Transactions of the Korean Nuclear Society Autumn Meeting
Jeju, Korea, May 12-13, 2016

Reduced Activation W Alloys for Plasma Facing Materials

Owais Ahmed Waseem[1], Seung Su Kim[1], Jun Ho Lee[2], Soon Hyung Hong[2], Ho Jin Ryu[1]*
[1] *Department of Nuclear and Quantum Engineering, Korea Advanced Institute of Science and Technology, 291 Daehakro, Yuseong-gu, Daejeon 34141, Republic of Korea*
[2] *Department of Materials Science and Engineering, Korea Advanced Institute of Science and Technology, 291 Daehakro, Yuseong-gu, Daejeon 34141, Republic of Korea*
**Co-corresponding Author: Tel.: +82–42–350–3812, Fax: +82–42–350–3810,*
E-mail address: hojinryu@kaist.ac.kr (Ho Jin Ryu)

1. Introduction

Due to refractory nature, high melting temperature, high strength, good conductivity and low erosion and sputtering [1], the tungsten (W) is capable of withstanding high temperature and plasma environment [2]. However, irradiant induced embrittlement, reduction in strength beyond 1000°C and the high ductile to brittle transition temperature of W are obstacles in commercial utilization of W [3]. In order to improve the in-service characteristics of W, thorough research on W-based composites [4], new W alloys [5] and functionally graded materials [6] are in progress.

The limitations of conventional W alloys such as such as high brittleness, high activation energy for dislocation's mobility [7], reduced mechanical strength due to reaction phases [8], poor fabricability [9], defective passive layer [9], poor workability [10] and unavailability of good number of alloy systems [11] have persuaded us to divert research focus towards W-based high entropy alloys [12] .

The core effects of high entropy alloys include enhanced mixing entropy, sluggish diffusion, severe lattice distortion and cocktail effects [13]. These key characteristics of high entropy alloys promote the formation of solid solution of 5 to 13 principle elements [14] with exceptionally high strength, hardness, thermal stability, wear resistance, fatigue and creep resistance [7].

Although the synthesis and exploitation of high entropy alloys have been in progress [15], this study comprises of our work in which we developed a reduced activation W-based high entropy alloy by not incorporating Mo, Cu, Nb and by reducing Ti content [16]. In this research a powder metallurgical technique has been successfully used for producing novel W-based alloy with enhanced mechanical properties.

2. Methods and Results

2.1 Experimental

The 99.9% pure elemental powders of W, V, Cr, Ta and Ti were mixed to develop the powder mixture of tungsten-based high entropy alloy containing 20 to 90at.% W with equal amount of remaining elements. Mixing was carried out in a plastic vial at a speed of 30 rpm up to 3 hours. The consolidation of powder mixture was carried out by spark plasma sintering (SPS) at 1500°C and 1600°C and 50 MPa pressure for 10 minutes. The samples were characterized by X-ray diffraction (XRD) and scanning electron microscopy - energy dispersive spectroscopy (SEM-EDS). The mechanical behavior of the samples was investigated by subjecting the samples to Vickers hardness tests.

2.2 Results

A significant effect of composition and sintering temperature were observed. The alloys prepared with ≥ 60% W showed full density when sintered at 1600°C.

The XRD examination of the powder mixture, see fig. 1, shows prominent peaks of all elemental powder with no distortion in amplitude and width, which distinguishes our methodology with conventional mechanical alloying.

The XRD of the sintered samples, shown in Fig. 2, exhibits successful development of solid solutions with simple mixing with a BCC lattice structure.

The microstructures of as-polished x%W(100-x)/4%(TaTiCrV) high entropy alloys, captured with backscattered electrons, as shown in Fig. 3, exhibit homogeneously distributed dark phases in a bright matrix. The chemical nature, analyzed via point energy dispersive spectroscopy (EDS) revealed the presence of dark Ti-rich phase in bright W-rich matrix.

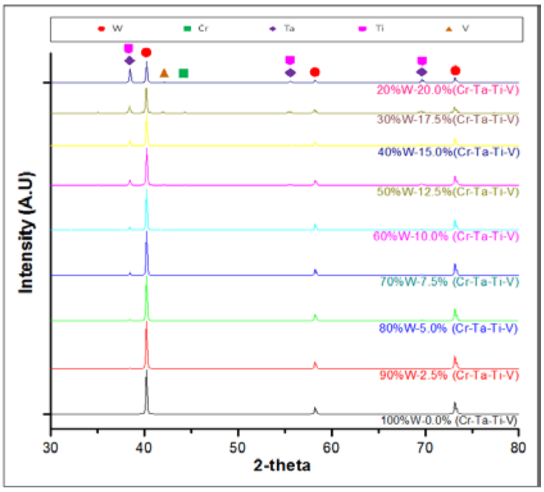

Fig. 1. XRD patterns of HEA powder mixture before sintering.

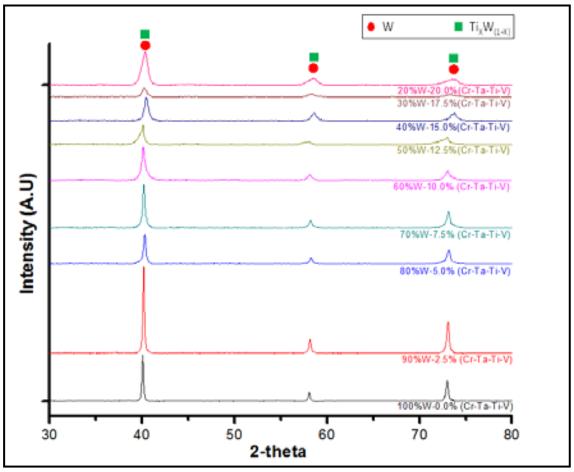

Fig. 2. XRD patterns of HEA samples sintered at 1500°C.

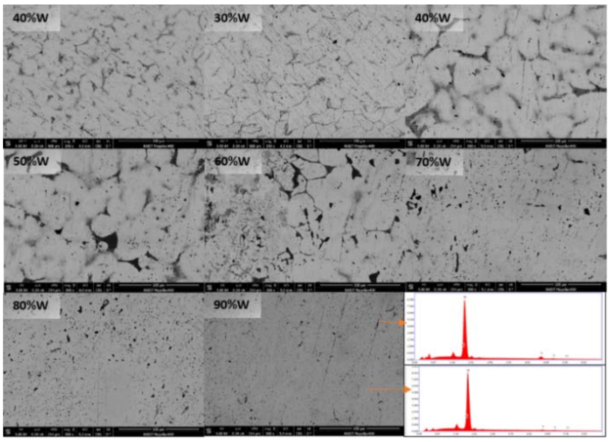

Fig. 3. SEM micrographs of x%W(100-x)/4%(TaTiCrV) samples sintered at 1600°C.

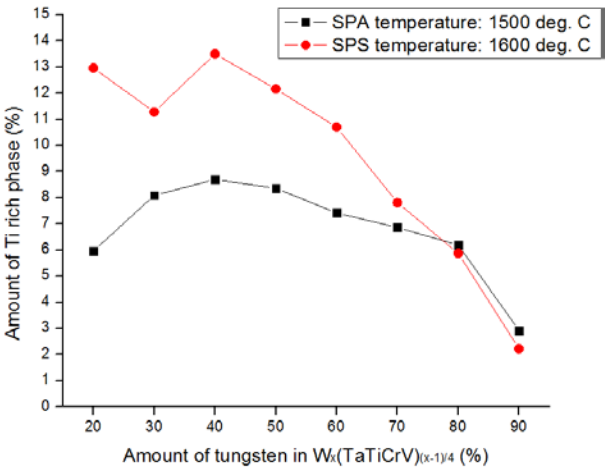

Fig. 4. Variation in volume fraction of Ti-rich phase with W content and sintering temperature.

The analysis of SEM images shows that the fraction of Ti-rich phase found increases in x%W(100-x)/4%(TaTiCrV) with decreasing W content up to 40%, where it reached to maximum, further reduction in W content, causes the fraction of Ti-rich phases to decrease. A significant addition of Ti rich phases to HEA samples can also be induced by increasing sintering temperature, as shown in Fig. 4.

The HEA samples were subjected to Vickers hardness tests to assess their mechanical behavior. The results of hardness tests, as shown in Fig. 5, illustrate enhanced hardness due to inhomogeneous stress and severe lattice distortion produced by the constituents of the HEA [86]. It can also be observed that by changing the composition by varying W content from 90at.% to 40at.%, the hardness can be increased to an impressive extent ~750 Hv (when sintered at 1500°C), which is much higher than the hardness of pure tungsten i.e. ~350 Hv.

The successful development of W-based high entropy alloys by simple mixing with enhanced physical and mechanical properties emphasizes upon the potential of this novel method in the development of materials for high temperature and fusion applications.

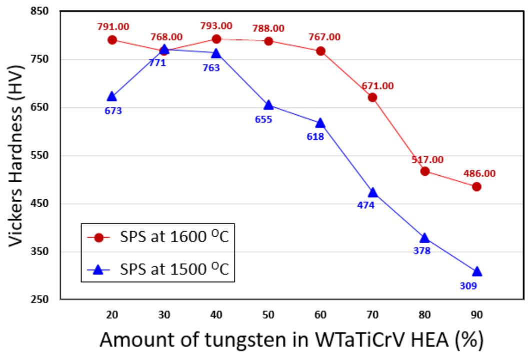

Fig. 5. Variation in hardness with W content and sintering temperature.

3. Conclusions

An undiscovered potential of an unusual and so far disregarded powder metallurgical technique i. e. simple mixing in the development of W based material for future fusion power plants has been exploited by fabricating and analyzing a novel tungsten based high entropy alloy system x%W-(100-x)/4%(TaTiCrV). Various compositions were prepared by varying 'x' from 20at.% to 90at.% in the powder mixture of HEA followed by spark plasma sintering. The density of the alloys was found increasing with decreasing W content and higher sintering temperature

The characterization of HEA samples by XRD showed BCC crystal structure of the alloys. Microstructural examination SEM-EDS revealed the presence of bright and dark regions as W and Ti-rich phases, respectively. The evaluation of microstructures by using image processing software and Vickers hardness via micro-Vickers hardness tester showed the similar dependency of phase fraction and hardness on composition and sintering temperature. The amount of Ti-rich phase was found to decrease in pre and post 40at.%W-HEA. An analogous behavior of hardness was observed when plotted against composition at different temperatures. The alloys sintered at 1600°C were found harder than previous samples which were sintered at 1500°C. This study revealed the potential role of simple mixing in the fabrication of reduced activation W-based alloys with enhanced physical and mechanical characteristics for high temperature and plasma facing applications.

Acknowledgments

This research was supported by National R&D Program through the National Research Foundation of Korea (NRF) funded by the Ministry of Science, ICT & Future Planning (NRF-2015M1A7A1A02002190)

REFERENCES

[1] M. Battabyal, P. Spätig, and N. Baluc, "Effect of ion-irradiation on the microstructure and microhardness of the W-2Y_2O_3 composite materials fabricated by sintering and hot forging," *Fusion Eng. Des.*, vol. 88, no. 9–10, pp. 1668–1672, 2013.

[2] B. I. Khripunov, V. S. Koidan, A. I. Ryazanov, V. M. Gureev, S. . N. Kornienko, S. T. Latushkin, A. S. Rupyshev, E. V. Semenov, V. S. Kulikauskas, and V. V. Zatekin, "Study of Tungsten as a Plasma-facing Material for a Fusion Reactor," *Phys. Procedia*, vol. 71, no. February, pp. 63–67, 2015.

[3] M. Xia, Q. Yan, L. Xu, H. Guo, L. Zhu, and C. Ge, "Bulk tungsten with uniformly dispersed La_2O_3 nanoparticles sintered from co-precipitated La_2O_3/W nanoparticles," *J. Nucl. Mater.*, vol. 434, no. 1–3, pp. 85–89, 2013.

[4] M. Battabyal, R. Schäublin, P. Spätig, M. Walter, M. Rieth, and N. Baluc, "Microstructure and mechanical properties of a W–2wt.%Y_2O_3 composite produced by sintering and hot forging," *J. Nucl. Mater.*, vol. 442, no. 1–3, pp. S225–S228, 2013.

[5] R. Mateus, M. Dias, J. Lopes, J. Rocha, N. Catarino, P. Duarte, R. B. Gomes, C. Silva, H. Fernandes, V. Livramento, P. a. Carvalho, E. Alves, K. Hanada, and J. B. Correia, "Blistering of W–Ta composites at different irradiation energies," *J. Nucl. Mater.*, vol. 438, pp. S1032–S1035, 2013.

[6] C. C. Ge, Z. J. Zhou, S. X. Song, J. Du, and Z. H. Zhong, "Progress of research on plasma facing materials in University of Science and Technology Beijing," *J. Nucl. Mater.*, vol. 363–365, no. 1–3, pp. 1211–1215, 2007.

[7] Y. Dong, L. Jiang, H. Jiang, Y. Lu, T. Wang, and T. Li, "Effects of annealing treatment on microstructure and hardness of bulk AlCrFeNiMo0.2 eutectic high-entropy alloy," *Mater. Des.*, vol. 82, pp. 91–97, 2015.

[8] F. Koch, J. Brinkmann, S. Lindig, T. P. Mishra, and C. Linsmeier, "Oxidation behaviour of silicon-free tungsten alloys for use as the first wall material," *Phys. Scr.*, vol. T145, p. 014019, 2011.

[9] P. López-Ruiz, F. Koch, N. Ordás, S. Lindig, and C. García-Rosales, "Manufacturing of self-passivating W-Cr-Si alloys by mechanical alloying and HIP," *Fusion Eng. Des.*, vol. 86, no. 9–11, pp. 1719–1723, 2011.

[10] P. López-Ruiz, N. Ordás, I. Iturriza, M. Walter, E. Gaganidze, S. Lindig, F. Koch, and C. García-Rosales, "Powder metallurgical processing of self-passivating tungsten alloys for fusion first wall application," *J. Nucl. Mater.*, vol. 442, no. 1–3 SUPPL.1, pp. S219–S224, 2013.

[11] L. C. Tsao, C. S. Chen, and C. P. Chu, "Age hardening reaction of the Al0.3CrFe1.5MnNi0.5 high entropy alloy," *Mater. Des.*, vol. 36, pp. 854–858, 2012.

[12] J. Y. He, W. H. Liu, H. Wang, Y. Wu, X. J. Liu, T. G. Nieh, and Z. P. Lu, "Effects of Al addition on structural evolution and tensile properties of the FeCoNiCrMn high-entropy alloy system," *Acta Mater.*, vol. 62, no. 1, pp. 105–113, 2014.

[13] B. Ren, Z. X. Liu, B. Cai, M. X. Wang, and L. Shi, "Aging behavior of a CuCr2Fe_2NiMn high-entropy alloy," *Mater. Des.*, vol. 33, no. 1, pp. 121–126, 2012.

[14] B. Schuh, F. Mendez-Martin, B. Völker, E. P. George, H. Clemens, R. Pippan, and A. Hohenwarter, "Mechanical properties, microstructure and thermal stability of a nanocrystalline CoCrFeMnNi high-entropy alloy after severe plastic deformation," *Acta Mater.*, vol. 96, pp. 258–268, 2015.

[15] X. Li, F. Tian, S. Schönecker, J. Zhao, and L. Vitos, "Ab initio-predicted micro-mechanical performance of refractory high-entropy alloys," *Sci. Rep.*, vol. 5, p. 12334, 2015.

[16] X. Xiao, G. Liu, B. Hu, J. Wang, and W. Ma, "Microstructure Stability of V and Ta Microalloyed 12%Cr Reduced Activation Ferrite/Martensite Steel during Long-term Aging at 650°C," *J. Mater. Sci. Technol.*, vol. 31, no. 3, pp. 311–319, 2015.