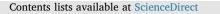
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## Abrasive-free polishing of tungsten alloy using electrochemical etching



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#### ABSTRACT

Tungsten alloy is a crucial engineering material for electrical and optical applications. However, damage-free and highly efficient polishing of tungsten has not been realized yet. We report the abrasive-free polishing of tungsten alloy using electrochemical polishing (ECP), which is an etching process. To achieve balance between polishing efficiency and surface quality, a two-step ECP process has been proposed. Current-driven ECP lasting for 3 min, as the first step, quickly removed the surface grinding marks and subsurface damage while the following potential-driven ECP lasted for 20 min, as the second step, improved the surface roughness and an ultra-smooth surface with an Ra roughness of 17.6 nm was finally obtained.

#### 1. Introduction

Tungsten, as a transition and refractory metal, has been used in various fields owing to its excellent physical, mechanical and chemical properties. Tungsten is widely used as the wiring material in fabrication of integrated circuits owing to its good electric conductivity and low coefficient of thermal expansion [1,2]. Tungsten has also been used in some high temperature applications such as SEM filaments or nozzles for high temperature fluids owing to its high melting point (3422  $\pm$  15 °C), which is the highest of all metals [3,4].

In recent years, glass molding, which is a newly emerging but promising application of tungsten, has been proposed [5]. Currently, most optical glass lenses are produced by molding using ultra-precision molds made of sintered tungsten carbide (WC) [6,7] or chemical vapor deposition-silicon carbide (CVD-SiC) [8,9]. WC and SiC have many excellent chemical and mechanical properties, like strong chemical inertness, high oxidation temperature, low coefficients of thermal expansion, high hot hardness and so on, making them superior as the mold materials for precision molding of glass lenses. However, these hard and brittle materials are difficult to machine using conventional ultra-precision machining methods like cutting, grinding and polishing [10]. These problems in machining of WC and SiC greatly limit the costreduction of the molding process. Thus, in recent years, tungsten alloy has been proposed to be used as the mold insert material for replacement of WC or SiC. Tungsten has lower hardness than WC and SiC but is hard enough for glass molding. Meanwhile, tungsten has better thermal properties such as a high melting temperature and a low coefficient thermal expansion. More importantly, tungsten alloy is machinable

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Received 12 July 2017; Received in revised form 25 July 2017; Accepted 31 July 2017 Available online 01 August 2017 1388-2481/ © 2017 Elsevier B.V. All rights reserved. using commercial carbide tools. These properties make tungsten a very economic mold material for glass molding.

For removal of subsurface damage and improvement of surface roughness, polishing is an indispensable step in the manufacturing process of tungsten alloy molds. Chemical mechanical polishing (CMP) using silica slurry has been widely used to polish tungsten substrates [11,12]. Even though excellent polishing characteristics of CMP have been demonstrated, the low polishing efficiency and large amount consumption of slurry make it a costly process. A highly efficient and slurry-free polishing technique, electrochemical polishing (ECP), has been widely used for planarization or polishing of metals or semiconductor substrates [13]. ECP is more cost-effective than CMP as there is no need to use slurry. Meanwhile, it is a damage-free process as there is no mechanical removal in ECP [14]. Recently, ECP has been applied to polish some difficult-to-polish metal materials like titanium alloy [15].

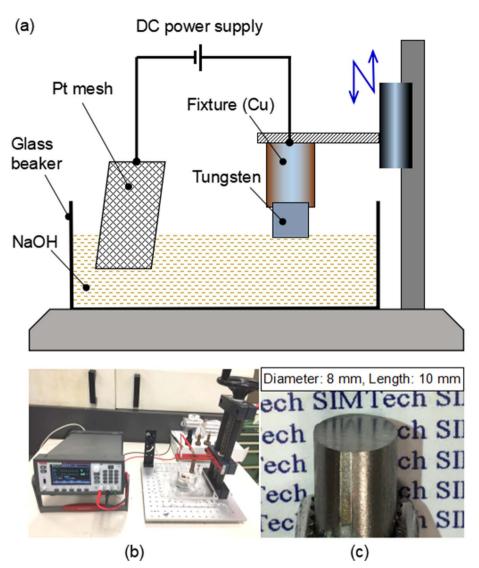
In this communication, an abrasive-free and highly efficient polishing process based on electrochemical etching is applied to tungsten alloy and the results are presented.

#### 2. Experimental

#### 2.1. Electrochemical polishing (ECP) of tungsten

The substrates used in this research are pure tungsten alloy (> 99.99%) with a diameter of 8 mm. The substrates were cut from a tungsten rod using electro-discharge machining and their end faces were ground using a diamond grinding wheel (#120). Fig. 1 shows the

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Fig. 1. Schematic (a) and photo (b) of experimental setup and photo (c) of tungsten substrate.

schematic and photo of experimental setup used in this work. Etching was performed in a glass beaker and NaOH solution was used as the electrolyte. A platinum mesh ( $20 \text{ mm} \times 20 \text{ mm}$ ) was used as the counter electrode and tungsten substrate as the working electrode. KEITHLEY 2280S provided electric current to perform electrochemical etching. To realize electric contact, the tungsten substrate was inserted into a copper fixture which was mounted on a vertical lifting platform. The distance between the substrate center and the counter electrode was 30 mm. During anodizing, the position of the substrate was immersed in the electrolyte and etched. After anodic etching, samples were rinsed in water and dried by blowing N<sub>2</sub>.

ECP of tungsten, which is an electrochemical etching process, is based on the simultaneous anodic oxidation and dissolution. The electrochemical reactions occurring on the cathode (platinum mesh) and anode (tungsten) can be expressed as follows:

Cathode:

$$6H_2O + 6e^- \rightarrow 3H_2(g) + 6OH^-$$
 (1)

Anode:

$$W(s) + 60H^{-} \rightarrow WO_{3}(s) + 3H_{2}O + 6e^{-}$$
 (2)

 $WO_3(s) + 2OH^- \rightarrow WO_4^{2-} + H_2O$  (3)

Anodizing as Eq. (2) occurs on the surface of tungsten and tungsten

is oxidized to WO<sub>3</sub>. The generated WO<sub>3</sub> reacts with the electrolyte (NaOH) and gets dissolved as Eq. (3). The flattening mechanism of ECP has been widely studied and several hypotheses have been proposed among which "viscous film theory" proposed by Jacquet is most widely accepted [16]. According to the viscous film theory, the dissolution products accumulate near the anode and form a viscous layer that increases the electrical resistance of the system and limits the current. This viscous layer over peaks is much thinner than over depressions. Consequently, the electric current density over peaks of substrate surface is higher than that over depressions, which results in the fact that peaks dissolved faster and the leveling effect can be expected.

#### 2.2. Characterization

The loose weights of tungsten substrates were measured to calculate the material removal rate (MRR) of ECP. Before and after ECP, the surface roughness was confirmed by measurements using a stylus profilometer (Talysurf 200) while the surface morphology was measured by a scanning electron microscopy (SEM, Hitachi S-4800). Surface composition of tungsten substrates before and after polishing was measured by X-ray photoelectron spectroscopy (XPS, ESCALAB 250Xi), in which AlKa (1486.6 eV) radiation served as the excitation source and the measuring area was 900 µm in diameter. Download English Version:

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