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Introduction

Oxide Dispersion Strengthened (ODS) steels are one of the most promising candidate for cladding materials in generation IV nuclear power plants. Their high swelling resistance induced by the ferritic matrix along with their high creep resistance given by the nano-sized oxides embedded in the matrix make them suitable for fast neutron reactor environment. However, even if ODS steels was widely studied, precipitation kinetics and mechanisms of nano-sized oxides are still misunderstood.

Topic and results

ODS steels with Fe-Cr matrix strengthened by Y-Ti-O nano-oxides are made by powder metallurgy processes. These processes are mandatory to ensure a finely dispersed oxide nano-precipitation due to the very low solubility of Y in a Fe-Cr matrix (which prevent casting elaboration). During the process, powders of Fe-Cr steels are mechanically alloyed with Y_2O_3 oxide and TiH_2 hydride in order to obtain a super-saturated solution of Y, Ti and O inside the matrix. Subsequent high temperature processing (hot extrusion or hot isostatic pressing around 1100°C) induces the precipitation of Y-Ti-O oxides with a typical size of 2nm in diameter leading to suitable mechanical properties.

This study focuses on this precipitation step where the kinetics and mechanisms of precipitation are still misunderstood. For this purpose, synchrotron Small Angle X-ray Scattering (SAXS) has been performed on as-milled cold pressed powders of an ODS steel – in order to avoid precipitation during consolidation – and subsequently annealed in-situ till 1100°C. These experiments allow to monitor the mean radius and the density of precipitates in real time during the thermal treatment as we showed in a preliminary study at 800°C and 900°C [1]. Additionally, the Y content of precipitates has also been probed along in-situ heat treatments using anomalous scattering [2].

In parallel, ex-situ observations using advanced microstructural characterization methods have been carried out to confirm the SAXS results and to obtain complementary observations. Atom Probe Tomography has been performed to access information on the particle chemistry (especially regarding O and Ti, inaccessible by the classical SAXS measurements), and orientation mapping in the transmission electron microscope has been carried out to characterize the evolution of the grain structure and of the density of crystalline defects.

Conclusion

We have achieved the characterization of the precipitation kinetics in different ODS steels and obtained information on chemical composition evolution over a thermal treatment representative of the industrial process. Those measurements were compared between the different ODS steels investigated and with classical manufactured ODS alloys. Those new insights of the precipitates evolution during the process could then represent a step forward on the elaboration process optimization.

References

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