Structural characterisation of $Y_2 Ti_2 O_7$ dispersoids in ODS alloys

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Oxide dispersion strengthened (ODS) steels are being developed as promising core component structural materials for future generation fast breeder and fusion reactors because of their better high temperature thermal stability and neutron irradiation void swelling resistance as compared to currently used austenitic steels. The type of oxide dispersoids, their size distribution in metallic matrix and stability at adverse service conditions (such as high temperature along with fast neutron irradiation) governs the physical and mechanical properties of the steel. Yttria (Y₂O₃), is the most preferred oxide dispersoid being used in the ODS steels, because of its superior thermal and neutron irradiation stability. However there are reports that show that these oxides either dissolve or dissociate or even become amorphous during mechanical milling and reprecipitate as coarse particles during high temperature consolidation process, in absence of Ti in a model Fe-15Y₂O₃ system [1]. It is believed that Ti can inhibit the growth of nano-dispersoid during annealing by formation of Y-Ti-O complex oxides such as Y₂Ti₂O₇ or Y₂TiO₅ or YTiO₃ [2]. The Y₂O₃ to Ti weight ratio in the alloy is critical in determining the chemical composition of the dispersoid and the Y2Ti2O7 oxides are finer and most stable oxide among all combinations of Y-Ti-O complex oxides whose size which varies in the range of 2-15 nm in ODS steel. Synchrotron XRD is used to characterize the dispersoids in the ODS steel, with 0.35 wt% of yttria and 0.2 wt% of Ti, due to the low volume fraction. TEM has been found more suitable for complete characterization of the nano-sized (~2-5 nm) dispersoids w.r.t size, distribution, morphology, chemical composition and crystal structure. However, characterisation of samples prepared by conventional methods for TEM studies continues to be difficult owing to magnetic nature of ferritic steel. Hence FIB was employed to extract electron transparent samples which are of micrometer dimensions. In order to understand the structural evolution of the Y2Ti2O7 oxide in ODS steel, a concentrated alloys of Fe-15wt%Y2O3-Xwt%Ti (X=0, 5, 10, 15) were synthesized by mechanical milling and subsequently annealed. Figure 1(a) and (b) represents the typical bright field (BF) TEM micrographs of Fe-15wt%Y2O3-15wt%Ti model ODS after 60h of milling and subsequent annealing at 1273K respectively, the corresponding SAD patterns are shown as inset. The analysis of SADP reveals amorphisation of yttria upon milling and recrystallisation of Y₂Ti₂O₇, in annealed alloy powder. Interestingly, it was observed when the Y₂O₃ to Ti weight ratio is 1:1, the oxide phase formed upon annealing is only Y₂Ti₂O₇ and are very finer in size (varies in the range of 2-30 nm). Details of these studies will be presented in the paper.

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