Effects of Zr and Al Additions on the Mechanical Properties of high Cr ODS Steels

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1. Introduction

Next generation nuclear systems require improved core structural materials to realize high efficiency and safety. Ferritic/martensitic steel capable of use at high temperatures is very attractive for the structural materials of fast fission reactors such as a sodiumcooled fast reactor (SFR). However, conventional ferritic- /martensitic steel has limited thermal creeprupture strengths at temperatures above 823K. To achieve higher operating temperatures, an improved elevated temperature strength is required for ferritic-/martensitic steel. Oxide dispersion strengthened (ODS) steel is considered one of the most promising candidate materials for next-generation nuclear systems because of its excellent elevated temperature strength, irradiation resistance, and corrosion resistance. Therefore, the development of Fe-based ODS alloys possessing higher strength and irradiation resistance at elevated temperature for structural materials such as the fuel cladding tube, wire, duct and so on may be inevitable.

The manufacturing of this high performance alloy essentially involves a meta-stable process called mechanical alloying (MA) to disperse the nano-oxide particles finely and uniformly in the matrix. Mixed powders are usually fabricated by hot extrusion (HE) or hot isostatic pressing (HIP) [1]. There have also been many works on optimal compositions based on physical metallurgy, thermal stability to improve the mechanical properties.

In this study, Fe-based ODS alloys were fabricated by HIP and hot rolling processes. Zr and Al were also added and the microstructures and mechanical properties were investigated.

2. Methods and Results

2.1 Experimental procedure

Fe-ODS alloys used in this study are Fe-10Cr-2W-0.35Y₂O₃ and Fe-14Cr-2W-0.25Y₂O₃ in wt% with some minor alloying elements including Ti, Al, and Zr. The ODS alloys were fabricated by mechanical alloying and a hot isostactic pressing (HIP) process. Metallic raw powders and Y₂O₃ powder were mechanically alloyed by a horizontal ball-mill apparatus, CM-08, under ultrahigh purity Ar gas (purity in 99.9999%) atmosphere. The mechanical alloying was performed at an impeller rotation speed of 300rpm for 40h with a ball-to-powder weight ratio (BPWR) of 10:1. After mechanical alloying,

the particle distribution was measured by a laser diffraction scattering method using a particle size analyzer. SEM was utilized to observe the surface morphology of MA powders. MA powders were sieved and charged in stainless steel capsule. Sealed capsules were then degassed at 773K below 5×10^{-3} torr for 2h. The HIP was carried out at 1423K for 4h at a heating rate of 5K/min and following furnace cooling. Hot rolling at 1423K was done in a fixed rolling direction for the plate shape, which resulted in a reduction to a 15mm thickness of the plate. 10wt% Cr ODS alloy was finally heat treated by normalizing at 1323K and tempering at 1073K for 1h to gain the tempered martensite structure. Ferritic ODS alloy with 14wt% Cr was heat treated at 1423K for 1h and followed aircooling. A schematic of the fabrication processing of the ODS alloy is shown in Fig. 1.

For a microstructural observation, ODS alloy was mechanically wet ground and finally polished with colloidal silica. The grain morphology was observed by FE-SEM. Hardness and miniaturized tensile tests were carried out to investigate the mechanical property.

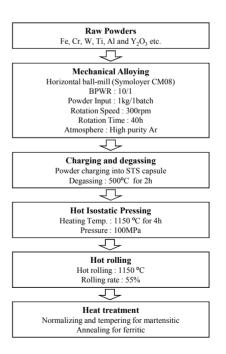


Fig. 1. The schematic of fabrication processing of the ODS alloy plate.

2.2 Microstructural and mechanical property

A powder morphology change depending on the mechanical alloying time, 2h, 4h, 8h, 16h, 32h, and 40h, was shown in Fig. 2. Powders milled for 2h was irregularly spherical and flake shapes, which means various raw powders with different particle diameter were not mixed and milled enough. As the MA time increased, the powders were clearly decreased into a spherical shape. Finally, the MA powders milled for 40h were quite uniform and spherical with 24 μ m of mean particle size.

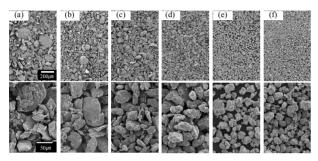


Fig. 2. SEM images of the powder after mechanical alloyed for (a) 2h, (b) 4h, (c) 8h, (d) 16h, (e) 32h and (f) 40h.

To fabricate Fe-based ODS alloy plates, hot isostatic pressing, hot rolling, and a heat treatment process were employed in this study. In Fig. 3, the hardness profile of each process was represented. After the HIP process, the hardness of ODS alloys were about 295Hv. The hardness of 10Cr ODS alloys were significantly increased over 500Hv owing to a martensitic structure formed by air-cooling. In mechanical alloying process, Zr was added as a minor alloying element and was very effective to strengthen the Fe-ODS alloy. When hot rolled, Zr added ODS alloy shows higher hardness than that without Zr. Although two 10Cr ODS alloys showed a similar hardness of about 285Hv after the final heat treatment, it is expected that martensitic ODS alloy with Zr potentially demonstrates the high strength. Consequently, the proper heat treatment condition should be optimized and a chemical composition modification is also necessary.

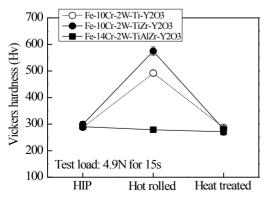


Fig. 3. Hardness changes of ODS alloys during fabrication process

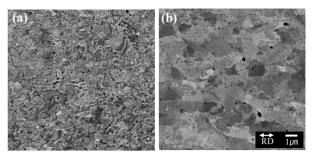


Fig. 4. Microstructures of Fe-14Cr ODS alloy (a) as HIPed and (b) hot rolled .

The hardness of the 14Cr ODS alloy was quite stable because of a full ferrite phase even during heat treatment. Finally, 14Cr ODS alloy with Zr exhibited 280Hv despite Al addition. Al is well known as a very effective element to improve the formability, while the strength inevitably decreased. Ohnuki et al. reported that the oxide particles were significantly decreased by a small addition of Zr in Al-added high Cr ODS ferritic steel, and it is responsible for the formation of a fine $Y_2Zr_2O_7$ complex oxide instead of coarse YAlO₃ [2]. In Fig. 4. microstructures of 14Cr ODS allov are shown. Comparably coarsened and equiaxed grains are uniformly distributed in as hipped 14Cr ODS alloy. After hot rolling, the grains were elongated toward the rolling direction with a fine grain size. To characterize the ODS alloys, more detailed investigations including grain morphology, oxide particle distribution, and the tensile property are on-going and will be presented.

3. Conclusions

Some Fe-based ODS alloys were fabricated by the HIP process and their microstructures and mechanical properties were investigated. Zr and Al are considered to be very effective alloying elements for high strength and formability in Fe-based ODS alloys. These preliminary results will be useful to develop advanced ODS steel.

Acknowledgements

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