

Study on the Hot Extrusion Process of Advanced Radiation Resistant Oxide Dispersion Strengthened Steel Tubes

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1. Introduction

The core structural materials of next generation nuclear systems are expected to operate under extreme environments, i.e., both high temperatures and dose rates. In a sodium cooled fast reactor (SFR), the coolant outlet temperature and peak temperature of the fuel cladding tube will be about 545°C and 700°C with 250 dpa of a very high neutron dose rate. To realize this system, it is necessary to develop an advanced structural material having high creep and irradiation resistance at high temperatures [1]. Austenitic stainless steel is considered to have good strength and corrosion resistances; however, irradiation swelling severely occurs at 120dpa at high temperatures. This eventually leads to a decrease of the mechanical properties and dimensional stability [2]. Ferritic/martensitic steel has a better thermal conductivity and swelling resistance than austenitic stainless steel. Unfortunately, the available temperature range of ferritic/martensitic steel is limited at up to 650°C [3]. Oxide dispersion strengthened (ODS) steels have been developed as the most prospective core structural materials for next generation nuclear systems because of their excellent high strength and irradiation resistance [1, 4]. The material performances of this new alloy are attributed to the existence of uniformly distributed nano-oxide particles with a high density, which is extremely stable at high temperature in a ferritic/martensitic matrix. This microstructure can be very attractive in achieving superior mechanical properties at high temperatures, and thus, these favorable microstructures should be obtained through the controls of the fabrication process parameters during the mechanical alloying and hot consolidation procedures.

In this study, a hot extrusion process for advanced radiation resistant ODS steel tube was investigated. ODS martensitic steel was designed to have high homogeneity, productivity, and reproducibility. Mechanical alloying and hot consolidation processes were employed to fabricate the ODS steels. A microstructure observation and creep rupture test were examined to investigate the effects of the optimized fabrication conditions.

2. Methods and Results

2.1 Experimental procedure

In order to obtain excellent mechanical properties, it is very important to fabricate microstructures with homogeneously distributed nano-oxide particles as well as fine grains. Advanced radiation resistant ODS steel has been design to have homogeneity, productivity, and reproducibility. For these characteristics, the martensitic phase is more favorable than the ferrite because of the availability of the isotropy microstructure by a transformation with a high density of lath grain structures. Many of the alloys for nuclear power plant application have 8~11wt% of Cr with a minor portion of C. Mo is known to be a good solid-solution element for high temperature strength in heat resistant alloys. Based on this design concept, ODS steels used in this study mainly contained Fe(bal.)-10Cr-1Mo-0.1C in wt%. Some minor elements such as Ti, Zr and Ni were added to obtain the phase stability and precipitate refinement. The ODS steel was fabricated by mechanical alloying and a hot consolidation processes. Mechanical alloying is an essential process in that the continuous collisions between the grinding media and raw powders with a high revolving energy makes repeated crushing and cold welding of the powders, which eventually create a homogenous mixing and alloying in the constitution elements. Metallic raw powders and Y₂O₃ powder were mechanically alloyed by a high energy horizontal ball-mill apparatus, a Simoloyer CM-20. Mechanical alloying atmospheres are thoroughly controlled in ultra-high purity argon (99.9999%) gas. The mechanical alloying was performed for 40h with a ball-to-powder weight ratio of 10:1. High strength carbon steel balls were used as a grinding media for the process. Milled powders were then sieved and charged in a stainless steel capsule. All powder handling processes for the weighing, collecting, sieving, and charging were conducted in a completely controlled high purity argon atmosphere to prevent the oxygen contamination during the process. Sealed capsules were then degassed at 400°C below 5×10^{-4} torr for 3h. The hot isostatic pressing was carried out at 1100°C for 3h at a heating rate of 5°C/min and following furnace cooling. Hot rolling at 1100°C was done in a fixed rolling direction for a plate shape with an 80% reduction of the total rate. Hot extrusion is also one of the consolidation methods. After annealing in the furnace at 1100°C for 2h, the capsules were extruded by a 600ton capacity press for several seconds with a 6.3:1 extrusion ratio. The grain morphology was observed through FE-SEM. Thin foil specimens fabricated using an electro-jet polishing

method were used to observe the precipitate distribution by a TEM.

2.2 Mechanical alloying and hot consolidation processes

The surface morphologies of the powder after the mechanical alloying process are shown in Fig. 1. Powders milled with a steady revolving condition were irregularly spherical and flake shapes, as shown in Fig. 1(a). The partial flake shape with various diameters normally gives a disadvantage in the uniformity of the constitution elements. This eventually leads to a partial large grain structure and deterioration of the mechanical properties. However, a modified mechanical alloying condition has been developed in advanced radiation resistant ODS steel. Continuous operation with low and high collision energy between raw powders and grinding media is a key technology. This process was successfully applied to fabricate mechanical alloying powder with very fine and homogeneous particles, as shown in Fig. 1(b).

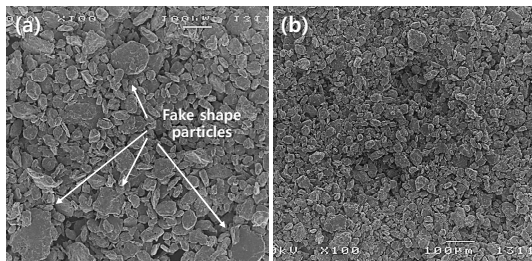


Fig. 1 Surface morphology of mechanical alloyed powders milled with (a) steady rotating condition and (b) modified continuous condition

Meanwhile, a hot consolidation process is also one of the important fabrication processes for high strength ODS steels. Preliminary study revealed that the optimum temperature for the hot consolidation of ODS steel with a favorable microstructure is 1100°C. The microstructures of advanced radiation resistant ODS steel are shown in Fig. 2. Martensitic grain was extremely fine and uniform below 1µm. Nano-oxide particles were also homogeneously distributed in the matrix and their diameters were measured below 10nm.

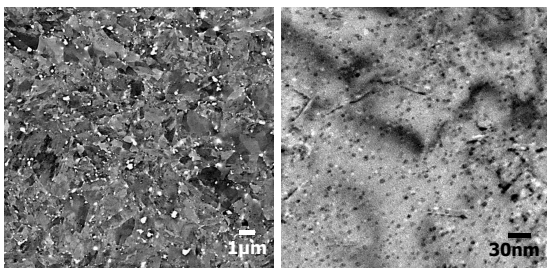


Fig. 2 Microstructure of advanced radiation resistant ODS steel.

2.3 Process development toward ODS steel tube fabrication

After normalizing heat treatment, advanced radiation resistant ODS steel showed high hardness of about 800Hv. This is too hard to perform the cold working process for the tubing. However, furnace cooling heat treatment with a diffusional transformation at austenitic temperature makes the process quite easy. The outward appearances of the hot extruded ODS steels and mother tubes are shown in Fig. 3. The ODS steel rods were hot forged for the axis straightening and followed furnace cooling heat treatment. Through this process, the hardness could be lowered to 250Hv, which is a sufficient hardness level for the tubing process. The

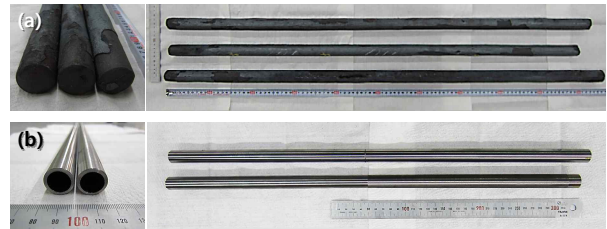


Fig. 3 Outward appearances of (a) hot extruded rods and (b) mother tubes of advanced radiation resistant ODS steel. fabrication process for the tubing is currently set up and has progressed in earnest.

3. Conclusions

Advanced radiation resistant ODS steel has been designed to have homogeneity, productivity, and reproducibility. For these characteristics, modified mechanical alloying and hot consolidation processes were developed. Microstructure observation revealed that the ODS steel has uniformly distributed fine-grain nano-oxide particles. The fabrication process for the tubing is also being propelled in earnest.

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