DE LA RECHERCHE À L'INDUSTRIE



Direction de l'Energie Nucléaire Direction déléguée aux Activités Nucléaires de Saclay Département des Matériaux pour le Nucléaire Service de Recherches Métallurgiques Appliquées

Simultaneous DAFS and XAFS analyses to evidence the Y- and Tispecies in nano-structured ODS steels

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What do we expect from DAFS ?

To characterize the microstructure of neutron irradiated materials, various experimental tools are available at the present time in nuclear environment: SEM, TEM, APT, RBS and NRA... However, these techniques are local probes and bring information for an extremely limited sample volume which could be not representative of the whole volume. Therefore, a good statistic can only be obtained by performing many examinations which are very time consuming.

To address this issue, Diffraction and Absorption analyses using synchrotron sources are of utmost interest:

-> X-Ray Diffraction (XRD) patterns allow us to identify the precipitates structural changes from line-profile analyses of diffraction peaks,

←at 4.9 keV

-at 16.9 keV

-> X-ray Absorption Fine Structure (XAFS) spectroscopy is particularly useful for studying, at the atomic scale, the local structure around a specific absorbing atom within the material that possess short-range (up to about 6 Å) or no translational order (amorphous structure).

However, the interpretation of XAFS data for industrial material where the absorbing atom studied is located in various secondary phases (showing different compositions, structures, densities, and grain sizes down to few nanometers), even in the matrix, is more challenging than for a quite homogeneous and "pure" material, where target species are clearly located in a specific phase.

DAFS combines into a single technique:

- The long-range order and crystallographic sensitivities of XRD with
- The spectroscopic and short-range order sensitivities of XAFS

by measuring the elastic Bragg reflection intensities *versus* photon energy.

It has enhanced capabilities compared to the separate techniques: **DAFS provides EXAFS- and XANES- like information** for the specific subset of atoms selected by the diffraction condition.

DIFFABS beamline setup

1-element Silicon Drift DAFS measuremen

fluorescence

		XRD analyses
400 -] α-Fe	
350 -	(110)	
<u>-</u> 300 -		

ODS steel grade : DY (SCK.CEN-Mol; 1975-1980)

Elaborated by mechanical alloying with the nominal composition (in Wt%): Fe-13Cr-1.5Mo-2.2Ti-0.5Y₂O₃

Material supplied as 9 mm diameter extruded rod. After a 15 min thermal



Elastic Bragg intensities Vs photon energy calibration for DAFS

at Ti K-edge (E₀: 4.964 keV) (the same procedure was used at Y K-edge (E_0 : 17.036 keV)





Comparison of DAFS analyses at Ti K-edge between the α -Fe

treatment at 1050°C, a final full day treatment was performed at 800°C to induce the χ -phase precipitation (Fe₃₆Cr₁₂Mo₃Ti₇)



P. Dubuisson *et al.*, ASTM STP 1325 (1999) 882-898 I. MONNET *et al.,* JNM 335 (2004) 311-321

Enhanced mechanical properties due to reinforcement by homogeneous dispersion of nano-sized Y-Ti-O hard particles (~0.2 to 0.6 Wt%) that precipitate from the Fe-Y-Ti-O solid solution during the consolidation

Mean particle radius: ~10 nm (< 25nm)			
	larger oxides ranging 25 – 500 nm		
Mean particle spacing: ~90 nm			

Their composition and structures are still not well understood

DY has been neutron irradiated in Phénix as fuel pin claddings

Perspectives

Information from other techniques such as APT, TEM and XRD may be



useful to help in analyzing the XAFS data.

DAFS evidences:

- That a significant part of **Ti atoms** are **located in** the α -Fe BCC matrix [S. Liu et al., J. Nucl. Mater. 445, 50-56 (2014); P. He et al., J. Mat. Chem. Phys. 136, 990-998 (2012)].
- Both **Ti and Y atoms** (in a lower proportion) are **observed in** the intermetallic **χ-phase** $(1 \ \mu m \ width \ and \ 10 \ \mu m \ length \ with \ a \ volume \ fraction \ estimated$ around 17%), thaught to be a quaternary phase of composition Fe₃₆Cr₁₂Mo₃Ti₇ [C. Zakine et Al., J. Nucl. Mater. **230**, 78-83 (1996)].
- However, DAFS measurements on Y-Ti-O nano-oxides were not possible due to their weak and broadened diffraction peaks.

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