Materials Research Bulletin 48 (2013) 5053–5057

Contents lists available at SciVerse ScienceDirect

Materials Research Bulletin

journal homepage: www.elsevier.com/locate/matresbu

Characterization of VPS-W coating layers on molybdenum after heat exposure

Gue Serb Cho *, Kyeong Hwan Choe, Soon Yeol Choi **

Korea Institute of Industrial Technology, 7-47 Songdo-dong, Yeonsu-gu, Incheon, Republic of Korea

ARTICLE INFO

Article history:
Received 3 January 2013
Received in revised form 26 April 2013
Accepted 27 April 2013
Available online 13 May 2013

Keywords:
Interfaces
Plasma deposition
Diffusion
Microstructures

ABSTRACT

Tungsten (W) coating layers were successfully deposited using a vacuum plasma spraying (VPS) technique on a molybdenum (Mo) substrate. Tungsten powder with a median size of 10 μm was applied to prepare coatings via a plasma spray system. For the VPS process, argon and hydrogen were used as plasma-forming gases, and the coatings were deposited in 35 mbar vacuum pressure. A coating with a thickness of 300 μm was obtained, and some unmelted W powders were observed in the coating layer. This heat exposure experiment was performed in a sapphire crystal growing furnace at 2100 °C up to 110 h. After heat exposure, the VPS-W coating layers were soundly bonded with the Mo substrate due to the interdiffusion between W and Mo.

1. Introduction

Pure molybdenum possesses a unique combination of physical properties including excellent high-temperature strength, high thermal conductivity, a high elastic modulus, and a low thermal-expansion coefficient. Because of these outstanding properties, pure Mo sheet has been used technically in a wide range of applications, including hot-zone materials such as heating elements, heat shields, and crucibles for high-temperature furnace construction [1]. In recent years, the global production of light-emitting diodes (LEDs) has increased rapidly, and the demand for sapphire (α-Al_2O_3) substrates has expanded to support the production of LEDs. The growth of sapphire single crystals from melt is performed in most cases using tungsten or molybdenum–tungsten crucibles in heating furnaces. The sapphire single crystal was contaminated with Mo^{3+} ion due to the complex reaction between the Mo crucible and Al_2O_3 melt during the crystal growing period [2,3]. Because of its high melting point (T_m = 2620 °C), pure Mo provides outstanding high-temperature strength and creep resistance. But, the Mo alloy was vaporized in a vacuum and react chemically with graphite heating elements or shielding material in a high-temperature vacuum furnace. Also, Mo alloy degrades under prolonged exposure to thermal radiation and decreased its high temperature mechanical properties due to the embrittlement of large recrystallized grains [4,5].

As a result, plasma-sprayed tungsten (PS-W) coatings have been successfully fabricated and studied to protect metallic and non-metallic substrates. It has been reported that tungsten is a promising candidate material for plasma-facing components in next-fusion experimental devices due to its low sputtering yield and good thermal properties [6,7]. Using the PS-W coating technique, the coating of tungsten onto a graphite plate with different intermediate layers was studied for use in sapphire-growing furnace materials [8]. The vacuum plasma spraying (VPS) process is done within the controlled low pressure of inert gas, which reduces the interaction between the plasma jet and the oxidative environment. Therefore, VPS technology has the advantages of reducing the oxidation of powder and sprayed deposits, and producing a more controlled coating with greater uniformity and less contamination. VPS technology is currently the favored method for preparing tungsten coatings [9].

This work investigates the influences of a VPS-W coating layer on the thermal resistance of a Mo substrate after a heat exposure experiment. Their microstructural changes were characterized by field emission scanning electron microscopy, X-ray diffraction, and energy dispersive spectrum. Microhardness tests were performed to evaluate the stability of the bonding interface of the VPS-W coatings.

2. Experimental procedure

A Mo substrate of 100 mm × 100 mm × 0.5 mm was coated by using the vacuum plasma spraying technique. The Mo specimens

* Corresponding author at: 7-47 Songdo-dong, Yeonsu-gu, Incheon 406-840, Republic of Korea. Tel.: +82 32 8500 445; fax: +82 32 8500 430.
** Corresponding author.
E-mail address: gscho@kitech.re.kr (G.S. Cho).

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http://dx.doi.org/10.1016/j.materresbull.2013.04.064

Table 1
Vacuum plasma spraying conditions for W coating.

Spraying parameter	Values
Current	1200–1400 A
Voltage	50–100 V
Input power	95 kW
Chamber pressure	35 mbar
Primary gas (Ar)	100 L/min
Secondary gas (H_2)	10 L/min
Spray distance	530 mm

used in this coating test were taken from high-purity Mo (Nilaco, purity 99.95 wt%) with a thickness of 0.5 mm. The thickness of the tungsten coating layer was measured on a fractured surface. Commercially available tungsten powder with a median size of 10 μm in a weight mean was applied to prepare the coatings via a plasma spray system (LVPS, Sulzer Metco). The Mo substrates were grit-blasted with alumina abrasive, and heated to 800–900 °C and cleaned with acetone prior to the plasma spraying process. VPS processing was performed in a controlled vacuum chamber equipped with an articulated robot. Prior to spraying, the chamber was first evacuated, and then a small amount of Ar gas was injected to provide a protective atmosphere from oxidation. The chamber was maintained at low pressure (35 mbar) during spraying. The spraying parameters are given in Table 1. In order to maintain uniformity as well as material properties of the coatings during VPS, it was important to regulate the temperature of the surface during the spraying process. Argon and hydrogen were used as the plasma-forming gases. A heat exposure experiment was performed in the sapphire single crystal growing furnace. The exposure samples were kept at sapphire-growing temperature of 2100 °C for hours and controlled by an optical pyrometer (300–3000 °C). Before and after heat exposure, the sample surface was observed with a scanning electron microscope (SEM). Microstructure and composition changes were also examined by a SEM equipped with an energy dispersion X-ray spectroscope (EDS). The Vickers microhardness tests of the VPS-W coating layers were performed on polished surfaces (cross-sections of the coatings) using a microhardness tester (FM-7, FUTURE, Korea) with a normal load of 100 g and a dwell time of 10 s. X-ray diffraction (MiniFlex II, Rigaku) was applied to measure the crystal structure of the VPS-W coatings.

3. Results and discussion

Fig. 1 shows the surface morphology and EDS result of the as-coated VPS-W layer. Fig. 1(a) shows the macroscopic surface image of the VPS-W coating layer and its EDS spectrum. Spherical W powders were observed on the coating surface. A magnified FESEM image of Fig. 1(a) is presented in Fig. 1(b). Fully melted W splats and spherical-shaped W powders were deposited onto the coating surface. From the EDS results, it was ascertained that a certain amount of oxygen was caused contamination during the vacuum plasma spraying of the W coating. The spraying chamber pressure of the VPS process was in the range of 35–50 mbar with inert argon gas. It can therefore be considered that the VPS-W coating layer may have been oxidized when the heated VPS-W coating sample was moved from the chamber to the ambient temperature. The cross-sectional FESEM images of the as-coated VPS-W layer are displayed in Fig. 2. The as-coated VPS-W coating layer was uniformly deposited onto the Mo substrate shown in Fig. 2(a). The

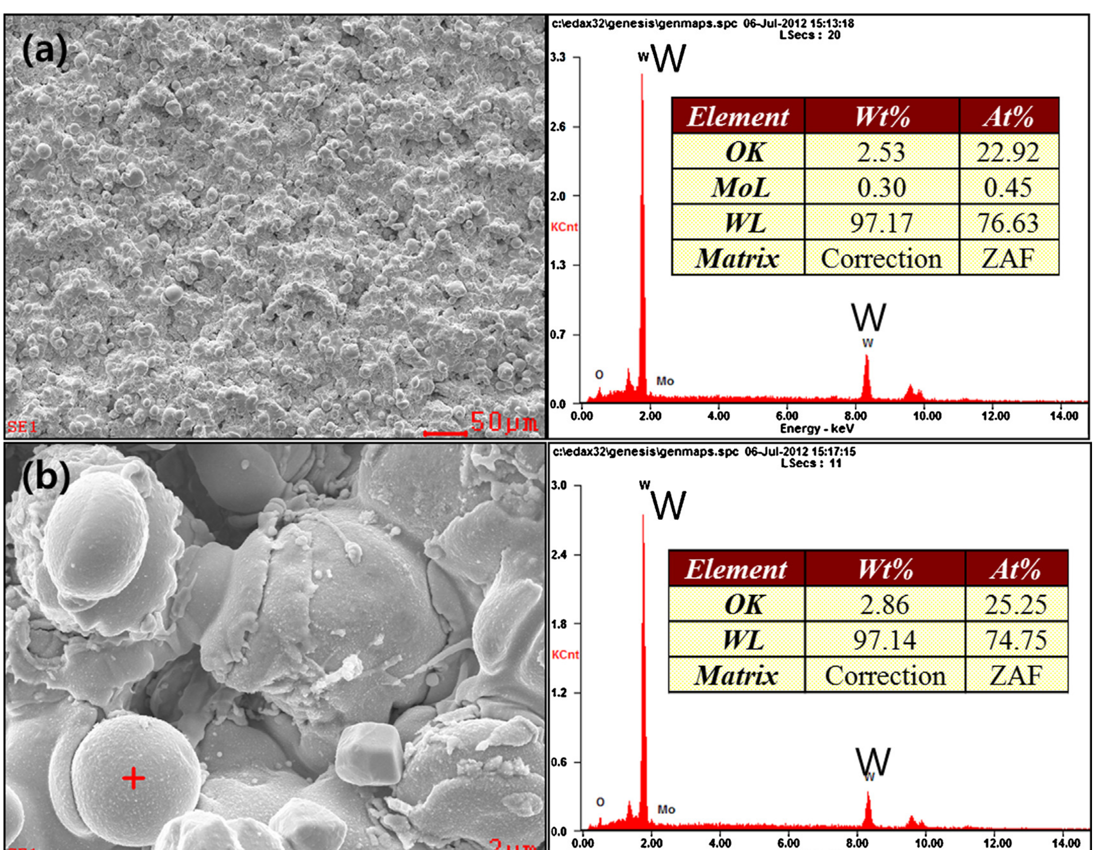

Fig. 1. Surface morphologies and EDS results of the as-coated VPS-W layer: (a) the macro FESEM image and EDS result and (b) the micro FESEM image and EDS result.