Nb_2O_5 AND TiO_2 THIN FILMS DEPOSITED BY PULSE MAGNETRON SPUTTERING OF CYLINDRICAL CERAMIC TARGETS

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ABSTRACT

Nb_2O_5 and TiO_2 thin films were sputtered on low-iron float glass with a cylindrical dual magnetron system equipped with substochiometric ceramic targets. The influence of the oxygen partial pressure and the pulse mode on the thin film properties was studied. Therefore the plasma excitation mode was varied between DC-DC, bipolar square wave and bipolar sine wave mode. The power density was set to approx. 25 kW/m. With increasing oxygen partial pressure and changing the pulse mode from bipolar to unipolar the films got a slightly columnar structure, thus leading to lower refractive indices and lower compressive film stress even up to tensile film stress.

EXPERIMENTAL SETUP

- vertical in-line sputtering plant ILA 900 (max. homogenous substrate width of 600 mm)
- cylindrical dual magnetron sputtering system (Bekaert, target length: 820 mm)
- power supplies: UBS-C2 (DC-DC, bipolar square wave), Hüttinger BIG 100/50P (bipolar sine wave)
- cylindrical ceramic targets (GfE Fremat, Freiberg, plasma sprayed substochiometric NbO_X and TiO_X)
- dynamic deposition (one pass)
- substrate pre-treatment RF plasma etching process (13.56 MHz)
- film thickness approx. 200 nm
- low-iron float glass with a thickness of 3.2 mm and thin glass slides for stress measurement

Vertical in-line sputtering plant ILA 900

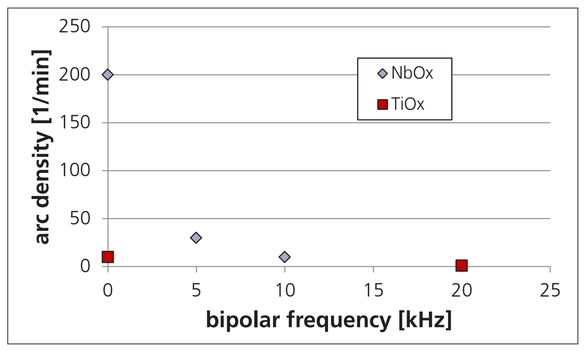
c-DMS with NbO_X-Targets

SPUTTERING PROCESS

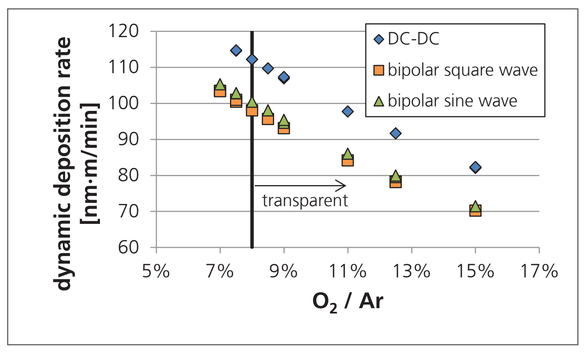
Process parameters	TiO_2	Nb_2O_5
power density	25 kW/m	
sputtering pressure	0.3 Pa	
additional oxygen	0 … 15% relative to argon	
bipolar frequency	20 kHz	
deposition rate [nm·m/min]	53 … 60	100 … 112

- arc density drops below 1 arc/min at 20 kHz
- arc density for TiO_X- lower than for NbO_X-targets
- DC-DC mode with moderate arc density (max. 4 arcs/s)
- deposition rate in bipolar mode below DC-DC mode due to losses in polarity switches
- deposition rate drops when oxygen added
- variation of pulse mode and oxygen content

Unipolar/Bipolar Switching Unit UBS-C2

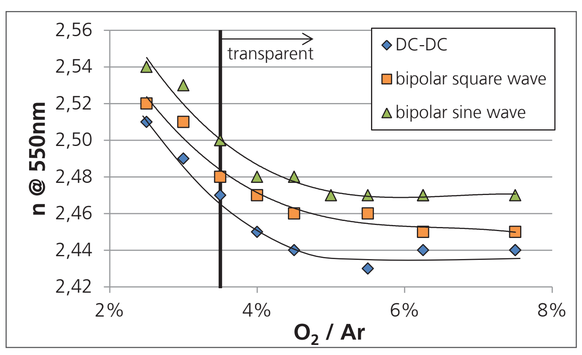


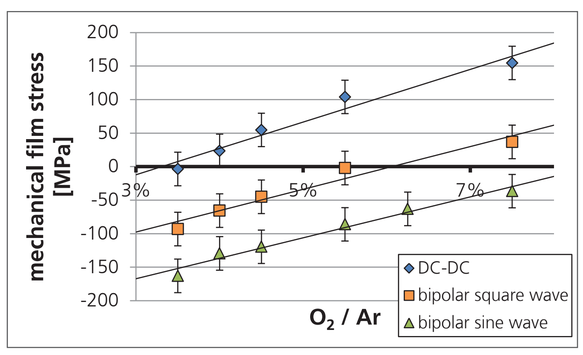
Arc density vs. bipolar frequency for NbO_x and TiO_x-targets at 25 kW/m, using UBS-C2 pulse unit

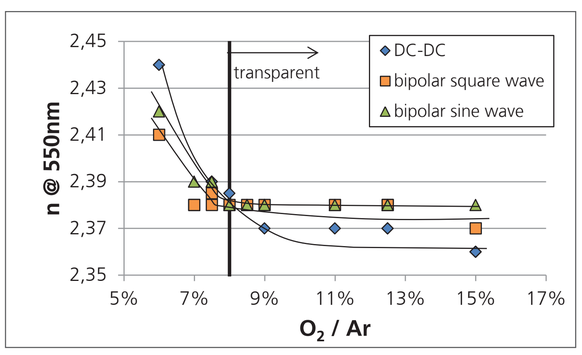


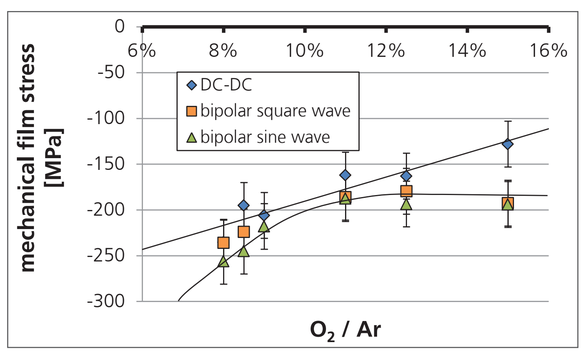
Deposition rate vs. ratio O_2/Ar for NbO_x-targets at approx. 25 kW/m, using different power supplies, above 8% the films are transparent (k < 1E-03)

OPTICAL AND STRUCTURAL PROPERTIES



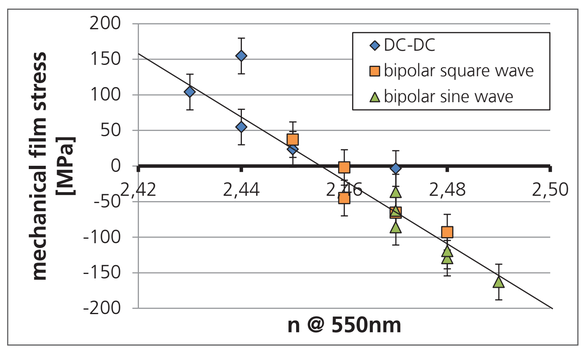


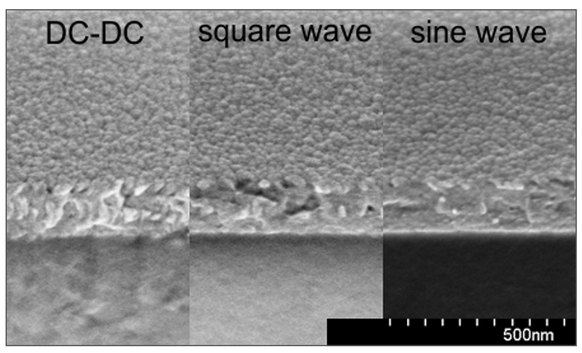




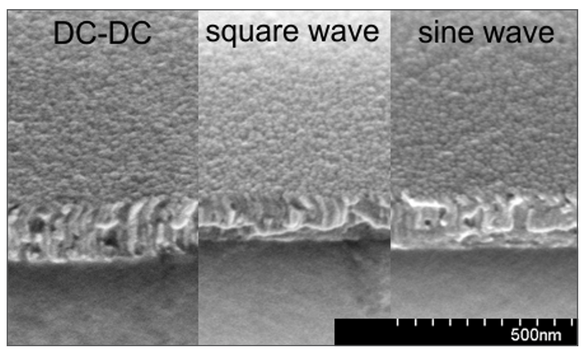
TiO_2

200 nm, refractive index and mechanical stress vs. ratio of oxygen to argon





3.5% O_2/Ar, SEM images of TiO_2 thin films

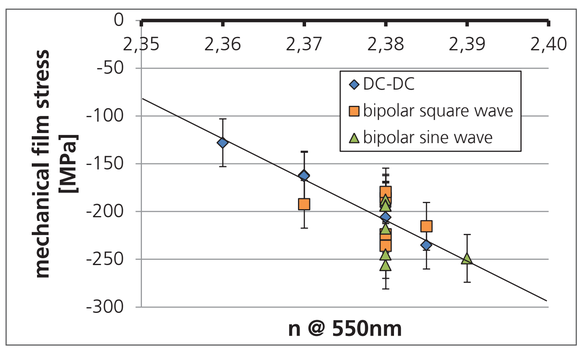


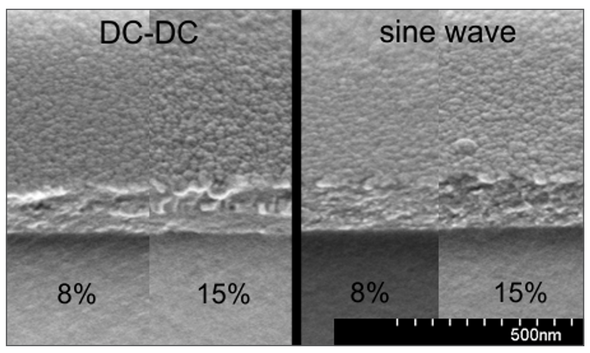
7.5% O_2/Ar, SEM images of TiO_2 thin films

- significant dependence of refractive index and film stress on pulse mode and oxygen content
  - refractive index and mechanical film stress decrease with increasing oxygen flow
  - highest refractive index and mechanical film stress observed in sine wave mode
- significant correlation between refractive index and mechanical film stress
- low film stress and low refractive indices can be attributed to a low film density and a more porous structure
- bipolar mode and low oxygen content results in dense film structure with a high refractive index and a high compressive film stress

Nb_2O_5

200 nm, refractive index and mechanical stress vs. ratio of oxygen to argon





Ratio O_2/Ar, SEM images of Nb_2O_5 thin films

- bipolar mode has higher energetic input than DC-DC mode
- for titania thin films stress changes from compressive to tensile when adding more oxygen in DC-DC mode an bipolar square wave mode
- AFM: average roughness varies from 4 to 5 nm and the RMS roughness from 1 to 1.6 nm

SUMMARY AND CONCLUSIONS

TiO_2 and Nb_2O_5 thin films were deposited by sputtering cylindrical ceramic targets using a c-DMS. The cylindrical targets were made of a substochiometric TiO_X and NbO_X, respectively. The films were deposited on low-iron float glass by pulse magnetron sputtering varying the oxygen partial pressure and the pulse mode. By SEM analyses it can be shown that increasing the oxygen flow and changing the pulse mode from bipolar to unipolar results in a more columnar film structure. The samples showing a columnar structure show a shift towards tensile film stress. Comparing the refractive index and the film stress high compressive stress can be attributed to dense high refractive films and tensile stress to films with lower refractive indices and density.

The conclusion is that at a higher oxygen partial pressure and at a lower energetic input (DC-DC mode) the films are characterized by:

- lower refractive indices
- lower compressive film stress even up to tensile film stress
- slightly more columnar appearance

In summary the investigated thin films are suitable for optical coatings due to their transparency, high refractive indices, surface morphology and adjustable film stress. The high observed deposition rates and the easy process handling makes the sputtering of the cylindrical ceramic targets attractive for highly productive processes.

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GfE