Effect of Silicon on the Microstructures and Tensile Properties of Austenitic ODS Stainless Steels for Fast Reactor Cladding

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1. Introduction

Oxide dispersion strengthened (ODS) steels have excellent high temperature mechanical properties due to the presence of thermally stable nano-scale oxides distributed in their matrix [1]. Therefore, ODS steels are being used for high temperature structural applications and ODS ferritic martensitic steels (FMS) have been considered as candidate cladding and structural materials for the Generation-IV fast reactors [2]. Generally, fabrication processes of ODS steels have incorporated a mechanical alloying (MA) process, in which repeated fracture and welding of mixed powders occur by a high energy impact of steel balls. Although MA has many advantages in forming a nano-scale microstructure, it is a very long-time expensive process and vulnerable to impurities contamination. High oxygen and carbon contents degrade the high temperature strength and creep strength of ODS steels [3].

To overcome the problem of MA process, AISI 316Lbased austenitic ODS steels were fabricated by a wet mixing of metallic salts [4]. This method dispersed oxide particles by thermal decomposition of metallic salt during fabrication process. Austenitic ODS steel could be fabricated successfully by a wet-mixing process of 316L stainless steel powder in yttrium containing salt solution. Wet-mixed ODS steel had lower carbon and oxygen contents than that of MA-ODs steel, because minimum inflow of carbon and oxygen during the manufacture process was kept in wet-mixed ODS steel. Ryu. *et. al* was reported that yttrium silicates were formed by reaction of silicon and yttrium oxide during the fabrication process.

In this study, we made simulated 316 stainless steel powder without adding silicon by using MA and then fabricated ODS steel by using a wet process in order to analyze the effect of silicon on the microstructures and tensile properties of the austenitic ODS stainless steels.

2. Experimental procedures

A flow chart of experimental procedures is shown in Fig. 1. Commercially available AISI 316L stainless steel powders were mechanically alloyed (MA) with 0.25 wt% Y_2O_3 powders by using a planetary-type ball mill with 200 rpm for 12 hours under the argon atmosphere, in order to fabricate a reference mechanically alloyed powder. ODS stainless steels by a wet mixing were fabricated by the following procedures. Yttrium nitrate (Y(NO₃)₃•6H₂O) was dissolved in

distilled water at room temperature. 316L stainless steel powder was wet-mixed in the yttrium containing solutions. Two kinds of 316L powder were used in order to investigate the effect of silicon on the oxide formation during the fabrication process. One is a commercially available 316L powder and the other is a mechanically alloyed 316L powder by using elemental powders without silicon. After drying the wet mixture, ball mixing was carried out for a homogeneous mixing of the 316L stainless steel powder and yttrium salts. Dried powders in each process were placed in an AISI 304L stainless steel container, sealed after a degassing process at 500°C and consolidated by a hot isostatic pressing (HIP) at 1150°C under a pressure of 103 MPa. HIPed bars were hot rolled at 1150°C and then heattreated for normalizing at 1150°C for 1 hour. Microstructures of the specimens were observed by using a transmission electron microscopy (TEM) and the elemental compositions of the interaction layers were measured by using an energy dispersive X-ray spectroscopy (EDS). High temperature tensile tests were carried out at 700°C to measure the mechanical properties such as yield strength and elongation.



Fig. 1. Fabrication process of the austenitic ODS steels without silicon.

3. Results and discussion

According to EDS analyses and size measurement of oxide particles from TEM images obtained from carbon replica samples of austenitic ODS steels, oxide particles were composed of yttrium, silicon and oxygen and have a size range of about $150 \sim 700$ nm. MA ODS steel showed little finer oxide particles than wet-processed ODS steel. But, the austenitic MA ODS steels formed a larger size of oxide particles when compared with previously reported ferritic martensitic ODS steels [5].

Fig. 2(a) shows a TEM image of oxide particles in an austenitic ODS steel without silicon. An austenitic ODS steel without silicon shows much smaller size of oxide particles when compared with previously manufactured austenitic ODS steels by using commercially available 316L stainless steel powder containing 0.8 wt% Si. As shown in Fig. 2 (b), oxide particle of austenitic ODS steel without silicon was composed of yttrium, chromium and oxygen. Fig. 3 shows a size distribution of oxide particles in the austenitic ODS steel without silicon. Most of the oxide particles in the austenitic ODS steel without silicon were less than 50 nm.

High temperature tensile tests of a hot-rolled specimen at 700°C showed that wet-processed ODS steels have tensile properties similar to the MA ODS steels. Because both steels were processed at the same HIP and hot rolling temperature, their grain structure and oxide distribution, which are key factors of their tensile properties, were not much different in each steels. Coarsening of a complex oxide of yttrium and silicon significantly affected the tensile properties in the 316L-based ODS steels. Wet-processed ODS steels showed a larger elongation when compared to mechanically alloyed ODS steels. In case of an austenitic ODS steel without silicon, higher yield strength was obtained due to the presence of fine oxides around 20 nm in average size. It is necessary to optimize silicon content to control the oxide size in austenitic ODS steel.



Fig. 2. An austenitic ODS steel without silicon: (a) a TEM image and (b) chemical composition of an oxide particle marked by a black dotted circle in (a).



Fig. 3. Size distribution of oxide particles in an austenitic ODS steel without silicon.



Fig. 4. Comparison of high temperature tensile properties of austenitic ODS steels at 700° C.

4. Conclusions

In case of 316L ODS steels containing 0.8 wt% silicon, Y-Si-O complex oxide particles formed and have a size of about $150 \sim 700$ nm. High temperature strength and elongation were not much increased when compared to commercial 316L steels because most oxide particles were too large to inhibit dislocation motion effectively. On the other hand, in case of 316 ODS steel without silicon, very fine complex oxide particles around 20 nm were formed after a fabrication process and higher yield strength can be obtained because of the presence of fine oxides.

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