Applied Surface Science 424 (2017) 290–298

Contents lists available at ScienceDirect

Applied Surface Science

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

Synthesis and characterization of sputtered titanium nitride as a nucleation layer for novel neural electrode coatings

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ARTICLE INFO

Article history:
Received 20 November 2016
Received in revised form 18 March 2017
Accepted 30 March 2017
Available online 8 April 2017

Keywords:
Titanium nitride
Nucleation layer
RF magnetron sputtering
Deposition rate
X-ray diffraction
Resistivity

ABSTRACT

A growing demand for chronically implantable electrodes has led to a search for the most suitable neural electrode interface material. Nobel metals such as platinum (Pt) are inadequate for electrode/neuron interfaces at small scales due to their poor electrochemical properties, low charge injection and high charge density per unit area. Titanium nitride (TiN) has been implemented in neural electrodes application due to its outstanding properties. In this work, TiN_x films were deposited by non-reactive radio frequency (RF) magnetron sputtering towards the development of a novel TiN nanowires (NWs) neural interface. Although, there is substantial work on this material, its growth using non-reactive RF magnetron sputtering has not been reported previously and optimised towards the growth of TiN NWs and their use in neural interface applications. The sputtering parameters of RF power and argon (Ar) flow rate were varied in order to investigate their effects on the structural, electrical and electrochemical properties of the TiN films. A dense film morphology was observed in the scanning electron microscopy (SEM) images of TiN thin films showing a columnar structure. The film preferential orientation was changed between (200) and (111) with Ar flow rate due to the variation of the kinetic energy (KE) of the sputtered atoms. The crystallites size obtained were in the range of 13–95 nm. Surface roughness was found to increase from 0.69 to 1.95 nm as Ar flow rate increased. TiN_x films showed a good electrical resistivity of 228 $\mu\Omega$ cm. Stoichiometry was found to vary with sputtering conditions in which the nitrogen content was found to deplete from the film at low Ar flow rate. The electrochemical behaviour of TiN films were characterised and the highest capacitance value obtained was 0.416 mF/cm^2. From the results, it can be suggested that TiN thin film can be easily optimised to act as a nucleation layer for the growth of nanowires.

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1. Introduction

Neural stimulation and recording electrodes are considered life-sustaining devices since they are used in applications such as Parkinson's disease [1]. Such applications impose special requirements on the electrodes to be able to chronically interface with tissues and deliver stimulus which are sufficient to trigger an action potential. Moreover, to enhance their efficacy, it would be of benefit to reduce the electrode dimensions to the microscale and below to minimise tissue trauma and invasiveness, hence achieving facile selectivity in recording and stimulation [2]. However, reducing the electrode dimension can limit the electrical, mechanical and biological performance [3]. For example, as the size of electrode is reduced the charges that must pass per unit area increases, causes an increase in electrode voltage and hence reducing the total charge which can be safely delivered [3]. Therefore such electrodes demand alternative neural interface materials that are capable of delivering adequate charge densities that are larger than the traditional materials such as platinum (Pt), iridium oxide (IrO) and platinum-iridium (Pt-Ir) [3]. A one way to compensate for the limited electrode performance caused by the reduction of electrode dimension is to have high surface area electrodes [4]. Modifying the electrode's surface by coating it with nanostructured materials has shown great potential for enhancing the efficacy of neural-electrode interfaces. Such modifications have led to improved electrochemical properties with an increased surface area facilitating increased charge transfer within the safe stimulation limit [4], and a reduction in the impedance of the electrodes. In addition, having a nanostructured coating on the electrode, such as nanowires or nanotubes, may help to minimise the stress at the cellular interface [4]. This is due to the fact that long nanowires or nanotubes have a degree of flexibility and can undergo deformation, which facilitates

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http://dx.doi.org/10.1016/j.apsusc.2017.03.277
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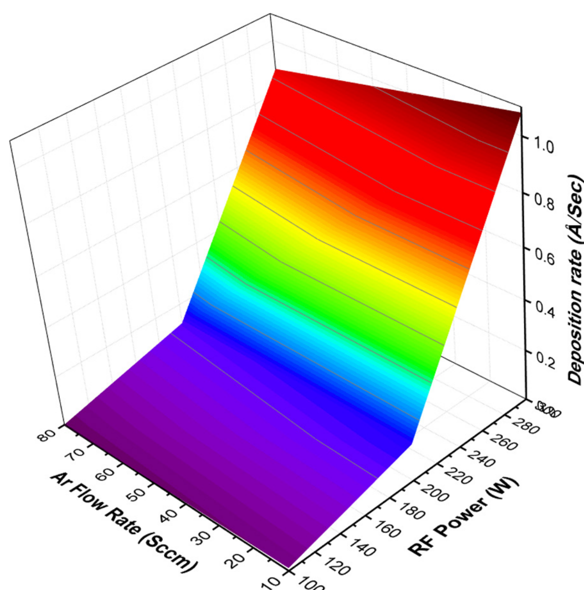

Fig. 1. Effect of RF power and Ar flow rate on deposition rate.

Table 1
Experimental conditions for the DoF optimisation of TiN.

	Level 1 (min)	Center point	Level 2 (max)
RF power	100 W	200 W	300 W
Argon flow rate	10 sccm	45 sccm	80 sccm

a better tethering mechanism with the tissues [5]. These improvements are considered key factors for safe stimulation and recording over a long period of time. The safe stimulation or recording of neurons is evidenced when there is no irreversible electrolysis of water taking place at the interface. If the charge injection through the electrode material is maintained between two voltage limits, called the water window, no irreversible electrolysis of water can occur. However, beyond this electrochemical limit, tissues around the electrode can be damaged and the electrode can degrade over time [6].

Titanium nitride (TiN) is a potential alternative material for neural stimulation and recording and has served as a neural electrode coating material for several decades [7]. It has been also employed in many industrial applications such as a diffusion barrier [8], an adhesion layer for electronic devices [9] and in solar cell applications [10]. TiN exhibits exceptional hardness, high thermal and chemical stability, low electrical resistivity and high resistance to wear and corrosion [11]. Indeed, of particular importance here, TiN has a higher charge injection as compared to Pt; the highest charge injection reported for TiN thin film is in the rage of 2.2–3.5 mC/cm^2 whereas Pt can vary between 0.02 and 0.15 mC/cm^2 [3].

In this study, TiN thin films were deposited by non-reactive RF sputtering which offers a simpler approach by only optimising RF power and Ar flow rate. This has not been reported previously or has the use of TiNWs as a viable neural electrode coating. One of the main aims of the application of this work is to utilise the sputtered TiN layer as a nucleation substrate for the growth of TiN NWs. Our previous work usingsputtered zinc oxide (ZnO) thin film as a nucleation layer for the production of nanostructured materials, demonstrated the validity in this approach [12].

Since the microstructural characteristics of the grown nanowires are likely to be influenced significantly by the structural and functional properties of the nucleation layer, it is essential to optimise the experimental parameters. There have been many deposition techniques implemented for the preparation of TiN

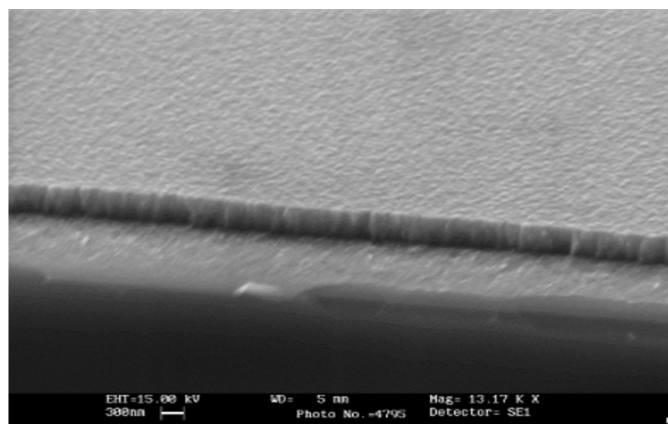

Fig. 2. SEM image of TiN deposited at 300 W 80 sccm showing columnar structure.

thin film including, chemical vapour deposition [13], reactive evaporation [14] and reactive sputtering [9]. In this study, radio frequency (RF) magnetron sputtering of a pure TiN target is used to deposit thin films of TiN and the effects of the sputtering parameters on the physical and electrochemical properties are discussed with relevance to its suitability as a nucleation layer for TiN NWs growth and their subsequent use as a neural electrode interface material.

2. Experimental

TiN_x was sputtered using RF magnetron sputtering and an eight inch high purity (99.99%) TiN target in an argon (Ar) gas atmosphere. Silicon wafers (100) and Corning glass (2855) were used as substrates (2 cm × 2 cm). For all depositions, the distance between the target and the substrate was kept constant at 7 cm, the reaction chamber was evacuated to a base pressure of 4×10^{-6} Torr and depositions were conducted at room temperature. The Ar flow rate was controlled by a mass flow controller. Prior to all depositions, silicon and glass substrates were ultrasonically cleaned with deionised (DI) water, acetone and isopropanol for 30 min each. The substrates were again rinsed with DI water and dried under nitrogen gas. The TiN target was pre-sputtered for 10 min to remove potential impurities from the target surface before performing any deposition.

The effect of RF power and Ar flow rate on crystallography, crystallite size, root mean square (RMS) roughness, stoichiometry, resistivity and electrochemical properties of the thin films were investigated by varying the sputtering power and Ar flow rate between 100–300 W and 10–80 sccm, respectively,. Moreover, to investigate the effect of thickness variation on the properties of TiN films, a series of films with various thicknesses were prepared.

2.1. Characterisation method

Film thicknesses were obtained by measuring the step heights of the sputtered films using atomic force microscopy (AFM) (Park AFM XE100). AFM was also used to characterise the topographical structure as well as surface roughness in non-contact mode. Scanning electron microscopy (SEM) (Leo S430) was used to examine the surface morphology of TiN. The crystallographic orientation of TiN films were characterised by X-ray diffraction (XRD) (Bruker D8 with Cu-Kα X-ray radiation having a characteristic wavelength of 1.5418 Å with 2°/min scanning speed. Using the full-width at half-maximum (FWHM) of diffraction peak of the XRD patterns,

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