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FKA195: Nanoscale Science & Technology: Thin Films & Materials.

Lab Demo
Physical Evaporation of Metals

1. Introduction.

Physical Vapor Deposition (PVD) employ the atomic vapor cloud formed by the vaporization of metal in a vacuum environment to cover all the surfaces along the straight line between the source and substrate.

PVD involves the following sequence of steps:

1) the material is converted into vapor by heating;
2) the vapor is transported from its source to the substrate in a vacuum; and
3) the vapor condense on the substrate and form the thin film.

The *advantages* of PVD by evaporation are:

1) high purity of the thin film thanks to the high-vacuum environment;
2) no substrate damage from impinging atoms during the thin-film formation (unlike sputtering that produces some damage because of high-energy particles);
3) high deposition rates;
4) relatively small substrate heating.

Heating of the material in PVD can be done in several ways. The simplest one is the resistive heating, when a wire of tungsten or molybdenum is resistively heated, so that the metal being in thermal contact to the wire melts and evaporates (sublimation can occur as well).

In electron beam evaporation, a high-energy beam of electrons is directed at the material. The electron energy is converted into thermal energy, thus heating up and evaporating the material. Heating of the material can also be done with RF energy (inductive heating).

Uniformity

- Lower deposition rate gives better uniformity, but increases risk for contamination in low-vacuum chambers.
- Larger distance from the source to the substrate improves uniformity, but also lowers the deposition rate.
- Rotating holder improves uniformity

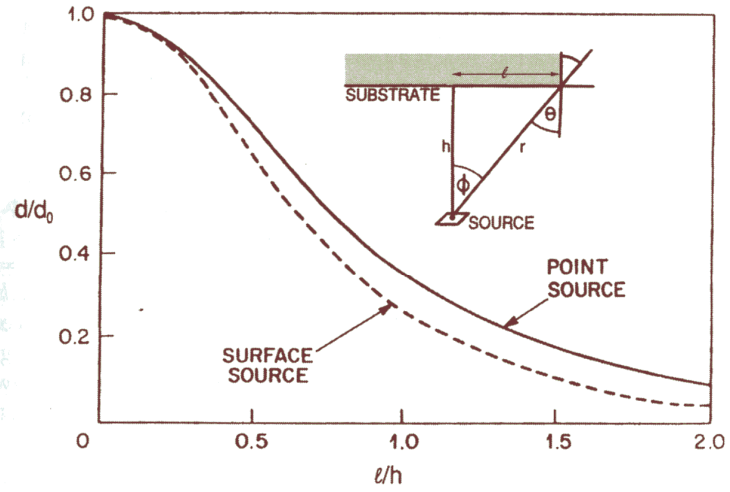


Figure 3-6 Film thickness uniformity for point and surface sources. (Insert) Geometry of evaporation onto parallel plane substrate.

(from Ref.1)

$$\frac{d}{d_0}=\left(1+\left(\frac{l}{h}\right)^2\right)^{-1.5}\text{ (point source), and }\frac{d}{d_0}=\left(1+\left(\frac{l}{h}\right)^2\right)^{-2}\text{ (surface source)}$$

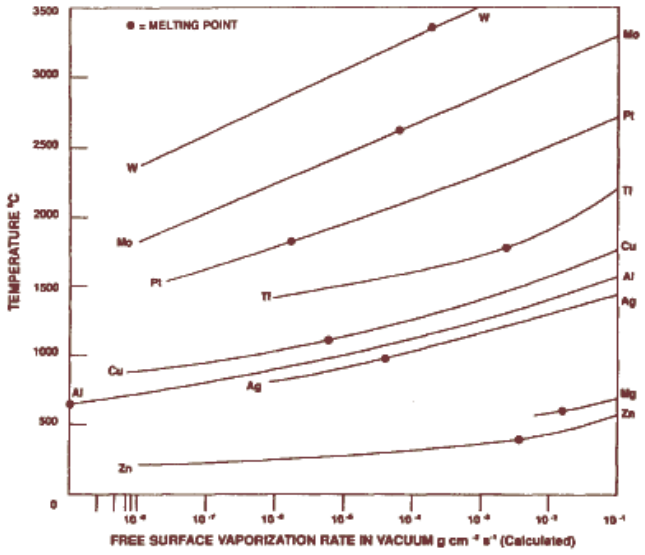
Deposition rate

- The deposition rate depends on the position and orientation of the substrate in the chamber.
- The evaporation rate Γ_e is the rate at which a material vaporizes. It can be calculated according to the equation :

  $\Gamma_e\left[\frac{g}{cm^2 s}\right]\approx 0.06\sqrt{\frac{M}{T}}P_v(T)[torr]$, where M is the molecular mass, P_v is the vapor pressure, and T is the temperature.

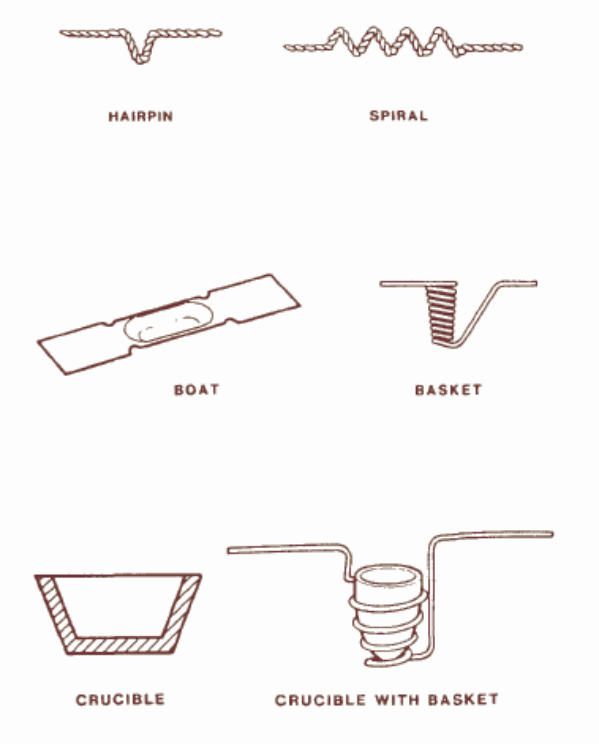
  A reasonable deposition rate (0.1 mg/cm^2 s) requires a vapor pressure above 10^{-2} torr. Refractory metals, such as W, need temperatures in excess of 3000 °C to reach such a vapor pressure. Other metals, for instance Au, Ag, Cu, In, etc. require much less heating.

Some examples:



(from Ref. 2)

Sources



Step Coverage

- Surfaces perpendicular to the view line connecting substrate and the source are not coated for evaporation PVD.
- Step coverage can be improved by tilting and rotation of the substrate.
- For some processes (lift-off), step coverage should be avoided.
- Sputtering will in general have better step coverage (for aspect ratios < 0.5: the aspect ratio is the ratio of the step height to its width) as sputtered atoms have random velocities.

Thickness monitor

1) Quartz crystals are used to measure deposition rates. The quartz crystal mechanically oscillates when ac voltage is applied to it (thanks to piezoelectric effect[1]). The resonance frequency of oscillations is dependent on the mass of the film deposited onto it. Quartz monitors can measure thickness of about a single atomic layer with the relatively high accuracy.
2) Quartz deposition meter must be programmed for each material it is used to measure (density, Z-ratio, and tooling factor)

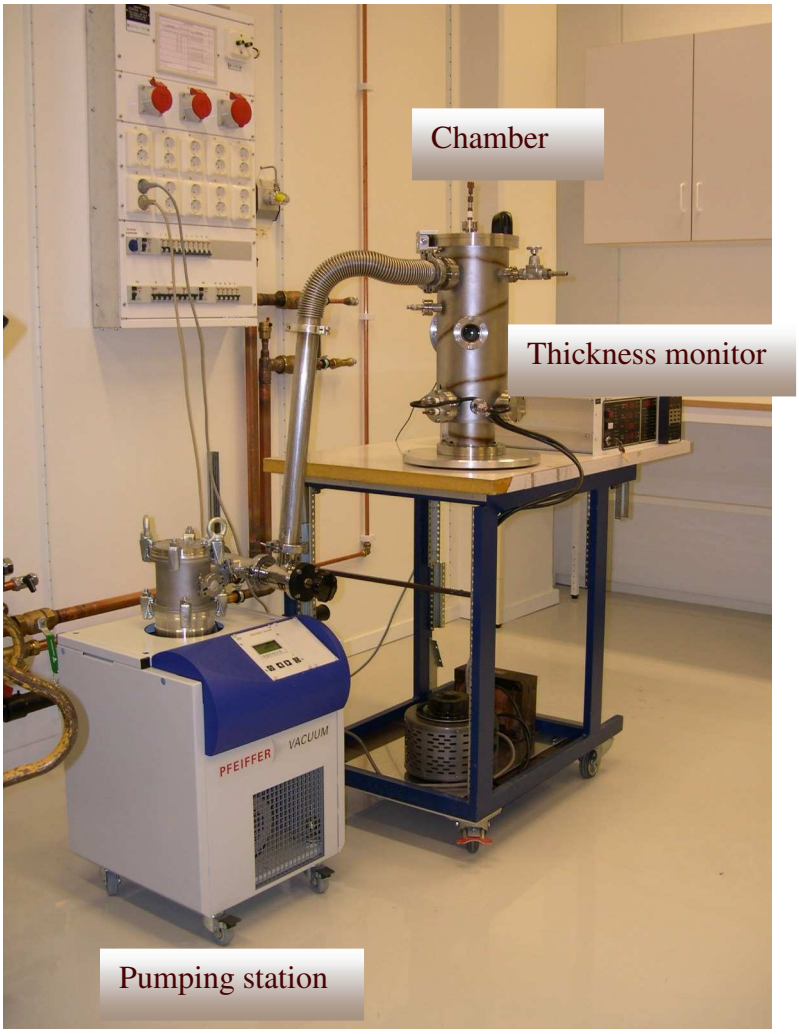
    - The *tooling factor* (TF) is used to calibrate the thickness meter to account for its position in the evaporation chamber (which is different from the position of a substrate)
    - The Z-ratio is a parameter that corrects the frequency-change-to-thickness transfer function for the effects of acoustic-impedance mismatch between the crystal and the deposited material. A table of densities and acoustic-impedances for common materials can be found below.

3) The quartz crystal must be replaced periodically for reliable results. Crystal life is dependent on the total amount of material deposited on it and also on the radiation heat from the source (forced cooling should be used in some cases).

Front panel of the thickness monitor used in the lab demo.

[1] In resonance, the amplitude of piezoelectric displacement dramatically increases.

2. The deposition system



3. Evaporation of a metal (Cu, In, or Ag)

In this lab demonstration there will be a Cu thin film deposited on a glass/Si substrate. The processing includes the following steps:

- Find all parts of the deposition system described in the previous chapter.
- Open the chamber, find the quartz crystal, the boat, and the mirror.
- Load metal-pieces in the boat (if there is not enough material in it).
- Mount substrates on the chamber cover using double-sticking tape.
- Close the cover. Make sure that the "vent" valve is closed, too.
- Start the pumping station. The pumping will take about 1-1.5 hour.
- Meanwhile, program the thickness monitor for the chosen material (Cu). Find the corresponding parameters in the table shown in Appendix 1.
- Plug in the mains connector of the variable transformer, making first sure that it is set to zero.

- By slowly rotating CW the handle of the transformer (less than 20% of the full turn), see how the source starts to glow.
- Increase the power further to achieve some reasonable deposition rate (of about 5 Å/s). Open the shutter and simultaneously set the thickness readings to zero.

- Wait until a required thickness will be achieved and close the shutter immediately after that.
- Turn CCW the handle of the transformer to zero. Wait a couple of minutes until the source cools down. Close pumping and open slowly the vent valve.
- Open cover and pick your samples. That's it.
- The samples will be patterned later on during the photolithography-lab exercise

Various qualitative questions may be asked during the demonstration, based on the lectures studied so far (PVD), the corresponding section of the M. Ohring's book (Chapter 3), and this PM, like:

- How would the tooling factor change if we moved the quartz-crystal head of the thickness monitor closer to the source?
- How could we estimate the thickness of the deposited thin film without using the quartz-crystal monitor?
- How would the uniformity of the deposited thin film change if we moved the substrate closer to the source? If we rotate the substrate around central axis of the system? Around the substrates central point?
- How does the deposition rate depend on the temperature of the boat? On the vacuum in the system? On the temperature of the substrate?
- Looking at the system, what should be done to improve vacuum?

References

1. *Materials Science of Thin films. Deposition and Structure* by M. Ohring (Academic Press © 2002.
2. *Handbook of Physical Vapor Deposition (PVD) Processing* by D.M. Mattox, (William Andrew Publishing/Noyes © 1998) ISBN: 0-8155-1422-0

Appendix

Acoustic impedance. Z-ratio.

The **acoustic impedance** (**Z**) of a material is defined as the product of density (**p**) and acoustic velocity (**V**) of that material.

$$Z = pV$$

Acoustic impedance is important in

1. the determination of acoustic transmission and reflection at the boundary of two materials having different acoustic impedance
2. the design of ultrasonic transducers.
3. assessing absorption of sound in a medium.

The Z-ratio is the ratio of the acoustic impedance of quartz (**$8.834 \cdot 10^5$ g/cm^2 s**) to that of the selected material (see the table below)

Bulk Densities and Acoustic Impedances for Common Materials

Material Name	Symbol	Density g/cc	Acoustic Impedance 10^5 g cm^{-2} s^{-1}
Aluminium	Al	2.70	8.17
Aluminium Oxide	Al_2O_3	3.97	- - -
Antimony	Sb	6.62	11.49
Arsenic	As	5.73	9.14
Barium	Ba	3.50	4.20
Berryllium	Be	1.85	16.25
Bismuth	Bi	9.80	11.18
Bismuth Oxide	Bi_2O_3	8.90	- - -
Boron	B	2.54	22.69
Cadmium	Cd	8.64	12.94
Cadmium Selenide	CdSe	5.81	- - -
Cadmium Sulphide	CdS	4.83	8.66
Cadmium Telluride	CdTe	5.85	9.00
Calcium	Ca	1.55	3.30
Calcium Fluoride	CaF_2	3.18	11.39
Carbon (Graphite)	C	2.25	2.71
Cerium (III) Fluoride	CeF_3	6.16	- - -
Cerium (IV) Oxide	CeO_2	7.13	- - -
Chromium	Cr	7.20	28.94
Chromium (III) Oxide	Cr_2O_3	5.21	- - -
Cobalt	Co	8.71	25.73
Copper	Cu	8.93	20.20
Copper(I) Sulphide(Alpha)	Cu_2S(Alpha)	5.60	12.80
Copper (I) Sulphide (Beta)	Cu_2S(Beta)	5.80	13.18
Copper (II) Sulphide	CuS	4.60	10.77
Cryolite	Na_3AlF_6	- - -	- - -
Erbium	Er	9.05	11.93
Gadolinium	Gd	7.89	13.18
Gallium	Ga	5.93	14.88
Gallium Arsenide	GaAs	5.31	5.55
Germanium	Ge	5.35	17.10
Gold	Au	19.30	23.17
Hafnium	Hf	13.09	24.53
Hafnium Oxide	HfO_2	9.63	- - -
Indium	In	7.30	10.49
Indium Antimonide	InSb	5.76	10.98
Indium Oxide	In_2O_3	7.18	- - -
Iridium	Ir	22.4	68.40
Iron	Fe	7.86	25.29
Lanthanum	La	6.17	9.60
Lanthanum Fluoride	LaF_3	5.94	- - -
Lanthanum Oxide	La_2O_3	6.51	- - -
Lead	Pb	11.3	7.81
Lead Sulphide	PbS	7.50	15.59
Lithium	Li	0.53	1.50
Lithium Fluoride	LiF	2.64	11.40
Magnesium	Mg	1.74	11.41
Magnesium Fluoride	MgF_2	3.00	- - -
Magnesium Oxide	MgO	3.58	21.47
Manganese	Mn	7.20	23.41

Material Name	Symbol	Density g/cc	Acoustic Impedance 10^5 g cm^{-2} s^{-1}
Manganese (II) Sulphide	MnS	3.99	9.39
Mercury	Hg	13.46	11.93
Molybdenum	Mo	10.20	34.34
Neodynium Fluoride	NdF_3	6.51	- - -
Neodynium Oxide	Nd_20_3	7.24	- - -
Nickel	Ni	8.91	26.66
Niobium	Nb	8.57	17.90
Niobium Pent Oxide	Nb_20_5	4.47	- - -
Palladium	Pd	12.0	24.72
Platinum	Pt	21.4	36.06
Potassium Chloride	KC1	1.98	4.30
Rhenium	Re	21.04	58.87
Rhodium	Rh	12.41	42.05
Rubidium	Rb	1.53	3.48
Samarium	Sm	7.54	9.92
Scandium	Sc	3.00	9.70
Selenium	Se	4.82	10.22
Silicon	Si	2.32	12.39
Silicon Monoxide	Si0	2.13	10.15
Silicon Dioxide	$Si0_2$	2.20	8.83
Silver	Ag	10.5	16.68
Silver Bromide	AgBr	6.47	7.48
Silver Chloride	AgCl	5.56	6.68
Sodium	Na	0.97	1.84
Sodium Chloride	NaCl	2.17	5.62
Sulphur	S	2.07	3.85
Tantalum	Ta	16.6	33.70
Tantalum (V) Oxide	Ta_20_5	8.20	29.43
Tellurium	Te	6.25	9.80
Terbium	Tb	8.27	13.38
Thallium	Tl	11.85	5.70
Thorium(IV)Fluoride	ThF_4	6.32	- - -
Tin	Sn	7.30	12.19
Titanium	Ti	4.50	14.05
Titanium (IV) Oxide	$Ti0_2$	4.26	22.07
Titanium Oxide	Ti0	4.9	- - -
Tungsten	W	19.3	54.14
Tungsten Carbide	WC	15.6	58.44
Uranium	U	18.7	37.08
Vanadium	V	5.96	16.65
Ytterbium	Yb	6.95	7.81
Yttrium	Y	4.34	10.57
Yttrium Oxide	Y_20_3	5.01	- - -
Zinc	Zn	7.04	17.17
Zinc Oxide	Zn0	5.61	15.87
Zinc Selenide	ZnSe	5.26	12.22
Zinc Sulphide	ZnS	4.09	11.39
Zirconium	Zr	6.51	14.72
Zirconium Oxide	$Zr0_2$	5.60	- - -

(the table is taken from the MODEL IL150 THICKNESS MONITOR INSTRUCTION MANUAL, see http://www.intellemetrics.com/15041.pdf)