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Evaluation of W–Si–C thick coating as a plasma facing material

Hyun Kwang Seok[a,*], Kyung Ho Jung[a], Yu Chan Kim[a], Jae-Hyeok Shim[a], Dong-Ik Kim[a], Seung-Hee Han[a], Kyeong Ho Baik[b], Pil-Ryung Cha[c]

[a] Korea Institute of Science and Technology, Seoul 136-791, Republic of Korea
[b] ChungNam National University, Deajeon 305-764, Republic of Korea
[c] School of Advanced Materials Engineering, Kooknin University, Seoul 136-702, Republic of Korea

ABSTRACT

We present tungsten alloy coating of 150–200 μm thickness with improved plasma erosion resistance fabricated by plasma spraying of granular W–SiC composite powders. During increasing the SiC concentration to 8 wt%, we observed the increase in the hardness of the coating from 250 to 440 Hv. The plasma erosion depth of the coating decreased by 10 times compared with pure tungsten in the same erosion environment.

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1. Introduction

Plasma facing materials (PFMs) are required to have high erosion resistance against the plasma or the ion bombardment, which is generated in Tokamak fusion reactor because the erosion products re-deposited on the PFM wall or penetrated into the core plasma can cause the degradation of the properties of PFM or the quality of the plasma. On this score, many researchers have searched new PFMs such as tungsten, carbon, vanadium, and beryllium [1]. Among those candidates for the PFMs, tungsten [2–5] is considered as an attractive PFM [6] in fusion devices due to its favorable physical properties, prominent thermal properties and the low sputtering yield.

In this study, we focus on the development of new tungsten alloys (or composite) with improved erosion resistance against the plasma and the ion bombardment compared with pure tungsten without sacrificing the mechanical and thermal properties. As an alloy component, SiC [7] was chosen because of its low activity, high melting temperature, and high thermal conductivity. W–Si–C alloy (or composite) coating was fabricated on Cu alloy substrate with the atmospheric plasma spraying process using composite granular powders composed of primary fine W and SiC powders.

2. Experimental procedures

W–SiC composite powders were prepared under the optimized spray drying conditions with varied SiC contents of 1, 2, 4, 8 wt% following conventional granulation route. At first, a fluidic slurry composed of distilled water, 1–2 μm sized W powder, 1–2 μm sized SiC powders, 0.8 wt% polyvinylpropylene as binder, and 0.2 wt% dispersant were prepared. Then, the slurry was spray dried with 180 °C hot air using high speed rotational nozzle, resulting in 5–50 μm sized granules containing polymeric binder inside. Then the spray dried granule was heat treated in vacuum furnace at an elevated temperature of 1450 °C for 1 h in order to remove polymeric binder as well as to increase the mechanical strength of powder not to be broken during plasma spraying.

The prepared composite powder was plasma spray coated under the conditions of 800 A, 41 V, Ar flow rate of 36 l/min, H_2 flow rate of 6 l/min, and spray distance of 120 mm in atmospheric surroundings to form 150 to 200 μm thickness coating on 7.62 cm diameter and 3 mm thickness Cu substrate which was designed to fit the DC magnetron sputter gun used in this study.

The microstructural evolution was estimated using scanning electron microspcopy (SEM) and phase transformation by X-ray diffraction (XRD), wavelength dispersive spectroscopy (WDS), and X-ray photoelectron spectroscopy (XPS). Micro Vickers hardness was measured under 1000 gf load. Plasma erosion rate was evaluated by using plasma sputter under the conditions of 0.48 A, 200 W, Ar gas flow rate of 0.046 l/min, 10 mtorr pressure for 1 h to simulate the erosion behavior of plasma facing materials of fusion reactor.

3. Results and discussion

Fig. 1 shows the XRD patterns of as-spray dried W–4 wt%(Si–C) powders (bottom), after heat treatment at 1450 °C for 1 h (middle), and plasma spray coating (top). As the atomic weight of Si and C are much lower than that of W, the relative intensity of SiC diffraction peaks are too weak to be distinguishable as shown in the bottom XRD pattern of Fig. 1. However, in the WDS investigation of the composite granular powder as shown in Fig. 2, Si and C elements are obviously observed, which means that SiC primary

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[*] Corresponding author.
E-mail address: drstone@kist.re.kr (H.K. Seok).

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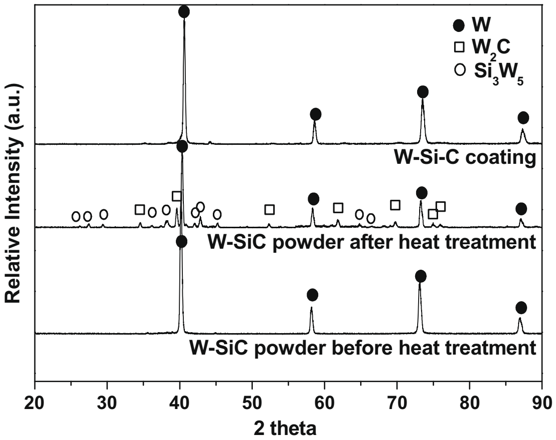

Fig. 1. XRD patterns recorded from as-spray dried W–4 wt%(Si–C) powder(lower), after heat treatment at 1450 °C for 1 h (intermediate), and plasma spray coating (upper).

powders exist in the composite granular powders. Hence, we can say that the spray dried granular powders composed of primary W and SiC powders were well prepared without loss of SiC powders from W powders. During the heat treatment of the granular powders at 1450 °C, Fig. 1 shows the occurrence of the intermixing between W and SiC powders and the formation of W–C and W–Si intermetallic compounds. After plasma spraying, however, the diffraction peaks corresponding to the intermetallics disappear as shown in the top plot of Fig. 1. The WDS investigation of W–Si–C coating shows the uniform distribution of Si and C through the entire coating film and shows no evidence of the formation of other intermetallics such as carbides or silicides. Considering the angle shift of diffraction pattern of W–Si–C coating as shown in the top plot of Fig. 1, the inter-planar distance between (1 1 0) planes of W coating with 4 wt% SiC decreased to 3.14 A, which is certainly smaller than that of pure W (3.16 A). The decrease in the inter-planar distance is due to replacing larger W atoms with smaller Si atoms. From the XRD and WDS studies of thermally sprayed W–Si–C coating, most Si and C atoms are thought to exist as substitutional or interstitial atoms in W–Si–C coating without the formation of the intermetallics.

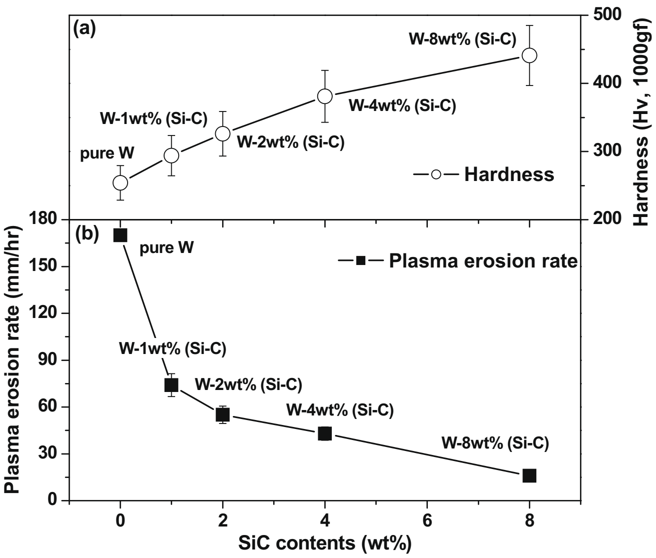

Fig. 3. Variation of (a) hardness and (b) plasma erosion rate measured from plasma sprayed W–Si–C coating prepared with different SiC contents.

Hardness of W–Si–C coating increased from 250 Hv to 440 Hv by increasing SiC contents up to 8 wt% as shown in Fig. 3(a). This result can be easily understood as the added Si and C atoms introduce crystalline lattice distortion by substitutional and interstitial atoms, resulting in retard of dislocation movement excited by external stress.

Plasma erosion rate of W–Si–C coating with different contents of SiC are shown in Fig. 3(b). During plasma sputtering test, intensive erosion of W–Si–C coating happens in the middle circular

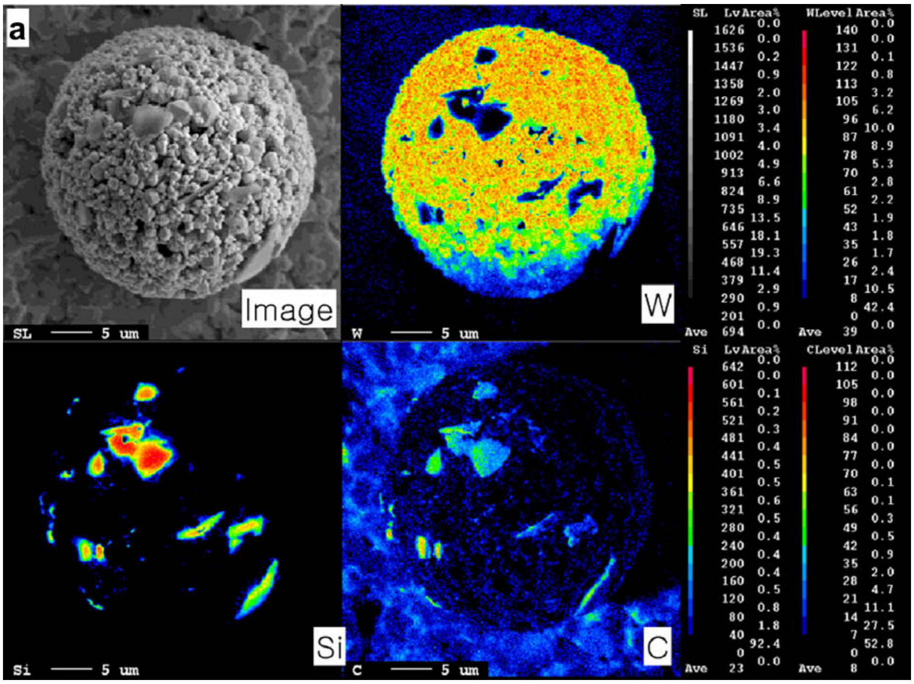

Fig. 2. Image and alloying elements distribution of as-spray dried W–4 wt%(Si–C) powder investigated by WDS.

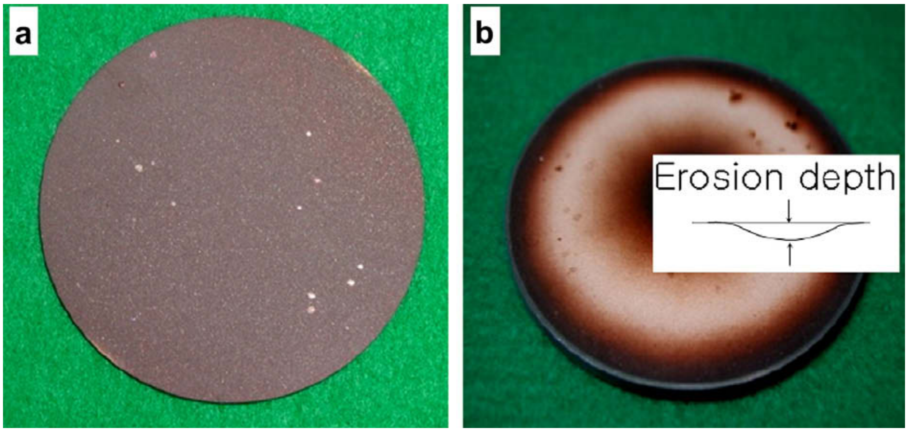

Fig. 4. Plasma spray coated W–Si–C sputtering targets, (a) before plasma erosion test and (b) after plasma erosion test.

region by concentrated plasma and ion bombardment as shown in Fig. 4(b), where plasma erosion depth was measured after 1 h exposure to plasma and ion. From the result shown in Fig. 3(b), drastic decrease of erosion rate from 170 μm/h down to 16 μm/h with increasing the additive SiC content up to 8 wt% are affirmed. Though W–Si–C coatings containing SiC over than 8 wt% are expected to reveal better erosion resistance but higher contents of SiC makes it easier to decompose to form carbon oxide gas during atmospheric plasma spraying, resulting in lots of pores in W–Si–C coating.

It is not clearly understood how C and/or Si dissolved in W improve the plasma erosion resistance of W coating. Gusev et al. [8] reported the sputtering coefficients of W, WC, and W–C mixed layer using deuterium ions. They revealed that the effect of C on the sputtering coefficient of W is not significant. Also, it is not expected that Si rapidly improves the erosion property with increasing Si content, since it is known that pure W and Si have very similar sputtering rates [9]. The protective layer of silicon oxide on the surface of W–Si–C coating might improve the sputtering-erosion property, as Koch and Bolt [10] recently indicated the presence of SiO_2 surface layer of plasma erosion resistant W–Cr–Si alloys. Actually, the formation of stable surface oxide is known to reduce the sputtering rate significantly [11]. In plasma spray coating of W–Si–C, SiO_2 formation in inter-splat boundaries is unavoidable because Si elements in molten powder can be easily oxidized by reaction with oxygen contained in atmosphere and/or carrier gas. Formation of SiO_2 was confirmed in XPS analysis in this study (not shown), where 99 eV corresponding to 2p orbital binding energy of Si in SiC, which was recorded before heat treatment of W–SiC mixture powder, was disappeared, however, 103.5 eV corresponding to 2p orbital binding energy of Si in SiO_2 was recorded in plasma sprayed W–Si–C coating.

Since the plasma sprayed W–Si–C coating is of meta-stable state, changes in the mechanical/thermal properties as well as plasma erosion resistance should be estimated after long time exposure to high temperature surrounding simulating continuous operation of fusion reactor. Nevertheless, in view point of plasma erosion resistance, plasma sprayed W–Si–C coating shows prominent characteristics compared to any reported refractory materials.

4. Conclusion

More improved plasma erosion resistant W–Si–C coating was successfully developed by plasma spraying of granular W–SiC composite powders. During heat treatment of W–SiC powder, W_2C and Si_2W_5 intermetallics were formed by decomposition of SiC. However, all the intermetallics disappeared and some SiO_2 were confirmed to be formed in W–Si–C coating.

By increasing SiC content up to 8 wt%, hardness of W–Si–C coating increased from 250 Hv to 440 Hv, and plasma erosion rate was reduced from 170 μm/h down to 16 μm/h. In view point of plasma erosion resistance, plasma sprayed W–Si–C coating shows prominent characteristics compared to any reported refractory materials.

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References

[1] H. Bolt, V. Barabash, G. Federici, J. Nucl. Mater. 307–311 (2002) 43.
[2] T.G. Wang, J.L. Chen, Y. Chen, Y.C. Wu, J. Nucl. Mater. 363–365 (2007) 1295.
[3] J.W. Davis, K.T. Slattery, D.E. Driemeyer, M.A. Ulrickson, J. Nucl. Mater. 233–237 (1996) 604.
[4] X. Liu, F. Zhang, S. Tao, Y. Cao, Z. Xu, Y. Liu, N. Noda, J. Nucl. Mater. 363–365 (2007) 1299.
[5] D.A. Buchenaur et al., J. Nucl. Mater. 290–293 (2001) 1165.
[6] C. Garcia-Rosales, Nucl. Mater. 211 (1994) 202.
[7] C.Y. Xie, J.L. Chen, J.G. Li, Q.F. Fang, J. Nucl. Mater. 363–365 (2007) 282.
[8] M.I. Gusev, S.N. Korshunov, A.L. Suvorov, N.E. Lazarev, J. Appl. Phys. 44 (1999) 1123.
[9] M. Yoshitake, Y. Yamaguchi, C. Bose, Surf. Interf. Anal. 36 (2004) 801.
[10] F. Koch, H. Bolt, Phys. Scr. T128 (2007) 100.
[11] M. Saidoh, J. Nucl. Mater. 128&129 (1984) 540.