High temperature creep strength of Advanced Radiation Resistant Oxide Dispersion Strengthened Steels

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1. Introduction

Sodium cooled fast reactor is considered to be one of the next generation nuclear systems with enhanced economics, stability, and reliability. For realization of this system, it is inevitable to develop the advanced structural material having both high strength and irradiation resistance at high temperatures [1]. Austenitic stainless steel may be one of the candidates because of good strength and corrosion resistance at the high temperatures, however irradiation swelling well occurred to 120dpa at high temperatures and this leads the decrease of the mechanical properties and dimensional stability [2]. Compared to this. ferritic/martensitic steel is a good solution because of excellent thermal conductivity and good swelling resistance. Unfortunately, the available temperature range of ferritic/martensitic steel is limited up to 650°C [3]. ODS steel is the most promising structural material because of excellent creep and irradiation resistance by uniformly distributed nano-oxide particles with a high density which is extremely stable at the high temperature in ferritic/martensitic matrix [1, 4]. In this study, high temperature strength of advanced radiation resistance ODS steel was investigated for the core structural material of next generation nuclear systems. ODS martensitic steel was designed to have high homogeneity, productivity and reproducibility. Mechanical alloying, hot isostactic pressing and hot rolling processes were employed to fabricate the ODS steels, and creep rupture test as well as tensile test were examined to investigate the behavior at high temperatures.

2. Methods and Results

2.1 Experimental procedure

ODS steels used in this study are Fe-9Cr and Fe-15Cr ODS steels. The former has a martensitic phase by a phase transformation from an austenitic temperature with a rapid cooling rate. The later has fully ferritic phase due to high Cr concentration more than 12wt%. The ODS steel were fabricated by mechanical alloying and hot isostactic pressing (HIP) and hot rolling processes. Mechanical alloying (MA) is essential process that the continuous collision between grinding media and raw powders with a high revolving energy

makes the repeated crushes and cold welding of powders, which eventually create the homogenous mixing and alloying in the constitution elements. Metallic raw powders and Y2O3 powder were mechanically alloyed by a high energy horizontal ballmill apparatus, Simoloyer CM-20. Mechanical alloying atmospheres are thoroughly controlled in ultra-high purity argon (99.9999%) gas. The mechanical alloving was performed at an impeller rotation speed of 240rpm for 40h with a ball-to-powder weight ratio of 10:1. Chemical composition and oxygen concentration of MA powders were analyzed by an ICP-AES and KS D 1778 methods, respectively. MA 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 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 HIP was carried out at 1150°C for 3h at a heating rate of 5°C/min and following furnace cooling. Hot rolling at 1150°C was done in a fixed rolling direction for a plate shape with 65% of a total reduction rate. The grain morphology was observed by FE-SEM. Specimens for mechanical property evaluations were taken out in rolling direction. Creep rupture test were carried out at 700°C.

2.2 Microstructures of advanced radiation resistant ODS steel

The optical microscopic image on advanced radiation resistant ODS steels and martensitic steel without Y₂O₃ are shown in Fig. 1. Microstructure of martensitic steel showed a typical tempered martensite with prior austenite grains and fine lath structures. This is a representative microstructure which is shown in 9Cr-1Mo or 9Cr-2W heat resistant ferritic/martensitic steels. The effect of Y₂O₃ addition by a mechanical alloying and hot consolidation processes gives dramatic evolution in the microstructures. Homogeneous grains were observed, however its grain size was too fine to determine as shown in Fig. 1(b). Precipitation status was also guite different between martensitic steel and ODS steel. In Fig. 2, bright field TEM micrographs of precipitate morphologies in two alloys are shown. The martensitic steel had carbides, M23C6 (M=Cr, Fe, W, Mo) in several tens of nm. This is usually precipitated in grain boundaries. Fine carbonitrides also precipitated as

forms of MX and M_2X (M=V, Nb, Ta) in the lath and lath boundary. Contrast to this, ODS steel has extremely fine oxide particles which precipitate uniformly in the grains as shown in Fig. 2(b). Grain refinement of ODS steels is attributed to grain growth and grain boundary migration suppression by uniformly distributed nanooxide particle which called as 'pinning effect'. Oxide particle plays an important role for nucleation site and suppress the grain growth during hot consolidation process.



Fig. 1 The optical microscopic images on the (a) Martensitic steel and (b) ODS martensitic steel.



Fig. 2 Bright field TEM micrographs of (a) $M_{23}C_6$ carbides in martensitic steel and (b) $Y_2Ti_2O_7$ complex oxides in ODS martensitic steel.

2.3 High temperature strength of advanced radiation resistant ODS steel

Tensile strengths of ferritic steel and ODS ferritic steel at room and elevated temperature were shown in Fig. 3. As temperature increased, all tensile strengths were decreased, however strength of ODS ferrtic steel was exceptionally higher than ferritic steel. This is due to effects of grain boundary and precipitation hardening. ODS steel normally has fine grains with nano-sized complex oxides as coincided with the results of microstructure observation. Especially, oxide particle is very stable even in the high temperature up to 1300°C and usually acts as an obstacle for the dislocation gliding when the material deforms [5]. In contrast with nano-sized complex oxide of ODS steels, carbides were known to be unstable at high temperature, which are coarsened or dissolved in the matrix.

Creep rupture tests of the ODS steels were performed at 700°C under various loads between 100 and 170MPa.

The test results are plotted on log-log scale in Fig. 4. The creep strength of ODS steels is more superior than the ferritic/martensitic steel. While creep strength of austenitic stainless steel before 10^{3} h is higher than ODS steel, this behavior reversed after 10^{4} h. The creep resistance of ODS steels overwhelmed ferritic/martensitic steel, austenitic stainless steel and even a conventional ODS steel. This is attributed to the presence of uniformly distributed nano-oxide particle induced by modified continuous mechanical alloying condition.



Fig. 3 Tensile strengths of ferritic steel and ODS ferritic steel.



Fig. 4 Creep strength of ODS steels at 700°C under various loads.

3. Conclusions

ODS steels were fabricated by a mechanical alloying and hot consolidation processes. Mechanical properties at high temperatures were investigated. The creep resistance of advanced radiation resistant ODS steels was more superior than those of ferritic/ martensitic steel, austenitic stainless steel and even a conventional ODS steel.

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