Effects of Heat treatment on Microstructure and Tensile Strength of 10Cr-ODS Steels.

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1. Introduction

An Oxide Dispersion Strengthened (ODS) steel has uniformly distributed nano scale oxide particles in microstructure. Due to their own microstructure, ODS steels have excellent creep and irradiation resistance at the high temperature under a neutron irradiation environment [1-3]. Because of these performance, ODS steels for nuclear power reactor is widely researched.

Advanced Radiation Resistant ODS Steel (ARROS) is being developed as a fuel cladding tube materials for future nuclear reactor system. ARROS with a chemical of Fe-10Cr-1Mo-0.5Mn-0.1V-0.25Ticomposition 0.35Y₂O₃ has higher strength and creep resistance than common Fe based ODS materials. ARROS is now researching about modifying composition and optimizing the fabrication process for commercialization in Korea Atomic Energy Research Institute (KAERI).

In this study, attempts are made to optimize the fabrication process for modified ODS steel which has better high temperature properties than existing ARROS. As rolled ODS steel samples were annealed at variety temperature. Their microstructure and tensile properties after annealed process were investigated.

2. Experimental Procedure

2.1 Materials

Bulk ODS steels were produced in a cylindrical shape by mechanical alloying and hot consolidation process, and their chemical composition is shown in Table I. In mechanically alloying process, metallic raw powders and oxide (Y_2O_3) powder were alloying themselves by a high energy horizontal ball milling equipment (Model; Simoloyer CM-20). The used powder of base material has mean diameter 85µm and 99% purity. Only the Y₂O₃ oxide powder with the size of 25~50 nm and 99.995% high purity was used for finely disperse in matrix during fabrication process. The mechanical alloving was progressed for 48 hours with a ball-topowder weight ratio 10:1 with 240 rpm at the argon atmosphere. After the mechanical alloying process, fabricated powders were charged in a 304 stainless steel capsule and sealed. Sealed capsules were degassed at 500°C with high vacuum condition below 10⁻⁴ torr. After the degassing process, high purity Ar gas flow was put into capsule for controlling oxide content by

reduction reaction. The hot isostatic pressing was carried out at 1150° C for 4 hours. The heating rate is 5° C /min and cooling in the furnace.

Table 1: Chemical composition of the ODS steels.

	Fe	Cr	Мо	Mn	V	Ti	С	Y_2O_3
ODS steel	Bal.	10	1.0	0.5	0.15	0.25	0.13	0.35

2.2 Rolling and Heat treatment

Hot rolling and annealed process were made to fabricated samples after HIP process. To stabilize the microstructure, solution treatment was given at 1150°C. Then cooling in air to 1050°C and hot rolled proceeded with holding the temperature. Rolled plate shape samples were annealed at variety temperature.

2.3 Microstructural Analysis

The ods steel specimens were polished and etched and microstructures were observed by an optical microscope and a scanning electron microscope. Their volume fractions were measured by an image analyzer (model; SigmaScan Pro ver. 4.0, Jandel Scientific Co., Erkrath, Germany). Electron back-scatter diffraction (EBSD) analysis (step size; 0.1 μ m) was conducted by a field emission scanning electron microscope, and the data were interpreted by an orientation imaging microscopy (OIM) analysis software provided by TexSEM Laboratories, Inc.

2.4 Tensile Tests

Plate type tensile specimens (gage width; 2 mm, gage length; 9 mm, gage width; 0.5mm) were prepared, and were tested 3 times at least for each condition at room temperature and 700 °C at a strain rate of 0.3×10^{-4} /s by a universal testing machine. The 0.2% offset stress was determined to be the yield strength.

3. Results and Discussion

3.1 Microstructure

Figure 1 is SEM micrographs of ODS steels after the normalizing at 1050 and 1150 °C. Two microstructures of ODS steels have both martensitic microstructure. The grain size with normalized at 1150 °C is larger than one with normalized at 1050 °C.



Fig. 1. Microstructure of ODS steels normalized at $(a)1050^{\circ}C$ and $(b)1150^{\circ}C$ with 1 hours.

Figure 2 (a) through (c) are SEM micrographs of ODS steels after the tempering with various conditions. All three microstructure show typical tempered martensite structure with dispersed carbides along grainboundaries. In Fig. 2(b), grain size increased although the tempering temperature decreased. This is because grain growth inhibition effect by precipitates is decreased in low tempering temperature.



Fig. 2. Microstructure of ODS steels tempered with (a) 780° C, 1 hour, (b) 720° C, 1 hour and (c) 780° C, 2 hours.

With increasing time for tempering, increasing the size of precipitates (Fig. 2(c)). It is expected that this type of precipitation (coarse) is less effective for precipitation hardening than fine and lot dispersed case.

3.2 Hardness and Tensile properties





Fig. 3. Hardness variation of ODS steels with changing heat treatment conditions.

In Fig. 3, hardness variation of ODS steels with changing heat treatment conditions. In the first diagram, at normalizing temperature increase to more than 1050°C, the hardness is decreasing. This results is correlated with microstructure in Fig. 1(b). With increasing normalizing temperature, grains size also increasing and because of this, the mechanical properties are decreased. The change of hardness by tempering temperature and time is also coincidence with prediction by microstructure. The hardness is decreased at low tempering temperature because of low precipitation. Meanwhile, time for tempering is more than 1 hour, the coarse carbide rather than fine carbide is precipitated. In this case also the hardness is decreased.

4. Conclusions

In this study, the microstructure change by varying heat treatment condition is analyzed with tests for mechanical properties. Microstructure is controlled by controlling variety variable for heat treatments (normalizing temperature, tempering temperature, etc.). It is expected this study is useful for optimizing the fabrication method of producing ODS steels.

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