Fusion Engineering and Design 88 (2013) 23–27

Contents lists available at SciVerse ScienceDirect

Fusion Engineering and Design

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

Preparation of ultrafine tungsten wire via electrochemical method in an ionic liquid

Xueqi Gao, Wencheng Hu*, Yushu Gao

State Key Laboratory of Electronic Thin Films and Integrated Devices, University of Electronic Science & Technology of China, Chengdu, 610054, PR China

HIGHLIGHTS

- The method of electrochemical corrosion is used to prepare ultra-fine tungsten wire less than 10 μm in diameter.
- Ionic liquid as a non-aqueous electrolyte was used in electrochemical corrosion experiments.
- The situation of anode polarization was different from the usual situation.
- Diameter of tungsten wire has been cut down to 8.5 μm uniformly under the optimized electric potential.

ARTICLE INFO

Article history:
Received 16 May 2012
Received in revised form
27 September 2012
Accepted 28 September 2012
Available online 25 October 2012

Key words:
Electrochemical corrosion
Ionic liquid electrolyte
Ultrafine tungsten wire
Electrochemical behaviors

ABSTRACT

Ultrafine tungsten wire less than 10 μm in diameter is often used as wire array load applied in Inertial Confinement Fusion (ICF) physical experiments. In order to obtain a higher yield of X-ray, both initial radius and line quality of metal wire were required to be of high quality simultaneously. This paper has studied the electrochemical method to corrode tungsten wires uniformly in an ionic liquid electrolyte containing 1 wt% sodium hydroxide. A three electrode system composed of a tungsten anode electrode, a stainless steel cathode and a saturated calomel electrode as a reference electrode, was used in the electrochemical experiments. Liner sweep voltammetry (LSV) and Tafel experiments were used to investigate the electrochemical behaviors of tungsten wires in ionic liquid and aqueous solution. Based on scanning electron microscope (SEM) observation, the morphologies of tungsten wire surface with uniform corrosion under different applied voltages have been demonstrated. X-ray diffraction (XRD) methods were employed to track the evolution of the crystal structure before and after corrosions, and there is an obvious difference in peak intensities. The ultrafine tungsten wire with a uniform diameter of 8.5 μm was obtained under the optimized electric potential (2.5 V) applied for decreasing diameter at 30 °C.

1. Introduction

In a Z-pinch experiment, a magnetic pressure is created by the azimuthal magnetic field associated with the axial flow of current through a cylindrically symmetric plasma that accelerates the plasma radially inward at high velocities [1]. X-rays are produced when the imploding plasma stagnates on the cylindrical axis of symmetry. In recent years, remarkable progress has been achieved in fast Z pinches, where effective conversion of the kinetic energy of imploding plasma into soft X-ray pulses has been achieved [2]. The key factor in this progress has been the use of cylindrical arrays of a large number (~400) of fine (~10 μm) metallic wires as a Z-pinch load [2]. Since the late of 1990s, X-ray output power and energy have been greatly improved in Z-pinch physics experiment due to the using of metal wires with micrometer-sized diameter applied in preparing cylindrical ICF capsule [1,3].

Tungsten is a metal with an extremely high hardness properties, high melting point, and high corrosion resistance. Hence, tungsten wire is one of most common choice applied in Z-pinch physical experiments [4]. For obtaining a higher yield of X-ray to convert electricity energy stored in the accelerator to implosion kinetic energy to the maximum, it was found in the study of Z-pinch that both initial radius and line quality of metal wire were of high quality simultaneous. As the main load wire, tungsten filament was required to be less than 10 μm in diameter, which is called ultrafine tungsten.

Tungsten wires purchased on the market in general are prepared by a drawing machine. The limitations of technologies on mold holes manufacturing, tungsten tensile strength, and running accuracy of drawing machine lead to the production of ultrafine tungsten process quite complex so that drawing process has much difficulty for volume production. Generally, ultrafine tungsten wires, which are below 10 μm in diameter, are obtained by

* Corresponding author.
E-mail address: huwc@uestc.edu.cn (W. Hu).

0920-3796/$ – see front matter © 2012 Elsevier B.V. All rights reserved.
http://dx.doi.org/10.1016/j.fusengdes.2012.09.022

chemical corrosion and electrochemical corrosion. During chemical corrosion process, it is required strictly to control the reaction rate, and it is also difficult to guarantee the consistency and concentricity of wire diameter. Therefore, chemical corrosion is rarely used in actual production. Electrochemical corrosion, as an alternative choice, is applied to produce ultrafine tungsten wires.

In 2005, Motoki et al. [5] used a 5 mol L^{-1} solution of KOH as an electrolytic solution to electropolish a 0.5 mm diameter tungsten wire, and the result showed that the polished tungsten wire has a tip radius on the order of 100 nm. In 2008, Bernal and Ávila [6] suggested that appropriate voltages for carrying out the electrochemical reaction are 2.5 V and 3 V for 2 mm and 3 mm depth of immersion in a 2 mol^{-1} solution of KOH, and they also found that the current was higher for an immersion of 3 mm, which implied the shorter fabrication time with the increasing immersion depth. In 2009, the DC electropolishing process was used by Kulakov et al. [7] to corrode tungsten wires in a 2 mol^{-1} solution of KOH. Reaction mainly occurred in the vicinity of the electrolyte–air interface in order to create two separate wire parts with nanosharp tips. All of the above study groups focused their interest on the corrosion of tungsten wire in alkaline solution to produce tungsten nano-cusps and the discussion of their properties [5–7]. However, few previous reports considered about decreasing tungsten wire in diameter uniformly by electrochemical corrosion.

In the present work, tungsten wires as anodes were decreased in diameter uniformly by electrochemical method in an ionic liquid containing ethylene glycol and choline chloride with a mole ratio of 2:1 and some additives. SEM analysis was also investigated the effect on the result of diameter decreasing under different voltage conditions.

2. Experimental procedures

Analytical-grade choline chloride (ChCl) and ethylene glycol (EG) were purchased from Chengdu Changzheng Chemical Corporation. Ethanol with a purity over 99.7% were from Chengdu Kelong Chemical Reagent Factory. Both analytical-grade Sodium hydroxide (NaOH) and calcium chloride anhydrous ($CaCl_2$) were also from Kelong Chemical Reagent Factory.

The ionic liquid electrolytes were prepared from a mixture of choline chloride and ethylene glycol with a mole ratio of ChCl:EG = 1:2 at 70 °C for 0.5 h [8], and then the mixture was magnetically stirred to form a homogeneous colorless liquid. Before electrochemical experiments, the ionic liquid was treated under a one-tenth atmospheric pressure at 100 °C for 8 h. Further purification of ethanol was a series of treatment involved adding $CaCl_2$ to ethanol, ultrasonic oscillation for 10 min, and then placed the mixture still for 24 h, followed by filtration. Finally, non-aqueous electrolyte solution was obtained from a uniform mixture of ionic liquids with 1 wt% ethanol solution of sodium hydroxide as a kind of additive.

Prior to the electrochemical experiment, tungsten wires were immersed in a 3 wt% NaOH solution to remove the oil stains and tungsten oxide in an ultrasonic cleaner, and then washed with water and ethanol in turn. After dried in atmosphere, the tungsten wire was placed in the electrolyte solution as an anode as shown in Fig. 1. The top part was connected to the positive of the power supply, and the opposite part was attached by a small PTFE ring to keep the tungsten electrode vertical. In this system, a cylinder-shaped steel counter electrode was used to surround the tungsten electrode, and a saturated calomel electrode as a reference electrode was applied during electrochemical experiment.

In order to reduce tungsten diameter uniformly, we used constant voltage electrolysis corrosion at 30 °C. Electrochemical behaviors of tungsten surface were characterized by Tafel and LSV tests recorded in an electrochemical workstation (CHI660D). XRD experiments were performed in a Philips X'pert X-ray diffractometer with Ni-filtered Cu Kα radiation and a 0.15406 nm wavelength operated at 40 kV and 40 mA. A field-emission scanning electron microscopy (FE-SEM, Inspect F Co.) was used to observe surface morphology of the corroded tungsten at 20 kV. As a supplement, the compositions of the tungsten surface were characterized by an EDAX Co. energy-dispersive X-ray spectroscopy (EDS) instrument.

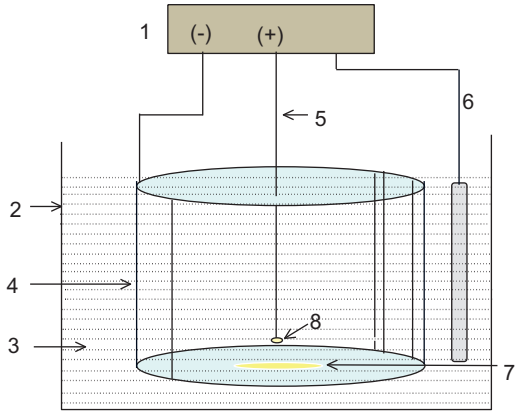

Fig. 1. A schematic diagram of experimental set up: (1) power supply, (2) glass electrolytic cell, (3) electrolytic solution + additives, (4) counter electrode, (5) working electrode, (6) reference electrode (SCE), (7) magnet and (8) a PTFE ring.

3. Analysis and discussion

3.1. Electrochemistry of the tungsten electrode in the ILs electrolytes

Fig. 2 shows a relationship curve of the corrosion voltage of tungsten wire with current value in ionic liquid. A tungsten wire working electrode, a steel counter electrode, and a SCE reference electrode were assembled to form a three-electrode system in electrochemical experiment. The applied potential is scanned from 0 V to 4 V to track the electrochemical reaction. From Fig. 2, there is no passivation interval observed, indicating a new mechanism of tungsten dissolved. As the applied voltage increases, the corrosion of tungsten atoms is transformed from a state of active dissolution to a state of rapid corrosion directly. Tungsten atoms on the surface

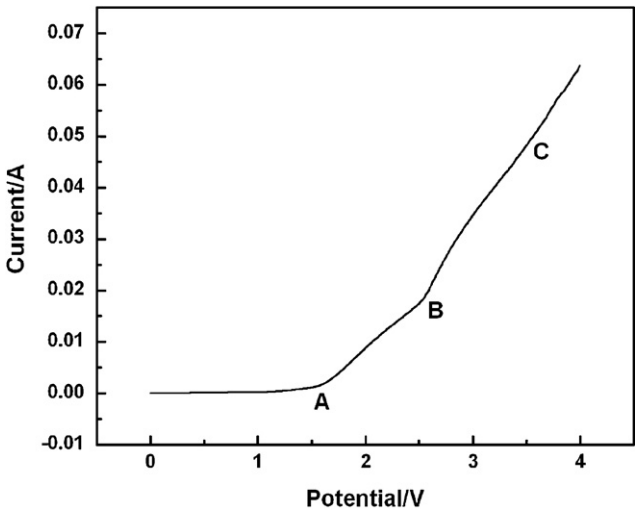

Fig. 2. curve of tungsten anodic polarization was performed in the ionic liquids at 30 °C, which sweep voltage was from 0 V to 4 V.