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## Thin Titanium Nitride films deposited using DC magnetron sputtering used for neural stimulation and sensing purposes.

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### Abstract

In recent times Platinum (Pt), Iridium (Ir), Iridium oxide (IrO) and Platinum-Iridium (Pt-Ir) are the favorable microelectrode materials. They show excellent electrical and mechanical properties suitable for neural stimulation and sensing/recording purposes. But their long term stability and performance is still in question especially working in conductive saline environment. Titanium Nitride (TiN) is advantageous than the mentioned noble metals used as microelectrode material for nerve stimulation and sensing. TiN films are generally used in biomedical implants due to their good mechanical and high corrosion resistance with extreme biocompatibility. Here we talk over the capabilities of sputtered TiN material as a microelectrode material with respect to its mechanical and electrical properties. Also, we discuss the initial results for sputtered TiN layers and its surface properties suitable for electrical stimulation and sensing neuronal activity. TiN was chosen because it readily lends itself to reactive sputtering method and provides significant charge injection rates  $23 \text{ mC/cm}^2$  with excellent corrosion and biocompatibility properties. Titanium (Ti) and TiN thin films of 200 nm thick were deposited by DC magnetron sputtering process on plain silicon substrates. Initial AFM and XRD characterization study was carried to study the crystal structural properties of these thin films.

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*Keywords:* Biomedical implants, Titanium Nitride, Neural stimulation and sensing, DC sputtering.

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### 1. Introduction

Biomedical implants such as brain stem implants, cardiac pacemakers, cochlear implants etc., intended to serve various application in human beings enforce special requirements on the microelectrode which

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are used for stimulation current delivery by stimulating the nerves. Microelectrodes are necessary to localize the volume by stimulation current sufficient enough to trigger an action potential in the nerve, mostly in the central nervous system. Micron size electrodes should be capable of managing larger current and charge densities than the traditional noble metals such as platinum and iridium oxide. It should exhibit low impedances, greater corrosion resistance especially against the conductive harsh and saline working environment and high reversible charge injection limit. This transfer process occurring at the electrode electrolyte interface is subjected to a charge balanced stimulation signal without leading to faradaic currents and thereby irreversible redox reactions, possibly causing the formation and release of toxic products in the electrolyte [1]. Noble metals like platinum (Pt), platinum-iridium (Pt-Ir), gold (Au), iridium (Ir), and iridium oxide (IrO) are commonly used as microelectrode materials. They are selected because of their ability to inject a considerable amount of charge with negligible electrode degradation. To avoid tissue damage and electrode degradation with long term device stability devices are operated at well below the charge injection limit of the electrode material. There are several techniques for depositing these materials such as thermal evaporation, physical vapour deposition (PVD) or magnetron plasma sputtering, ion beam coating, atomic layer deposition (ALD), electroplating and chemical vapour deposition. These methods are well established for microelectrode fabrication. Among these the sputtering techniques are considered the most suitable methods which are being extensively used for metal deposition. Although these materials have demonstrated their robust capabilities, they still have some drawbacks out of which the important one is metal degradation and corrosion [2], especially in conductive saline environment where the limits of reversible charge injection capabilities have been exceeded.

Titanium Nitride (TiN) is one of the material which can be looked upon in future for such type of these applications. TiN in general application is used as diffusion barrier layers in semiconductor industry, in glass and solar industries as reflecting materials and also as protective coatings in ornament industry. It's use in modern microelectronics is because of its excellent electrical and mechanical properties, low electrical resistivity and metallurgical stability at high temperatures [3].

## 2. Fabrication procedure.

TiN thin layers are generally deposited by physical vapor deposition (PVD) techniques which results in a micro-columnar structure rendering an increased effective available surface area which is ideal for nerve stimulation purposes. TiN was chosen in our study because of its extensive use in medical industry for bio-medical applications [4] as an capable microelectrode material and it suits easily itself to reactive sputtering method in micro-fabrication technologies and provides significant charge injection capacities.

TiN metal layers are selectively patterned by lithographic techniques on silicon substrates. These films were sputter deposited on p-type Si(100) wafers by DC reactive magnetron sputtering from a titanium target of 332 mm diameter with 99.999% purity, using Sigma 204 SPTS deposition system. The distance between target and specimen during deposition was 27.5 mm. By roughening and cryo-pump, fitted to deposition chamber, a base pressure of  $2.106 \times 10^{-8}$  mbar is achieved. At this pressure the chamber was purged with pure Argon (100 sccm) and Nitrogen (300 sccm). The working pressure during deposition is between 0.0066 mbar to 0.0133 mbar. Prior to sputtering, titanium target was cleaned at 5 kW by pre-sputtering method for 2 minutes in argon atmosphere to avoid oxidation and nitriding of the target surface. Before deposition the wafers were cleaned by standard nitric acid cleaning. As the substrate temperature between 27 and 400°C has insignificant effect on mechanical properties, sputtering was carried at 300°C. To study the RF power effects on the crystallography, stress and surface roughness we deposited TiN at 0.5, 2.5 and 5 kW with different time to achieve 200 nm thickness. The substrate bias voltage was kept to 0 V for optimum sputtering conditions.

### 3. Results and Discussions.

To study the microstructural surface difference, Ti and TiN of 200 nm thick were deposited with above sputtering conditions. These layers were subjected to stress measurement before and after deposition by using wafer curvature technique. From Fig. 1 it's clear that TiN layers exhibit low tensile stress for low power and compressive stress for layers deposited at high power.

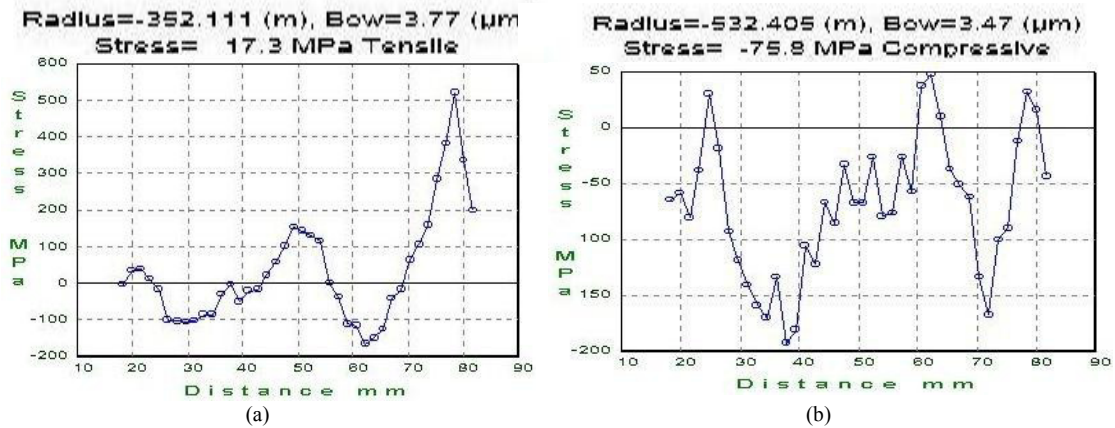


Fig. 1. Stress graph for sputtered (a) Titanium (200 nm) and (b) Titanium Nitride (200 nm) at 0.5 KW power.

The sheet resistance was  $73.45 \Omega/\square$  which is well within the requirements and further process parameter changes did not cause deviation from the required electrical properties.

#### 3.1. AFM characterization

Surface topographical characterization was done by Atomic Force Microscopy (NTEGRA Aura AFM). The AFM scan was carried with semi-contact mode on both sputtered Ti and TiN for a scan area of  $5 \mu\text{m} \times 5 \mu\text{m}$  on the surface. From the AFM images (refer Fig. 2) titanium surface shows more hillock type structure with less porous surface, on the other hand titanium nitride surface shows porous structure consisting of tapered crystallites with less hillocks than the titanium surface.

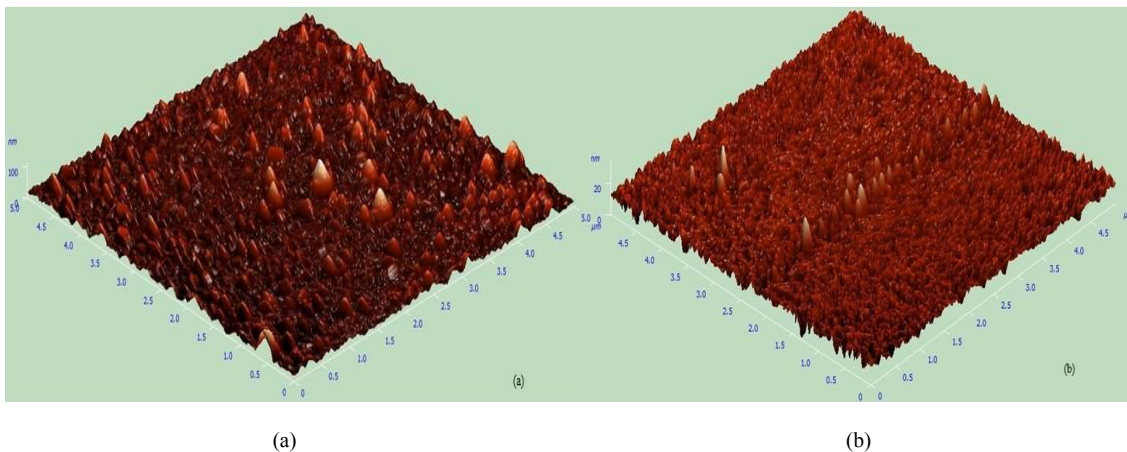


Fig. 2. AFM topographical images of (a) Titanium (200 nm) and (b) Titanium Nitride (200 nm).

### 3.2. AFM characterization

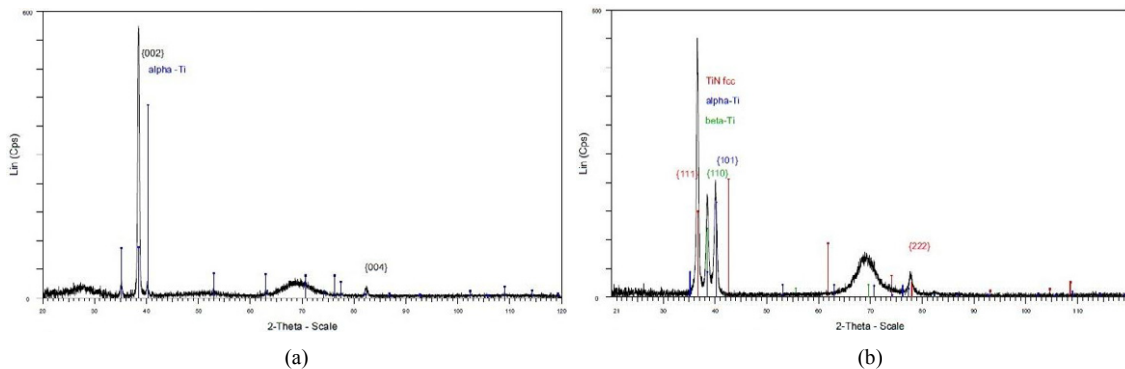


Fig. 3. XRD pattern for (a) 200 nm Titanium and (b) 200 nm Titanium Nitride on Silicon substrate.

The crystal structure of the films was examined using a fixed angle X-ray diffraction (XRD) using a parallel beam of  $\text{CuK}_\alpha$  radiation operating at 40 kV and 40 mA. After sample alignment, two diffraction patterns were recorded between  $20$  and  $120^\circ$   $2\theta$  with a fixed omega offset of  $2^\circ$ , step size  $0.02^\circ$  and step time of 0.5 sec. Figure 3 shows the measured XRD pattern for Ti and TiN. The coloured sticks give the peak positions and intensities of the identified phases of Titanium and Titanium nitride. The blue sticks in figure 3 (a) and (b) shows the peak positions and relative intensities of Ti with random orientation of all hkl planes.  $\alpha$ -Ti  $\{001\}$  is highly textured. For TiN sample cubic fcc TiN  $\{111\}$  phase is textured. The titanium is multiphase with presence of  $\alpha$  and  $\beta$  phases [refer Fig. 3(b)]. The “bump” in both XRD patterns seen above around  $69^\circ$   $2\theta$  is due to the underneath silicon substrate.

### Conclusions

Thin Ti and TiN films are prepared by PVD methods. Initial characterization of films by AFM and XRD was done. Future work will include deposition of TiN films of various thicknesses with altering sputtering parameters and a detailed X-ray diffraction study. In-vitro experiments are planned to check the material performance for stimulation/sensing and its biocompatibility in saline solution.

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