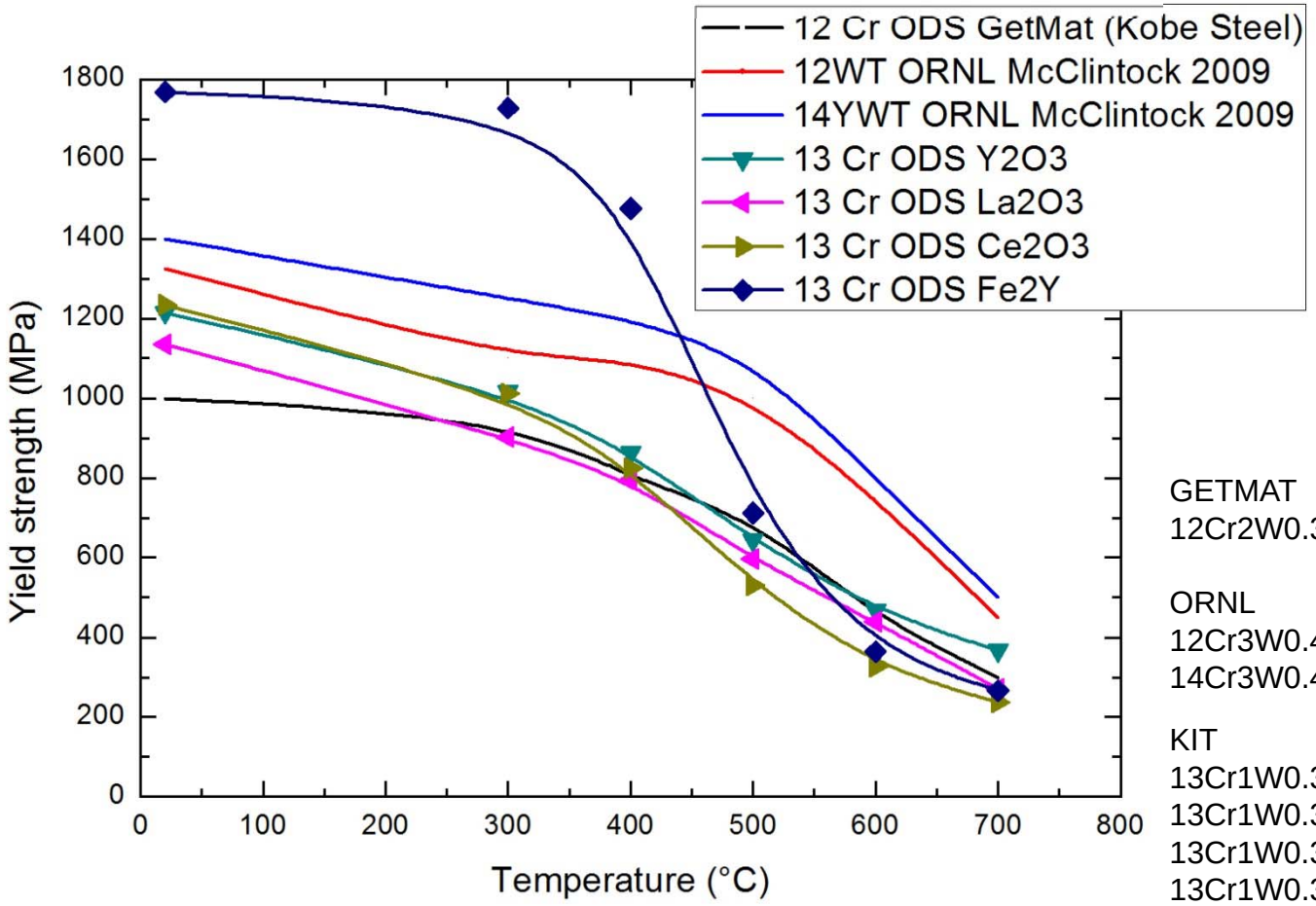


- Y-containing alloys show the best results
- Most alloys perform in a similar way

mechanical tests (tensile tests)



Comparison of 12Cr, 13Cr and 14Cr ODS



GETMAT
12Cr2W0.3Ti 0.2 Y_2O_3

ORNL
12Cr3W0.4Ti 0.25 Y_2O_3
14Cr3W0.4Ti 0.25 Y_2O_3

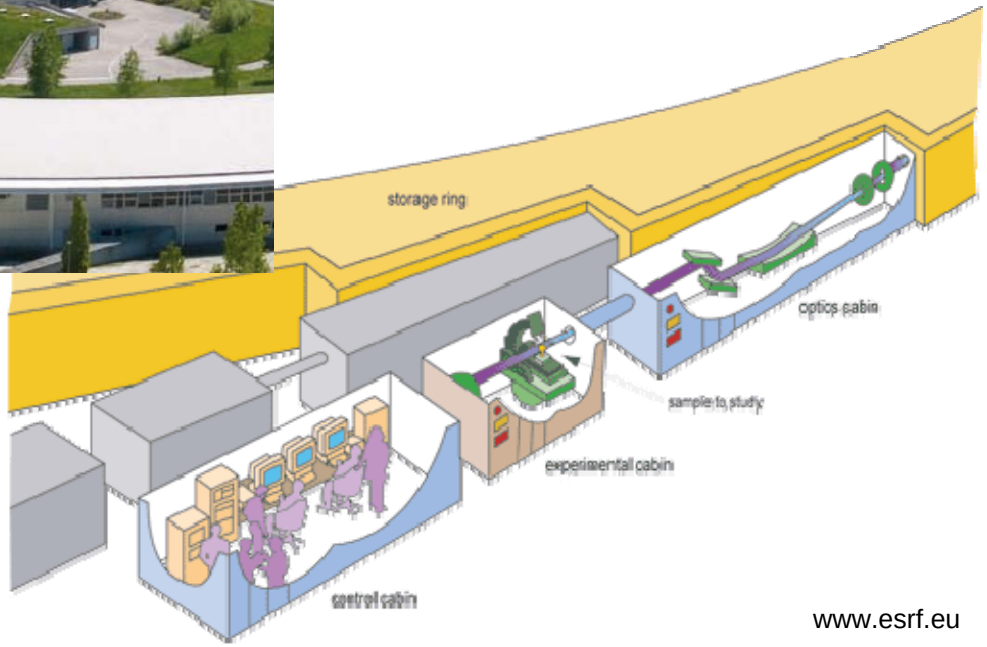
KIT
13Cr1W0.3Ti 0.3 Y_2O_3
13Cr1W0.3Ti 0.3 La_2O_3
13Cr1W0.3Ti 0.3 Ce_2O_3
13Cr1W0.3Ti 0.3 Fe_2Y

- Performance in tensile tests is comparable to alloys produced at other facilities

XAFS experiments - Introduction

ESRF

European
Synchrotron
Radiation
Facility



www.esrf.eu

XAFS experiments - Introduction

XAFS characteristics:

- Used to determine the **oxidation state**, **bond length** and **coordination number** aswell as species of the atoms surrounding the absorbed atom
- High accurancy: bond length (0,005A), coordination no. (5-10%)
- Very good statistics, due to measurements in bulk materials
- Applicable to dilute elements in materials (several ppm → Y_2O_3 and Ti in ODS steels)

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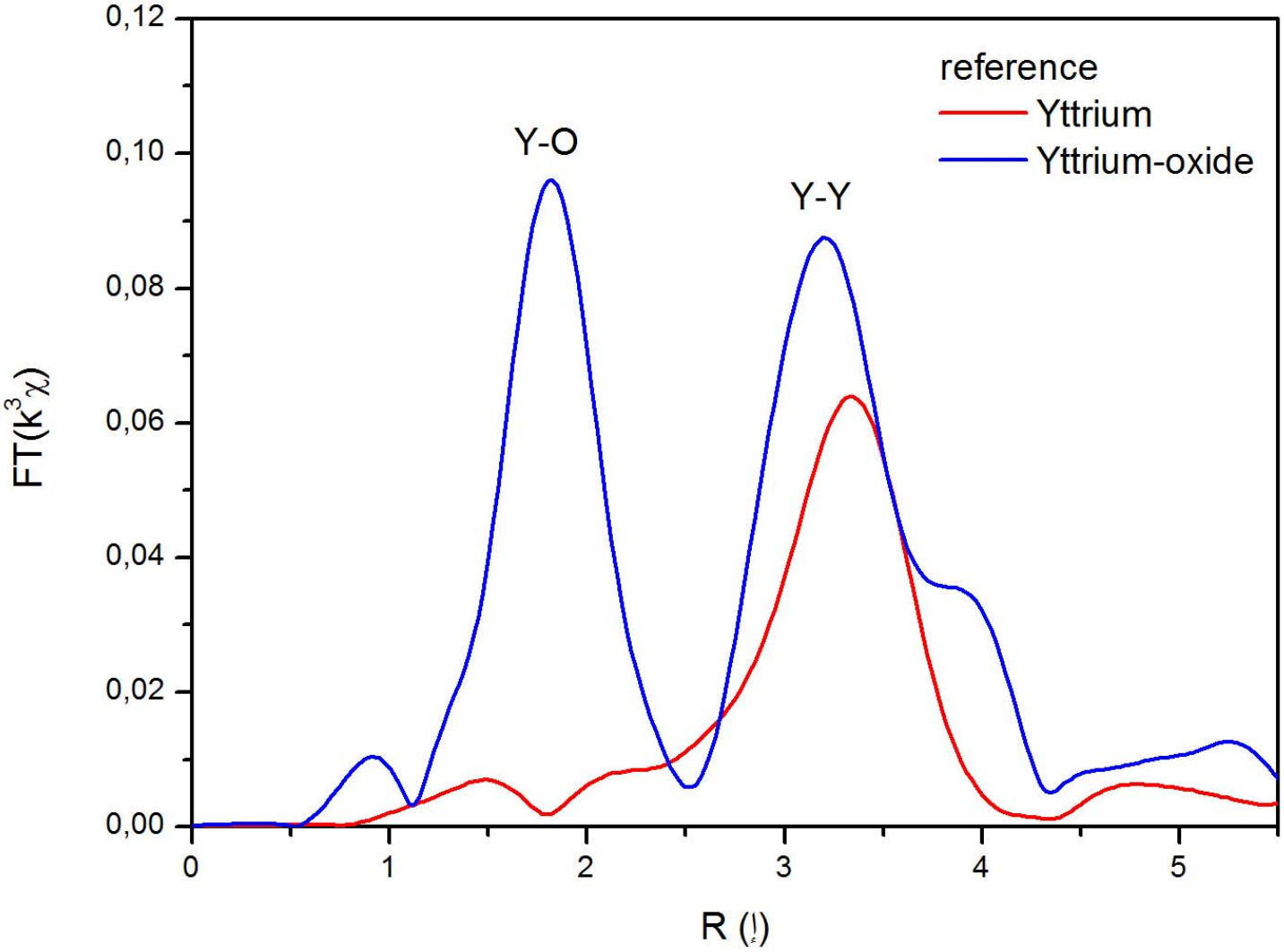
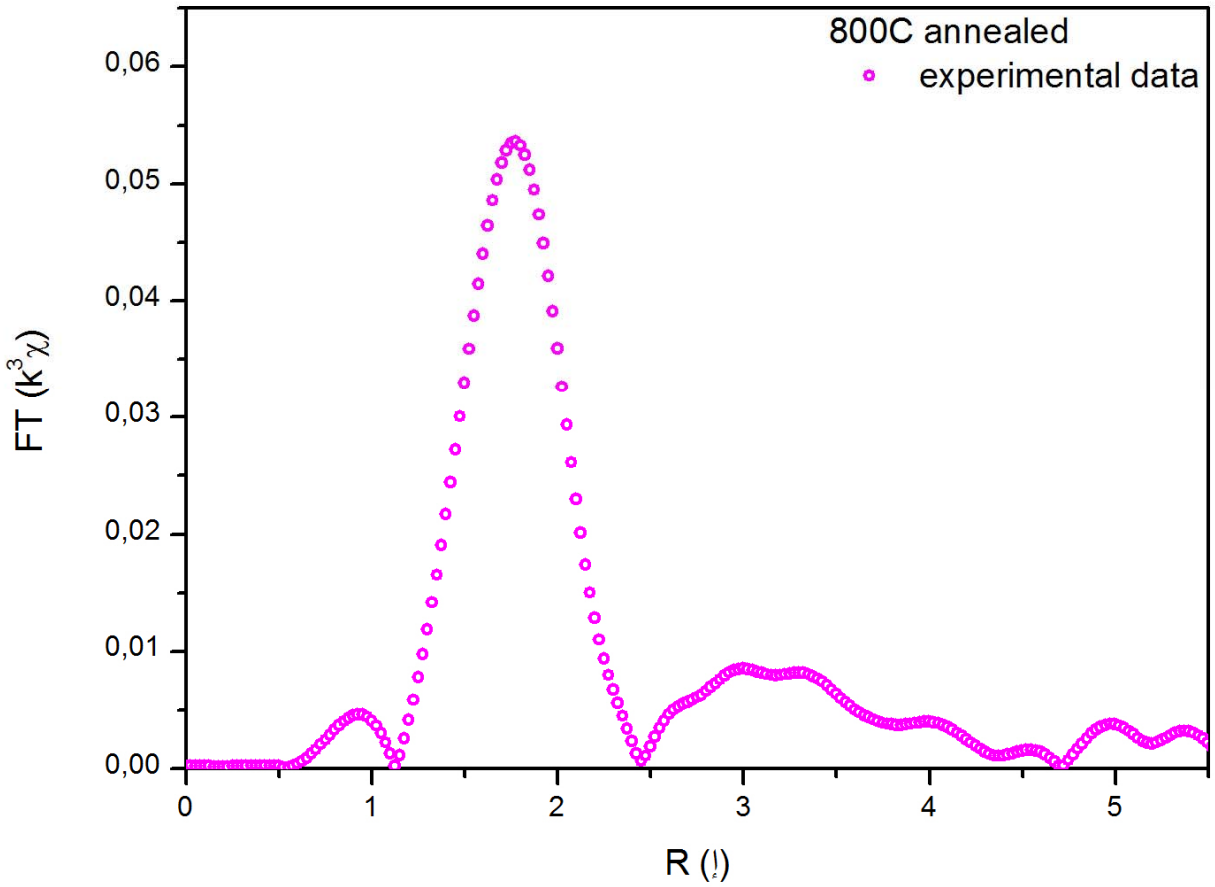
Goal → knowledge about the precipitation of ODS particles during thermal-treatment

XAFS experiments (reference measurement)



XAFS experiments (powder 800°C 2h annealed)



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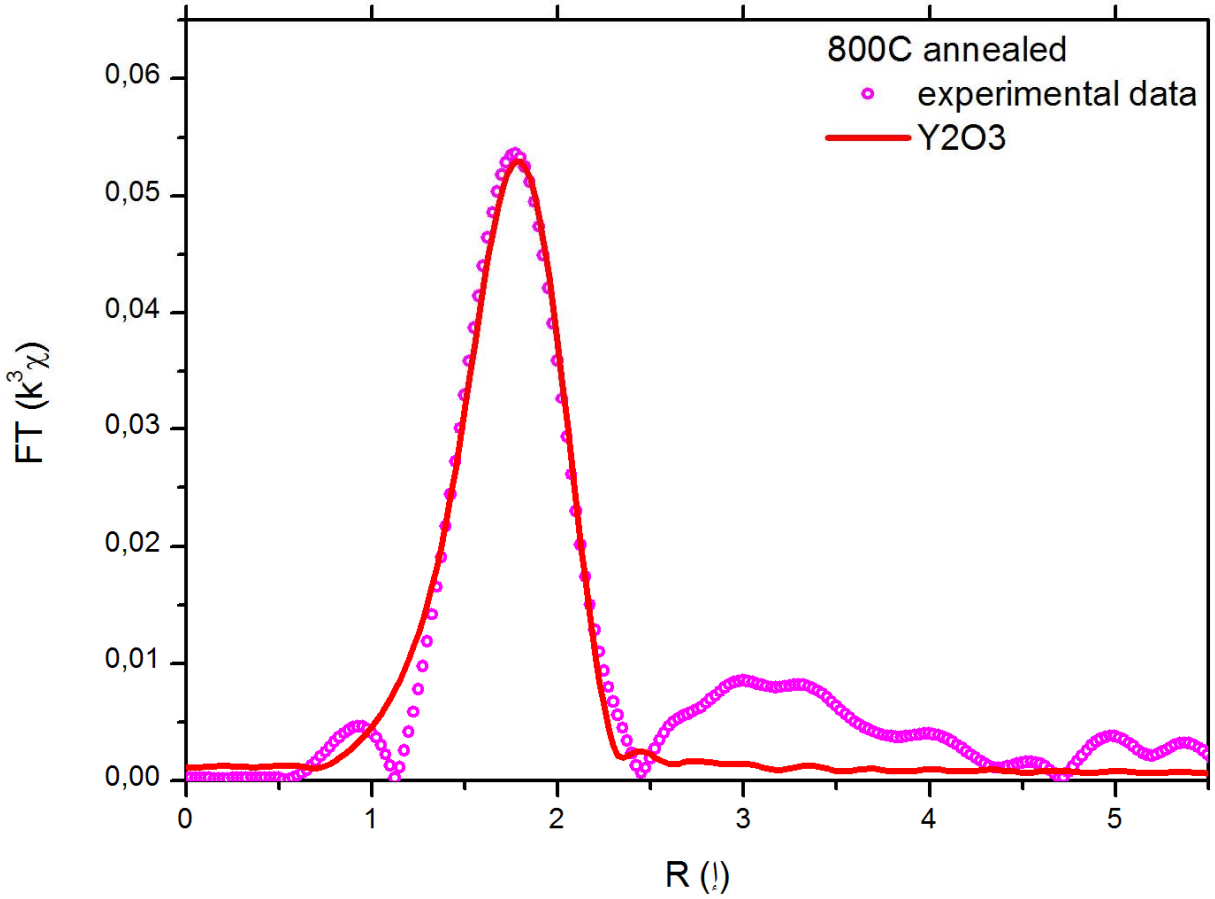

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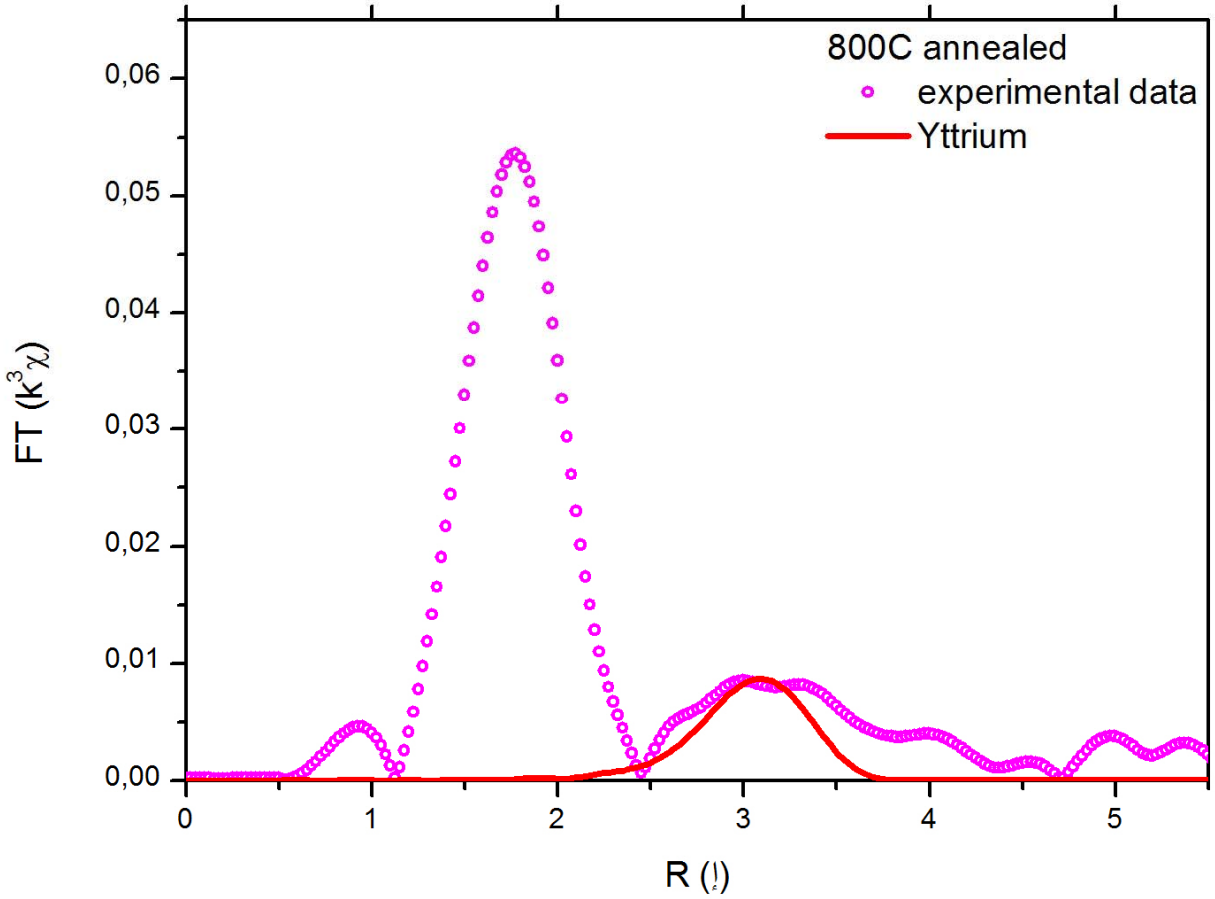

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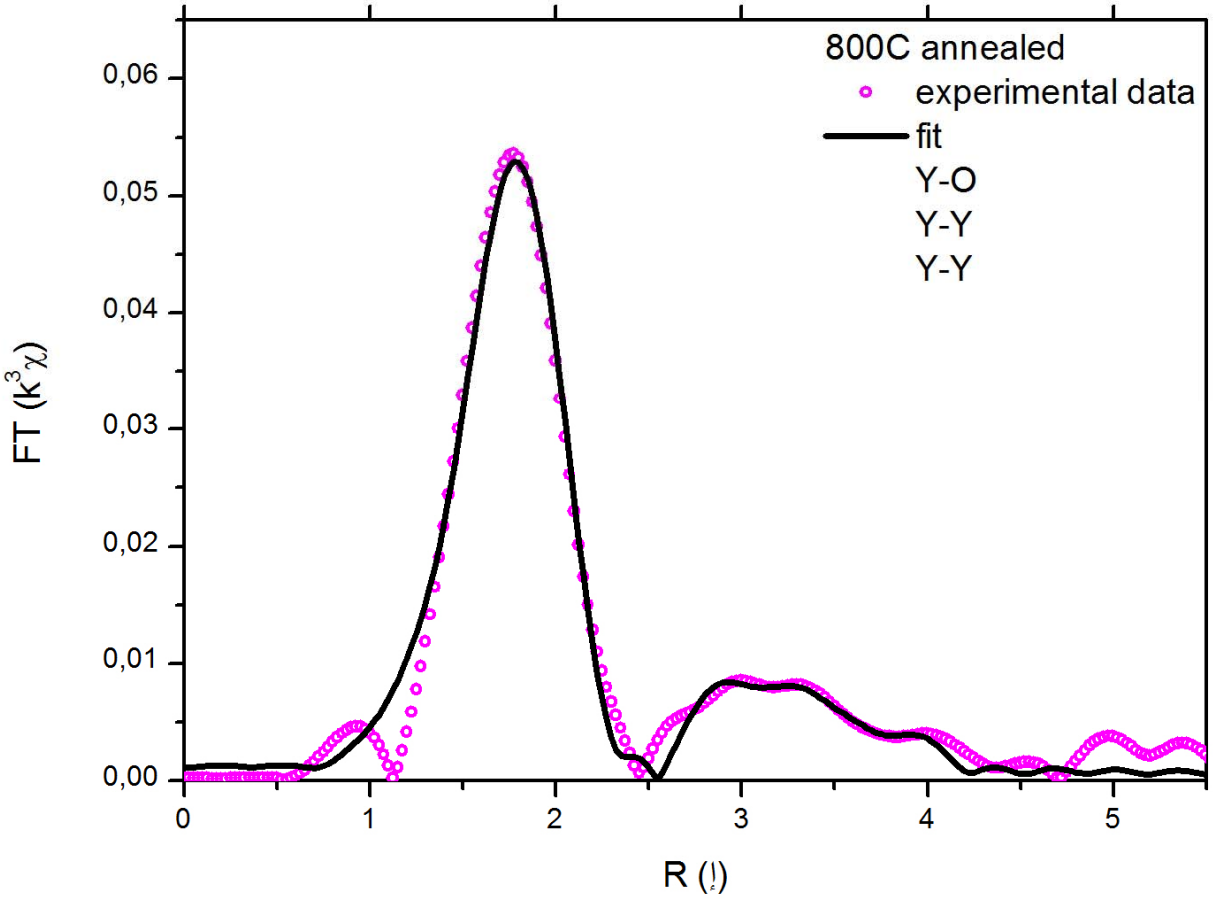
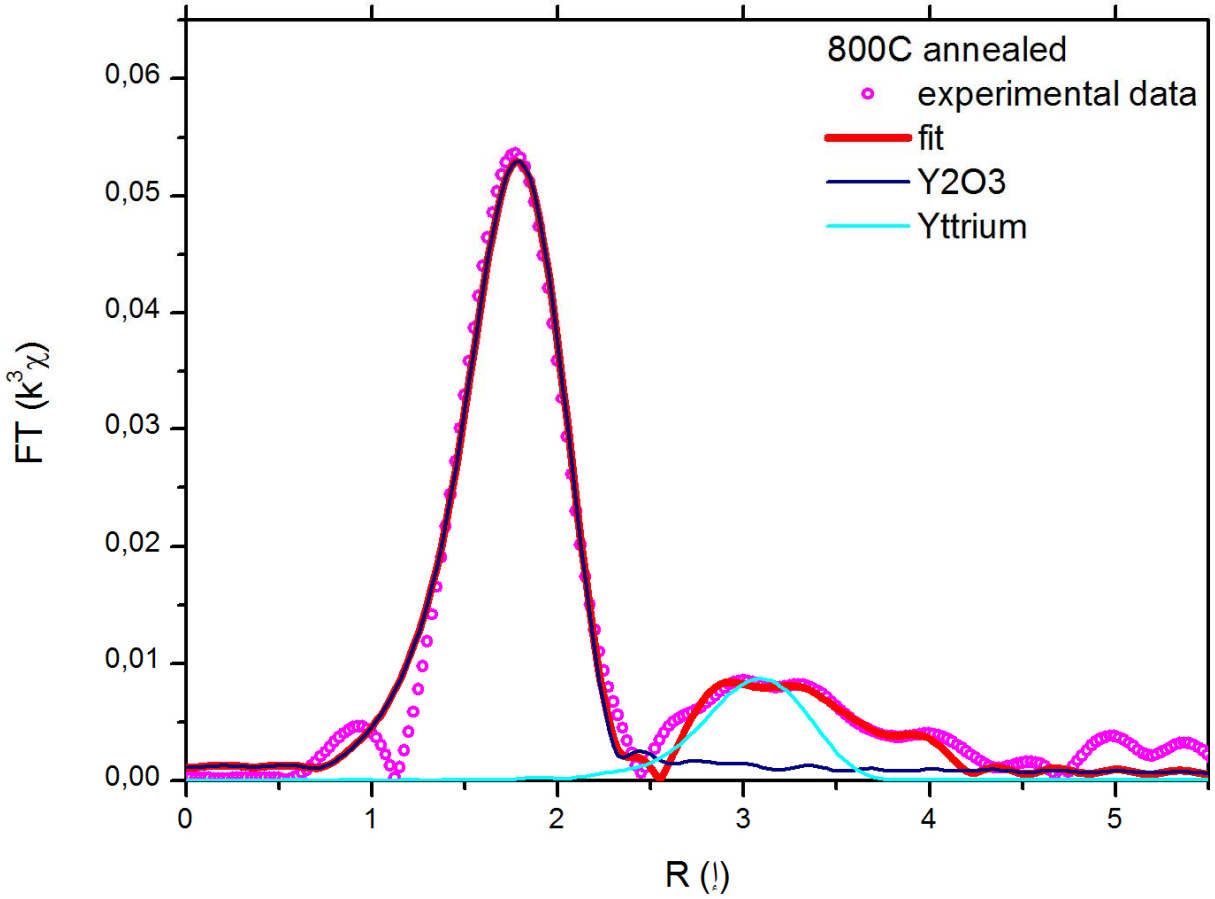
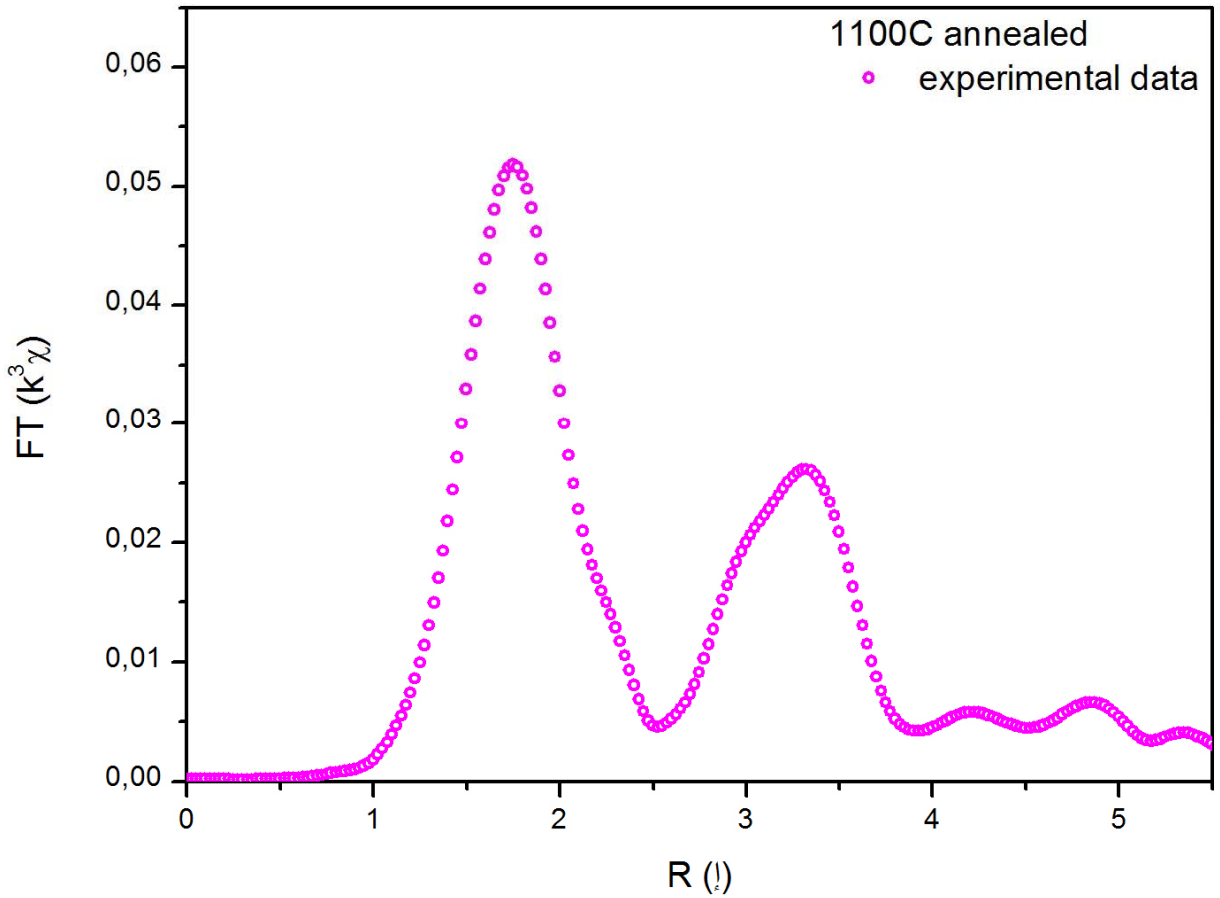


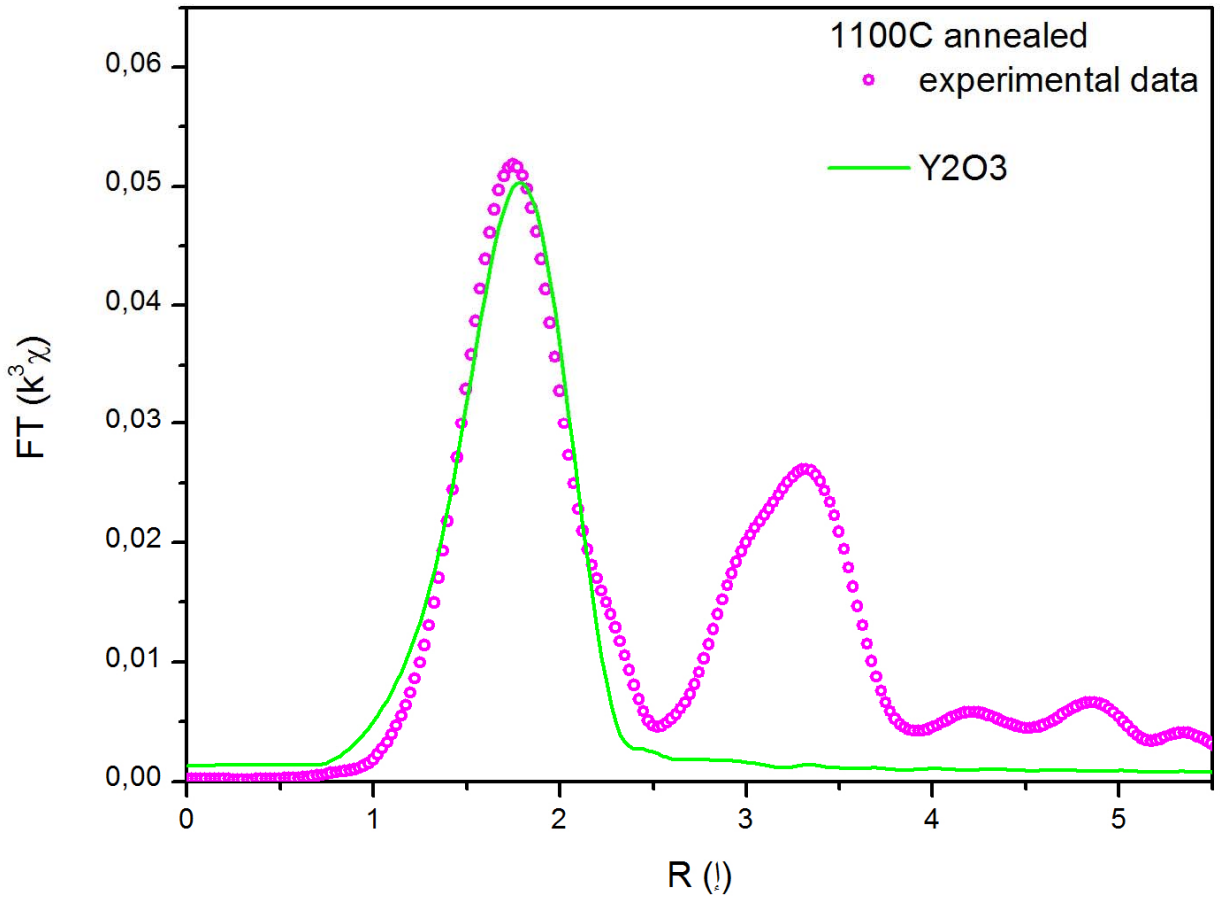
XAFS experiments (powder 800°C 2h annealed)



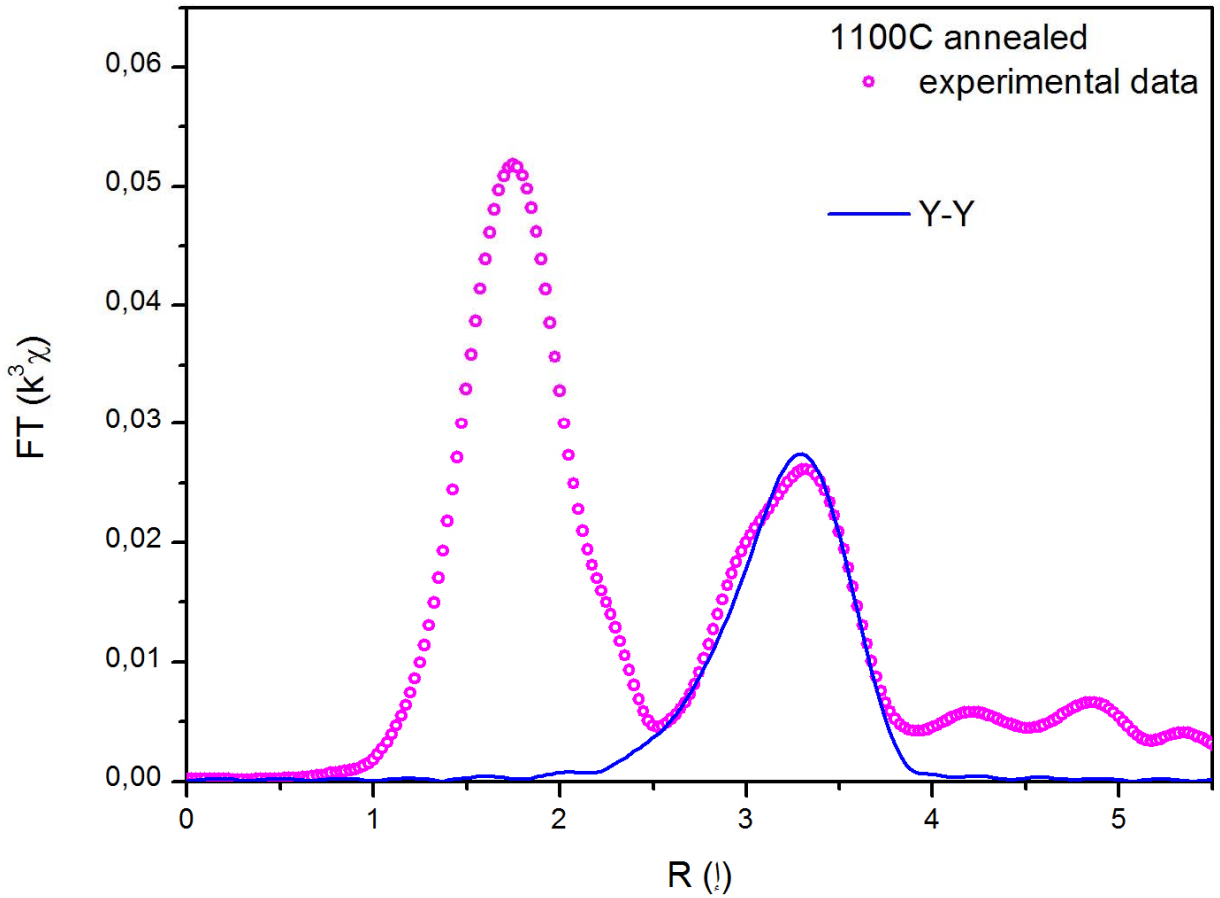
XAFS experiments (powder 1100°C 2h annealed)



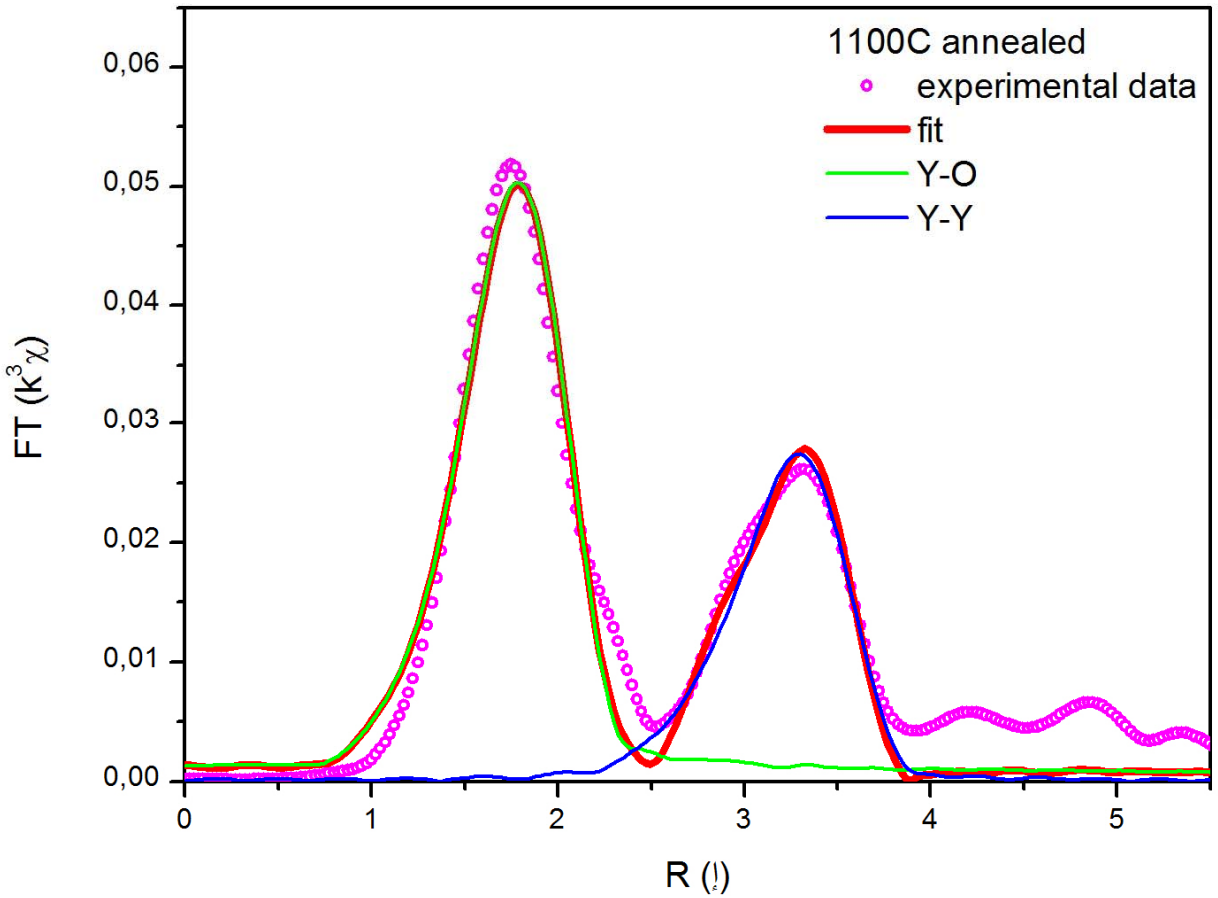
XAFS experiments (powder 1100°C 2h annealed)



XAFS experiments (powder 1100°C 2h annealed)



XAFS experiments (powder 1100°C 2h annealed)



XAFS experiments - summary

Annealing at 800°C

- No ordered yttria (dispersed particles) detected after annealing
- Peaks match metal-Yttrium, which is in solution

Annealing at 1100°C

- Major increase in ordered yttria(Y_2O_3) nano-particles
- Precipitation of ODS particles inside the powder during annealing

Conclusion and Outlook

Alternative oxides for ODS steels

- Formation of nano-oxides is possible with alternative oxides
- Tensile properties of different oxides are comparable to yttrium-alloys
- Improved charpy-impact properties for Ce_2O_3 and MgO

Outlook

- TEM Characterization of nano-oxides is still in progress
- Detailed analysis of XAFS results

Thank you for your attention!