

Stability of Y-Ti-O phases of ODS ferritic steels after neutron irradiation: X-ray diffraction and absorption analyses at synchrotron.

D Menut^{a*}, S. Cammelli^b, J-L Béchade^a, I. Monnet^c, M. Morales^c, D. Chateigner^d, L. Lutterotti^d, B. Sitaud^b, J. Ribis^a, Y. de Carlan^a

^a CEA, DEN, Service de Recherches Métallurgiques Appliquées, 91191 Gif-sur-Yvette, France (*correspondence : denis.menut@cea.fr)

^b Synchrotron SOLEIL, Ligne de lumière MARS, L'Orme des Merisiers, Saint Aubin, BP 48, 91192 Gif-sur-Yvette, France

^c CIMAP-ENSICAEN, UMR 6252, 6 Bd Maréchal Juin, 14050 Caen, France

^d CRISMAT-ENSICAEN, UMR 6508, 6 Bd Maréchal Juin, 14050 Caen, France

The development at CEA of new ferritic/martensitic (F/M) ODS materials for the cladding for GENIV Sodium Fast Reactors and fusion reactor is a key issue [1]. F/M ODS exhibit very low dimensional changes (creep and void swelling) under irradiation up to high doses, higher than 150 dpa. They contain a very high density ($>10^{24} \text{ m}^{-3}$) of Y-Ti-O nanosized particles, responsible of the excellent behavior at high temperature, and favoring the trapping of He in the case of fusion reactor. Nano-phases have been extensively studied by SANS, APT and TEM. However, their exact nature is not yet well understood. They appear to range from coherent solute enriched clusters with complex shell structures as found by APT, to near stoichiometric complex oxides, such as Y_2TiO_5 and $\text{Y}_2\text{Ti}_2\text{O}_7$, as found from SANS and TEM analyses. Nevertheless, for these nano-oxides, the main point is to remain stable under irradiation up to high fluence and high temperature.

Here, we focus on the use of X-ray synchrotron-based techniques operated at the MARS beamline to detect and characterized precisely the Y-Ti-O clusters [2]. The MARS beamline offers unique opportunities for characterizing radioactive materials (α , β , γ and n emitters) with high activities and relative high dose rates. A subassembly with the DY alloy as cladding material was irradiated in the French Sodium Fast Reactor Phenix. DY is a $\text{Fe-13Cr-1.5Mo} + 1\text{TiO}_2 + 0.5\text{Y}_2\text{O}_3$ ODS, developed by SCK/CEN Mol and elaborated by DOUR Metal in the seventies [3]. The irradiation dose was between 0 to 81 dpa and temperatures went from 400 to 580°C along the fuel pin. Four locations along the fuel pin were investigated; with a reference for the non-irradiated state [4]. Thanks to Mars beamline, we combine the long-range order and crystallographic sensitivities of Transmission-XRD with the spectroscopic and short-range order ($\sim 6 \text{ \AA}$) sensitivities of XAFS techniques at the Y-K edge. That allows us to study the evolution of minor yttrium titanate oxide phases versus the irradiation dose using both techniques. Finally, thanks to TEM observations already performed on these samples [4], these analyses allow to get a new insight into the local atomic configuration of the Y-Ti-O nanoclusters.

[1] P. Dubuisson, *et al.*, J. Nucl. Mater. 428, issues 1-3 (2012) 6-12.

[2] J-L Béchade, *et al.*, J. Nucl. Mater. 428, issues 1-3 (2012) 183-191.

[3] A. De Bremaecker, *et al.*, J. Nucl. Mater. 428 (2012) 13-30.

[4] I. Monnet, *et al.*, J. Nucl. Mater. 335 (2004) 311-321.