Transactions of the Korean Nuclear Society Autumn Meeting
Gyeongju, Korea, October 25-26, 2012

Effects of oxygen concentration on the characteristics of oxide particles in ODS steels

Tae Kyu Kim*, Chang Hee Han, Byoung-Kwon Choi, Ki-Baik Kim, Young-Bum Chun, Sanghoon Noh, Suk Hoon Kang, Jinsung Jang, Yong-Hwan Jeong

Nuclear Materials Division, KAERI, 1045 Daedeok-daero, Yuseong, Daejeon 305-353, Korea
** tkkim2@kaeri.re.kr*

1. Introduction

Oxide dispersion strengthened (ODS) steel is being considered as a candidate structural material in future nuclear fission and fusion reactor systems mainly due to its excellent creep resistance and irradiation resistance at high temperatures [1,2]. ODS steel normally contains an exceptionally high oxygen concentration owing to the elemental powders themselves, as well as to the contamination during mechanical alloying (MA) and consolidation. It has been reported that excess oxygen concentration affects the mechanical properties of ODS steel. However, little attention has been paid to the effect of oxygen concentration on the characteristics of oxide precipitates.

This study focuses on the effects of oxygen concentration on the characteristics of oxide precipitates in ODS steel.

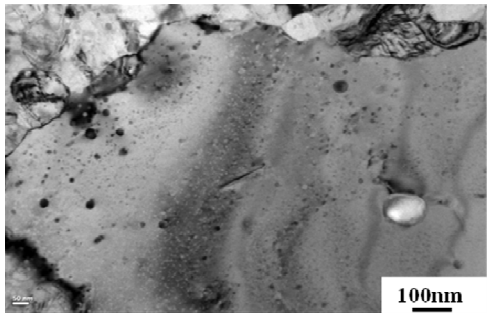
2. Methods and Results

2.1 Experimental procedure

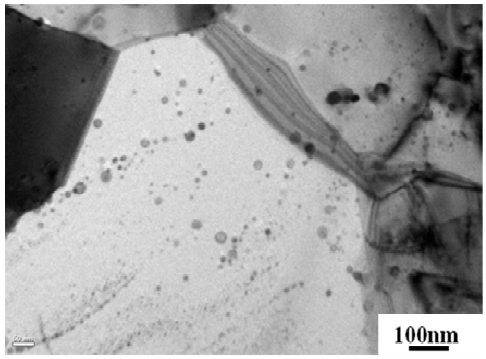
12Cr ODS steel was designed and fabricated by mechanical alloying and hot isotropic pressing (HIPing) processes. The metal powders along with Y_2O_3 powders were mechanically alloyed using a Pulverisette-5 planetary mill at 200 rpm in a high purity argon gas atmosphere for 12 hr. A ball to powder ratio of 15:1 was applied. After the ball milling process, the MA powders were transferred to a steel can. The oxygen content in one ODS steel sample A was about 7000 ppm, while that in the other sample B was controlled to be about 3000 ppm by a hydrogen reduction process prior to consolidation. Degassing was then performed at 500ºC for 2 h, and hipping at 1150^{o}C under a pressure of 100 MPa for 4 h followed. The precipitates taken from the carbon extraction replicas were examined by using a transmission electron microscope (TEM) with an energy dispersive spectroscope (EDS). The carbon extraction replicas were prepared by means of mechanical polishing and etching with a mixed solution of 5% perchloric acid and 95% ethanol, carbon coating, and removing the replicas by electrochemical etching with a mixed solution of 90% methanol and 10% hydrochloric acid. The size distribution of the precipitates was measured by using an image analyzer.

2.2 Microstructure

Fig. 1 shows the TEM images of the precipitates obtained from carbon extraction replica samples in ODS steel samples A and B. Sample A was fabricated without a reduction process, while sample B was reduced by 5vol.% H_2 and 95vol.% Ar mixed gas with a flow rate of 2 liter/min at 500ºC for 1 h. Sample A appeared to have a small grain size while sample B exhibited equiaxed ferritic grains with a mean size of 1 μm.



(a)

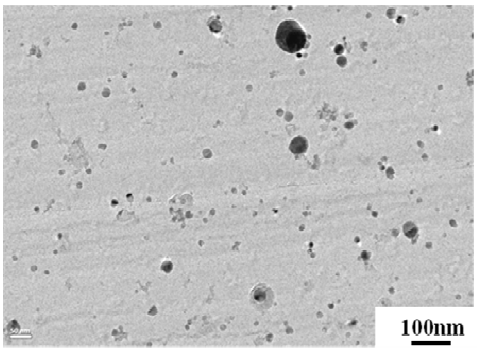


(b)

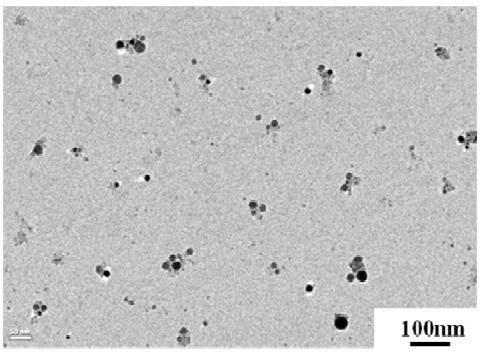
Fig. 1. TEM images of the as hipped 12Cr ODS steel samples (a) A and (b) B.

The TEM images of oxide precipitates taken from carbon replica samples are shown in Fig. 2. Fine Y-Ta-Cr-O and large Cr-V-O type oxide particles were found in

sample A, but mostly fine Y-Ta-Cr-O type oxide particles were found in sample B. These results indicate that the oxygen concentration has a significant effect on the characteristics of oxide precipitates in ODS steel. The precipitate size distribution was calculated from the particles in the area of analysis from TEM images. Excess oxygen is an important alloying element of ODS steel and controlling excess oxygen concentration turned out to be a key point to achieve high mechanical properties. These results show that hydrogen reduction by flowing gas is an effective method to control the excess oxygen concentration in ODS steel.



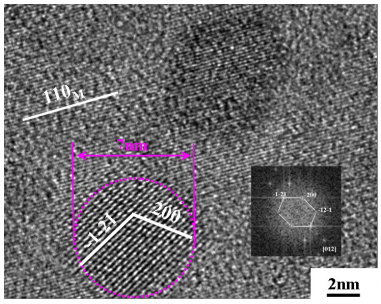
(a)



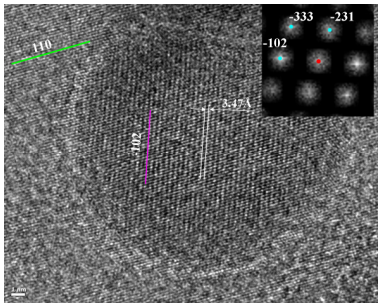
(b)

Fig. 2. TEM images of oxide precipitates in the as hipped 12Cr ODS steel samples (a) A and (b) B.

The HRTEM analysis results of fine oxide particles in the as-hipped sample B are shown in Fig. 3. These fine $YTaO_4$ and $YCrO_3$ particles with a size less than about 10 μm were revealed to be coherent with the matrix. In many types of ODS steel, fine oxide particles such as Y_2O_3 have been found to be coherent with the matrix [3].



(a)



(b)

Fig. 3. HRTEM analysis results of the oxide particles (a) $YTaO_4$ and (b) $YCrO_3$.

3. Conclusions

Effects of oxygen concentration on the characteristics of oxide particles in ODS steel were evaluated. The observation of oxide precipitates in ODS steel prepared with and without hydrogen reduction indicated that the hydrogen reduction process significantly reduced the mean particle size of the precipitates and eliminated large Cr-rich oxide particles in the ODS steel, which is prospective for preparing ODS steel of high mechanical properties.

Acknowledgements

This work was supported by the National Research Foundation(NRF) grant funded by the Korea government(MEST) (No. 2012M2A8A1027872).

REFERENCES

[1]. S. Ukai, T. Nishida and T. Okuda, R&D of oxide dispersion strengthened ferritic martensitic steels for FBR, Journal of Nuclear Materials Vol. 258–263, p. 1745, 1998

[2]. S. Ukai, M, Harada and H. Okada, Alloying design of oxide dispersion strengthened ferritic steel for long life FBRs core materials, Journal of Nuclear Materials, Vol. 204, p. 65, 1993

[3]. M. Klimiankou, R. Lindau, A. Moslang, J. Cryst. Growth, Vol. 249, p. 381, 2003.