



## Full Length Article

## Interface and stability analysis of Tantalum- and Titanium nitride thin films onto Lithiumniobate

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## ARTICLE INFO

## Article history:

Received 17 March 2017

Received in revised form 28 June 2017

Accepted 29 June 2017

Available online 30 June 2017

## Keywords:

TaN

TiN

Interface

LiNbO<sub>3</sub>

ARXPS

Stability

SAW

## ABSTRACT

Modern surface-acoustic-wave (SAW) devices are characterized by their trend to higher frequencies, power densities and new applications. For this, a shrinking of the dimensions is necessary resulting in an increased power density and temperature within the metallization.

To reduce the emerging damaging effects (acustomigration, diffusion etc.) additional barrier layers between substrates and electrodes are necessary, especially for high temperature applications.

In this context, we present results of detailed chemical interface analysis of sputtered TaN and TiN thin films as potential barrier layers onto SAW-substrate material (LiNbO<sub>3</sub>) with respect to their temporal (up to 8 h) and thermal stability up to 600 °C in vacuum. We report good stability of both systems. The main technique for analysis was non-destructive and surface sensitive angle-resolved X-ray photoelectron spectroscopy (AR-XPS).

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## 1. Introduction

Since the beginning of the 1970s an effect based on high frequency crystal deformation due to electric stimulus, called surface acoustic wave (SAW) is used for electronic devices, e.g. as frequency filters [1,2]. Recently, SAW devices are also used in sensor applications and for the transport/nebulisation of fluids [3–5].

The implementation of these technologies in future devices requires a shrinking in dimensions parallel to an increase in power density. However, these new requirements lead to challenges for the lifetime of the whole device. The main failure mechanism of SAW devices is the damage of the (aluminium) electrodes caused by the electro-mechanical stress at the interface between the substrate and the electrodes [6–9] which is called acustomigration [10]. For the reduction of this effect, the implementation of a thin adhesion and barrier layer based on TaN and TiN resp. seems to be suitable due to their temperature stability [11–13], Fig. 1. The present work was stimulated by similar previous studies of our

group on the behaviour of Ti- and Ta-based barrier layers on Si and SiO<sub>2</sub> substrates [14–17].

The scope of this work is the chemical analysis of the interface formation of TaN and TiN thin films deposited onto the LiNbO<sub>3</sub> substrate material and the subsequent study of their stability against time (up to 8 h) and thermal treatment (up to 600 °C), based on the authors' previous work on surface preparations for such depositions and pure Ta and Ti thin films [18–20]. The upper temperature limit is chosen due to starting degeneration of Al electrodes (melting temperature at 660 °C) in a targeted complete metallization stack (not shown). The work is to understand as a basic study of interface reactions that occur with the highly reactive metal Ta and Ti in combination with N onto complex oxide and piezoelectric substrates. The characterisation of the thin films and interfaces will be mainly performed using angle-resolved X-ray photoelectron spectroscopy (ARXPS) which is approved as a non-destructive method for combined chemical and morphological investigation method for thin films and interfaces in the nm-thickness region.

## 2. Experiments

For the *in situ* analysis of the deposited thin films, a PHI 5600 CI (Physical Electronics) XPS system with a standard non-monochromatized Mg K $\alpha$  X-ray source and a pass energy of

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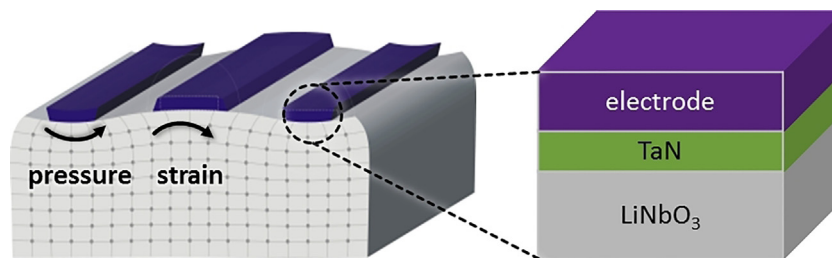


Fig. 1. Schematic sketch of a SAW device metallization with a thin TaN barrier layer.

29.35 eV was used. Due to its combination of chemical analysis with non-destructive depth-profiling up to 5 nm [21], interface studies were done by ARXPS. A motorised stage tilt mechanism was used for angle-resolved measurements. The measurement spot size had a diameter of about 800  $\mu\text{m}$ . Because this depth-profiling is based on a mathematical processing of the measured XPS concentrations, the final analysis for the layer model was done using complex mathematical algorithms based on the electron attenuation length within the surface materials [18,22]. The experimentally determined concentrations for each angle were provided by the MultiPak<sup>®</sup> [23] software with the included sensitivity factors for each pure element (RSF). Directly connected to the XPS system was a deposition chamber; both with base pressures below  $1 \times 10^{-7}$  Pa and  $1 \times 10^{-6}$  Pa, resp. The removal of surface contamination prior to deposition was done using a low energy rf-plasma treatment with He ions that results in a residual carbon based contamination below 1 at.-% [18].

The TaN and TiN depositions were inspired by previous work [15,18] and done in the above mentioned vacuum chamber that is directly connected to the XPS analysis chamber, with a 1 in. (diameter) target and a DC sputter source at 100 nW with a distance of 30 cm to the substrate. This setup enables to reach low deposition rates, which are necessary for studying the first stages of interface formation. Because of the very low base pressure the comparability with standard deposition parameters are ensured. Deposition was done at a working pressure of 1 Pa and a gas flow mixture of 30 sccm (1:2 – Ar:N<sub>2</sub>) controlled by two MFC (mass flow controller) at an approx. deposition rate of 0.1 nm/s (TaN) and 0.07 nm/s (TiN) measured with a quartz crystal monitor. Film thickness homogeneity was measured by X-Ray fluorescence spectroscopy (XRF) utilizing a 5  $\times$  3-point matrix across the whole surface with a relative deviation from the mean value of  $\pm 5\%$ . The LiNbO<sub>3</sub> samples had a crystal surface orientation of 128° YX-rotated and were cut to 15 mm  $\times$  8 mm  $\times$  0.4 mm pieces.

The achieved stoichiometry was verified using WDX (Wavelength-dispersive X-ray spectroscopy, “IncaWave 700” Oxford Instrumens) at prepared films of 100 nm thickness by comparison with certified TaN and TiN reference samples. It should be mentioned that XPS quantification of even these reference films did not result in an exact 1:1 ratio of the metal and nitrogen due to RSF.

Deposition series were done using the nominal deposition parameters for WDX-verified stoichiometric TaN and TiN with stepwise increasing film thicknesses and a subsequent ARXPS analysis for each deposition step. In this way it is possible to determine the interface layer structure until approx. 5 nm overall film thickness, when it is limited by the vanishing substrate signal due to limited photoelectron escape depth. All depositions were performed without any additional temperature management of the substrates at room temperature. Both Ar and N<sub>2</sub> gases had a purity of 5N.

Thermal treatment (30 min) was done in the same vacuum chamber as used for deposition using an electron emission heater up to 600 °C for heating from the backside of the sample carrier.

The temperature was measured and calibrated by a thermocouple. The heating-up to nominal temperature took approx. 2 min.

### 3. Results

The deposited TaN and TiN films were first characterised with respect to their specific interface formation onto the substrate surfaces and followed by a comparison of their temporal and thermal stability in ultra-high vacuum.

#### 3.1. Interface formation of deposited thin films

The binding energies of the relevant elements O1s, N1s, Ta4f and Ti2p were determined by XPS and are listed in Table 1. They are in good agreement with the literature values given in brackets.

Although these values are well-known, the detailed analysis of the chemical bonding at the interfaces is challenging. Due to the fact that the metals have a doublet peak in the used emission lines (Ta4f, Ti2p, Nb3d) even simple metal-oxide-nitride composites need at least six (partially constrained) complex peakfit curves. Additionally, the Mg K $\alpha$  X-ray source leads to a relatively large minimum linewidth of 0.7 eV (Ag3d<sub>5/2</sub>) and the deviation of the individual binding energy in reference data can be as large as  $\pm 0.4$  eV. All these effects can accumulate and increase the uncertainty in the detection of specific chemical binding energies. Iterative Shirley background is used in all spectra.

Additionally, the ARXPS layer model has certain intrinsic systematic errors (straight line approximation for electron trajectory, uniform layers, island formation, insignificant surface roughness etc.) that can influence the values for element concentration and layer thickness [18,21,22].

In light of this, the main focus of the following analysis lies in the classification of plausible and consistent trends in the thin film and interface growth and not in the determination of the exact binding energies, chemical states and atomic concentrations.

##### 3.1.1. Tantalum-nitride

For the analysis of the TaN films, Fig. 2 shows exemplarily the N1s (Fig. 2a), Ta4f (Fig. 2c) and the O1s (Fig. 2e) spectra with peak fits for a 20 s deposition and the resulting angle-dependent chemical bonding state portions in (Fig. 2b), (d) and (f). Within the N1s spectra the TaN can be seen at 397.2 eV. Additionally there are N–O bondings (398.8 eV) present that originate in part from the residual oxygen within the process gas (5N) and from the residual gas in the vacuum chamber and was incorporated during sputtering.

Looking at the Ta4f spectra, there is also a small amount of oxygen bound to TaO (27.9 eV) besides TaN (25.3 eV). The steady increase of the TaO<sub>x</sub> signal in Fig. 2d points to an oxygen-rich top layer. The effect of the oxidation of deposited metallic Ta using the LiNbO<sub>3</sub> substrate as a source of oxygen and thereby reducing the Nb in the LiNbO<sub>3</sub> which can also be seen in Fig. 2e at 531.9 eV and was already observed in previous work [18]. Even though the actual deposition utilizes reactive sputtering of TaN, partial oxidation to

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