Thin Films Lecture 2

Vacuum Technology

Jari Koskinen



Vacuum system















 \wedge

School of Chemical Technology

Large surfaces, upscaling



www.scheuten.com

Residual gas



Composition of atmospheric air

	% by weight	% by volume	Partial pressure mbar
N ₂	75.51	78.1	792
02	23.01	20.93	212
Ar	1.29	0.93	9.47
CO ₂	0.04	0.03	0.31
Ne	1.2 · 10 ^{−3}	1.8 · 10 ⁻³	1.9 · 10 ^{−2}
Не	7 · 10 ^{–5}	7 · 10 ^{−5}	5.3 · 10 ⁻³
CH₄	2 · 10 ⁻⁴	2 · 10 ⁻⁴	2 · 10 ^{−3}
Kr	3 · 10 ⁻⁴	1.1 · 10 ⁻⁴	1.1 · 10 ^{−3}
N ₂ O	6 · 10 ^{−5}	5 · 10 ⁻⁵	5 · 10 ⁻⁴
H ₂	5 · 10 ^{−6}	5 · 10 ⁻⁵	5 · 10 ⁻⁴
Xe	4 · 10 ^{−5}	8.7 · 10 ^{_6}	9 · 10 ^{−5}
0 ₃	9 · 10 ^{−6}	7 · 10 ^{−6}	7 · 10 ^{−5}
Ť	Σ 100 %	Σ 100 %	Σ 1013
50 % RH at 20 °C	1.6	1.15	11.7

Note: In the composition of atmospheric air the relative humidity (RH) is indicated separately along with the temperature. At the given relative humidity, therefore, the air pressure read on the barometer is 1024 mbar.



Units of pressure

	Ра (N m ⁻²)	mbar	Torr (mm Hg at 0 °C)	Technical Atmospheres (at)	Physical Atmospheres (atm)
Ра (N m ⁻²)	1	1.0 x 10 ⁻²	7.5 x 10 ⁻³	1.02 x 10 ⁻⁵	9.87 x 10 ^{.6}
mbar	1.0 x 10 ²	1	7.5 x 10 ⁻¹	1.02 x 10 ⁻³	9.87 x 10 ^{.4}
Torr (mm Hg at 0 °C)	1.33 x 10 ²	1.33	1	1.36 x 10 ⁻³	1.32 x 10 ⁻³
Technical Atmospheres (at)	9.80 x 10 ⁴	9.80 x 10 ²	7.36 x 10 ²	1	9.68 x 10 ⁻¹
Physical Atmospheres (atm)	1.01 x 10 ⁵	1.01 x 10 ²	7.60 x 10 ²	1.03	1

http://vacuumtech.blogspot.com/2009/06/in-international-system-of-unit-units.html



Sources of residual gas

Limiting factors

•High vacuum

- pumping speed
- leak
- Good High vacuum
 - desorption from walls
 - baking
- Ultra high vacuum
 - impurities
 - internal leaks
 - material selection
 - diffusion
 - permeation





Average mean free path (distance between collission) in nitrogen residual gas



Phases of residual gas

- d = diameter of chamber
- Viscotic <λ> < d/100
- Intermediate
- Molecular $\langle \lambda \rangle >> d$







Time to form one molecular layer on surface

average molecule mass m diameter of molecule

$$\tau = \frac{(2 * \pi * m * k * T)^{1/2}}{\zeta^2 * P}$$

ζ

15 vrk 21 min 1,3 s 1,3 ms 1,3 μs 1,3 ns



Use of High Vacuum

- Ultra High vac. UHV $\tau \sim \text{hour}$
 - Clean surface during slow experiment
 - Atomic clean surface
 - Ion accelerators
 - MBE-processes
 - Surface analysis
 - XPS, ESCA
 - SIMS

- Good High Vacuum $\tau \sim \min$
 - Sufficient for electron gun
 - Clean surfacea during slow process
 - Several thin film processes
 - Crystal growth
 - Electron microscopy
 - Mass spectroscopy
 - Lithography

- High vacuum $\tau \sim s$
 - Clean surface during process
 - Several thin film processes
 - Ion implantation



Critical temperatures for some residual gasses

	1	
Gas or vapor		<i>T</i> _c (°C)
Helium	He	268
Hydrogen	H ₂	
Nitrogen	N ₂	
Carbon monoxide	CO	
Argon	Ar	122
Oxygen	O ₂	
Methane	CH₄	
Carbon dioxide	CO ₂	31
Chlorine	Cl_2	144
Ether	$(C_2H_5)_2O$	195
Ethanol	C₂H₅OH	243
Carbon tetraclor.	CCl ₄	283
Water	H ₂ O	374
	1	

above T_c no liquid





Vacuum Pumping

• Mechanical pumping \rightarrow 0.1 Pa

 High Vacuum pumps with backing pump → HV, Good HV (VHV)



• High Vacuum closed \rightarrow UHV



From Jyrki Målarius

Vacuum

- Pump throughput Pa m³/s
- Pumping speed m³/s, l/s, m³/h

$$Q_p = p \frac{\Delta V}{\Delta t}$$
$$S = \frac{\Delta V}{\Delta t}$$
$$Q_p = Q_d + Q_v$$

• Final pressure







From Jyrki Målarius

• m³/s

• m³/h

• 1/s

Vacuum

Pumping speed and the ultimate pressure



Adsorption



Kuva 12.1. Lennard-Jones-diagrammi.



Adsorption

Physisorption

Chemical bonding:

polaroization (van der Waals)

Bonding energy ≈ 0.001 – 0.5 eV
Bond length ≈ 3 – 10 Å
For example: nobel gas or molecules on materials
Possibly precursion state before chemisorption





Kuva 12.1. Lennard-Jones-diagrammi.

Adsorption

Chemisorption

Chemical bonding:

charge exchange

Bonding energy ≈ 0.5 – 5 eV
Bond length ≈ 1 – 3 Å
For example: H, O, N, CO on metals
Dissociation of molecule
Final absorption





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Kuva 12.1. Lennard-Jones-diagrammi.

Desorption

Adsorbed molecule must receive energy E_D in order to leave surface

thermal

radiation

- photons
- electrons
- ions
- electric field



2. Excitation of the substrate-adsorbate complex



Vacuum pumps

Positive displacement (mechanical pumps)

Momentum transfer (molecular pumps)

• Entrapment







Mechanical pumps





Very common fore vacuum-and general vacuum pump.

- •Typically 1 or 2 stage configuration.
- •Gas is moved by rotating vanes.
- •Oil is used as seal, lubricant, and coolant.

+ High capacity from 10³ to ~10⁻²mbar.
- Potential back streaming of oil into vacuum chamber.



Mechanical pumps



Counter rotating blades moves gas volume.

•No contact between surfaces \rightarrow oil free operation.

Roots



ZJP-1200C

•Runs very hot without fore vacuum pump.

+ High capacity from 10 to ~10⁻⁴ mbar.
(Medium capacity from 1000 to ~10 mbar)
+ Oil free

- Works best together with fore vacuum pump.



Mechanical pumps



Moving scroll orbiting a fixed scroll.

•Compressed gas volume pushed towards center outlet.

+ Oil free

- + Reliable, low maintenance.
- Low to medium capacity (10³to ~10⁻²mbar)

Scroll pump





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Momentum transfer - Turbo pump



Turbo molecular



•Fast moving rotor (30k to 90k rpm) with several stages and many blades per stage.

•High efficiency in the molecular regime where gas molecules collide with rotor blade and not each other.

•Some modern pumps have magnetic, non-contact, bearings.

- + High capacity from 10⁻³to ~10⁻⁸ mbar.
- + Low maintainance.
- Sudden large gas loads may cause severe, expensive damage.



Momentum transfer in turbo molecular pump





Momentum transfer - oil diffusion pump



•Hot dense oil vapor is forced through central jets angled downward to give a conical curtain of vapor.

•Gas molecules are knocked downwards and eventually reach the backing vacuum pump.

- + Simple pump without moving parts.
- + High capacity from 10⁻³ to ~10⁻⁸ mbar.
- + Low maintenance.
- Needs cooled baffle to reduce oil contamination of vacuum chamber.

https://vacaero.com/information-resources/vac-aero-training/170466-the-fundamentals-of-vacuum-theory.html



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Entrapment



cryo pump



Cool head with several plates (stages).

The metal top side of the cool (12K) plates traps gas molecules by cryocondensation.

The bottom side of the plates are coated with active charcoal and traps gas molecules by cryo-adsorption.

The cooling is done with a Helium filled refrigerator loop.

- + Very High capacity down to ~10⁻⁹ mbar.
- + No backflow contamination.
- Pump saturates if exposed to high pressure or continuous gas flow.
- Need periodic regeneration of cool head.



Entrapment



Free electrons move in helical trajectories towards the anode, ionizing gas molecules upon collisions.

•Gas ions strike the Ti cathodes and some get buried.

•Sputtered Ti deposits inside the tubes and getters gas molecules through chemical reactions.

- + Simple pump without moving parts.
- + Can work at very low pressure ~10⁻¹¹mbar.
- + Oil free.
- Not suitable for gas loads.



Pumps and vacuum ranges



Fig. 9.16: Common working ranges of vacuum pumps

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Vacuum gauges

- Mechanical diaphragm
- Electronic
 - Piezoresitive (strain gauge)
 - Capacitive
 - Magnetic
 - Piezoelectric
 - Optical
 - Potentiometric
 - Resonant
- Thermal conductivity Pirani
- Ionzation gauge
- Hot cathode
- Cold cathode (Penning)





Gauges







ionization gauge hot filament



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Residual gas analyser









Figure 3-1 cont. A quadrapole mass spectrometer.

https://vacaero.com/information-resources/vac-aero-training

Residual gas analyser



https://vacaero.com/information-resources/vac-aero-training/6884-residual-gas-analyzers.html



Vacuum systems





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Figure 3-8. Vacuum/plasma processing system.

Handbook of Physical Vapor Deposition (PVD) Processing







in parallel:

$$C_{\rm sys} = C_1 + C_2 + C_3 + \cdots$$



Conductance of various geometries

M. Ohring

(A)
$$C = 3.64A \left(\frac{T}{M}\right)^{1/2} = 11.7A$$

(D) D (A) $C = 6.18 \frac{A^2}{DL} \left(\frac{T}{M}\right)^{1/2} = 12.2 \frac{D^3}{L}$

rersity Chemical 3y

At room temperature									
Standard values1 (mbar · I · s ⁻¹ · cm ⁻	-2)		Metal 10 ⁻⁹ ·	s 10 ⁻⁷			Nonmet 10 ⁻⁷ ·	tals 10 ^{−5}	
Outgassing rates (standard values) as a function of time									
Examples: Ag Al Cu Stainless steel ¹ All values depend	1/2 hr. 1.5 · 10 ⁻⁸ 2 · 10 ⁻⁸ 4 · 10 ⁻⁸	1 hr. 1.1 · 10 ⁻⁸ 6 · 10 ⁻⁹ 2 · 10 ⁻⁸ 9 · 10 ⁻⁸ etreatment!	3 hr. 2 · 10 ^{−9} 6 · 10 ^{−9} 3.5 · 10 ^{−8}	5 hr. 3.5 · 10 ⁻⁹ 2.5 · 10 ⁻⁸	Examples: Silicone NBR Acrylic glass FPM, FKM	1/2 hr. 1.5 · 10 ⁻⁵ 4 · 10 ⁻⁶ 1.5 · 10 ⁻⁶ 7 · 10 ⁻⁷	1 hr. 8 · 10 ⁻⁶ 3 · 10 ⁻⁶ 1.2 · 10 ⁻⁶ 4 · 10 ⁻⁷	3 hr. 3.5 · 10 ⁻⁶ 1.5 · 10 ⁻⁶ 8 · 10 ⁻⁷ 2 · 10 ⁻⁷	5 hr. 1.5 · 10 ⁻⁶ 1 · 10 ⁻⁶ 5 · 10 ⁻⁷ 1.5 · 10 ⁻⁷

Table X: Outgassing rate of materials in mbar \cdot I \cdot s^{-1} \cdot cm^{-2}



Table 3.2. Pressure (in Pascal and Torr), impingement rate, and monolayer formation time for selected vacuum and process conditions

<i>p</i> (Pa)	p (Torr)	$J_{\rm g}~({\rm m}^{-2}~{\rm s}^{-1})$	τ (s)
1 10 ⁻¹ 10 ⁻²	7.5×10^{-3} 7.5×10^{-4} 7.5×10^{-5}	Nitrogen 2.9×10^{22} 2.9×10^{21} 2.9×10^{20}	3.5×10^{-4} 3.5×10^{-3} 3.5×10^{-2}
10 ⁻³ 10 ⁻⁴ 10 ⁻⁵	7.5×10^{-6} 7.5×10^{-7} 7.5×10^{-8}	water vapor 3.6×10^{19} 3.6×10^{18} 3.6×10^{17}	0.28 2.8 28



Vacuum Baking

- In order to obtain UHV
- heated to high temperatures (100 300 ° C or so)
- mostly water is adsorbed to the chamber walls
- very long time at room temperature.

- Electrical heating tapes (shown in the picture below).
- Then everything is covered with aluminium foil for insulation and heat distribution.

http://philiphofmann.net/ultrahighvacuum/ind_bakeout.html



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Basics 5:Outgassing Resources

The outgassing table below is a bit dated but still useful as an introduction. Check the N.A.S.A. link below for a really large data base on the subject.

	Outgass	ing Data Tab	ole			
Outgassing Rate in Torr Liters Per Square Cm. Per S						
Material	Condition	1 Hour	10 Hours	100 Hours	Source	
Aluminum	Cleaned	_	8X10e-09	_	1	
Aluminum	Anodized	_	1X10e-07	_	1	
Aluminum	Anodized	_	1X10e-07	_	<u>3</u>	
Aluminum	degassed	1.7X10e-07	2.7X10e-08	4.6X10e-09	4	
Aluminum	none	1.3X10e-06	_	_	<u>4</u>	
Aluminum 6061-T6	none	_	2.5X10e-09	_	5	
Aluminum 6061-T6 @ 200 deg. C	hot	_	4.5X10e-09	_	5	
Aluminum 6061-T6	Bake 13.5 hr. @ 200 deg. C	_	3.7X10e-10	_	5	
Aluminum 6061-T6 @ 300 deg. C	hot	_	1.4X10e-08	_	5	
Aluminum 6061-T6	Bake 15 hr. @ 300 deg. C	_	1.6X10e-10	_	5	
Brass	Cast, cleaned	_	3X10e-07	_	1	
Copper	_	2.3X10e-06	_	_	3	
Copper, 450 Deg.C	None	1.6X10e-06	_	_	<u>4</u>	
Copper, 450 Deg.C	degreased, pickled	2.6X10e-07	_	_	4	
Copper, 450 Deg.C	degreased	1.4X10e-06	_	_	4	
Molybdenum	_	7X10e-07	_	_	3	
Nickel	_	6X10e-07	_	_	3	
Silver	_	6X10e-07	_	_	3	
Silver	_	6X10e-07	_	_	3	





Basics 5:Outgassing Resources

The outgassing table below is a bit dated but still useful as an introduction. Check the N.A.S.A. link below for a really large data base on the subject.

	Outgassi	ng Data Tab	ole		
		Outgassing Rate	Outgassing Rate in Torr Liters Per Square Cm. Per Second		
Material	Condition	1 Hour	10 Hours	100 Hours	Source
Steel, Mild	Shot-blasted		6X10e-08	_	1
Steel, mild	_	5X10e-07	5X10e-08	_	3
Steel, mild	degassed	5.3X10e-08	1X10e-08	1.9X10e-09	4
Steel, mild	none	_	1.9X10e-9	4X10e-10	5
Steel, mild @ 200 deg. C	hot		8.6X10e-9	_	5
Steel, mild	baked 15 hrs. @ 200 deg. C	_		4.3X10e-11	5
Steel, mild @ 400 deg. C	hot	_	8.4X10e-9		5
Steel, mild	Baked 15 hrs. @ 400 deg. C	_	1.2X10e-11	_	5
Steel, mild	none	4.2X10e-07	_	_	4
Steel, mild	none	4.2X10e-07		_	4
Steel, chrome plated	Polished & vapour degreased	1X10e-08	9X10e-10	_	3
Steel, nickel plated	Polished & vapour degreased	5X10e-07	1X10e-09	_	3
Steel, stainless		2X10e-07	2X10e-08	_	1
Steel, stainless	Polished & vapour degreased	_	1.4X10e-09	_	3
Steel, stainless	none	6.4X10e-07		_	4
Steel, stainless	degreased	4X10e-07			4
Steel, stainless	annealed	5.3X10e-08	_	_	4
Steel, stainless	none	7.6X10e-10	_	1.1X10e-10	5
Steel, stainless	none	_	1.2X10e-08	_	5
Steel, stainless	bake 24 hr, 200 deg. C	_	1.5X10e-10		5
Steel, stainless	bake 12 hr., 400 deg. C	_	9.3X10e-13	_	5
Steel, stainless @ 400 deg. C	hot	_	1.4X10e-09	_	5
Tantalum	_	9X10e-07	_	_	3
Tungsten	_	2X10e-07	_	_	3





Basics 5:Outgassing Resources

The outgassing table below is a bit dated but still useful as an introduction. Check the N.A.S.A. link below for a really large data base on the subject.

Outgassing Data Table						
		Outgassing Rate in Torr Liters Per Square Cm. Per Second				
Material	Condition	1 Hour	10 Hours	100 Hours	Source	
"Araldite D"	_	_	1X10e-06	3X10e-07	1	
Neoprene	_	3X10e-05	1.5X10e-05	_	<u>1</u>	
PVC	_	_	8X10e-07	1.3X10e-07	1	
Mylar	outgassed	2X10e-07	_	_	2	
Neoprene	As received	2X10e-04	_	_	2	
Silicone rubber	As received	3X10e-05	_	_	2	
Teflon	As received	5X10e-06	_	_	2	
PVC	As received	9X10e-07	_	_	<u>2</u>	
Textolite	As received	7X10e-06	_	_	<u>2</u>	
Mylar	As received	3X10e-06	_	_	2	
Zirconium	_	1.3X10e-06	_	_	3	
Butyl rubber	_	1.5X10e-06	_	_	3	
Kel F	_	4X10e-08	_	_	3	
Plexiglass	Outgassed	1X10e-06	_	_	3	
Polyethylene	_	2.6X10e-07	_	_	3	
Nylon	_	1.2X10e-05	_	_	3	
Porcelain	Glazed	6.5X10e-07	_	_	<u>3</u>	
Steatite	_	9eX10e-08	_	_	<u>3</u>	
Epon 828	degassed	6.7X10e-07	5.9X10e-08	9.4X10e-09	4	
Teflon	degassed	4.6eX10e-07	2.1X10e-07	9X10e-09	<u>4</u>	



Oxygen contamination



Figure 4.3. Variation in the as-deposited sheet resistance of cosputtered Ta-Si deposits (on SiO_2) as a function of Si:Ta atomic ratio for several oxygen concentration ranges. Oxygen was incorporated as a contaminant during sputtering.



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Case NiTi

- Nickel Titanium shape memory alloy
- MEMS devises
- Based on reversible austenitemartensite transformation, which is temperature driven
- Transformation temperature depends on stoichimetry
- oxygen reacts with Ti forming TiO_x, which changes Ni/Ti ratio in alloy



Thin Solid Films 370 (2000) 18-29



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Case NiTi

- RGA control in UHV vacuum
- Before baking H₂, H₂O, CO₂ and CO
- By baking H₂O, CO₂ and CO gases were kept below 10⁻⁸ Torr
- Sputtering with Ar partial pressure of 2 mtorr during film growth
- Stoichimetry and transition temperature as in bulk NiTi
- without careful ambient control transition temperature changes radically. (Oxygen detection al low contents difficult in metallic thin films)





Vacuum systems and sample loading





Handbook of Physical Vapor Deposition (PVD) Processing



Planar Load Lock Assemblies



ENLARGE



- Connected to main chamber via gate valve
- transferring samples
- its volume can be pumped and vented without disturbing the main chamber pressure
- chamber clean from water vapor or other contaminant's
- increased sample throughput.
- Components
- viewport
- o-ringed door
- ports for pumping and gauging





Sputter cleaning effect



Figure 3.5. Schematic depiction of the energetic particle bombardment effects on surfaces and growing films.



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⁵³ Sputter cleaning using oxgen plasma or Ar-plasma





Self-sputtering of Ni ions





7. Rate of deposition or etching as a function of bias voltage for aluminum arc (Adapted from [67])



Self sputtering of selected metals Yield of self-sputtering



Fig. 8.16. Self-sputtering yield for selected metals as a function of ion energy (calculated by T-DYN Monte Carlo code; the apparent scatter is due to the statistics). The energy scale of up to 4 keV is quite appropriate considering the typical bias of 1 kV and the presence of multiply charged ions



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8700 CONTINUOUS FEED LOAD LOCK

Isolated Chamber Layout and Backside/Edge Wafer Transfer System



275-B7



A

⁵ 7 In-situ fabrication and characterization



Figure 8.11. Top view of special dual-chamber system for in-situ fabrication and characterization. Courtesy of A. Kaloyeros, SUNY, Albany, NY.



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Figure 8.12. Schematic representations of multichamber integrated processing with chambers for substrate cleaning, remote PECVD deposition, and analysis (AES and RHEED or LEED). Both systems provide for substrate introduction into load-lock chambers. From Lucovsky et al [21].



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⁵ Schematic of in-situ UHV processing system



Figure 8.13. A schematic drawing of possible all-UHV in-situ processing system.



6 0 MBE system



Figure 8.14. A typical sophisticated MBE system.



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Vacuum system design

- Access—how large and heavy are the parts and fixturing?
- Do the parts need to have *in-situ* processing? e.g. outgassing, heating, plasma treatments, etc.
- System cleaning—is there a lot of debris generated in the process? Does the debris fall into critical areas such as valve sealing surfaces? How often will system cleaning be necessary?
- Cycle time for the system—production rate.
- How often do fixtures and tooling need to be changed?
- Is the processing sensitive to the processing environment?
- Sophistication of the operators—operator training.
- Maintenance.
- Safety aspects—high voltage, interlocks.
- Fail safe design—short or long power outages, water failure.
- Environmental concerns exhaust to the atmosphere, traps.

