Arch. Metall. Mater. **65** (2020), 4, 1265-1268

DOI: 10.24425/amm.2020.133681

CHUN WOONG PARK[1], WON JUNE CHOI[1], JONG MIN BYUN[2**], YOUNG DO KIM[1,3*]

EFFECTS OF ADDING NIOBIUM AND VANADIUM TO Fe-BASED OXIDE DISPERSION STRENGTHENED ALLOY

In this study, the effects of adding niobium and vanadium to Fe-based oxide dispersion strengthened alloys are confirmed. The composition of alloys are Fe-20Cr-1Al-0.5Ti-0.5Y_2O_3 and Fe-20Cr-1Al-0.5Ti-0.3V-0.2Nb-0.5Y_2O_3. The alloy powders are manufactured by using a planetary mill, and these powders are molded by using a magnetic pulsed compaction. Thereafter, the powders are sintered in a tube furnace to obtain sintered specimens.

The added elements exist in the form of a solid solution in the Fe matrix and suppress the grain growth. These results are confirmed via X-ray diffraction and scanning electron microscopy analyses of the phase and microstructure of alloys. In addition, it was confirmed that the addition of elements, improved the hardness property of Fe-based oxide dispersion strengthened alloys.

Keywords: Fe-based ODS, vanadium, niobium, superalloy, hardness

1. Introduction

The usable temperature range, stability under high temperatures, and irradiation resistance of Fe-based oxide dispersion strengthened (ODS) alloys are higher than those of conventional Fe-based superalloys [1-5]. In a Fe-based ODS alloy, fine oxide particles are uniformly dispersed in a matrix, these particles not only provide excellent stability under high temperatures, but they also effectively inhibit the dislocation movement, thereby considerably improving the high-temperature mechanical properties such as tensile and creep resistances [6,7]. Major factors that determine the properties of ODS alloys include the size and density of the dispersed oxide particles, grain size, and dislocation density during the ball milling process. For the same amount of added oxide particles, smaller the size of the oxide particles, larger the number of oxide particles per unit area, which improves the properties [8,9].

ODS alloys cannot be manufactured via casting process because the oxides have to be finely and uniformly dispersed. Therefore, ODS alloys are manufactured via powder metallurgy that includes mechanical alloying. Typically, the powder of each element is added. Mechanical alloying requires a large amount of impact energy, thus high-energy milling processes are carried out using a mill such as the planetary mill or the attritor mill [10,11].

Among the various ODS alloys, Fe-based ODS alloys are applied in the nuclear reactors such as the nuclear fission and fusion reactors. These fields require extreme environments, and various studies are being conducted to satisfy these properties [12,13].

In this study, the effects of niobium and vanadium on Fe-based ODS alloys were examined. The addition of niobium to a Fe-based ODS alloy increases the strength and the diffusion activation energy of the alloys [14]. As the activation energy increases, the diffusion rate decreases, which slows the grain growth maintaining the properties of the alloy for a longer time. The addition of vanadium, which is a high-radioactive element, to a Fe-based ODS alloy improves the irradiation resistance of the alloy [15]. Vanadium is an additive element that can retain the properties of the alloy for a prolonged period by suppressing the grain growth through the reduction in the diffusion rate.

2. Experimental procedure

In this study, 0.3 wt.% of vanadium and 0.2 wt.% of niobium were added to the basic composition, Fe-20Cr-1Al-0.5Ti-

1 HANYANG UNIVERSITY, DEPARTMENT OF MATERIALS SCIENCE AND ENGINEERING, SEOUL 04763, KOREA
2 SEOUL NATIONAL UNIVERSITY OF SCIENCE AND TECHNOLOGY, DEPARTMENT OF MATERIALS SCIENCE AND ENGINEERING, SEOUL 01811, REPUBLIC OF KOREA
3 THE RESEARCH INSTITUTE OF INDUSTRIAL SCIENCE, HANYANG UNIVERSITY, SEOUL 04763, KOREA

* CORRESPONDING AUTHOR: ydkim1@hanyang.ac.kr
** Co-CORRESPONDING AUTHOR: byun@seoultech.ac.kr

$0.5Y_2O_3$, to confirm the change in the properties of Fe-based ODS alloys. When 0.3 wt.% or more of vanadium was added, carbides were formed. Niobium has a solid solubility of 0.5 wt.%; however, composition of 0.2 wt.% was set in consideration of the amount of vanadium added.

The alloy powder was prepared via mechanical alloying using the elemental powder of each element. Mechanical alloying was carried out using a planetary mill (PM 400, Retsch GmbH), for 20 h at a rotation speed of 200 rpm. The bowl and the ball used in the process were made of stainless steel. The diameter of the ball was 4.7 mm, and the ball to powder ratio was charged at 10:1. In addition, to prevent the oxidation of the powder during the milling process, Ar gas was injected to form an inert atmosphere. To prevent the agglomeration of the powder during the mechanical alloying process, cooling was performed for 10 min after the milling process that was carried out for 20 min. Next, a magnetic pulsed compaction (MPC, nanotech) equipment was used to achieve a pressure of 5 GPa for producing a molded specimens and that were then sintered for 30 min at a temperature of 1150°C to obtain the sintered specimens. Etching was performed to confirm the grain size. The etching solution composed of 5 g copper chloride, 100 mL hydrochloric acid, and 100 mL ethyl alcohol.

Phase analysis was performed on the mechanical alloyed powder and the sintered specimens using an X-ray diffractometer (XRD, D/MAX 2500/PC, RIGAKU). Scanning electron microscopy (SEM, JSM-6701F, JEOL) analysis was performed to analyze the microstructure of the alloys. In addition, micro-Vickers (VH-3100, Buehler) analysis was performed to obtain the hardness of the sintered specimens based on the composition.

3. Results and discussion

Fig. 1 shows the results of the XRD analysis performed on the mechanically alloyed powder and the sintered specimens. XRD peaks of the mechanically alloyed powder are illustrated in Fig. 1(a). In the results, no other phase was identified except for the Fe-Cr matrix phase; it was confirmed that the peaks of the compositions containing niobium and vanadium and those of the conventional composition without the addition of elements were similar. This result is considered an effect of the broadening of the peak due to the refinement of the powder. The results of the XRD analysis performed on the sintered specimens are presented in Fig. 1(b). In addition to the known Fe matrix phase, the complex oxides such as $Y_2Ti_2O_7$ and $Y_3Al_5O_{12}$ phase was identified; however no other phases such as the precipitated phase were identified. The $Y_2Ti_2O_7$ and $Y_3Al_5O_{12}$ phase, which is a complex oxide, was formed through the reaction of the added Y_2O_3 and titanium and aluminum during sintering. These complex oxides play an important role in improving the properties of the alloy; it has a finer particle size and better stability under high temperature than the conventional Y_2O_3. These phase analysis results indicate that the added niobium and vanadium did not form the precipitated phases such as the carbides, and they acted as solid solutions.

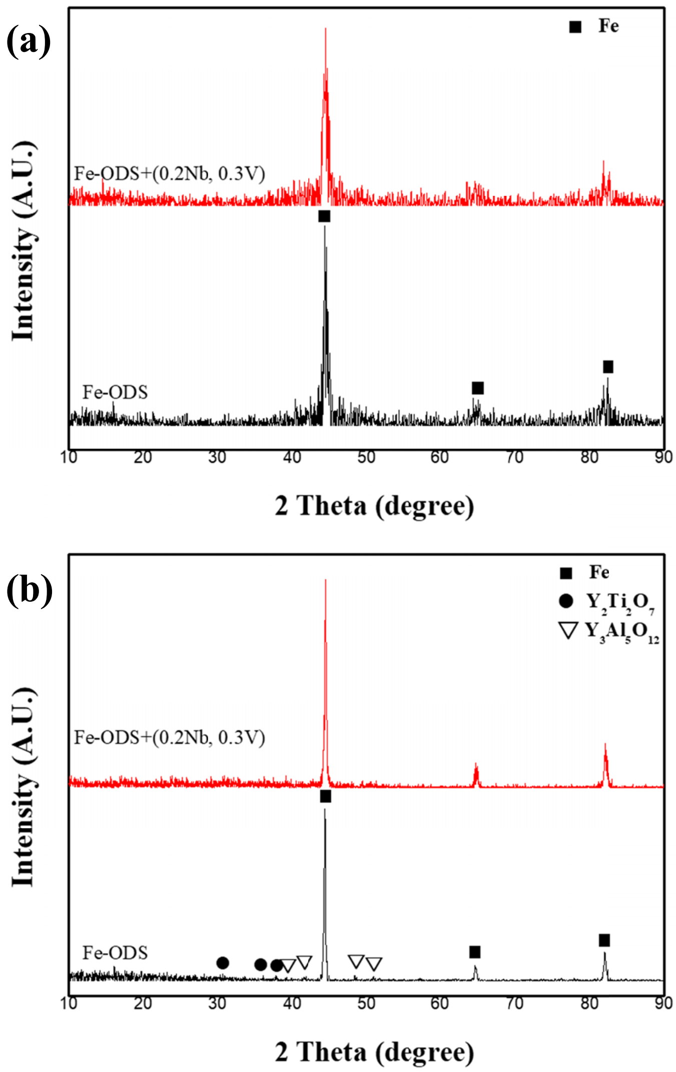

Fig. 1. XRD patterns of the Fe-ODS and Fe-ODS+(Nb, V); (a) mechanical alloyed powders, (b) sintered specimens

SEM analysis was performed to confirm the difference in the microstructures after the addition of niobium and vanadium. The results are presented in Fig. 2. The results of the microstructure analysis confirmed that the Fe matrix phase and the complex oxide phase were distributed, and other phases such as the precipitated phase were not identified. Relatively bright phases such as Fe matrix phase and dark spot complex oxide phases were identified. The surface morphologies of the sintered specimens are presented in Fig. 2(a) and Fig. 2(b).

Fig. 2(c) and Fig. 2(d) are surface images after etching the sintered specimens. As a result of obtaining the average grain size by analyzing the microstructure of the etching specimen, the average grain size in Fig. 2(c) was 3.84 μm and the average size in Fig. 2(d) was 2.91 μm. The grain was more finely distributed in the case with the addition of niobium and vanadium. In addition, the grain boundaries are indicated by a blue arrow. This result was because the diffusion activation energy of Fe increases with the addition of niobium and vanadium; This results in slower diffusion during the sintering process and slower grain growth. Through these results, it was confirmed that the

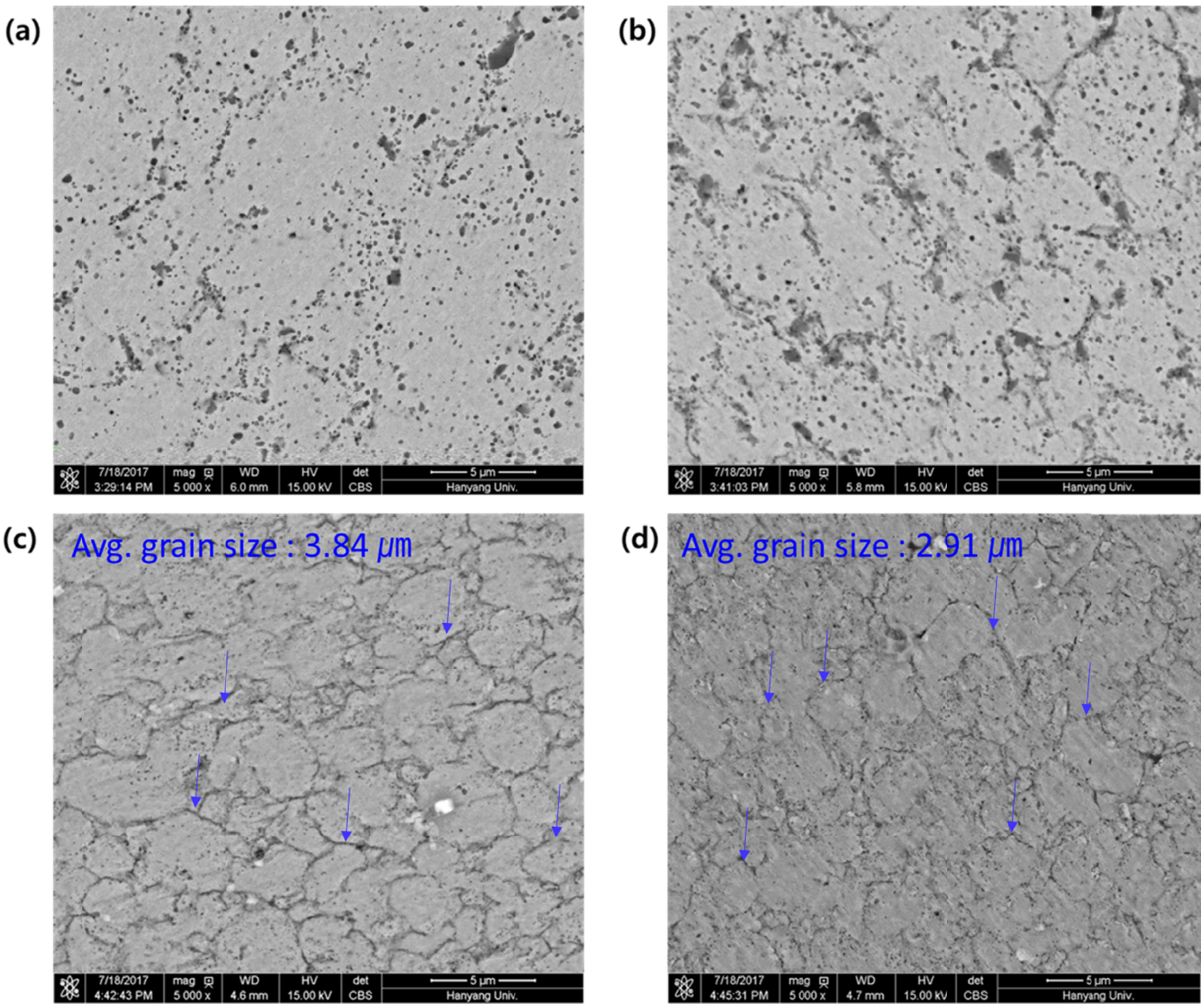

Fig. 2. Surface morphologies of the sintered (a) Fe-15Cr-1Al-0.5Ti-0.5Y_2O_3, (b) Fe-15Cr-1Al-0.5Ti-0.5Y_2O_3-0.2Nb-0.3V and etched surface images of (c) Fe-15Cr-1Al-0.5Ti-0.5Y_2O_3, (d) Fe-15Cr-1Al-0.5Ti-0.5Y_2O_3-0.2Nb-0.3V

addition of niobium and vanadium suppresses the grain growth in the alloy. In addition, grain growth inhibition maintains the alloy properties for a prolonged period.

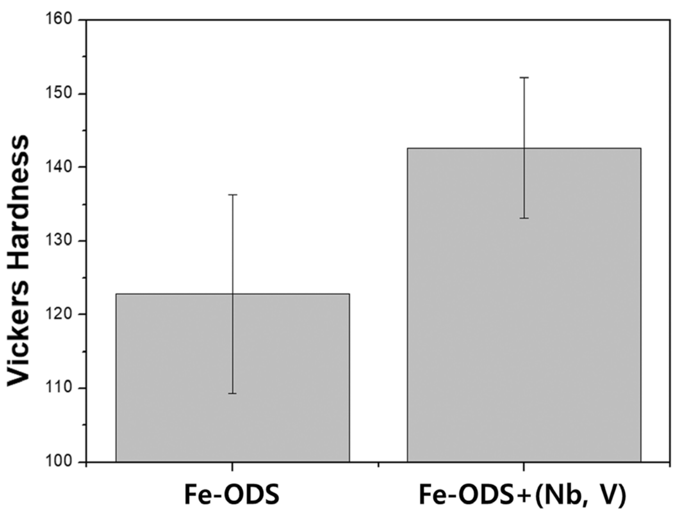

Fig. 3. Vickers hardness test results of sintered specimens

To confirm the changes in the hardness after the addition of niobium and vanadium, the micro-Vickers measurement was performed, and the result is presented in Fig. 3. As a result of the micro-Vickers hardness analysis, the average hardness of the conventional Fe-based ODS alloy was estimated to be 122 Hv, and that of the composition containing niobium and vanadium was 143 Hv, thereby indicating that the hardness improved by approximately 18%. This result confirmed that the addition of niobium and vanadium improve the hardness of the alloy. The improvement of hardness can be decision by the effect of grain refinement due to the increase of diffusion activation energy and the effect of improving the mechanical properties of the alloy by effecting as a matrix solid solution.

TABLE 1

Chemical composition of Fe-based ODS alloy

Sample name	Elements (wt.%)						
	Fe	Cr	Al	Ti	Nb	V	Y_2O_3
Fe-ODS	Bal.	20	1	0.5	—	—	0.5
Fe-ODS+(Nb, V)	Bal.	20	1	0.5	0.2	0.3	0.5

4. Conclusion

In this study, the differences in phase formation, microstructure, hardness between the Fe-based ODS alloys containing niobium and vanadium and the conventional Fe-based ODS alloys were examined. In the phase analysis results of the mechanically alloyed powder and the sintered specimen, the precipitated phase was not confirmed; however the effect of the additive element were confirmed to be similar to those of a solid solution element. The result of the microstructure analysis; indicated that the grains of niobium and vanadium added specimen were finely formed. This is because the diffusion activation energy increased due to the solid solution of the additive elements and the grain growth rate decreased. The result of the micro-Vickers hardness analysis confirmed that the hardness of niobium and vanadium added specimen was 143 Hv, which is approximately 18% higher than that of the specimen without the added element. In conclusion, it was confirmed that when niobium and vanadium were added to Fe-based ODS alloy, the grain growth of the alloy was suppressed and the hardness was improved.

Acknowledgement

This study was supported by a Basic Science Research Program through the National Research Foundation of Korea (NRF) funded by the Ministry of Education (2016R1A6A1A03013422). This research was also supported by Basic Science Research Program through the National Research Foundation of Korea (NRF) funded by the Ministry of Education (2019R1A6A3A13097242).

REFERENCES

[1] J.R. Davis, ASM Speciality Handbook: Heat Resistant Materials, ASM Speciality Handbook, ASM International (1997).
[2] R.-W. Cahn, A.-G. Evans, M. McLean, High-Temperature Structural Materials, Chapman & Hall for The Royal Society (1996).
[3] J.M. Byun, C.W. Park, Y.D. Kim, Met. Mater. Int. **24**, 1309 (2018).
[4] D. Andy, R. Jones, Historical Perspective – ODS alloy Development, University of Liverpool, Liverpool (2010).
[5] J.R. Rieken, I.E. Anderson, M.J. Kramer, G.R. Odette, E. Stergar, E. Harney, J. Nucl. Mater. **428**, 65 (2012).
[6] Q. Tang, T. Hoshino, S. Ukai, B. Leng, S. Hayashi, T. Wang, Mater. Trans. **51**, 11 (2010).
[7] S. Pasebani, A.K. Dutt, J. Burns, I. Charit, R.S. Mishra, Mater. Sci. Eng. A **630**, 155 (2015).
[8] C. Suryanarayana, Prog. Mater. Sci. **46**, 1 (2001).
[9] H.J. Jin, S.H. Kang, T.K. Kim, J. Korean Powder Metall. Inst. **21**, 271 (2014).
[10] J.S. Benjamin, Metall. Trans. **1** (1970) 2943.
[11] C. Capdevila, H.K.D.H. Bhadeshia, Adv. Eng. Mater. **3**, 647 (2001).
[12] Z. Oksiuta, P. Olier, Y.D. Carian, N. Baluc, J. Nucl. Met. **393**, 114 (2009).
[13] S.J. Zinkle, J.L. Boutard, D.T. Hoelzer, A. Kimura, R. Lindau, G.R. odette, M. Rieth, L. Tan, H. Tanigawa, Nucl. Fusion **57** (9), 092005 (2017).
[14] V.L. Sordi, L.O. Bueno, J. Phys. Conf. Ser. **240**, 012088 (2010).
[15] Q. Zhang, Y. Zhao, G. Yuan, W. Yang, Results Phy. **15**, 102335 (2019).