H3. SMALL ANGLE NEUTRON SCATTERING INVESTIGATION OF ODS MARTENSITIC STEELS

M.H. MATHON[1], Y. DE CARLAN[2], P. OLIER[3], L. CHAFFRON[4], C. CAYRON[5], S. UKAI[6], A. ALAMO[2]

[1]LLB(CEA-CNRS), [2]DEN/SRMA, [3]DRT/S3ME, [4]DEN/SRMP, CEA/Saclay, 91191 Gif-sur-Yvette, France
[5]CEA/Grenoble, DTEN/SMP, 17 rue des Martyrs, 38054 Grenoble, France
[6]Nuclear Fuel Research Group, O-arai Engineering Center, JNC 4002, Narita, Ibaraki, 311-1393, Japan

The materials reinforced by oxide dispersion, usually called ODS (Oxide Dispersion Strengthened), have a vast applicability because of their excellent mechanical resistance at medium and high temperatures. In general, ODS alloys are manufactured by mechanical alloying from elementary powders and consolidated by hot extrusion or HIP (High Isostatic Pressure). Iron based ODS could be used for nuclear applications between 550°C and 900°C. Indeed, they present good dimensional stability and excellent resistance to swelling under irradiation due to their body centered cubic structure and also good creep resistance because of the dispersion of nanometric Y_2O_3 oxide particles.
The main objective of this work is to study the evolution of the oxide dispersion during the different stages of the fabrication, that is, after mechanical alloying, consolidation process (extrusion or HIP) and after thermal treatments. For this purpose, Small Angle Neutron Scattering (SANS) experiments were used to characterize the nanometric Y_2O_3 oxide distribution in the matrix. Also, the A ratio of the magnetic and nuclear SANS contrasts between matrix and particles gives information on the chemical composition of the particles.

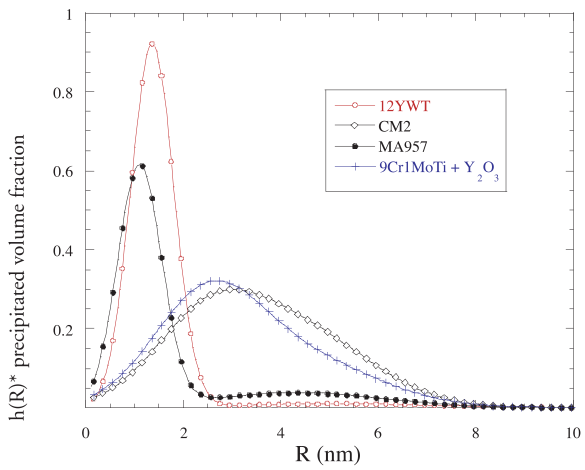

Figure 1 : Y_2O_3 clusters size distribution in consolidated ODS alloys.

In the present study, several commercial or experimental ODS martensitic/ferritic materials have been investigated at consolidated state: MA957 manufactured by INCO metal and the Japanese steel 12YWT (12%Cr) produced by Kobe containing 12% of chromium, and two experimental martensitic alloys presenting a lower chromium content (9%). The SANS experiments show the existence of nanometric (<10 nm) oxides in all materials but their volume fraction depends strongly of the alloy (see figure 1). The ferritic alloy 12YWT which presents exceptional creep-rupture properties, contains the most homogeneous and fine oxide distribution. For the MA957, the volume fraction of very small oxides (radius of 1 nm) is lower. Concerning the low Cr material, the size distribution is larger in relation with their worse creep properties.
In the aim to be able to reproduce and improve the 12YWT steel, different mechanical alloying conditions and thermal treatments were tested.
The volume fraction of small oxides observed after ball milling is higher if the yttrium oxides are introduced as a mixture of Fe_2O_3 and Fe_2Y intermetallics than as Y_2O_3 micro powder. Those small oxides could be the first step of a new precipitation of nano-phases or "the residues" of the mechanical alloying (MA). A heat treatment during 1 hour at 850°C and at 1100°C, induces the precipitation of new nano-oxides with a different chemical composition from the one observed in the MA powder. This result proves that the mechanical alloying produces a partial solid solution super-saturated in yttrium, titanium and oxygen and that a new precipitation occurs during the consolidation treatment. After 1h at 1100°C, the size distribution is quite similar to the Y12WT one…

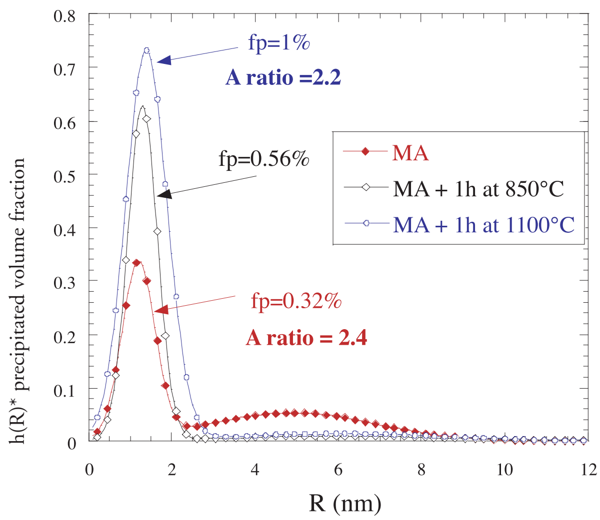

Figure 2 : Y_2O_3 size distribution evolution under thermal treatment.