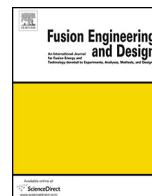




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Effect of pulse parameter on preparation of W coating on V alloy

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HIGHLIGHTS

- Tungsten coatings were electroplated on vanadium alloy by pulse plating.
- The influence degree of current parameters was investigated by orthogonal experimental.
- The pulse parameters affected the performance of tungsten coatings.
- The effects of duty cycle on morphology were investigated.
- The effects of period on morphology were investigated.

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ABSTRACT

The tungsten coatings were prepared on vanadium alloy substrate by pulse electroplating in $\text{Na}_2\text{WO}_4\text{--WO}_3$ molten salt. A series of tungsten coatings with compact and smooth morphologies were successfully obtained under various conditions. Orthogonal experimental design method was used to analysis the influence degree of current density, duty cycle and period on tungsten grain size, coatings thickness and current efficiency. The results demonstrated that current density was the most important factor influencing tungsten grain size and tungsten coatings thickness, which all had a positive correlation with current density. The pulse duty was the most important factor influencing current efficiency; the result also showed a positive correlation between current efficiency and pulse duty factor.

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1. Introduction

Tungsten has the highest melting point among all metals and offers good thermal stability for high temperature applications [1]. Tungsten has been widely used in fusion industry because of its high physical sputtering threshold energy, and low erosion rate under plasma loading [2]. However, the main drawbacks of tungsten, such as low temperature brittleness and difficulty in machining make it expensive for direct application [3]. A promising method for industrial applications of tungsten is to coat the designated part with a thin tungsten layer.

V–4Cr–4Ti alloy is considered as a promising structure material because of its good creep resistance, high thermal stress factor and superior corrosion resistance [4–7]. Compared with low-activation ferritic/martensitic steels, V–4Cr–4Ti alloy exhibits better mechanical properties at high temperature [5]. Thus, V–4Cr–4Ti alloy with

tungsten coating can widely used as a novel and promising material in fusion application. Nowadays, plasma spray (PS) [8], physical vapor deposition (PVD) [9] and chemical vapor deposition (CVD) [10] have been applied in preparing tungsten coatings on various substrates. Nagasaka et al. successfully prepared tungsten coating on V–4Cr–4Ti alloy substrate by vacuum plasma spray (VPS) and brazing and investigated the mechanical properties of W coatings [11,12]. Ueda et al. investigated D retention in a VPS–W coated V–4Cr–4Ti and demonstrated D retention was significant for VPS–W coated V alloy [13]. However, there is no experimental study about fabricating tungsten coatings on V–4Cr–4Ti substrate.

Electrodeposition from molten salts is a promising method for fabricating dense coatings on products with complex shape [14]. Senderoff and Mellors [15] prepared coherent electro-deposition tungsten coatings from fluoride melts at the temperature of 973–1123 V. V. Malyshev and A.I. Gab [14] electrodeposited tungsten coatings on molybdenum substrates from NaF--NaCl--WO_3 melts at 1113–1193 K. Nitta et al. [16] successfully obtained smooth tungsten -films from a $\text{Li}_2\text{WO}_4\text{--Na}_2\text{WO}_4\text{--K}_2\text{WO}_4\text{--LiCl--NaCl--KCl}$ melt by addition of KF at 873 K. Whereas, all electroplating processes mentioned above were conducted in inert atmosphere which would increase the complexity of electroplating equipments.

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Compared to other salt systems, the $\text{Na}_2\text{WO}_4\text{--WO}_3$ melt is non-volatile, non-hygroscopic, chemically stable and easily prepared. An investigation of direct current density on microstructure of tungsten coating has been made by Li [17]. Pulse current (PC) plating has been demonstrated to produce a greater influence on crystal structure and microstructure of coatings as compared to direct current (DC) plating in the solution system [18]. However, studies of pulse current parameters on morphology of tungsten coating on V–4Cr–4Ti substrate have been rarely reported in papers. In this work, an effort was made to produce tungsten coating on V–4Cr–4Ti alloy substrate by electrodeposition in the $\text{Na}_2\text{WO}_4\text{--WO}_3$ system in atmosphere under various conditions. And the effects of the deposition condition including current density, duty cycle, and period on the morphology of tungsten coating were investigated through orthogonal experiment. The influence degree of each factor on performance of tungsten coating was also investigated.

2. Material and methods

2.1. Preparation of tungsten coating

Na_2WO_4 and WO_3 were dried in a furnace at 773 K for 24 h. The dried chemicals were well mixed into a eutectic composition ($\text{Na}_2\text{WO}_4\text{:WO}_3 = 0.6\text{:}0.2$, by mole ratio) and then molten in an electric resistance furnace. The working electrode was a vanadium based alloy plate (V–4Cr–4Ti, 25 mm $\times$ 25 mm $\times$ 5 mm). A tungsten plate (purity: 99.95%, 25 mm $\times$ 25 mm $\times$ 6 mm) was employed as a counter electrode. Prior to electrodeposition, the electrodes' surfaces were mechanically polished to obtain high quality surfaces and then cleaned in acetone and distilled water by ultrasonic cleaning. Tungsten coatings were electrodeposited on V–4Cr–4Ti alloy substrate from the molten salt in an open bath at 1173 K for 1 h using a pulse power supply (HPMCC-5). After the electroplating, the samples were immediately immersed in a 5 M NaOH solution to remove adherent salts.

2.2. Characterization

The surface morphologies of the deposits were characterized by scanning electron microscopy (SEM, JSM 6480LV). The cross section morphologies were observed by scanning electron microscopy (SEM, JSM 6480LV) with line analysis metallurgical microscope (LEICA DMR). The average grain size and coating thickness were calculated using specialist computer software named Image-Pro

Table 1
Levels and factors affecting the properties of the tungsten coatings.

Level	Factors		
	A Current density (mA cm^{-2})	B Duty cycle	C Period (ms)
1	20	0.1	2
2	40	0.25	10
3	70	0.4	50
4	100	0.8	100

Plus 6.0. The tungsten coatings were peeling from the substrate by nitric acid picking, and then washed in acetone and acetone by ultrasonic cleaning. Archimedes method was applied to measure the density of tungsten coating by using analytical balance (YDK01-C, Sartorius, Germany). The oxygen content was measured by the Nitrogen/Oxygen Analyzer (TC600, LECO, USA). The adhesive strength of the coatings was tested by the coating-pull-off test performed according to ISO 4624:2002(E) (2011) and thermal shock test. The coating-pull-off test and thermal shock test were measured three times, and the minimum figure was available. The adhesive was E7 glue according to QJ 1992.14–1990. The thermal shock test was carried out in a furnace. The heating time was 3 min at 800 °C and then it was soon cooled in water at 25 °C for 2 min. The electric current efficiency (η) was calculated by the following formula:

$$\eta (\%) = \frac{m}{CIt} \times 100 \quad (2-1)$$

where m is the mass of tungsten coatings, C is the electrochemical equivalent (1.143 g/(A h)), I is the average cathode current, t is the deposition time.

2.3. Orthogonal experimental design

In this experiment, the effects of electrodeposition parameters were investigated based on an orthogonal method. The theory and methodology of orthogonal have been described design in detail in relevant literature reported by Hosseini and Eslami [19]. Table 1 listed the schedule of the orthogonal array where three key variables with four levels were analyzed: current density (factor A), duty cycle (factor B) and period (factor C). The orthogonal design table $L_{16} (3^4)$ was used and test program was given in Table 2. The grain size, coating thickness and electric current efficiency were taken as evaluation indices. The average and corresponding extreme values were calculated.

Table 2
Performance of samples obtained under different experimental conditions.

Experimental number	Factors			Evaluation indexes		
	A Current density (mA cm^{-2})	B Duty cycle	C Period (ms)	X Average grain size (μm)	Y Thick-ness (μm)	Z Electric current efficiency (%)
1	20	0.4	100	6.58	14.00	89.53
2	20	0.1	2	7.17	10.08	59.65
3	20	0.8	50	7.51	13.26	84.84
4	20	0.25	10	5.42	9.59	76.97
5	40	0.4	10	6.31	44.72	76.71
6	40	0.1	50	7.51	49.80	75.18
7	40	0.8	2	8.26	25.17	79.46
8	40	0.25	100	10.74	42.22	88.56
9	70	0.1	100	5.26	22.70	52.85
10	70	0.4	2	7.88	48.09	61.68
11	70	0.25	50	11.25	39.10	53.25
12	70	0.8	10	12.22	53.71	84.19
13	100	0.1	10	12.88	35.28	56.69
14	100	0.4	50	19.67	64.27	79.46
15	100	0.25	2	11.83	59.11	67.14
16	100	0.8	100	13.06	41.11	78.26

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