# On the History of ALD and the VPHA project

The 14th International Baltic Conference on Atomic Layer Deposition

**Riikka L. Puurunen,[1] Yury Koshtyal,[2] Henrik Pedersen,[3] J. Ruud van Ommen,[4] Oksana Yurkevich,[5] Jonas Sundqvist[6]**

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*[2] Ioffe Institute, St. Petersburg, Russia*

*[3] Linköping University, Linköping, Sweden*

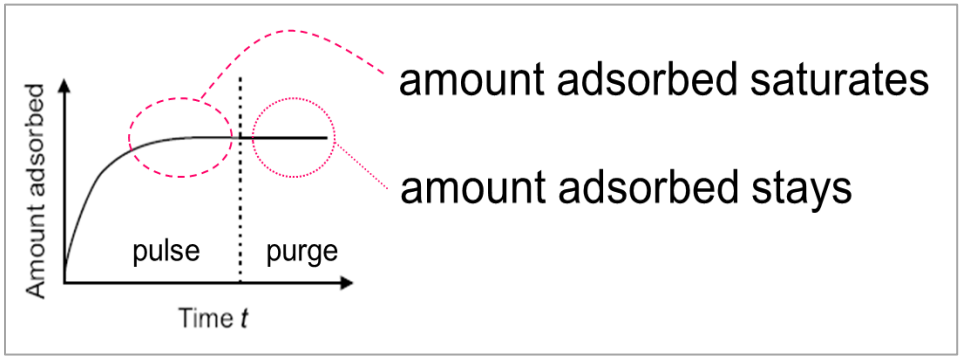
*[4] Delft University of Technology, Delft, the Netherlands*

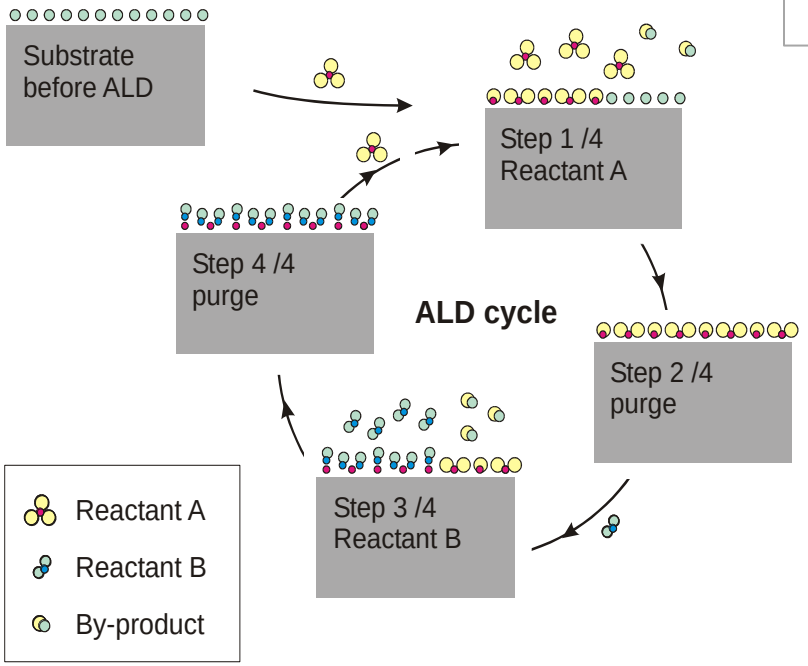
*[5] Immanuel Kant Baltic Federal University, Kaliningrad, Russia*

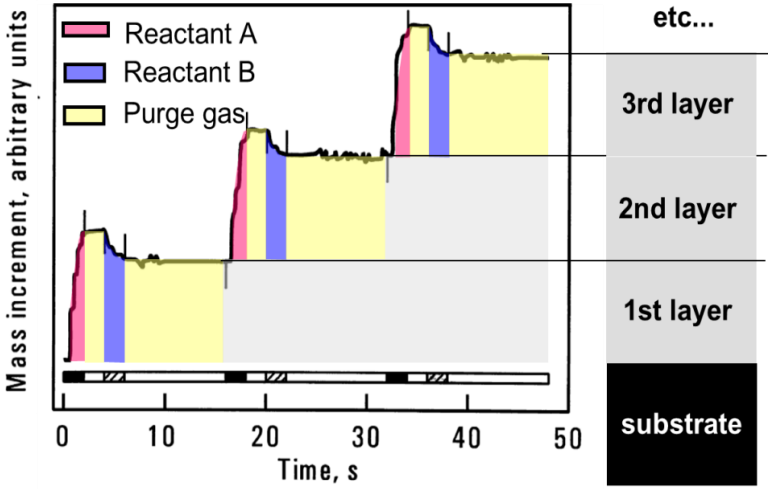
*[6] System Integration and Technology Transfer, Fraunhofer Institute for Ceramic Technologies and Systems IKTS, Dresden, Germany*

# *Atomic Layer Deposition (ALD): a chemical gas-phase technique for thin film growth*

*** saturating, irreversible chemisorption reactions**
*** repeated**

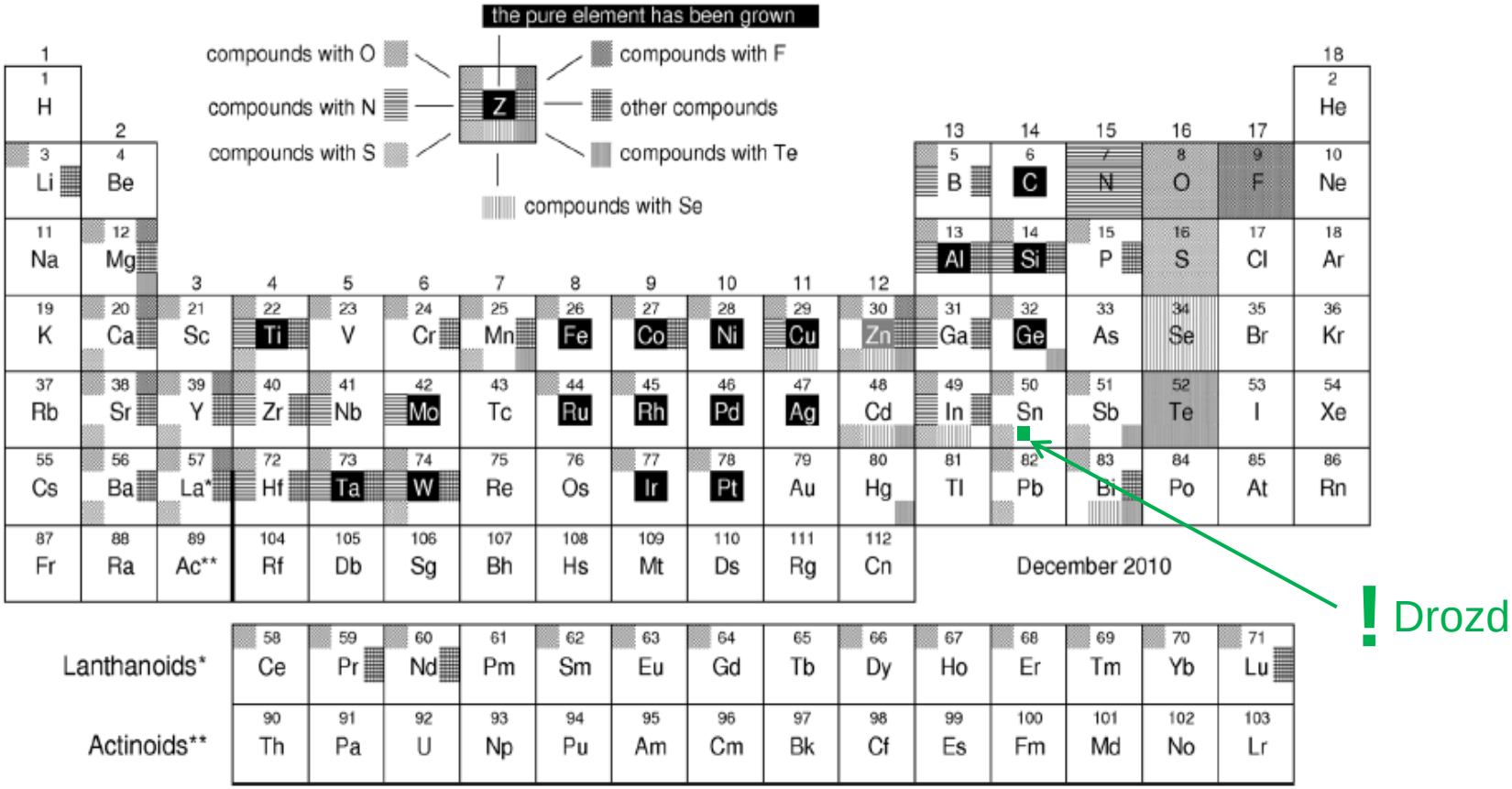



Adapted with permission from: Aarik et al., Thin Solid Films 340 (1999) 110.

# *Many classes of materials have been studied by ALD: oxides, nitrides, sulfides, metals, …*




Source: review by Miikkulainen, Leskelä, Ritala, Puurunen, J. Appl. Phys. **113**, 021301 (2013); >2000 references.
http://dx.doi.org/10.1063/1.4757907

# *ALD discovered & developed independently under two names*

- Atomic layer epitaxy (ALE), Finland, 1974 →
- Molecular layering (ML), Soviet Union, 1960s →
  - Молекулярное наслаивание (MH), transliterated as molekulyarnoye naslaivanie

- Especially ML has remained poorly known and cited

→ Virtual Project on the History of ALD (VPHA)

*Goal of this presentation: summarize the of key achievements in VPHA 2013-2016*

ALD history

Web of Science,
Sept. 30, 2016

Knez, Nielsch, Niinistö, Adv. Mat. 2007

- "Atomic layer deposition (ALD), originally called Atomic layer epitaxy (ALE), was developed in the 1970s by Suntola and Antson…"

(Times cited: 426)

George, Chemical Reviews 2010

- "The history of ALE and ALD dates back to the 1970s in Finland."

(Times cited: 1340)

ALD history

Puurunen, Doctoral thesis, Helsinki University of Technology, 2002

Several terms are used in the literature to describe processes that are based on repeating separate, saturating gas–solid reactions. In addition to ALD, terms such as atomic layer epitaxy (ALE),[23–25,55] atomic layer chemical vapour deposition (ALCVD™),[67] molecular layering,[68] atomic layer growth,[49] atomic layer processing[69] and chemical surface coating[70] can be found. Atomic layer epitaxy was the original term, used by Suntola and co-workers, who developed the technique in the 70's for the preparation of ZnS thin films for electroluminescent devices.[71] During the 90's, the ALE technique was extended to catalyst preparation.[24,25,28,72] In catalyst applications, and most often also in thin film applications, the processed materials are amorphous or polycrystalline, not epitaxial, in nature,[23,25,26,73] and the original term, atomic layer epitaxy, does not well describe the processed materials. Although the term atomic layer deposition is now preferred,[26,56,57,63,74,75] some groups[66,76] prefer to use the original term, mainly for historical reasons.[76] In this thesis, the term atomic layer deposition (ALD) is used.

Table I. Alternative Names to the ALD Method

| Name | Acronym | Comments |
|---|---|---|
| Atomic layer deposition | ALD | General, covers all kinds of films In a close connection with the original name |
| Atomic layer epitaxy | ALE | The original name, but should be reserved for epitaxial films only |
| Atomic layer growth | ALG | Like ALD but less used |
| Atomic layer chemical vapor deposition | ALCVD | Emphasizes the relation to CVD |
| Molecular layer epitaxy | MLE | Emphasizes molecular compounds as precursors |
| Digital layer epitaxy | DLE | Emphasizes the digital thickness control |
| Molecular layering | ML | Dates back to old Russian literature |
| Successive layerwise chemisorption | | |
| Sequential surface chemical reaction growth | | |
| Pulsed beam chemical vapor deposition | | |

Ritala, M. & Leskelä, M. Nalwa, H. S. (Ed.) Atomic Layer Deposition Handbook of Thin Film Materials, Academic Press, 2002, 1, 103-159

"Early work on Atomic Layer Deposition cited"
Malygin, Smirnov, Solid State Technology, 2002, March, p. 14

Puurunen, J. Appl. Phys. 2005

- "TABLE I. Some Soviet–Russian ALD investigations."

ALD history

Puurunen, J. Appl. Phys. 2005

- "According to the more commonly acknowledged origin, ALD was developed under the name "atomic layer epitaxy (ALE)" in Finland by Suntola and co-workers. "
- "The less commonly acknowledged origin of ALD dates back to … the Soviet Union … 1960s … group of Professor Aleskovskii."

(Times cited: 1017)

Web of Science, Sept. 30, 2016

Knez, Nielsch, Niinistö, Adv. Mat. 2007

- "Atomic layer deposition (ALD), originally called Atomic layer epitaxy (ALE), was developed in the 1970s by Suntola and Antson…"

(Times cited: 426)

George, Chemical Reviews 2010

- "The history of ALE and ALD dates back to the 1970s in Finland."

(Times cited: 1340)

ALD history

# LinkedIn question May 6, 2013

**Riikka Puurunen**
Senior Research Scientist at VTT

3y

## What are the "Molecular layering" papers by Koltsov from "early 1960's"?

I noticed that the "hall of fame" of aldpulse.com has been updated with a few faces, among them Aleskovskii and Koltsov.

The question is: what are these papers published "early in 1960's" by Koltsov?

So, if anyone has info on this, please share, for the common benefit. --- The references should preferably be detailed anough so that a skilfull librarian gets a copy orderd with that info. Show less

Like Comment 1 25

LinkedIn ”ALD - Atomic Layer Deposition” group
https://www.linkedin.com/groups/1885076/1885076-238399494

## LinkedIn question May 6, 2013

## VPHA opened July 25, 2013 + ALD 2013 San Diego

Atomic layer deposition (ALD) has become a technique that, for its part, changes the world we live in. As for any significant tec
tec
Wh
wh

1. First, we should generate a complete list of early ALD publications.
2. Second, interested individuals should pick up some of the early publications, read them, and comment on the work. For example: was ALD made (i.e., do you recognize the work as ALD), and if yes, which process it was; and other noteworthy things.
3. Third, the individual should share their comments with others, and the comments of different people should be collected together.

*Atmosphere of Openness, Respect, and Trust*

http://vph-ald.com/Introduction%20and%20invitation%20to%20participate.html

# VPHA opened July 25, 2013 + ALD 2013 San Diego



**ALD-history-evolving-file**
Early ALD literature up to 1986
Curretly: ~300 pages

https://docs.google.com/document/d/1AlJg29dJM2if4SGzMJSSmwZskCNaAMQMO9LU6UYPios/

# VPHA opened July 25, 2013 + ALD 2013 San Diego

http://www2.avs.org/conferences/ALD/2013/index.html


**"Virtual project on the history of ALD"**

- Goal: generate a common view on the early evolution of ALD in a collaborative project by the whole ALD community
  - "Unknowns" especially in the Molecular Layering work (Soviet Union)
- Invitation to participatepublishedJuly 25, 2013, aldpulse.com (Riikka Puurunen, AzizAbdulagatov, Jonas Sundqvist and Annina Titoff)
  - Everyonewelcome to join, differentbackgroundsbeneficial
- List of early ALD publications will be created
- Participation: read & comment on the significance of at least one historical publication that interests you (not much work!)
- Result to be published, names of all contributors announced
- Plan: project will be open until the end of 2013

More info: invitation at aldpulse.com; LinkedIn "ALD History" subgroup; or Riikka Puurunen at the ALD 2013 conference (Tue: poster 369)

Announcement made by the conference chair Prof. Kim

# VPHA opened July 25, 2013 + ALD 2013 San Diego

Puurunen, J. Appl. Phys. 2005
- "According to the more commonly acknowledged origin, ALD was developed under the name "atomic layer epitaxy (ALE)" in Finland by Suntola and co-workers. "
- "The less commonly acknowledged origin of ALD dates back to … the Soviet Union … 1960s … group of Professor Aleskovskii."

(Times cited: 1017)

Web of Science, Sept. 30, 2016

Knez, Nielsch, Niinistö, Adv. Mat. 2007
- "Atomic layer deposition (ALD), originally called Atomic layer epitaxy (ALE), was developed in the 1970s by Suntola and Antson…"

(Times cited: 426)

George, Chemical Reviews 2010
- "The history of ALE and ALD dates back to the 1970s in Finland."

(Times cited: 1340)

Parsons, Elam, George, Haukka, Jeon, Kessels, Leskelä, Poodt, Ritala, Rossnagel, JVST A 2013
- "The ALD principle… was first published under name "molecular layering" in the early 1960s"
- **"A planar thin film was not produced or evaluated."**

Published: 16.8.2013
(Times cited: 12)

ALD history

LinkedIn question May 6, 2013

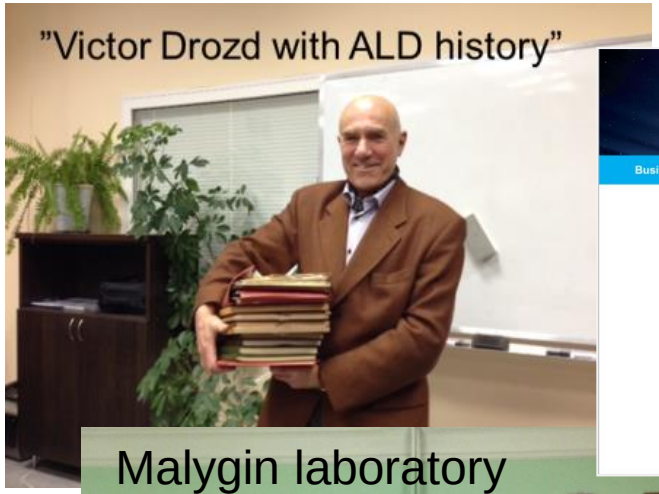
VPHA opened July 25, 2013 + ALD 2013 San Diego

Publication Plan October 18, 2013 (updated Jan 13, 2014 & April 5, 2015)

1) Poster at Baltic ALD 2014
2) Poster at ALD 2014, Kyoto
3) Presentation at ALD 2014, Kyoto
4) ALD history tutorial at ALD 2014, Kyoto
5) Essay on the early history of ALE-ALD
6) Website for ALD history and VPHA
7) Exhibition: 40 years of ALD in Finland - Photos, Stories (FinALD40)
8) Review article/essay on the early history of ML-ALD
9) Presentation at ALD 2016
10) Optional: general ALD history review article
11) Updating wikipedia
12) Closing the VPHA

http://vph-ald.com/Publication%20Plan.html

- LinkedIn question May 6, 2013
- VPHA opened July 25, 2013 + ALD 2013 San Diego
- Publication Plan October 18, 2013 (updated Jan 13, 2014 & April 5, 2015)
- Puurunen visit to St. Petersburg November 2013



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- Publication Plan October 18, 2013 (updated Jan 13, 2014 & April 5, 2015)
- Puurunen visit to St. Petersburg November 2013
- Baltic ALD 2014 Helsinki: VPHA poster + FinALD40 exhibition


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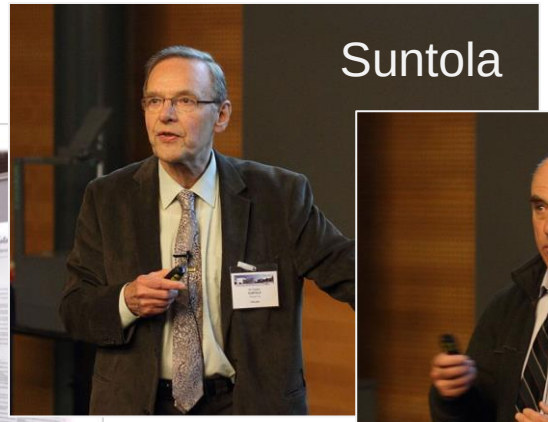


https://commons.wikimed
File:Tuomo_Suntola,_20


Photo by Riikka Puurunen

Access through:
http://vph-ald.com/ALD-history-publications.html

- LinkedIn question May 6, 2013
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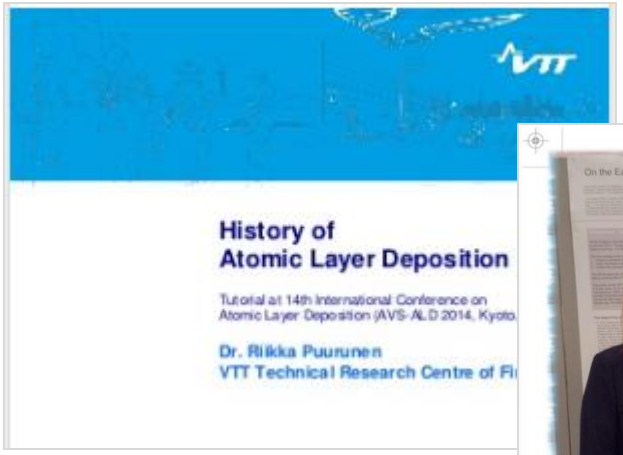
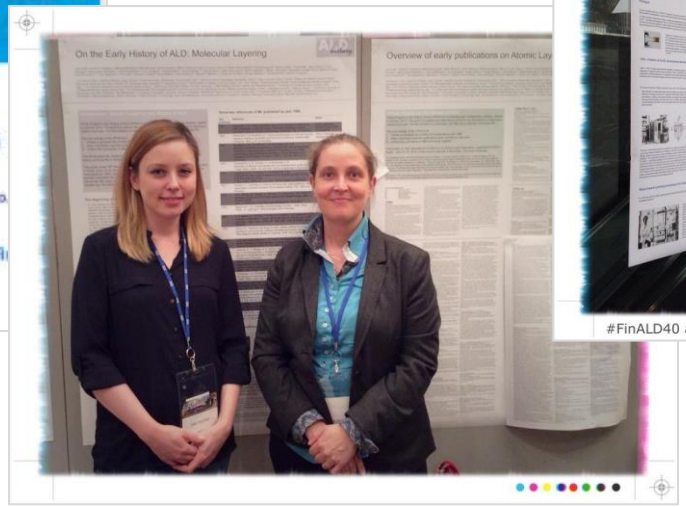
- ALD 2014 Kyoto: history tutorial + two posters

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https://twitter.com/rlpuu/status/478367321572913152

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- ALD 2014 Kyoto: history tutorial + two posters
- ”Suntola’s ALE essay” published Oct 15, 2014 & FinALD40 release

Chemical Vapor Deposition *Explore this journal >*

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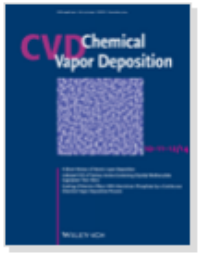
Essay

## A Short History of Atomic Layer Deposition: Tuomo Suntola's Atomic Layer Epitaxy†

Riikka L. Puurunen

**First published:** 15 October 2014 Full publication history

**DOI:** 10.1002/cvde.201402012 View/save citation

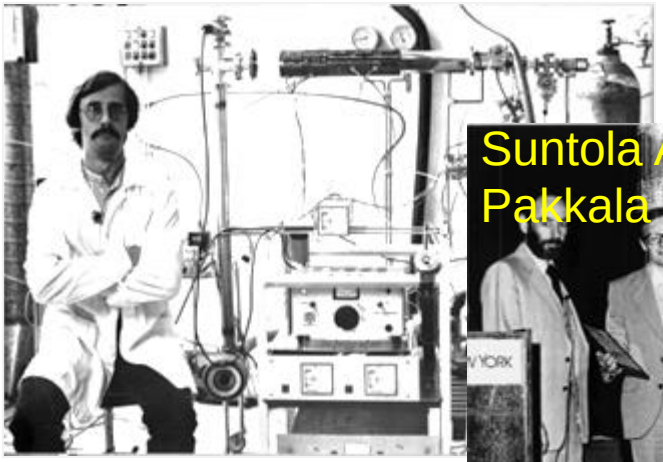


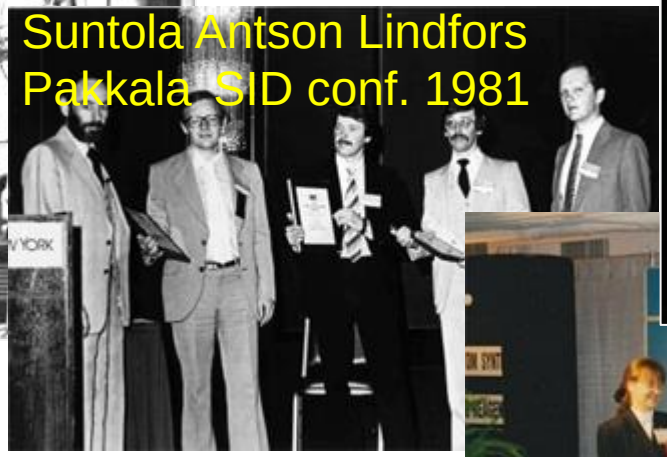

View issue TOC
Volume 20, Issue 10-11-12
December 2014
Pages 332–344

10.1002/cvde.201402012/full

# ”Suntola’s ALE essay” published Oct 15, 2014 & FinALD40 release

1978 Sven Lindfors & flow reactor

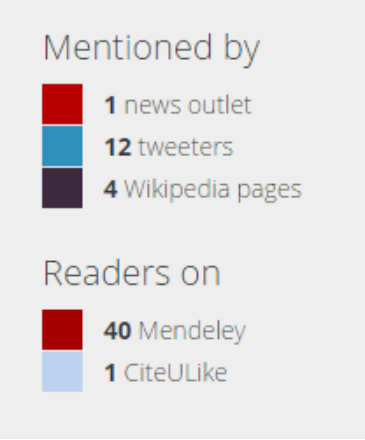






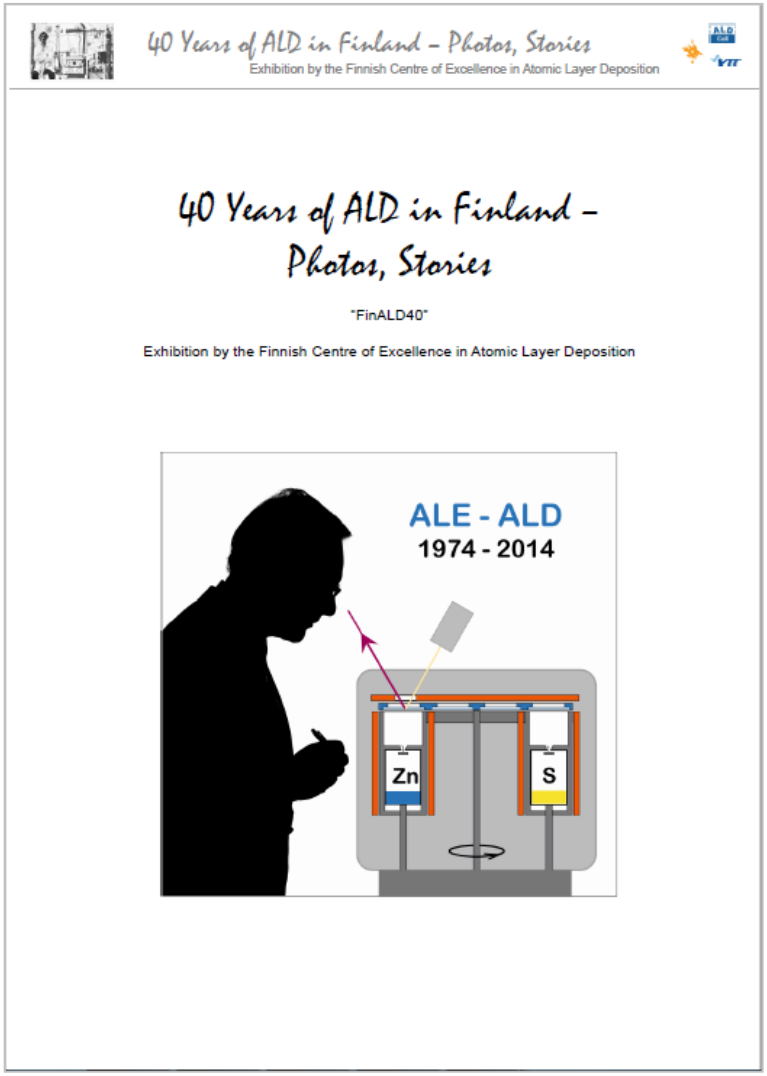
Microchemistry's at the MRS 1994, Boston

10.1002/cvde.201402012/full


Times cited: 7

# ”Suntola’s ALE essay” published Oct 15, 2014 & FinALD40 release

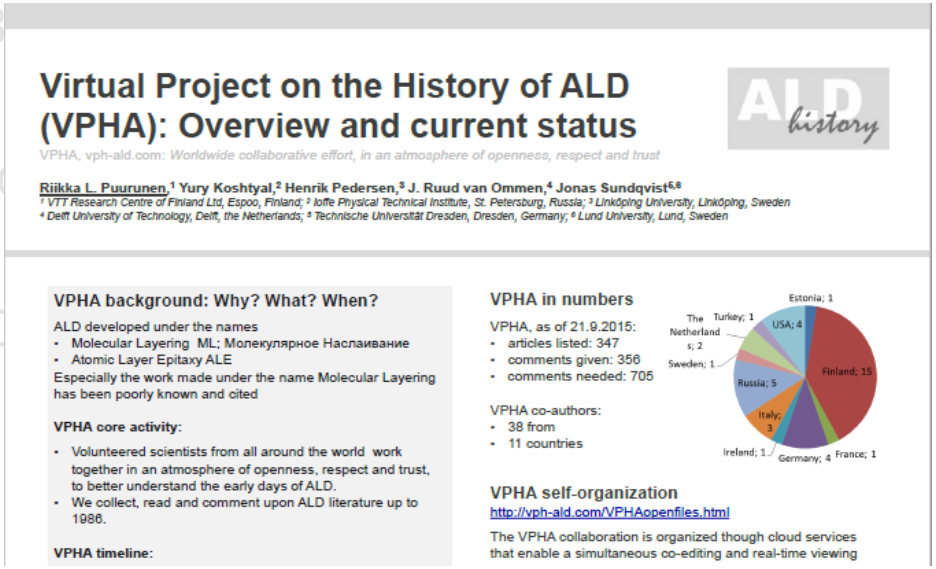



- Released Nov 29, 2014
- Preface Markku Leskelä
- Tuomo Suntola’s ALE in Short
- ALD research at Helsinki University of Technology
- ALD research at University of Helsinki
- Photos related to ALD research
- Academic ALD theses in Finland
- Organizations active with ALD in Finland
- Stories: Story on ZyALD

Access through:

- http://aldcoe.fi
- http://vph-ald.com

- LinkedIn question May 6, 2013
- VPHA opened July 25, 2013 + ALD 2013 San Diego
- Publication Plan October 18, 2013
- Puurunen visit to St. Petersburg N
- Baltic ALD 2014 Helsinki: VPHA p
- ALD 2014 Kyoto: history tutorial +
- ”Suntola’s ALE essay” published C
- ALD Russia Moscow
  + Baltic ALD Tartu Sep-Oct 2015



## Virtual Project on the History of ALD (VPHA): Overview and current status

VPHA, vph-ald.com: *Worldwide collaborative effort, in an atmosphere of openness, respect and trust*

Riikka L. Puurunen,[1] Yury Koshtyal,[2] Henrik Pedersen,[3] J. Ruud van Ommen,[4] Jonas Sundqvist[5,6]
[1] VTT Research Centre of Finland Ltd, Espoo, Finland; [2] Ioffe Physical Technical Institute, St. Petersburg, Russia; [3] Linköping University, Linköping, Sweden
[4] Delft University of Technology, Delft, the Netherlands; [5] Technische Universität Dresden, Dresden, Germany; [6] Lund University, Lund, Sweden

**VPHA background: Why? What? When?**

ALD developed under the names
- Molecular Layering ML; Молекулярное Наслаивание
- Atomic Layer Epitaxy ALE

Especially the work made under the name Molecular Layering has been poorly known and cited

**VPHA core activity:**

- Volunteered scientists from all around the world work together in an atmosphere of openness, respect and trust, to better understand the early days of ALD.
- We collect, read and comment upon ALD literature up to 1986.

**VPHA timeline:**

**VPHA in numbers**

VPHA, as of 21.9.2015:
- articles listed: 347
- comments given: 356
- comments needed: 705

VPHA co-authors:
- 38 from
- 11 countries



**VPHA self-organization**

http://vph-ald.com/VPHAopenfiles.html

The VPHA collaboration is organized though cloud services that enable a simultaneous co-editing and real-time viewing

| | |
|---|---|
| 11.00 – 11.20 | Oral presentation: Virtual Project on the History of ALD: overview and current status, ***Riikka Puurunen, VTT, Finland*** |

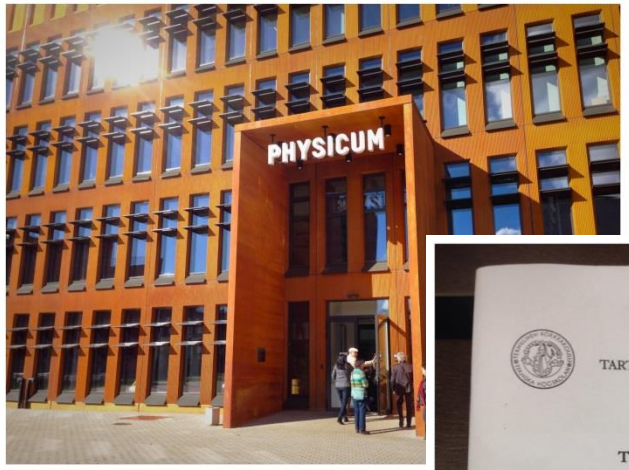
Access through:
http://vph-ald.com/ALD-history-publications.html

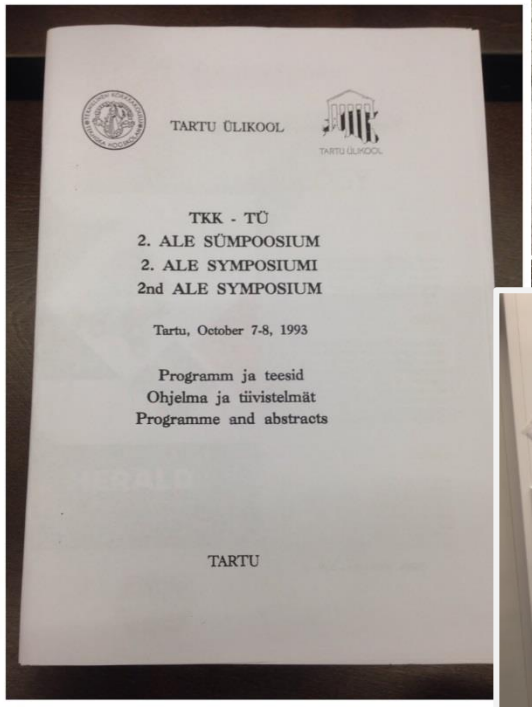
# ALD Russia Moscow + Baltic ALD Tartu Sep-Oct 2015




http://www.ald-mipt.ru/ald-russia.html
http://aldhistory.blogspot.fi/2015/11/ald-russia-2015-travel-report-Puurunen.html

# ALD Russia Moscow + Baltic ALD Tartu Sep-Oct 2015



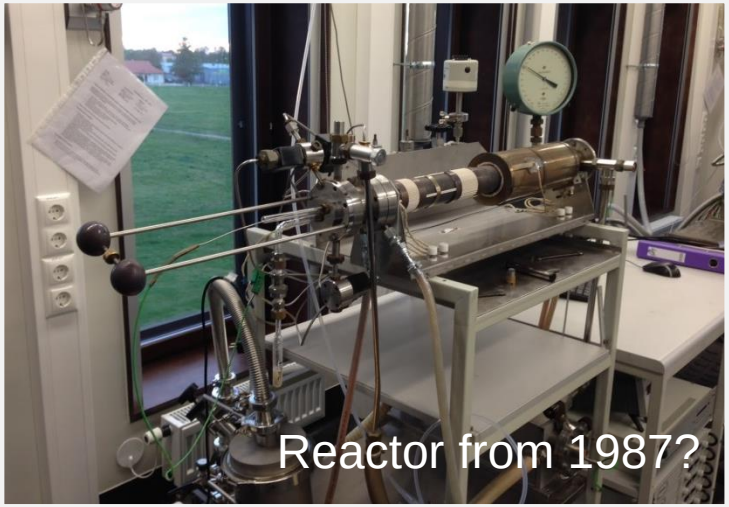
TARTU ÜLIKOOL

TKK - TÜ
2. ALE SÜMPOOSIUM
2. ALE SYMPOSIUMI
2nd ALE SYMPOSIUM

Tartu, October 7-8, 1993

Programm ja teesid
Ohjelma ja tiivistelmät
Programme and abstracts

TARTU


http://www.bald2015.ee/
http://aldhistory.blogspot.fi/2016/01/baltic-ald-2015-tartu-travel-notes-puurunen.html

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- ”Suntola’s ALE essay” published Oct 15, 2014 & FinALD40 release
- ALD Russia Moscow + Baltic ALD Tartu Sep-Oct 2015
- “ML essay” published Dec 17, 2015

Chemical Vapor Deposition *Explore this journal >*

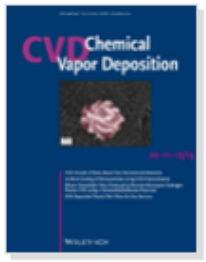
Essay

## From V. B. Aleskovskii's “Framework” Hypothesis to the Method of Molecular Layering/Atomic Layer Deposition†

Anatolii A. Malygin, Victor E. Drozd, Anatolii A. Malkov, Vladimir M. Smirnov

**First published:** 17 December 2015 Full publication history

**DOI:** 10.1002/cvde.201502013 View/save citation

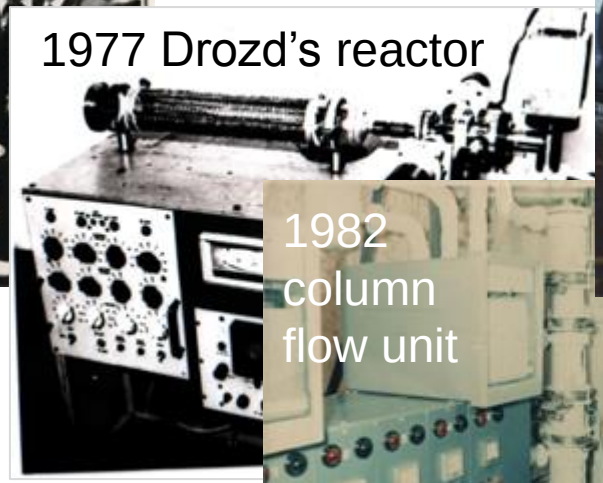



View issue TOC
Volume 21, Issue 10-11-12
December 2015
Pages 216–240

10.1002/cvde.201502013/abstract

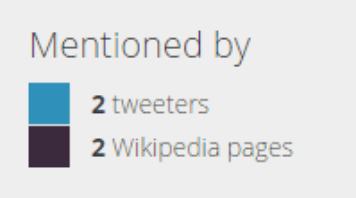
# “ML essay” published Dec 17, 2015

ALE-4 Linz 1996: George, Suntola, Drozd, Niinistö, Leskelä







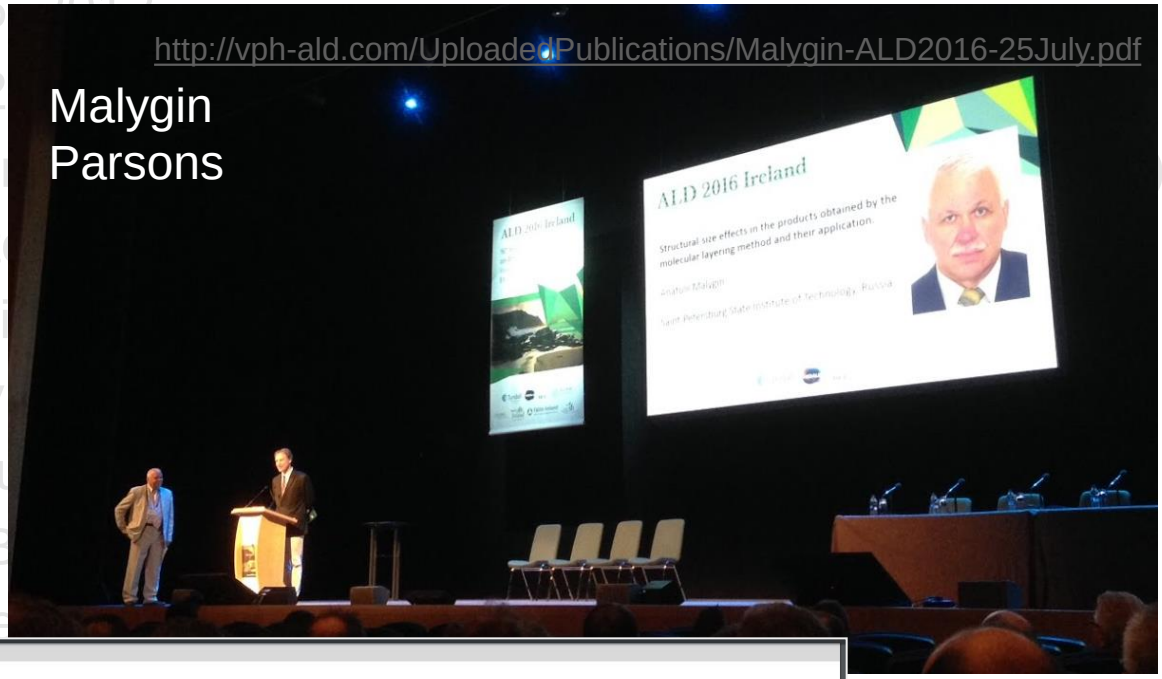

Times cited: 0

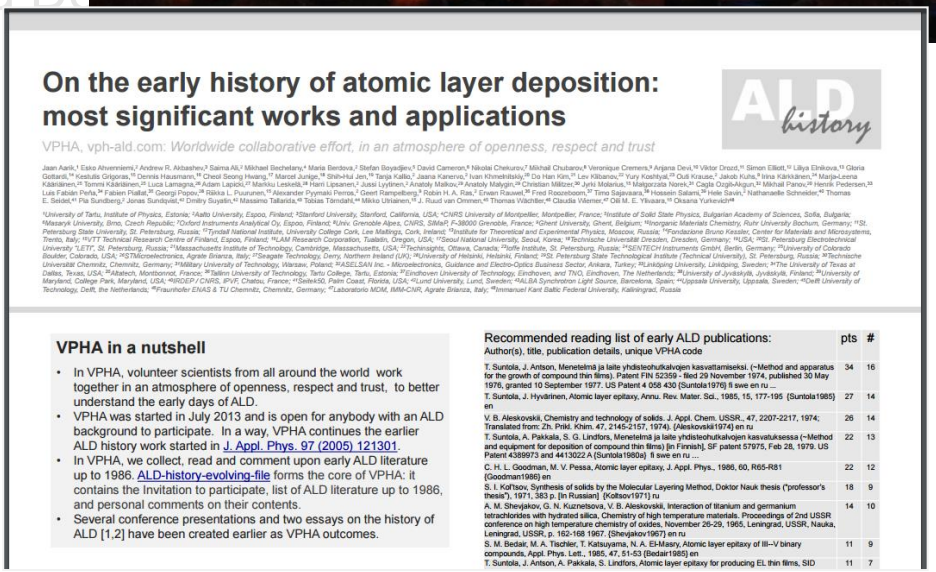
10.1002/cvde.201502013/abstract

ALD history

- LinkedIn question May 6, 2013
- VPHA opened July 25, 2
- Publication Plan October
- Puurunen visit to St. Pet
- Baltic ALD 2014 Helsinki
- ALD 2014 Kyoto: history
- ”Suntola’s ALE essay” pu
- ALD Russia Moscow + E
- “ML essay” published De
- **ALD 2016 Dublin: Malygin invited talk + VPHA poster**

http://vph-ald.com/UploadedPublications/Malygin-ALD2016-25July.pdf



## On the early history of atomic layer deposition: most significant works and applications

VPHA, vph-ald.com: *Worldwide collaborative effort, in an atmosphere of openness, respect and trust*

### VPHA in a nutshell

- In VPHA, volunteer scientists from all around the world work together in an atmosphere of openness, respect and trust, to better understand the early days of ALD.
- VPHA was started in July 2013 and is open for anybody with an ALD background to participate. In a way, VPHA continues the earlier ALD history work started in J. Appl. Phys. 97 (2005) 121301.
- In VPHA, we collect, read and comment upon early ALD literature up to 1986. ALD-history-evolving-file forms the core of VPHA: it contains the Invitation to participate, list of ALD literature up to 1986, and personal comments on their contents.
- Several conference presentations and two essays on the history of ALD [1,2] have been created earlier as VPHA outcomes.

Recommended reading list of early ALD publications:

| Author(s), title, publication details, unique VPHA code | pts | # |
|---|---|---|
| T. Suntola, J. Antson, Menetelmä ja laite yhdisteohutkalvojen kasvattamiseksi. (~Method and apparatus for the growth of compound thin films). Patent FIN 52359 - filed 29 November 1974, published 30 May 1976, granted 10 September 1977. US Patent 4 058 430 (Suntola1976) fi swe en ru ... | 34 | 16 |
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LinkedIn question May 6, 2013

**Recommended reading list of early publications on Atomic Layer Deposition—outcome of the Virtual Project on the History of ALD**

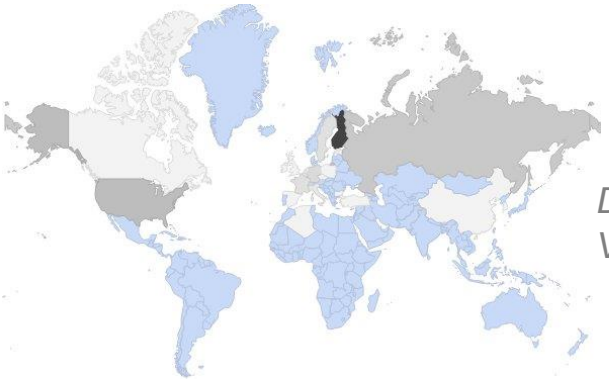
Running title: Recommended reading list of early ALD publications

Running Authors: Ahvenniemi et al.

62 authors, submitted Oct 30, 2016

ALD 2016 Dublin: Malygin invited talk + VPHA poster

VPHA manuscript + Baltic ALD 2016 St. Petersburg



*Darker grey → more VPHA contributors*

http://aldhistory.blogspot.fi/search/label/JVS

# VPHA manuscript + Baltic ALD 2016 St. Petersburg

| Title | Authors | Year |
|---|---|---|
| Some characteristics of molecular layering reactions | Aleskovskii, Koltsov | 1965 |
| Interaction of titanium and germanium tetrachlorides with hydrated silica | Shevjakov, Kuznetsova, Aleskovskii | 1967 |
| Measuring thicknesses of ultra-thin silicon oxide films deposited by molecular layering on the surface of single crystal silicon using polarization method | Sveshnikova, Koltsov, Aleskovskii | 1969 |
| Synthesis of solids by the Molecular Layering Method | Koltsov | 1971 |
| Chemistry and technology of solids | Aleskovskii | 1974 |
| Method and apparatus for the growth of compound thin films | Suntola, Antson | 1974* |
| Synthesis and study of oxide coatings obtained by molecular layering on semiconductor surfaces | Drozd | 1978 |
| Atomic layer epitaxy for producing EL thin films | Suntola, Antson, Pakkala, Lindfors | 1980 |
| Atomic layer epitaxy | Suntola, Hyvärinen | 1985 |
| Atomic layer epitaxy of III-V binary compounds | Bedair, Tischler, Katsuyama, El-Masry | 1985 |
| Molecular Layer Epitaxy | Nishizawa, Abe, Kurabayashi | 1985 |

Shortened from: VPHA manuscript (62 authors), submitted to JVSTA (2016).

# VPHA manuscript + Baltic ALD 2016 St. Petersburg



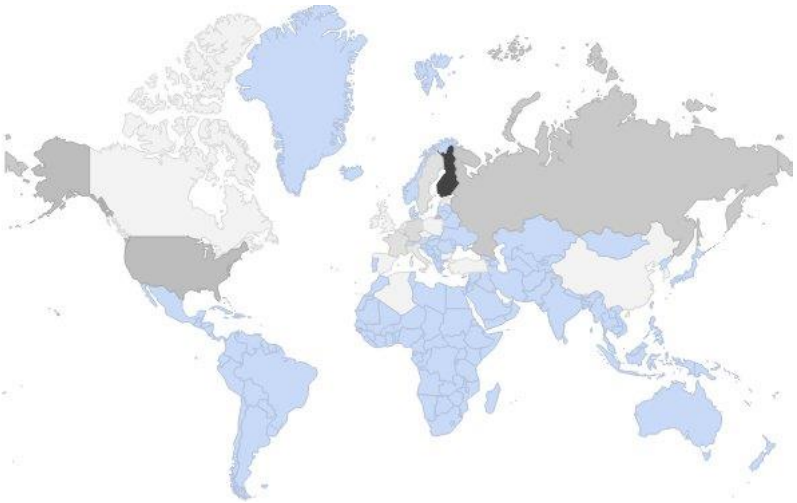
TABLE I. Some Soviet–Russian ALD investigations.

| Z[a] | Material | Reactant A | Reactant B | Substrate[b] | Refs. |
|---|---|---|---|---|---|
| 5 Boron | | | | | |
| | $B_2O_3$ | $BBr_3$ | $H_2O$ | $SiO_2$ gel | 85 |
| | $B_xP_yO_z$ | $B(OMe)_3$ | $POCl_3$ | $SiO_2$ gel | 88 and 89 |
| 13 Aluminum | | | | | |
| | | | | $SiO_2$ gel | 83, 90, and 91 |
| | | | | $Al_2O_3$ gel | 90 |
| | | | | Si, Al | 91 |
| | | | | Si | 92 |
| | | | | Si | 93 |
| | | | | $SiO_2$ gel | 94 |
| | | | | Si | 93 |
| | | | | Si | 78 |
| | | | | Ge | 95 |
| | | | | $SiO_2$ gel | 90 |
| | | | | $Al_2O_3$ gel | 90 |
| | | | | SiC | 96 |
| | | | | $SiO_2$ gel | 97–99 |
| | | | | $SiO_2$ gel | 94 |
| | | | | $SiO_2$ gel | 88 |

catalysts by ALD from 1972,[54] geometrical modelling of surface site filling during ALD from 1975,[55] particle coating in a fluidized bed from 1979,[56] ALD with an electric field to guide the growth from 1981,[57] quantum chemical modelling from 1983,[58] amine-catalyzed $SiO_2$ ALD from 1984,[59] ALD on polymers from 1984,[60] and the proposal of photo ALD from 1984.[61]

From: VPHA manuscript (62 authors), submitted to JVSTA (2016).

# *Conclusion & outlook*



- ALD discovered & developed independently twice
- VPHA reading still continues, new volunteers welcome
  - >300 comments still needed
- ALD-history-evolving-file: growing reference source
- Worldwide ALD-related lists under construction
  - Conferences, theses (PhD + higher), review articles
- More info: http://vph-ald.com & ALD History Blog http://aldhistory.blogspot.fi
  Contact: info@vph-ald.com

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- VPHA co-authors (as of 1.10.2016): Jaan Aarik, Esko Ahvenniemi, Andrew R. Akbashev, Saima Ali, Mikhael Bechelany, Maria Berdova, Stefan Boyadjiev, David Cameron, Nikolai Chekurov, Rong Chen, Mikhail Chubarov, Veronique Cremers, Anjana Devi, Viktor Drozd, Simon Elliott, Liliya Elnikova, Gloria Gottardi, Kestutis Grigoras, Dennis Hausmann, Cheol Seong Hwang, Marcel Junige, Shih-Hui Jen, Tanja Kallio, Jaana Kanervo, Ivan Khmelnitskiy, Do Han Kim, Lev Klibanov, Outi Krause, Jakob Kuhs, Irina Kärkkänen, Marja-Leena Kääriäinen, Tommi Kääriäinen, Luca Lamagna, Adam Lapicki, Markku Leskelä, Harri Lipsanen, Jussi Lyytinen, Anatoly Malkov, Anatoly Malygin, Abdelkader Mennad, Christian Militzer, Jyrki Molarius, Małgorzata Norek, Cagla Ozgit-Akgun, Mikhail Panov, Luis Fabián Peña, Fabien Piallat, Georgi Popov, Alexander Pyymaki Perros, Geert Rampelberg, Robin H. A. Ras, Erwan Rauwel, Fred Roozeboom, Timo Sajavaara, Hossein Salami, Hele Savin, Nathanaelle Schneider, Thomas E. Seidel, David C. Smith, Pia Sundberg, Dmitry Suyatin, Massimo Tallarida, Tobias Törndahl, Mikko Utriainen, Thomas Wächtler, Claudia Wiemer, Oili M. E. Ylivaara
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- HERALD COST MP1402 Action

# Thank you, questions? СПАСИБО, ВОПРОСЫ?

атомно-слоевое осаждение 原子层沉积

Deposição por Camadas Atômicas Осадження атомних шарів

Atomikerroskasvatus Atomik Katman Biriktirme Aatomkihtsadestus

आण्विक थर लेप Atomlagenabscheidung परमाणु परत निक्षेपण

원자층증착 **Atomic Layer Deposition** Atoomlaagdepositie

Atomlagerdeponering Depositación de Capas Atómicas

Deposizione a Strati Atomici Parmanu Parat Nishepan

Молекулярное Наслаивание εναπόθεση ατομικού στρώματος

Atomlagsdeponering שכבות אטומיות השקעת Dépôt de Couches Atomiques

Dépôt Chimique en Phase Vapeur à Flux Alternés 原子層堆積

*ALD name collection in LinkedIn ALD – Atomic Layer Deposition*

ALD history