

# Systematic XAFS investigation of the structural evolution of yttrium-enriched oxides in ODS ferritic steels by advanced synchrotron source at ESRF

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## Abstract

The speciation and structural evolution of nanoscale yttrium-enriched oxides in numerous reduced activation oxide dispersion strengthened (ODS) ferritic steels have been investigated by systematic X-ray absorption fine structure (XAFS) spectroscopy (including X-ray absorption near-edge structure and extended X-ray absorption fine structure). The local structure and speciation of Y-enriched oxides during the fabrication process have been traced by Yttrium (Y) K-edge XAFS in fluorescence mode for both mechanical alloyed (MA) powders and compacted ODS alloys. After 24 h of milling, only 10%-14% of the initially added 0.3 wt. %  $Y_2O_3$  dissolves into the steel matrix and titanium (Ti) exhibits a minor influence on the Y solid solution during the MA. The EXAFS analysis for compacted ODS alloys indicates the formation of new Y-enriched oxides rather than initial  $Y_2O_3$ . The addition of Ti exhibits an evident influence on the consolidation process rather than in the MA.

Table 1 Chemical composition, sample geometry and fabrication parameters of 13.5Cr ODS steels

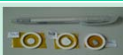

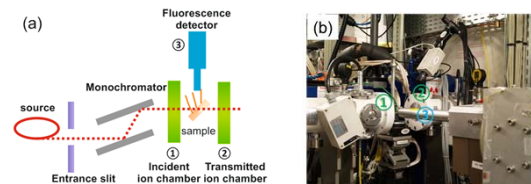
Sample	Composition	Mass & Geometry	Remarks
Reference	Y foil	25*5*0.025 mm <sup>3</sup>	
	$Y_2O_3$	Pellet (d=10mm)	
MA steel powders	0 Ti		MA: 24 hours; H <sub>2</sub> ;
Fe-13.5Cr-2W	0.2 Ti		Ball-to-powder ratio= 10:1
	0.3 Ti		
	0.4 Ti		
Compacted samples	0 Ti	27*3*4mm <sup>3</sup>	MA + HIP +HT
Fe-13.5Cr-2W	0.2 Ti		HIP: 1150 °C, 100 MPa, 2.5 hours
	0.3 Ti		HT: 1050 °C, 2 hours, Vacuum
	0.4 Ti		

Fig.1 XAFS experimental setup at BM26A of ESRF



Y-K edge is measured. ( $E_0=17.038$  keV)

-Transmission mode: reference compounds; measured by ion chambers

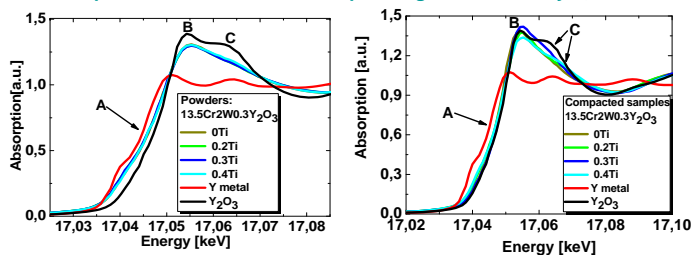
-Fluorescence mode: dilute samples; measured by a multi-elements Ge-detector

$$\text{Beer's law: } I = I_0 e^{-\mu t}$$

$\mu$  is absorption coefficient, which gives the probability that x-rays will be absorbed as a function of energy.

$$\mu(E) \propto I_f / I_0$$

Fig.2 Y K-edge XANES spectra of the MA powder and compacted samples together with the reference samples, metal Y and  $Y_2O_3$ , the absorption edge, the main peak and the shoulder at the post edge are marked by A, B and C.



All the curves for MA powders overlap and the Y K-edge lies between Y and  $Y_2O_3$ .

• Overlap indicates no obvious influence of Ti during MA

• The curve position indicates partial dissolution of  $Y_2O_3$  during MA.

XANES spectra for compacted samples shows pronounced differences

• The chemical shift (A) for compacted samples almost coincides with that of  $Y_2O_3$ .

• A bump (C) in pure  $Y_2O_3$  is still absent in all compacted sample.

Fig.3 Y K-edge XANES of MA powders (0 wt. % Ti) and a linear fit by (a) Y metal and  $Y_2O_3$ , (b) by Y metal and compacted sample with 0.4 wt. % Ti.

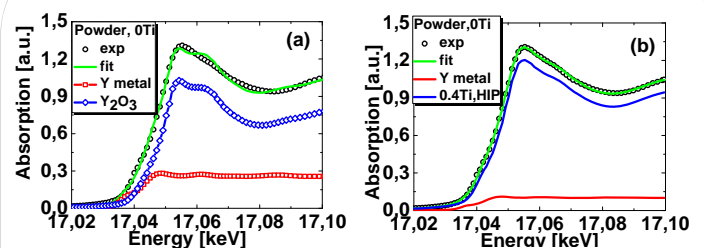
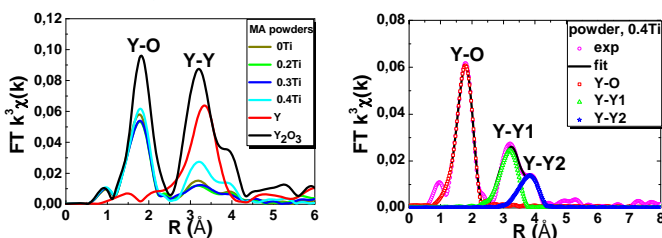


Table 2 Relative fraction of the metallic Y component in the MA powders determined from the linear fit of XANES.

Composition	0 Ti	0.2 Ti	0.3 Ti	0.4 Ti
%	10.2	12.8	13.7	11.5

After 24 h of milling, 10%-14% of the initially added 0.3 wt. %  $Y_2O_3$  dissolves into the steel matrix. It indicates that longer milling time is required for complete Y dissolution.

Fig.4 Fourier transform of EXAFS functions of MA steel powders and metal Y,  $Y_2O_3$  references, phase shift was not corrected. The two peaks corresponding to Y-O and Y-Y are marked.

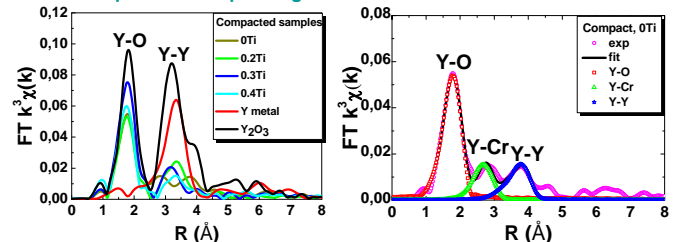


• The MA powders are characterized by Y-O and Y-Y peaks, close to  $Y_2O_3$ .

• The much smaller peak height of the Y-Y indicates that the  $Y_2O_3$  in the MA powders is much more disordered because of severe plastic deformation.

• The fitting reveals that the yttrium oxides after the MA are different from the initially added  $Y_2O_3$  powders, with a decreased coordination number, especially for the second Y-Y shell.

Fig. 5 Fourier transform of EXAFS functions of MA steel powders and metal Y,  $Y_2O_3$  references, phase shift was not corrected. The two peaks corresponding to Y-O and Y-Y are marked.



The local structure of Y varies significantly with Ti content

- 3 shells observed for the sample without Ti, better fit with  $YCrO_3$  than  $Y_2O_3$
- 2 shells observed for the samples with Ti, the intensity and the distance of Y-Y shell are different
- Ti-containing samples can not be well fit with each single phase indicating they have a mixture of various nano phases.