Research - Services

ALD – Atomic Layer Deposition

Atomic Layer Deposition is a deposition process for assembling of thin films on the nanometer scale. The self-limiting deposition of atomic monolayers occurs trough a cyclic injection of volatile and highly reactive precursors. The several gases are induced alternately into the process chamber and are separated by a purge pulse respectively.

Advantages of the ALD-coating:

- deposition of homogenous coatings on any formed substrate
- accurate film thickness monitoring
- defined layer composition
- high layer quality

News

Current Research:

- ALD – Internal Coating of X-ray Optics

  The rapid development both in nanotechnology and photovoltaics leads to new standards for more accurate spatial resolutions in X-ray analytics. Single capillary optics and X-ray micro-mirrors are of particular interest.

Funded by the European Union and the Free State of Saxony

Gefördert aus Mitteln der Europäischen Union

Europa fördert Sachsen.
EFRE
Europäischer Fonds für regionale Entwicklung

Workshops & Conferences

Fischer D., Eschner M., Glühmann J., Lorenz C, Neidhardt A, Reinhold C., Reinhold U., Schnabel H.-D., Schneeweiß M., *Protective Layer on Indexable Inserts*, DPG Frühjahrstagung der Sektion Kondensierte Materie (SKM) 76. Jahrestagung der DPG, Berlin, Germany, March 2012

Lorenz C., Schenk T., Fischer D., Eschner M., Neidhardt A., Reinhold C., Reinhold U., Schnabel H.-D., *ALD Process for Tungsten Films in Model-like Glass Capillaries,* ALD-AVS & Baltic-ALD conference 2012, Dresden, Germany, June 2012

C. Lorenz, M. Neuber, A. Neidhardt, C. Reinhold, U. Reinhold, H.-D. Schnabel *Adhesion Strength of Low Temperature Tungsten ALD-Films*, *13th International Conference on Atomic Layer Deposition*, San Diego, July 2013

Staff

Professors (from left to right)

Prof. Dr. rer. nat. habil. Andreas Neidhardt
Tel: (0375)536 1507
E-Mail: andreas.neidhardt@fh-zwickau.de

Prof. Dr. rer. nat. Ullrich Reinhold
Tel: (0375)536 1508
E-mail: ullrich.reinhold@fh-zwickau.de

Prof. Dr. rer. nat. Christel Reinhold
Tel: (0375)536 1500
E-mail: christel.reinhold@fh-zwickau.de

Prof. Dr. Ing. Hans-Dieter Schnabel
Tel: (0375) 536 1530
E-Mail: hans.dieter.schnabel@fh-zwickau.de

Fax: (0375) 536 1503

Co-workers:
Dipl.-Ing. (FH)Mario Eschner

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Services

Producible films:

- Aluminium oxide (Al_2O_3)
- Titanium aluminium nitride (Ti(Al)N)
- Titanium nitride (TiN)
- Titanium oxide (TiO_2)
- Metallic tungsten (W)

Both thermal and the plasma enhanced ALD (PEALD) processes allow the deposition of coatings ranging between 1 and 100 nm on a variety of substrates (e.g. metals, glasses).

The precision of the high-grade coatings with self-limiting growth is implemented through multi-stage, cyclic processes.

The deposition process of insulating or conducting thin films on complex, 3D and large-scale structures improves the surface properties and advances the further miniaturization of components in the field of nanotechnology.

Other coating equipment at the WHZ:

- Cavity magnetron coating system
- Hollow-cathode arc coating system
- Plasma-enhanced chemical vapour deposition system

Equipment

Laboratory equipment with modern technology which is compliant with the safety instructions

The ALD system, which started up in 2011, consists of:

- System (process chamber with merchant system)
- Vacuum pump
- Dry bed absorber
- Switchboard
- Gas control unit for liquid and gaseous precursors

Measurement and testing equipment to characterize thin films:

- Spectral ellipsometer SE800 (SENTECH)
- Analysis system (UHV-STM, XPS, UPS) (SPECS)
- Scanning electron microscope (SEM) with quantitative EDX analysis MIRA FE-REM (TESCAN)
- X-ray diffractometer (XRD) D8 Discover with GADDS (Bruker)
- Scratch tester from home development

Westsächsische Hochschule Zwickau
University of Applied Sciences

Protective Layer on Indexable Inserts

D. Fischer, M. Eschner, J. Glühmann, C. Lorenz, A. Neidhardt
C. Reinhold, U. Reinhold, H.-D. Schnabel, M. Schneeweiß
Westsächsische Hochschule Zwickau
University of Applied Sciences, Dr.-Friedrichs-Ring 2a, 08056 Zwickau, Germany
contact: dustin.fischer@fh-zwickau.de

Abstract

The use of uncoated indexable inserts and micro tools is a common procedure for cutting metals. In order to achieve an effective removal of material, a uniform abrasion is required at the cutting edge of the tool. Despite the great thermal resistance of the tool there is a high risk of oxidation at high temperatures so that the sealing of the inserts is indicated. An aluminium oxide (Al_2O_3) coating performed by Atomic Layer Deposition is a method to positively influence the cutting parameters as well as the wear and tear.

Motivation

Coatings with hard materials are able to considerably extend the lifetime of cutting tools. Chemical and Physical Vapor Deposition (CVD, PVD) are common techniques to coat these tools since the 1970s. Thereafter, no comparable progress was achieved and the potential of CVD and PVD seems exhausted. With growing thickness of coatings an edge rounding effect occurs, which is a serious problem for both finishing (e.g. indexable inserts) and micro cutting tools (e.g. drillers). Due to miniaturization, conformality becomes another problem. Since ALD is expected to provide thin, conformal, and dense films its potential for future cutting tool production is to be examined.

Background

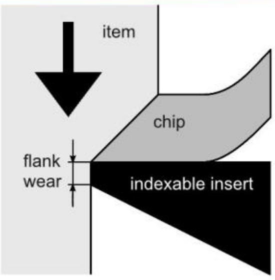


Fig. 1: Schematic diagram for the machining.

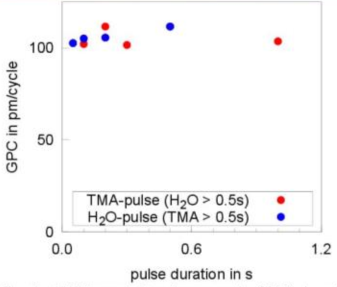


Fig. 2: ALD-Process: Growth per cycle (GPC) is not depended on the gas flow. In contrast to Chemical Vapor Deposition (CVD) the highly reactive precursors are separated by inert gas pulses to make uses of a self limiting surface reaction that allows only a monolayer to grow.

Indexable Inserts

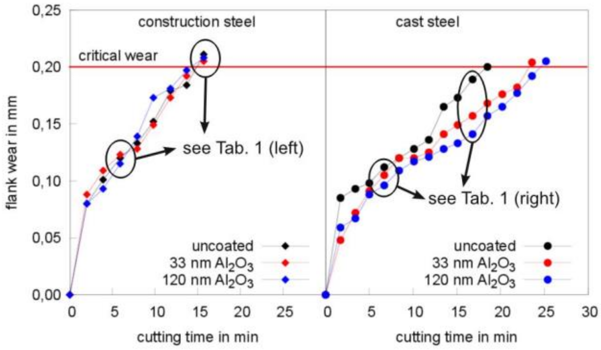


Fig. 3: Wear-time-diagram for the machining of construction (left) and cast steel (right). The sintered carbide substrate was TTM09 for construction steel and THM* for cast steel. (* Co. Kennametal)*

Tab. 1: Front and top view on indexable insert during the measurement

		after 8.4 min	after 18.5 min	after 5.9 min	after 15.7 min
uncoated	front view				
	top view				
140 nm Al_2O_3	front view				
	top view				

- low cutting speeds: ALD coatings cause more even wear at major flank and cutting face; built-up edge formation is reduced
- high cutting speeds: no significant improvement
- no significant influence of film thickness on wear behavior could be observed with the used cutting parameters
- ALD coating does not lead to any process-related performance loss

Drillers

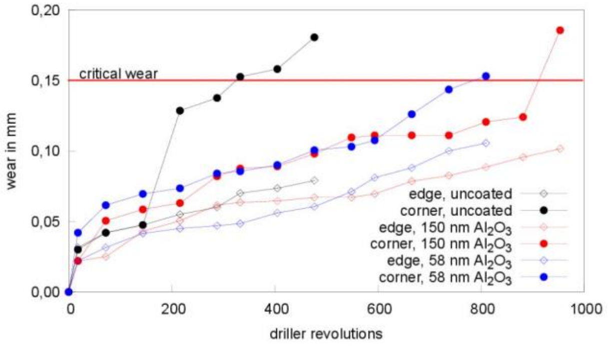
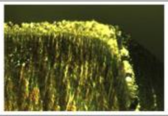
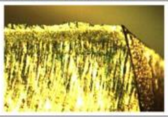
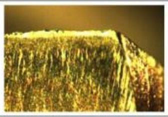


Fig. 4: Wear-time-diagram for the machining of cast steel. The sintered carbide substrate was HM-K20-K40. (* Co. GESAU)*

Tab. 2: Front view on driller's cutting edge

	after 549 turns	after 882 turns
uncoated		failed after 549 turns
150 nm Al_2O_3		

- wear of major flank is reduced and edge wear is drastically decreased
- a 170 % better performance was observed for drilling cast iron (abort criterion: width of wear mark = 0.15 mm)
- film thickness showed only little influence; the thicker coating (120 nm) tends to be favorable
- ALD coating does not lead to any process-related performance loss at used cutting speed of 120 m/min

Conclusion

It was proven that ALD provides a repeatable deposition process for Al_2O_3 as one of the fundamental hard materials for today's metal cutting applications. Abrasive wear of used tools (indexable inserts, drillers) is reduced without downgrading the cutting performance. Further ALD experiments have to show if other common hard coatings like e.g. TiN, AlN, and multi-layers of these are able to improve cutting tools as well.

Acknowledgement

This work was financially supported by the Saxon State Ministry for Higher Education, Research and the Arts.

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ALD Process for Tungsten Films in Model-like Glass Capillaries

C. Lorenz, T. Schenk, D. Fischer, M. Eschner, A. Neidhardt, C. Reinhold, U. Reinhold, H.-D. Schnabel
– Westsaxon University of Applied Sciences, Zwickau, Germany –
Contact: christine.lorenz@fh-zwickau.de

Aim of Investigation

Standard deposition methods, like PVD and CVD, are commonly applied on planar and low structured substrates. For structures with high aspect ratios, buried structures, or large scale substrates these deposition techniques fail.

Several applications, like photonic crystals or capillaries, require very thin, conformal and smooth (rms roughness below 1 nm) films of metals or metal compounds on silicon, glass or polymers.

These sophisticated requirements comply well with properties of an atomic layer deposition (ALD).

The aim of our institute's ALD-group is the achieving of an inner tungsten coating on model-like silicon capillaries. These studies serves as background for the inner coating of glass capillaries.

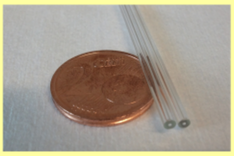

Fig. 1: Glass capillaries (diameter: 500 µm) compared to a 2 cent coin.

ALD System

Characteristic of ALD 150:

- Thermal ALD and PEALD possible
- Radial flow channel reactor
- Dual chamber system with a load lock module and a process chamber
- Precursor: TMA, H_2O, WF_6, Si_2H_6, $TiCl_4$, NH_3
- Coating materials: Al_2O_3, TiN, TiO_2, Ti(Al)N, W

Fig. 2: ALD system ALD 150 of Co. FHR.

Background

Molecular flow of reactants in capillaries

- Rectangular cross-section with that a >> b and the aspect ratio is found to be depth to slot thickness l / b (discussed in Gordon et. al. /1,2/)
- Transmission probability for rectangular cross sectional capillaries $P_{RCS}(x)$, where x is depth in the capillary: $$P_{RCS}(x)=\frac{1+\ln(0{,}433\cdot x/b+1)}{x/b+1}$$

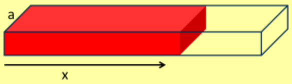

Fig. 3: Penetration depth l in capillary.

- Transmission probability for real circular cross-section capillaries $P_{CCS}(x)$, where d is the diameter: $$P_{CCS}(x)=\frac{14+4\cdot x/d}{14+18\cdot x/d+3(x/d)^2}$$
- Correlation between exposures required for complete coating and depth l of penetration is obtained by numerical integration, where U denotes the perimeter, A the cross-sectional area of the capillary tube, *exposure*$_{capillary}$ is the exposure of a capillary tube and *exposure*$_{flat\ surface}$ is the minimum exposure of a flat surface: $$\frac{(exposure)_{capillary}}{(exposure)_{flat\ surface}}=\frac{U}{A}\cdot\int_0^l P(x)^{-1}\,dx$$

Exposure in dependence on aspect ratio of different capillaries

- Circular capillary results agree with kinetic model of Gordon et. al. /1,2/
- Significant differences for rectangular aspect ratio at same exposures
- At same exposures the quotient of the aspect ratios between circular capillaries to the rectangular capillaries is about 1 to 3

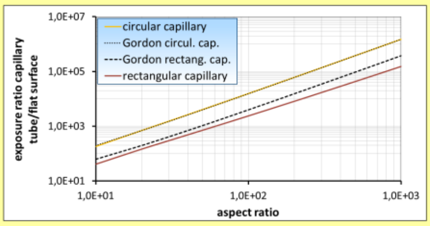

Fig. 4: Exposure ratio versus the aspect ratio.

Coating Process

- Model capillaries (see fig. 5) used to determine the process parameters
- Deposit ALD-Al_2O_3 first to advance the nucleation of ALD-Tungsten
- Layer properties (thickness, density, roughness, ...) are measured with spectroscopic ellipsometry and x-ray reflectometry
- Penetration depth is measured visually
- Experimental penetration depth results can be applied on real circular cross-section capillaries

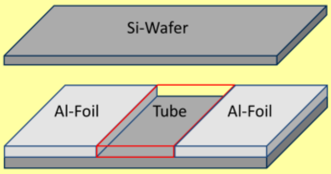

Fig. 5: Assembly of a model capillary with a 5 mm slit, in which the precursors can penetrate, embedded between 50 µm thick aluminum foil.

Result + Discussion

- Precursor penetration depth in a capillary increases with $t^{1/2}$, where t is the pulse duration, agreeing with theory of gas diffusion
- Results are consistent with the theory of gas diffusion limiting the flux of reactants into the capillaries
- Wider capillary forms facilitate deeper coating penetration (see Fig. 8)
- Tungsten coating require much longer pulse durations as Al_2O_3 coatings due to inertial reaction behaviour

coating material	process parameter			characteristic	
	pulse duration in s	purge time in s	GPC in nm/cycle	density in g/cm³	roughness in nm
Al_2O_3	0.5 - 3.0	10 - 30	0.1	3.15	0.67
W	2.0 - 48.0	10 - 60	0.4	16.73	2.31

Table 1: An overview of the used process parameters and the obtained film characteristic.

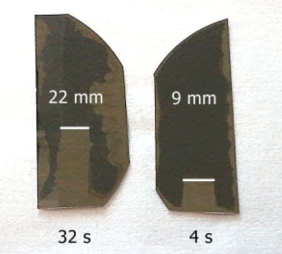

Fig. 6: Tungsten coated model capillaries. The white lines mark the deposition's end. During the process the capillaries are clamped. Emerging tensions permit an intrusion of coating at the edges of the substrate.

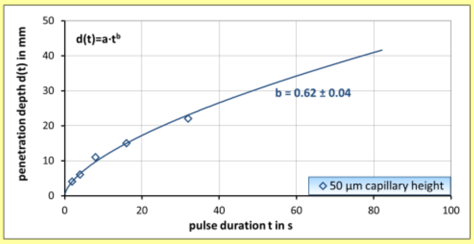

Fig. 7: Penetration depth – pulse duration diagram for a tungsten-coating, constituting a root function.

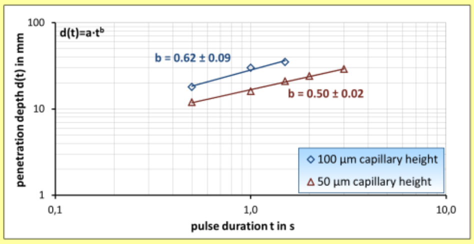

Fig. 8: Penetration depth – pulse duration diagram for an aluminium-oxide-coating with two different capillary heights.

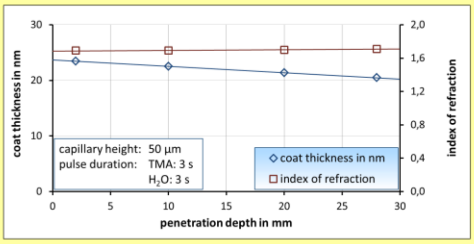

Fig. 9: Distribution of the coating thickness and the index of refraction over the penetration depth of Al_2O_3. The distribution of both parameters could be maintained nearly constant.

Conclusion

The repeatable deposition of aluminium oxide and tungsten films within capillaries is possible with an optimized ALD-coating process. Therefore, the correlation between precursor pulse duration and penetration depth agrees with the theory of gas diffusion.

The possibility to transfer the deposition results from model to real glass capillaries will be tested by us soon.

References

/1/ Gordon, R. G. et al. Chem. Vap. Deposition 2003,9,73,
/2/ Jousten, K., in "Wutz-Handbuch der Vakuumtechnik",Vieweg+Teubner, Wiesbaden 2010 (G)

Acknowledgement

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Adhesion Strength of Low Temperature Tungsten ALD-Films

C. Lorenz, M. Neuber, A. Neidhardt, C. Reinhold, U. Reinhold, H.-D. Schnabel,
- Westsaxon University of Applied Sciences, Zwickau, Germany
Contact: christine.lorenz@fh-zwickau.de

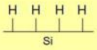
Motivation

Atomic Layer Deposition (ALD) is a coating technique which provides ultra-thin, conformal layers of metals and metal compounds. Important quality properties to characterize the generated films are not only thickness and density but also adhesion strength, which shows difficulties in measurement due to its small film thickness. ALD provides only few opportunities to enhance the growth velocity or the emerging layer qualities of tungsten films. One is the substrate surface condition. Our studies focus upon the effects of changed functional groups at the surface on the adhesion strength of tungsten films on Si-wafers.

Pretreatment of Silicon

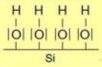
a) Wet etching with HF to etch native SiO_2 on the Si surface.

HF leads to:



b) Wet etching with KOH to etch native SiO_2 on the Si surface.

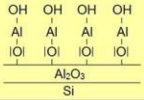
KOH leads to:



c) Precoating for W ALD: silicon coated with Al_2O_3 ALD at the same temperature

- 20 cycles of Al_2O_3 and the precursor are trimethyl aluminium (TMA) and water

The last water pulse leads to:



Low Temperature ALD of Tungsten (130 °C)

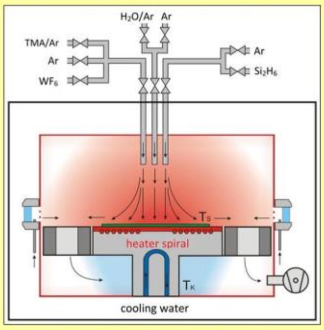


Fig. 1: The structure of radial flow channel reactor with W-precursor (WF_6 and Si_2H_6) and purge gas lines (Argon).

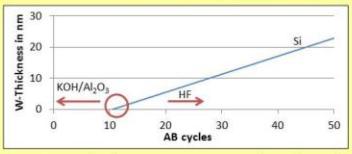


Fig. 2: W-Thickness versus AB cycles show the typically delayed start the W- nucleation.

Delayed tungsten nucleation

- Common with Si-wafer
- Intensified delay with HF wet etching

$I\text{-}Si\text{-}H + WF_6 \rightarrow I\text{-}Si\text{-}WF_5 + HF$ **improbable**

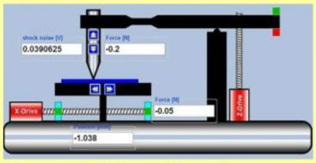
- Reduced delay with hydroxyl group by KOH or Al_2O_3- treatment

$I\text{-}Si\text{-}OH + WF_6 \rightarrow I\text{-}Si\text{-}O\text{-}WF_5 + HF$ **probable**

Scratch Adhesion Test

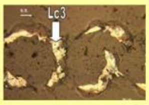
- Incrementally increasing load on the coating surface by a Rockwell C scratching point, while the sample is displaced at constant speed
- Critical load determination by the microscope observation and force recording (normal and tangential force)

Fig. 3: Scratch Tester.



shock noise [V] 0.0390625
Force [N] -0.2
Force [N] -0.05
Position [mm] -1.038

Fig. 4: Virtual image of scratch tester.

First failure $L_{C,1}$: initial cracking	
Second failure $L_{C,2}$: extensive cracking	
Final failure $L_{C,3}$: partial delamination of the coating from the substrate	

Tab. 1: Optical micrographs of critical failure points along a load scratch on HSS (High Speed Steel).

Results

	W on Si with native SiO_2 (few OH-groups)	W on Si (etched by HF, few OH-groups)	W on Si (etched by KOH, many OH-groups)	W on Si precoated with Al_2O_3 ALD (many OH-groups)
Critical failure $L_{C,1}$ in N	15.9	6.9	19.0	19.2
Thickness of SiO_2 in nm	3.0	< 1.0	7.0	3.0

Tab. 2: Overview of chemical pretreatments of silicon substrates with their first critical failure and thickness of SiO_2. X-ray reflectivity is used to measure the thickness of SiO_2.

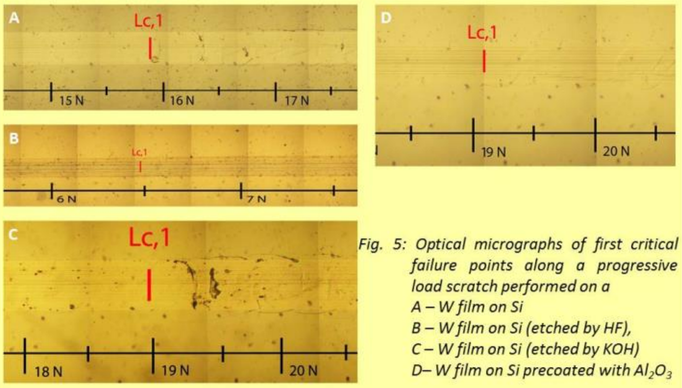
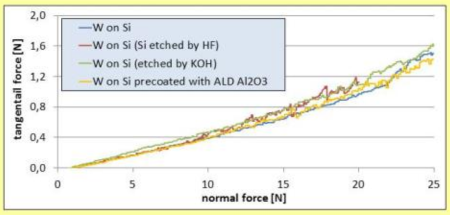


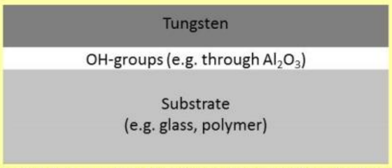
Fig. 5: Optical micrographs of first critical failure points along a progressive load scratch performed on a
A – W film on Si
B – W film on Si (etched by HF),
C – W film on Si (etched by KOH)
D– W film on Si precoated with Al_2O_3

Fig. 6: Tangential force versus normal force.



Discussion

- Significant increase of the critical failure by W-ALD-Coating with stable layers of SiO_2 or Al_2O_3
- W film on Si/SiO_2 wet etched by HF shows the smallest adhesion strength
  - ➢ Pure Si offers only few adsorption traps
- Depositions with high SiO_2 thicknesses show good adhesion strengths due to the increased concentrations of hydroxyl groups in the reactions
  - ➢ Connection between W and O is stable as between W and Si at low grown temperatures
  - ➢ Selection of the substrate pretreatment must be considered



Tungsten

OH-groups (e.g. through Al_2O_3)

Substrate
(e.g. glass, polymer)

Fig. 7: Ideal structure for W coatings on different substrate.

The results supported coating polymers or glass capillaries with tungsten. Investigations about stable coatings on such surfaces with OH-groups at low temperatures is the next challenge of our ALD Group.

References

/1/ Sekler, The Scratch Test; Surface and Coatings Technology, 36 (1988) 519-529

Acknowledgement

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Europäischer Fonds für regionale Entwicklung

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First results are presented at the 12th international conference on Atomic Layer Deposition (ALD)

International researcher/scientist, specialists and 29 exhibitors came to „AVS Topical Conference on Atomic Layer Deposition“ (ALD 2012) from 17 to 20 June 2012 in Dresden.

The conference supported by the American Vacuum Society takes place once a year alternately in America, Asia or Europe. This year the conference was assisted by 21 sponsors and was organized by NaMLab gGmbH in Dresden.

The AVS Topical Conference on Atomic Layer Deposition was dedicated to the science and technology of atomic layer controlled deposition of thin films. Atomic layer deposition (ALD) is used to fabricate ultrathin and conformal thin film structures for many semiconductor and thin film device applications. A unique attribute of ALD is the sequential self-limiting surface reaction to achieve control of film growth in the monolayer or sub-monolayer thickness regime. ALD is receiving attention for its potential applications from advanced electronics, microsystems, display manufacturing, energy capture and storage technologies, solid state lighting, biotechnology, security, and consumer products - particularly for any advanced technologies that require control of film structure in the nanometer or sub-nanometer scale.

The process and its application is an important main research topic for the Saxon research facilities, which were joined to become „ALD Lab Dresden“. Since that time the ALD principles are applied in the semi-conductor industry in Saxony and the Saxon equipment-industry offers tailored process plants and research facilities.

Poster

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International ALD Conference 2013 in San Diego

Already the thirteenth time, scientists and research groups from the industry and academic research field met to exchange insights of their research on the annual international conference of “Atomic Layer Deposition”. This year, the conference took place in the gorgeous ambience of San Diego from the 28th to the 31st of July.

The Atomic Layer Deposition is a technique that is used for the production of ultra-thin films. A very important feature is the self-limiting reaction of the precursors (gaseous compounds that take part in the reaction) on the surface of the substrate, leading to a step by step growth (in monolayers) of the compound. This way, thin films can be deposited precisely and reproducible.

Markus Neuber presents a poster of the ALD research group of the UAS Zwickau and answers questions of the interested audience.

Despite the many benefits and its high potential, the Atomic Layer Deposition is still not very much common in industrial applications. Therefore, this year’s conference was focused on the industrialization of the ALD.

The University of Applied Sciences (UAS) Zwickau was also involved in this exchange of knowledge. It was represented by Professor Hans-Dieter Schnabel, the current head of the ALD research group at the UAS Zwickau, and Markus Neuber, a master student who is doing an internship abroad. They showed the results of their research with the topic “Adhesion Strength of Low Temperature Tungsten ALD-Films” and answered the questions of the audience. A lot of them had suggestions that will be considered in the further work of research in Zwickau.

Furthermore, ideas for more projects with pioneering character were gathered. That is because the ALD gains more and more influence in research and industry.

Next year the international ALD conference be held in Osaka (Japan). Hopefully again with a contribution of the UAS Zwickau.

Especially the lunch time was used to establish contacts and to discuss different results and presentations.

Poster

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