Depth profiling protective coating titanium nitride based on glow discharge optical emission spectrometry

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Figure 1 – Profile GD crater, obtained on the sample №1 with protective coating TiN. GD Phofiler-2 parameters are pressure 650 Pa and power 40 W.

| $\frac{25 \times 10^3}{25 \times 10^3}$ | | | | | | | | | |
|---|-------|--------|--------|---------|--------|-------|------------|--------------------|---|
| | C KLL | N KLJ | L | O KLL | | O 1s | Ti 2p N 1s | C 1sAr 2p1/2 | O 2s |
| | MM | ר | Гi LMM | | | Ti 2s | Ti 2p1/2 | Ar $2s$ Ar $2p3/2$ | Ti 3s O 2ı |
| | Name | Pos. | FWHM | Area | At% | | Ti 2p3/2 | Ar 2p | |
| | O 1s | 531.10 | 2.815 | 1774.2 | 16.404 | | | | $\begin{array}{c} 0 & 2 \\ C & 2 \end{array}$ |
| 20 | C 1s | 285.70 | 2.240 | 340.6 | 8.976 | | | | $C 2_{\rm F}$ |
| 20 | N 1s | 397.30 | 1.384 | 2459.3 | 36.614 | | | | $C 2_{\rm F}$ |
| | Ti 2p | 455.20 | 2.579 | 11047.9 | 36.852 | | | | |

Table 1. Sputtering rate of the samples with protective coating TiN.

| Sample | № 1 | № 2 | № 3 | № 4 | № 5 |
|----------------------------|------------|------------|------------|------------|------------|
| Sputtering time, s | 45 | 105 | 85 | 105 | 65 |
| Depth crater, µm | 0,8 | 2,4 | 1,7 | 3,0 | 1,4 |
| Sputtering rate, µm/min | 1,1 | 1,4 | 1,2 | 1,7 | 1,3 |



Figure 2 – SEM image sample N_{21} with protective coating TiN.

Table 2. Result XPS analysis is mass concentration of the samples with protective coating TiN, calculated in program CasaXPS.

| Sample | № 1 | № 2 | № 3 | № 4 | № 5 |
|--------|------------|------------|------------|------------|------------|
| O 1s | 9,4 | 17,6 | 15,1 | 19,4 | 15,7 |
| C 1s | 3,9 | 2,5 | 7,7 | 2,5 | 3,1 |
| N 1s | 18,3 | 10,9 | 14,1 | 9,4 | 13,5 |
| Ti 2p | 66,8 | 67,1 | 61,4 | 65,5 | 64,0 |
| Ar 2p | 1,6 | 1,9 | 1,7 | 3,2 | 3,7 |





Figure 3—XPS spectra of the sample №1 with protective coating TiN and atom concentration, obtained in program CasaXPS.

This poster presents elemental depth profile analysis based on glow discharge optical emission spectrometry for titanium nitride protective coating is created ion-plasma method. Solved the problem required standard samples with a high concentration of nitrogen more then 2% needed for calibration of the spectrometer glow discharge.

For calibration of the spectrometer GD Profiler-2 (Horiba Jobin Yvon) was made 5 samples with different concentrations of nitrogen in ion-plasma protective coating. The coating was formed on polished steel substrates, AISI 304.



Figure 4 – Depth profile signals High Dynamic Detectors of the sample №1 with protective Figure 5 – Quantitative depth profile of the sample №1 with protective coating TiN.

Preliminary analysis samples was of the spectrometer GD Profiler-2. Depth profile signals High Dynamic Detectors of the sample N_1 with protective coating TiN was obtained on mechanical profilometer Dektak 150 (Fig.1). Sputtering rate was calculated based on sputtering time and depth crater (Tabl.1).

Elemental concentration of the protective coatings was analyzed by X-ray Photoelectron Spectroscopy (XPS). XPS spectra was obtained on module for electron-ion spectroscopy based on Nanofab 25 platform. Result XPS analysis was elemental concentration of the samples with protective coating TiN , obtained in program CasaXPS (Fig.3, Tabl. 2). Depth profile calibration was made by values sputtering rates and elemental concentration protective coatings TiN. This calibration was checked at sample №1 with protective coating TiN calculated in the program Quantum (Horiba Jobin Yvon) presents on the Figure 5. Conclusions. The proposed depth profile analysis based on glow discharge optical emission spectrometry for titanium nitride protective coating. Solved the problem required standard samples with a high concentration of nitrogen at range 9-20% needed for calibration of the spectrometer glow discharge.