Properties of optical thin films produced by reactive low voltage ion plating (RLVIP)

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Investigations are done in valuable cooperation with

- Fraunhofer IOF, Jena, Germany – S. Yulin
- University of Ulm, Electron Microscopy Group for Material Sciences – U. Kaiser
- Instiute of Mineralogy, R. Tessadri
- Institute of Physical Chemistry, E. Bertel

Outline

1. Introduction
2. Deposition process
3. Film properties
4. Conclusion

Introduction

Thin Film:

- material layers with thickness of several nm or µm
- metals, alloys, chemical compounds can be deposited by different physical vapour deposition (PVD) technologies under high vacuum conditions

Applications:

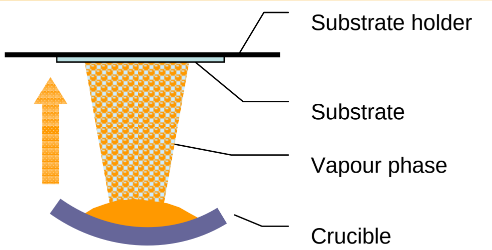
- Electronics, Optics, Optoelelctronics
- Decorative films, Barrier films, Hard coatings

"Ideal" Thin Film:

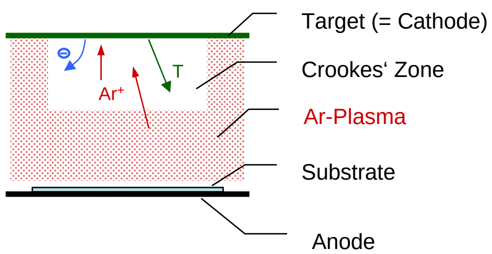
- isotropic and homogeneous
- absolutely flat surfaces and interfaces
- optical thin films: refractive index is constant within the film
- no optical losses, no film stress

HOWEVER: requirements not fulfilled by presently developed deposition technologies (e.g. birefringence, different thermal expansion, high mechanical film stress)

Introduction

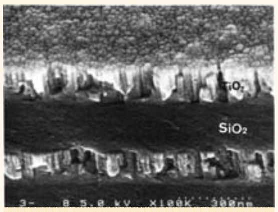


Principle of conventional evaporation under vacuum conditions[1]



Principle of conventional sputtering under vacuum conditions[1]

Depositions by conventional reactive evaporation as well as conventional sputtering yield films which have insufficient properties for high-quality optical interference coatings:



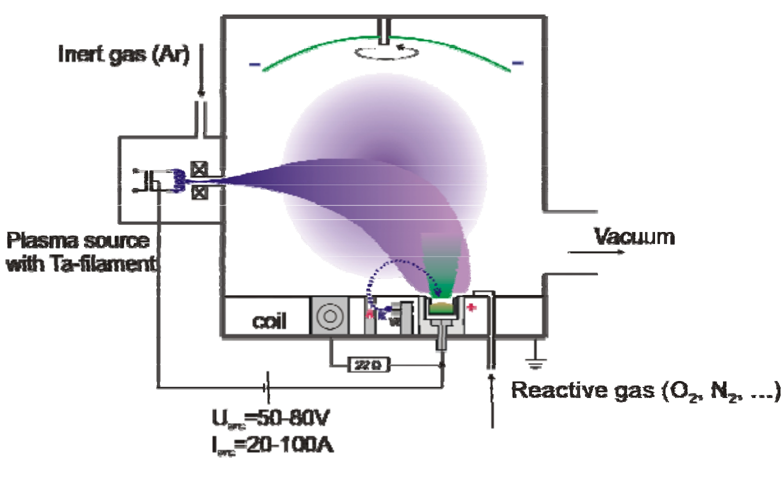
Columnar microstructure of TiO_2 on SiO_2 [2]

- porous microstructure
- rough surface
- low film refractive index
- mechanical properties (e.g. adhesion, abrasion, hardness) not sufficient

- Ion- or plasma-assisted as well as pulsed magnetron sputtering show clear improvement of the film properties
- RLVIP: film properties are environmental stability, high mechanical film resistance

[1] *Dünnschichttechnologie, H. Frey, G. Kienel, VDI-Verlag, Düsseldorf, 1987*
[2] *Q. Tang, K. Kikuchi, S. Ogura, A. Macleod, J. Vac. Sci. Technol. A 17 (1999) 3379*

Reactive low-voltage ion-plating (RLVIP)



Schematic of industrial ion plating plant Balzers/Evatec BAP 800[3]

- Coating materials:
Me, Me_xO_y, Me_xN_y, Me_xC_y, MeO_xC_y, ...

Parameters influencing film properties:

- total gas pressure & composition (Ar + O_2)
- discharge arc current
→ *process specific:* increasing arc current leads to an increase of particle energy
- Energy input resulted in very dense, meta-stable constrained amorphous film structure
- Deposition rate: 0.3 – 0.7nm/sec monitored by quartz crystal

[3] US Patent; 4,619,748; (1986) Balzers AG, E. Moll, H. K. Pulker, W. Haag

Film properties

- detailed investigations of the film properties of metal oxide layers
- relevant properties:

Refractive Index
(Spectrophotometric measurements & calculations)

Optical Absorption
(Photothermal deflection spectrometry)

Mechanical Film Stress
(Laser interferometric method)

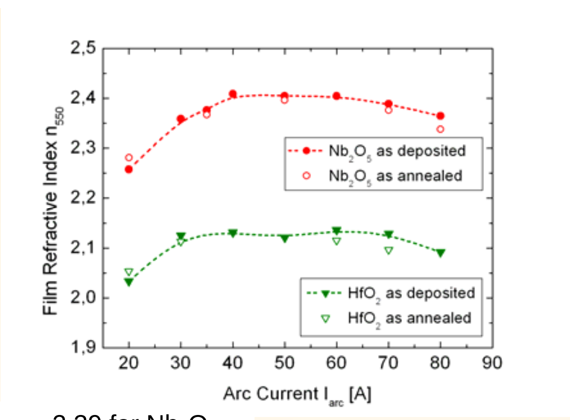
Film Density
(X-ray reflection)

Surface Topography & Roughness
(Atomic force microscopy using tapping mode)

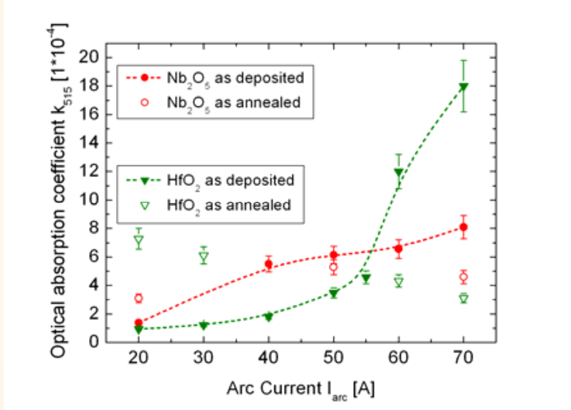
Film structure
(X-ray diffraction, HR-Transmission electron microscopy)

Optical film properties

Film thickness ~200nm



p_{O2} = const. = $8{\cdot}10^{-4}$mbar (Nb_2O_5), $11{\cdot}10^{-4}$mbar (HfO_2)
p_{Ar} = const. = $4{\cdot}10^{-4}$mbar



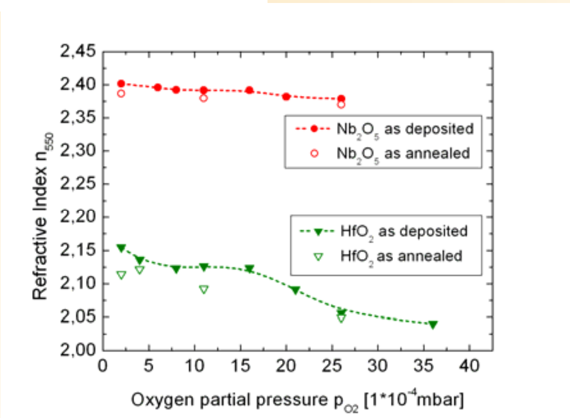
n_{550} = 2.39 for Nb_2O_5

n_{550} = 2.12 for HfO_2

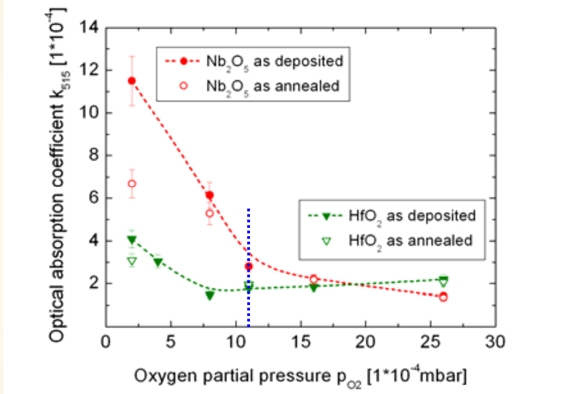
- **highest refractive indices obtained between 40-60A**
- **However, under these conditions higher residual optical film absorption**
- **annealing (4h, 350° on atmosphere) caused slight drop of refractive index and reduction of optical absorption**

Optical film properties

Film thickness ~200nm



I_{arc} = const. = 40A (HfO_2), 50A (Nb_2O_5)
p_{Ar} = const. = $4{\cdot}10^{-4}$mbar



$k_{515} < 3{\cdot}10^{-4}$

- **Increase of oxygen pressure resulted in acceptable low optical absorption - beginning with $p_{O2} = 11{\cdot}10^{-4}$mbar**
- **but also a slight decrease of film refractive index between 0.5-1%**
- **In this case - annealing caused practically no effects**

Films are dense and environmental stable!

[4] *A. Hallbauer, D. Huber, G. N. Strauss, S. Schlichtherle, A. Kunz, H. K. Pulker, Thin Solid Films, 2006, submitted*
[5] *A. Kunz, A. Hallbauer, D. Huber, H. K. Pulker, Vakuum in Forschung und Praxis 18 (2006) 12*

Mechanical Film Stress

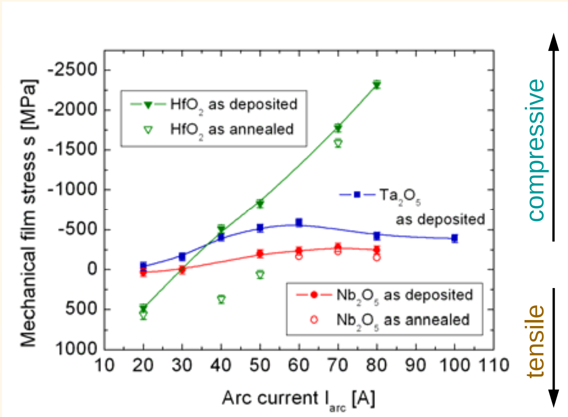
– due to higher energetic ion bombardment - the residual mechanical film stress is always compressive [4,5]

– values can increase to some hundreds MPa and higher

→ bending of the substrate, delamination

– elimination of the bending by stress-compensating layers [6,7]

– reduction of the stress by post deposition heat treatments at some hundreds °C or in-situ by increasing total pressure during deposition [4,5]



HOWEVER:
also influences on optical properties !

[6] F. Richter, H. Kupfer, P. Schlott, T. Gessner, C. Kaufmann, Thin Solid Films 389 (2001) 278
[7] H. Kupfer, T. Flügel, F. Richter, P. Schlott, Surf. Coat. Technol. 116-199 (1999) 116

Why are we doing these investigations?

Deposition parameters are based on empirical correlations to film properties

- high film refractive index, low optical absorption, moderate film stress
- nearly atomically flat surfaces can be obtained (rms ~0.5nm)
- investigations concerning density and structure showed: film density (obtained by XRR) can be higher than bulk material & structure is mostly amorphous with a short range order of ~1nm (obtained by HR-TEM) [8]
- Knowledge about atomic arrangements about the *constrained high-dense amorphous states* are very poor – in contrast to crystalline states as well as amorphous metals (e.g. a-Si)
- Importance to clarify the physical connection between the intrinsic structural parameters and macroscopic film properties with the aim to find deposition conditions for producing the “ideal” thin film

[8] A. Hallbauer, D. Huber, F. Klauser, A. Kunz, R. Tessadri, U. Kaiser, S. Yulin, H. K. Pulker, Plasma Processes and Polymers, 2006, submitted

Thank you for your attention!

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