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

Denis Menut¹, Jean-Luc Béchade¹, Dominique Thiaudière²

¹CEA, DEN, Service de Recherches Métallurgiques Appliquées, 91191 Gif-sur-Yvette, France

²Synchrotron SOLEIL, Ligne de lumière DIFFABS, L'Orme des Merisiers, Saint Aubin BP48, 91192 Gif-sur-Yvette, France

ODS materials are promising candidates for structural components of GEN IV and fusion reactors. Enhanced mechanical properties, especially high creep rupture strength, are due to reinforcement by homogeneous dispersion of nano-sized Y-Ti-O hard particles, ranging from 0.2 to 0.6 Wt%. The detailed characters of the nano-oxides, that precipitate from the Fe-Y-Ti-O solid solution during the consolidation at high temperature of the mechanically alloyed powders, are still not well understood. They are formed with different densities, sizes, compositions, and structures depending both the alloy composition and on thermo-mechanical treatment imposed during the fabrication process. However, the oxides in the Y-Ti-O system that are the most relevant are Y_2O_3 , TiO, TiO_2 , and complex oxides Y_2TiO_5 and $Y_2Ti_2O_7$. Further, Ti has also to be considered in solid solution in the Fe-Cr matrix, which is not the case for Y. Thus, the interpretation of XAFS results for nano-oxides is more challenging than for a homogeneous material, or when the target species are clearly partitioned to a specific phase.

Diffraction Anomalous Fine Structure (DAFS) [1], which measures the elastic Bragg reflection intensities versus photon energy, combines the long-range order and crystallographic sensitivities of x-ray diffraction with the spectroscopic and short-range order sensitivities of x-ray absorption techniques. The kappa diffractometer end-station of the DIFFABS beamline was set-up in reflection-XRD geometry using an energy-resolved Silicon Drift Detector to perform Near Edge (DANES) and Extended (EDAFS) DAFS measurements. These analyses were done both at the Ti K- (4.966 keV) and Y K-edge (17.069 keV). XAFS spectra were recorded simultaneously in X-Ray Fluorescence mode, which is very important for energy calibration during data collection and, in addition, in the case of highly absorbing material, for absorption correction.

In the DY ODS steel analysed (Fe-13Cr-1.46Mo-0.1Al+ 1.0 TiO_2 + 0.5 Y_2O_3 , Wt%) [2], the difference observed between DAFS and XAFS proves that a significant part of Ti atoms are located in the Fe-Cr BCC matrix [3, 4]. Likewise, both Ti and Y atoms were observed in the intermetallic χ -phase (1 μm width and 10 μm length with a volume fraction estimated around 17% [5]) that precipitates mainly on grain boundaries. Unfortunately, DAFS measurements on Y-Ti-O nano-oxides were not possible due to the weak and broadened diffraction peaks.

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