Optimisation of deposition parameters of Ionised Jet Deposition (IJD) created Titanium nitride thin films

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Abstract

Titanium nitride is often used as a protective coating due to its hardness. This material is mostly deposited by pulsed laser deposition or magnetron sputtering. Ionized jet deposition has a potential to improve and diversify ways of making titanium nitride thin films. We optimised the nitrogen pressure in the IJD chamber to suppress concentration of titanium oxide in films. We manufactured a set of films using different deposition pressures. Based on the XRD data, higher gas pressure is preferable. In contrast, lower gas pressure contributes to higher film growth, based on AFM data.

Keywords: IJD, TiN, thin films

Introduction

Titanium nitride is a material with wide range of applications. Thanks to its toughness, TiN is used as a protective layer for multitude of tools from drills to medical devices[1]. In electronics, TiN is used as diffusion barrier[2] or in newly emerging field of plasmonics[3].

Ionised Jet Deposition as a new method of thin film deposition has potential to create TiN films with better characteristic compared to older techniques. Thanks to high ionisation[4] of deposition gas it could be also possible to use titanium target instead of TiN target. IJD is also cheaper than other methods and could be easily scalable, so it is a perfect candidate for industrial application[5].

The aim of this research is to investigate deposition using titanium target, deposit series of thin films with different deposition pressures and analyse it by qualitative XRD analysis and AFM analysis to optimise deposition.

Materials and Methods

Initially AISI304 steel sheets were washed with water and soap solution, isopropyl alcohol with acetone, and pure acetone in ultrasonic bath respectively.

Thin titanium nitride layers on AISI304 steel sheets were deposited using IJD JetDep100 system in Applied photonics and quantum technologies laboratory of Department of Solid State Engineering Faculty of Nuclear Sciences and Physical Engineering, Czech Technical University in Prague. Ultra-pure (99.99%) titanium target was used.

4 samples were deposited on room temperature for 30 minutes with 5 minutes conditioning of the target and IJD settings being: voltage 17 V, frequency 50 Hz, IJD-target distance 3 mm, IJD-substrate distance 110 mm. Nitrogen pressure varied between 1.9 10^{-5} - 4.0 10^{-5} mbar, see table 1.

Table 1: Gas pressure in IJD vacuum chamber

sample	TiN-1	TiN-2	TiN-3	TiN-4
Pressure before deposition [10^{-5} mbar]	4.2	6.0	3.5	3.4
Deposition gas pressure [10^{-4} mbar]	1.9	2.4	3.7	4.0

Thin film thickness was determined by measuring substrate-thin layer transition with Atomic Force Microscopy (AFM) using Akyama probe at laboratories of Applied photonics and quantum technologies. Phase composition was analysed by qualitative XRD analysis and by annealing of the samples. XRD patterns were obtained using grazing incidence geometry.

Results and Discussion

AFM

By analysing the substrate-thin layer transition from AFM using Gwyddion software the film thickness was determined, see table 2. There is a clear dependency of deposition growth rate on gas pressure, see fig. 1. By lowering gas pressure the deposition growth rate increases.

Table 2: Thin layer thickness

sample	TiN-1	TiN-2	TiN-3	TiN-4
Deposition gas pressure [10^{-4} mbar]	1.9	2.4	3.7	4.0
Thickness [nm]	250	210	200	190

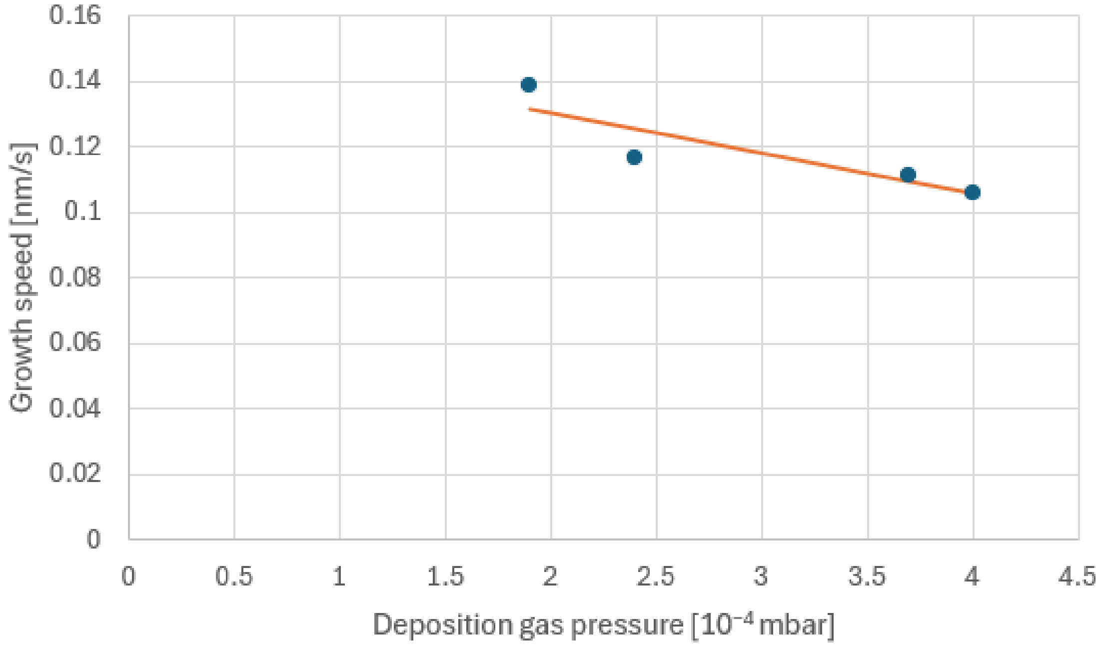

Figure 1: Dependency of layer growth rate on deposition gas pressure

XRD and Annealing

Qualitative phase analysis of the diffraction record of the TiN-1 sample, see figure 6 revealed that the most intense peaks belong to the steel substrate. Peaks were also found that may belong to both TiN and TiO, as both crystallise in the same lattice with very similar interplane distances [6][7]. The probable occurrence of hexagonal titanium in the sample was also revealed.

On the diffraction records of samples TiN-2, TiN-3 and TiN-4, see figures 3,4 and 5 respectively peaks from the steel substrate and peaks belonging to TiN or TiO were found.

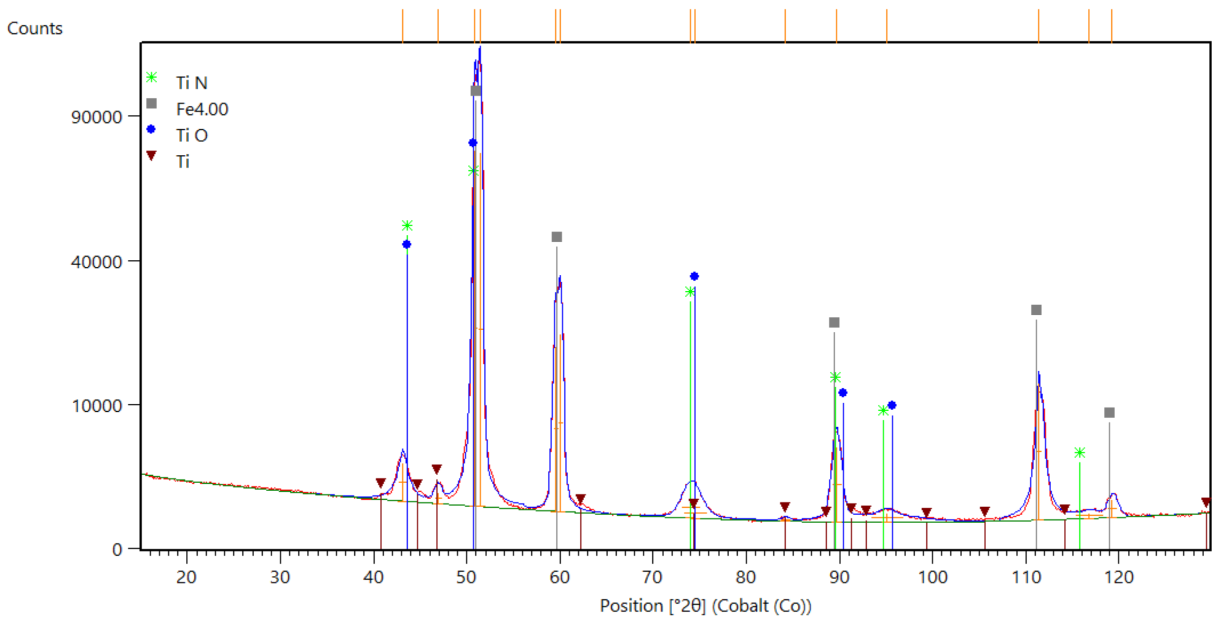

Figure 2: Diffraction record of sample TiN-1 measured under 3° angle for 9 h

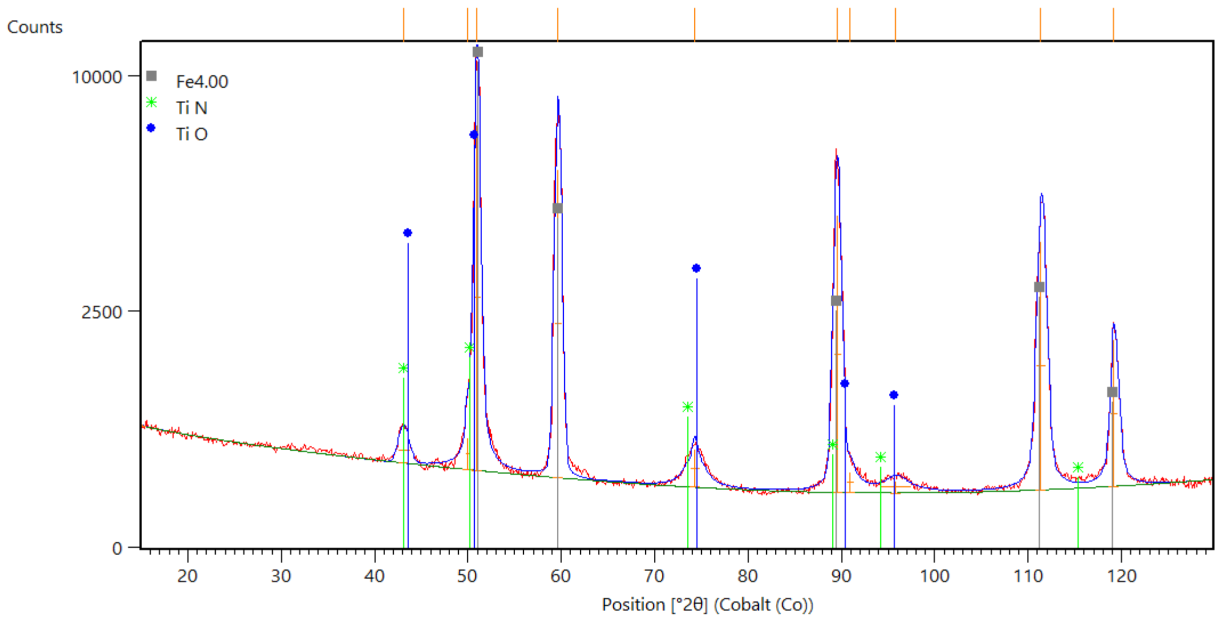

Figure 3: Diffraction record of sample TiN-2 measured under 3° angle for 5.75 h

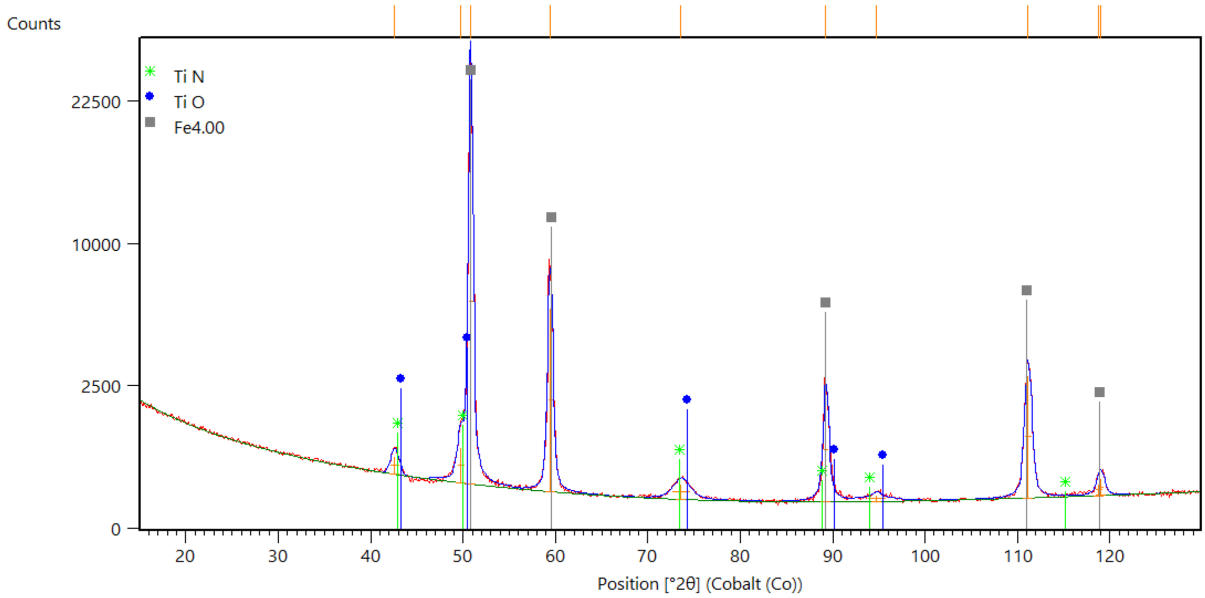

Figure 4: Diffraction record of sample TiN-3 measured under 1° angle for 3 h

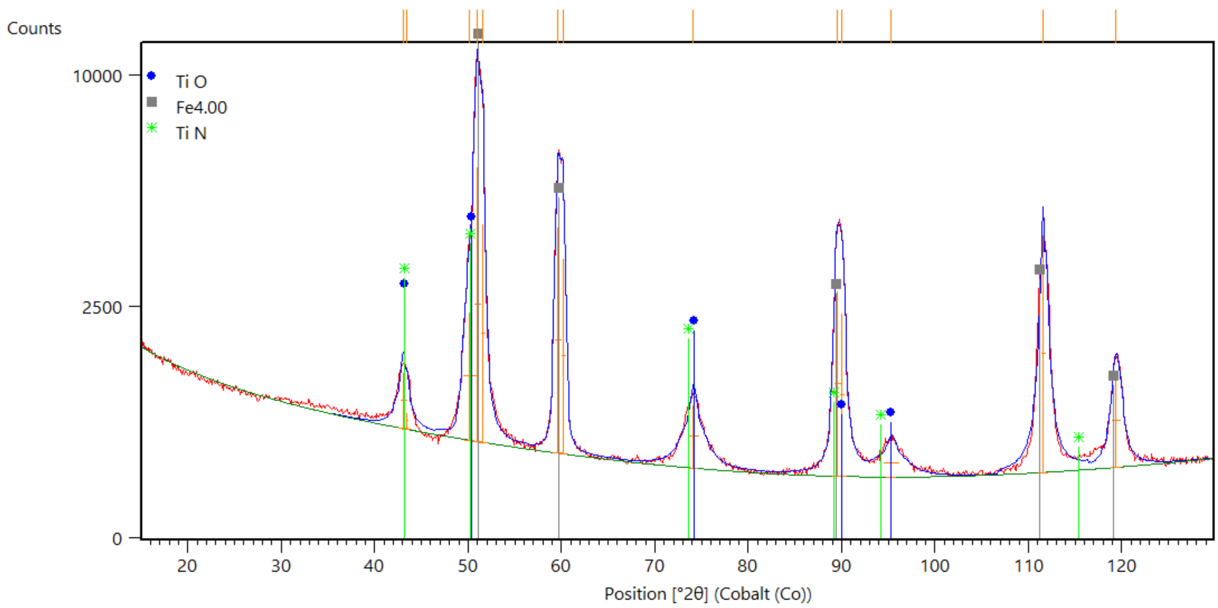

Figure 5: Diffraction record of sample TiN-4 measured under 3° angle for 3 h

After annealing the TiN-1 sample, there was a significant change in the color of the layer, see figure 6 and 7. On the diffraction record of the annealed sample, see image 11, steel from the substrate, TiN, TiO, hexagonal titanium and, newly TiO_2 were found. Other samples also changed colour after annealing, see pictures 8-10.

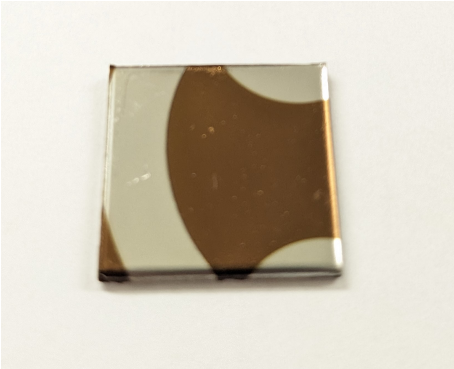

Figure 6: Not annealed TiN-1 thin film

Figure 7: Annealed sample TiN-1

Figure 8: Annealed sample TiN-2

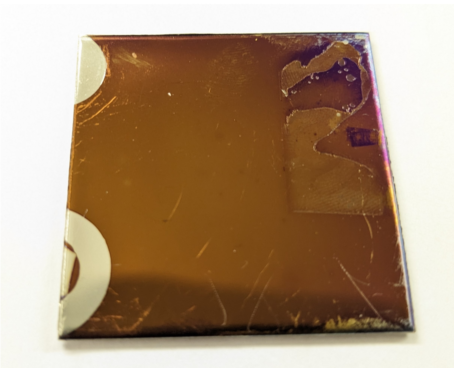

Figure 9: Annealed sample TiN-3

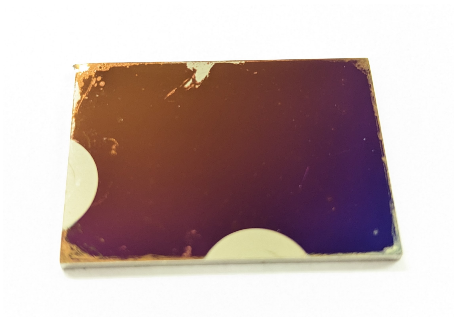

Figure 10: Annealed sample TiN-4

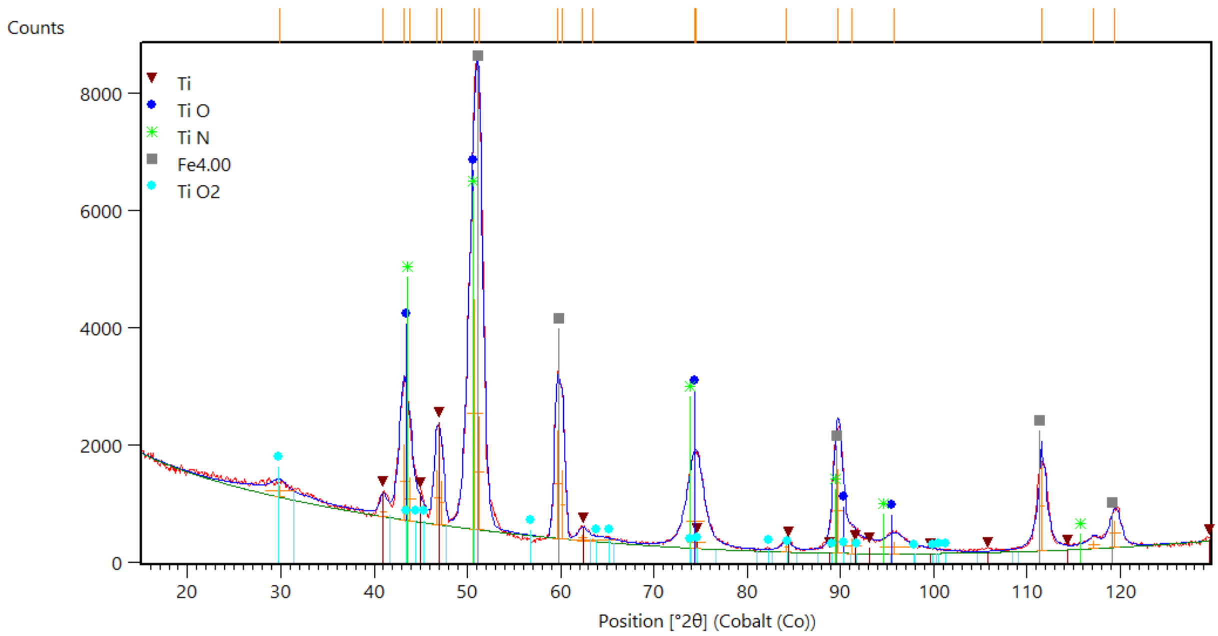

Figure 11: Diffraction record of annealed sample TiN-1 measured under 1° angle for 3.75 h

Hexagonal titan was only found in sample TiN-1 and after annealing TiO_2 peaks showed up. Also the colour of samples TiN-1 and TiN-2 changed more than of samples TiN-3 and TiN-4 probably because of lower concentration of TiN in samples TiN-3 and TiN-4 as TiN oxidises slower then titanium. Higher nitrogen pressure during deposition of samples TiN-3 and TiN-4 probably led to higher nitridation of samples.

Conclusions

Totally 4 samples were prepared. Thin film thickness analysis by AFM revealed indirect dependency of layer growth rate on deposition gas pressure. Qualitative XRD analysis together with annealing of the samples revealed that the nitridation of the thin films S directly proportional to the deposition gas pressure.

Problem with similar diffraction peaks of TiN and TiO need to be addressed in the next research. Use of silicon or glass as substrate would be preferred as it would allow use of more methods for analysis.

Reference

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