Analysis of W reinforced High Entropy Alloy-based Composites Developed by Spark Plasma Sintering

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1. Introduction

Tungsten (W) is a potential candidate material for high temperature and plasma facing applications because of its high melting point, high strength, adequate conductivity and low erosion and sputtering [1]. These properties equip W with capability of sustaining high heat load, mechanical stresses and plasma environment [2]. However, W shows high ductile to brittle transition temperature, it suffers with irradiation induced embrittlement and reduction in strength at high temperature (above 1000 ^{O}C) as well, therefore its commercial applications in fusion power plants are still constrained [3]. These limitations ask for the innovations in the field of W-based materials [4] for application in future fusion reactors.

Several new W-based alloys are being investigated. But the limitations of conventional alloys for instance defective passive layer [5], poor workability [6], and high activation energy for dislocations mobility [7] diverts the research focus towards high entropy alloy (HEA) based materials [8] which offers extraordinary strength, hardness, wear resistance, fatigue and creep resistance [7].

W-fibers reinforced W-matrix composites, produced by chemical vapor deposition (CVD) and hot isostatic pressing (HIP) have been analyzed as they reduce inherent brittleness of matrix material due to ductile fracture of W-fibers [9][10][11][12]. But low density and premature stopping of deposition of W-matrix in CVD [9] and secondary grain growth in HIP [12] emphasizes upon the requirement of developing this sort of composite material by spark plasma sintering (SPS).

This manuscript presents the study of spark plasma sintered reduced activation HEA (WTaTiCrV)/W_{mesh} and HEA (WTaTiCrV)/$W_{whiskers}$ composites. Comparison of composites having different geometrically different reinforcements have been done for the selection of better candidate material for fusion applications.

2. Methods and Results

2.1 Experimental

The bulk composite samples were produced by spark plasma sintering (SPS), at 1600 ^{O}C, of powder mixture having W, Ta, Ti, V and Cr. In order to reinforce the matrix with W_{mesh}, $W_{whiskers}$ and $W_{3D\text{-}mesh}$, 10wt.% mesh, whiskers and 3D-mesh (named to denote the composite having random network of meshes) were incorporated in powder mixture prior to sintering. Fig. 1 shows schematics of the sintered samples.

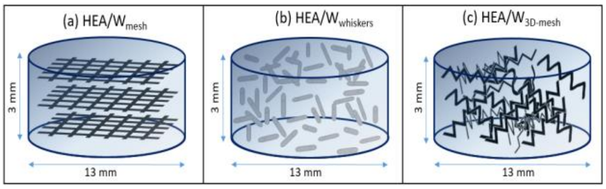

Fig. 1. Schematics of (a) W_{mesh} (b) $W_{wiskers}$ and (c) $W_{3D\text{-}mesh}$ reinforced HEA (WTaTiCrV) based composites.

In order to impart the composite material with reduced activation characteristics, the addition of Mo, Cu and Nb was restricted [13]. Spark plasma sintering (SPS) of the powder mixture reinforced with W-meshes was done at a temperature of 1600 ^{O}C. Sintered samples were characterized by x-ray diffraction (XRD), scanning electron microscopy (SEM). Mechanical properties were compared by carrying out Vickers hardness test.

2.2 Results

The XRD examination of fully dense $W_{mesh,}$ $W_{whiskers}$ and $W_{3D\text{-}mesh}$ reinforced HEA (WTaTiCrV) composites reveals development of homogeneous solid solution matrix with BCC lattice, as shown in Fig. 2. Overlapping BCC peaks of matrix and reinforcements were observed due to W-based chemical nature of both of them.

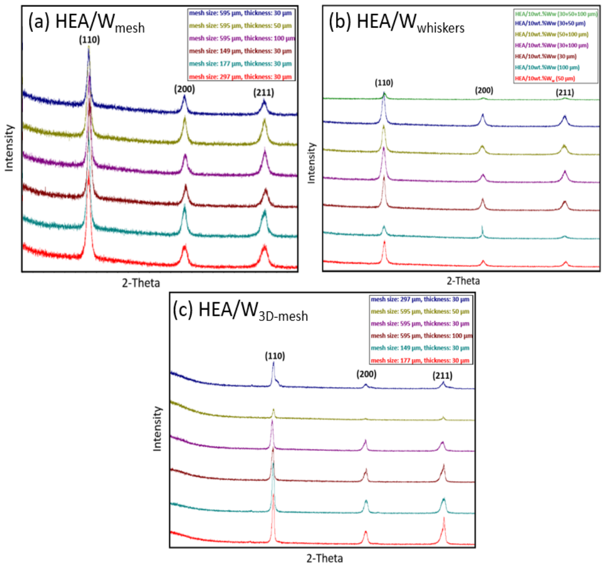

Fig. 2. XRD patterns of (a) W_{mesh} (b) $W_{wiskers}$ and (c) $W_{3D\text{-}mesh}$ reinforced HEA (WTaTiCrV) based composites sintered at 1600 ^{O}C.

The microstructural examination, carried out via SEM-EDS of the composites, shows uniform distribution of constituent elements and Ti rich phase in W rich HEA matrix. Proper adhesion of matrix material with the reinforcements is also evident, see Fig. 3.

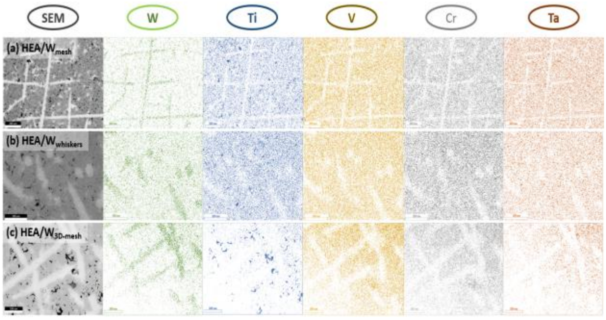

Fig. 3. Microstructures and elemental maps of (a) W_{mesh} (b) $W_{wiskers}$ and (c) $W_{3D\text{-}mesh}$ reinforced HEA (WTaTiCrV) based composites sintered at 1600 ^{O}C.

The comparison of mechanical behavior of W_{mesh}, $W_{whiskers}$ and $W_{3D\text{-}mesh}$ reinforced HEA composites is presented in Fig. 4. Because of anisotropic nature of HEA/W_{mesh} and HEA/$W_{3D\text{-}mesh}$ samples, both surfaces (top surface and cross-section) were examined under hardness tester. The minor and abrupt variation in hardness values with respect to mesh size, as shown in Fig. 4 (a) & 4 (c) can be related to experimental fluctuations. However, a slight but clear decreasing trend in hardness with increasing mesh and whickers' thickness can be observed in Fig. 4 (b), 4 (d) & 4 (e).

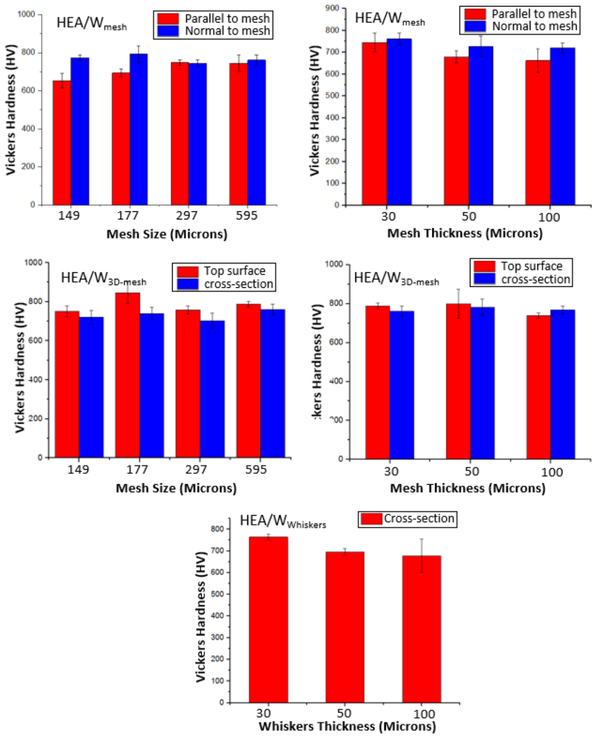

Fig. 4. Vickers hardness of (a & b) W_{mesh}, (c & d) $W_{3D\text{-}mesh}$ and (e) $W_{wiskers}$ reinforced HEA (WTaTiCrV) based composites sintered at 1600 ^{O}C.

Overall Vickers hardness data shows the values ranging from 690 HV to 800 HV of various samples, which is almost two times higher than pure W.

3. Conclusions

The 3D mixing and SPS successfully development reduced activation high entropy alloy based composite samples. Fully dense composite materials exhibited enhanced mechanical properties with slight variation with size and thickness of reinforcements. The presence of thin reinforcement i.e. W mesh, whiskers and fibers, which provides brittle matrix with toughness, forecasts the possible future application of this material in fusion applications.

Acknowledgment

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 $W\text{-}2Y_2O_3$ Composite Materials Fabricated by Sintering and Hot Forging, Fusion Engineering and Design, Vol. 88, p. 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, Physics Procedia, Vol. 71, p. 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, Journal of Nuclear Materials, Vol. 434, p. 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, Journal of Nuclear Materials, Vol. 442, p. S225-S228, 2013.

[5] 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 Engineering and Design, Vol. 86, p.1719-1723, 2011.

[6] 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, Journal of Nuclear Materials, Vol. 442, p. S219-S224, 2013.

[7] Y. Dong, L. Jiang, H. Jiang, Y. Lu, T. Wang, and T. Li, Effects of Annealing Treatment on Microstructure and Hardness of Bulk $AlCrFeNiMo_{0.2}$ Eutectic High-Entropy Alloy, Materials and Design, Vol. 82, p. 91-97, 2015.

[8] 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 Materialia, Vol. 62, p. 105-113, 2014.

[9] J. Riesch, M. Aumann, J. W. Coenen, H. Gietl, G. Holzner, T. Höschen, P. Huber, M. Li, C. Linsmeier, and R. Neu, Chemically Deposited Tungsten Fibre-Reinforced Tungsten - The Way to a Mock-Up for Divertor Applications, Nuclear Materials and Energy, Vol. 0, p. 1–9, 2015.

[10] J. Riesch, J. Y. Buffiere, T. Höschen, M. Di Michiel, M. Scheel, C. Linsmeier, and J. H. You, In situ Synchrotron Tomography Estimation of Toughening Effect by Semi-Ductile Fibre Reinforcement in a Tungsten Fibre Reinforced Tungsten Composite System, Acta Materialia, Vol. 61, p. 7060–7071, 2013.

[11] B. Jasper, J. W. Coenen, J. Riesch, T. Höschen, M. Bram, and C. Linsmeier, Powder Metallurgical Tungsten Fiber Reinforced Tungsten, Materials Science Forum, Vol. 825-826, p. 125–133, 2015.

[12] J. Riesch, Y. Han, J. Almanstötter, J. W. Coenen, T. Höschen, B. Jasper, P. Zhao, C. Linsmeier, and R. Neu, Development of Tungsten Fibre Reinforced Tungsten Composites Towards Their Use in DEMO —Potassium Doped Tungsten Wire, Physica Scripta, Vol. T167, p. 014006, 2016.

[13] 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 ^{O}C, Journal of Materials Science and Techonology, Vol. 31, p. 311-319, 2015.