STRUCTURE, MECHANICAL AND TRIBOLOGICAL PROPERTIES OF VANADIUM NITRIDE FILMS DEPOSITED BY REACTIVE MAGNETRON SPUTTERING

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1 Introducion

Because of solid state lubricious properties of vanadium oxides, wear resistant coatings based on that metal are still of interest for research teams. Special attention is paid on tribochemical oxidation of nitride layer yielding lubricious oxide to tribo-contact, particularly at elevated temperature.

The intention of our team is to apply hard coatings on tools for wood machining. Vanadium nitrides or carbides are considered as a component of complex composite coatings. The knowledge about structure and tribological properties of pure nitrides is necessary for conscious development of such coatings.

2 Experimental

2.1 Deposition system

Vanadium nitride films were obtain by pulsed magnetron, reactive deposition. Magnetron sputtering source was driven by pulsed power supply (1 kHz with 100 kHz modulation) and power dissipated at the source was 0.9 kW. The magnetron gun is planar (100 mm of target diameter) and was operated in unbalanced mode using external coils.

The samples are placed in the distance of 70 mm from the target and are heated up with resistance heater. The vacuum chamber is pumped down before process to the ultimate vacuum 10^{-3} Pa (10^{-5} mbar) by standard system equipped with a diffusion pump.

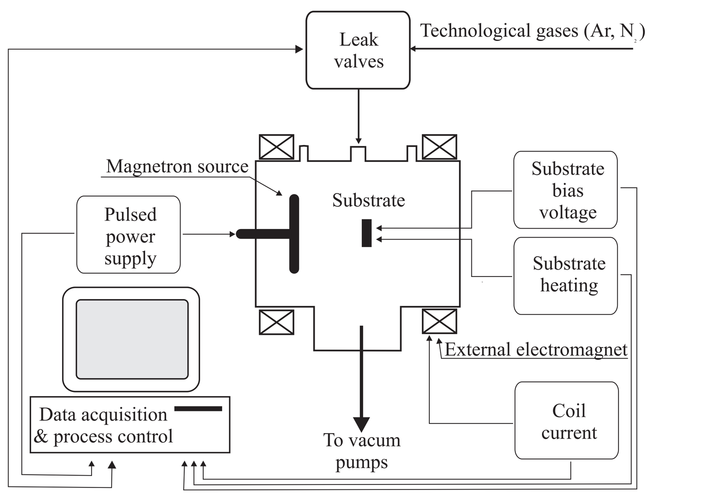

Fig. 1. *Schematic diagram of deposition system*

2.2 Sample preparation

The films have been deposited on mirror polished (R_a = 35 nm), 440C steel substrates, 28 mm in diameter, hardened to 4 GPa (4000 N/mm^2) of Martens hardness (2 N normal load)

Before deposition substrates are heated up to 350±10°C and ion cleaned for 15 minutes in argon DC glow discharge at 0,6 kV, 35 Pa ($3.5{\cdot}10^{-3}$ mbar).

The films were deposited in Ar/N_2 atmosphere at total pressure 0.75 Pa. There were two kinds of process carried on:

A) with N_2 constant flow giving *before process* N_2 partial pressure (0.1, 0.2 and 0.4 Pa)

B) with N_2 flow varying (namely increasing) during process, yielding *during process* the same, this time constant, N_2 partial pressure (0.1, 0.2 and 0.4 Pa).

Deposition rate was about 70 nm/min giving during 30 min process layers 2.1–2.2 μm thick. Substrates were negatively biased at voltage −100 and −70 V.

3 Results and discussion

3.1 Atomic composition and structure

Applying constant nitrogen flow (**A**) films containing bcc-V, tetragonal $VN_{0.09}$ (ICDD 71-1230), hexagonal $VN_{0.5}$ (ICDD 71-0618) and fcc-VN (ICDD 78-1315) was obtained according to magnitude of nitrogen flow. The last phase was observed in unexpected orientation, not mentioned in the literature. Applying constant pressure (**B** type of process), samples with gradually changing orientation were deposited. At the highest flow films were highly oriented with planes (200) parallel to substrate surface – diffractograms were taken in Bragg-Bretano geometry.

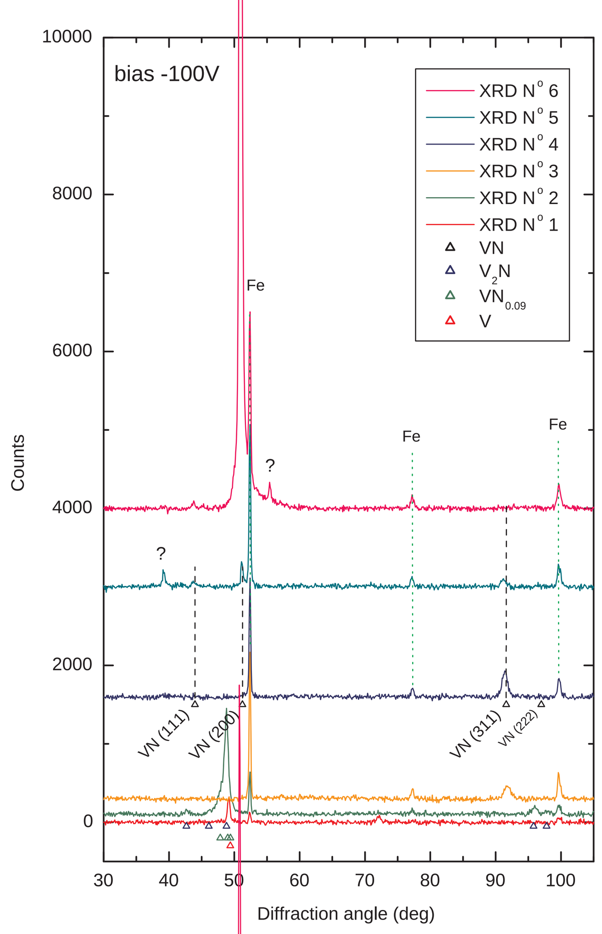

Fig. 2. *XRD spectra obtained in processes at bias voltage −100 V.*

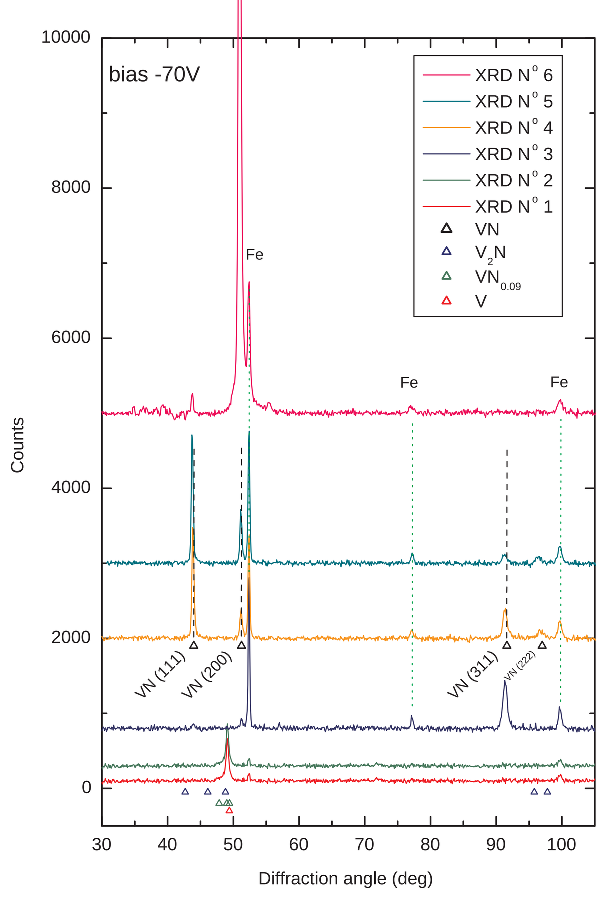

Fig. 3. *XRD spectra obtained in processes at bias voltage −70 V.*

3.2 Peak positions and lattice parameter

It was expected distinct changes in composition with nitrogen flow. Surprisingly, except lowest nitrogen flows, samples contain 60–67 atomic percent nitrogen (measured with WDS method) with no correlation to nitrogen flow. The value seems to be very high but other measurement with EDX (less appropriate for light elements) method gave values also very high – above 55 at. %.

Estimated lattice parameter for VN phase, obtained with XRD spectra fitting are generally higher then that presented in ICDD files.

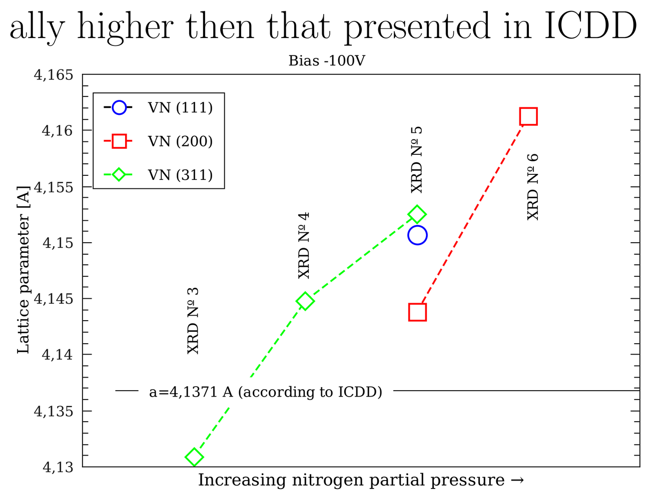

Fig. 4. *Lattice parameter calculated from XRD peaks positions, arranged by nitrogen flow in deposition process of the sample.*

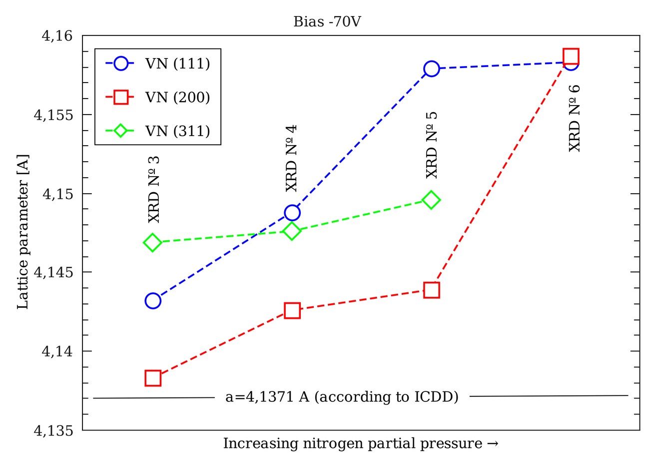

Fig. 5. *Lattice parameter calculated from XRD peaks positions, arranged by nitrogen flow in deposition process of the sample.*

3.3 Hardness

Hardness of the samples were measured using depth sensitive method with Fisheroscope 2000 apparatus. Using different loads it was concluded that measurements with loads less or equal 25 mN were not affected by softer substrate and these results are presented bellow.

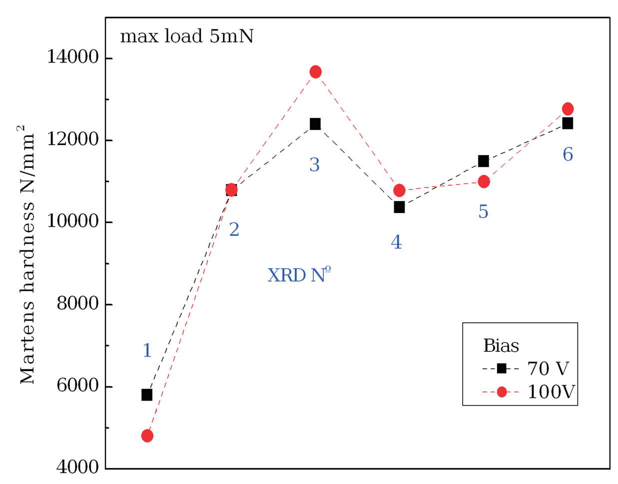

Fig. 6. *Martens hardness for samples deposited at both −70 and −100 V bias measured with maximal load 5mN (0.5 G).*

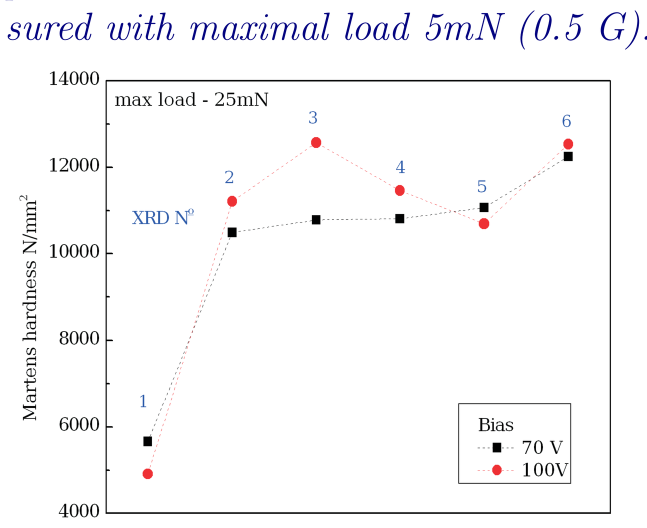

Fig. 7. *Martens hardness for samples deposited at both −70 and −100 V bias measured with maximal load 25 mN (2.5 G).*

3.4 Friction

Friction tests were carried out in ball-on-disc geometry at normal load of 1 N, sliding speed 40 mm/s. Tests were done in ambient atmosphere and lasted 1 hour (5000 turns).

As a counterpart polished 10 mm alumina balls having roughness of $R_a < 0{,}03$ μm was applied. The Hertzian mean pressure in this arrangement was about 300 MPa.

Wear in applied conditions was almost not observed except samples containing minimal amount of nitrogen – they were completely worn after half an hour of the test.

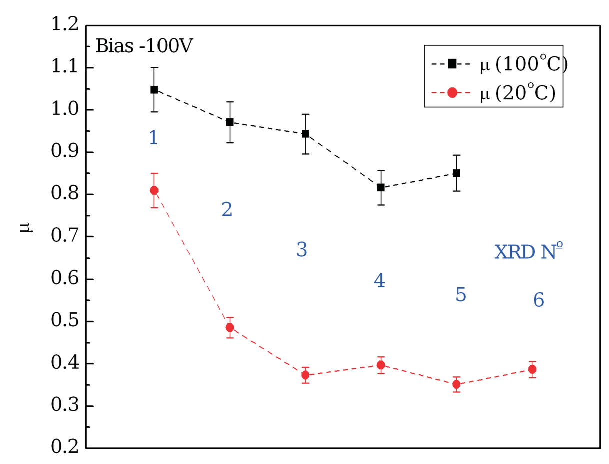

Fig. 8. *Friction coefficient obtained from friction tests in temperature 20 and 100° C, arranged by nitrogen flow in deposition process.*

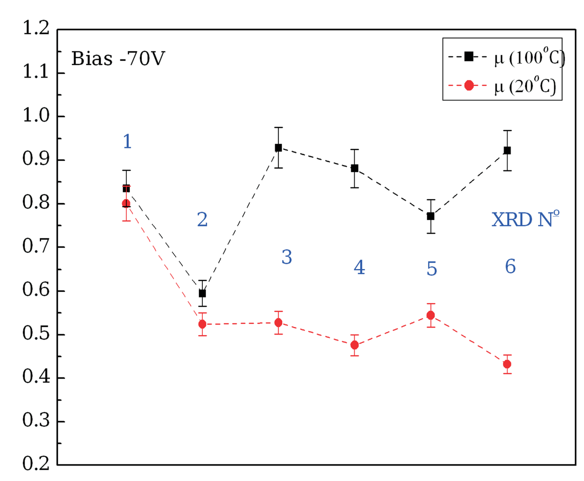

Fig. 9. *Friction coefficient obtained from friction tests in temperature 20 and 100° C, arranged by nitrogen flow in deposition process.*

4 To do

- The uncertainties related to nitrogen content should be explain.
- Friction and wear tests will be continued.
- Vanadium carbides are planed as next step of research.
- Vanadium nitride as a component of composite coatings is considered

Acknowledgements

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