1. Introduction

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Outline - chapter 1. Introduction

- 1.1 Fields of Expertise, Suggested Literature
- 1.2 Overview of Material Processing
- 1.3 Introduction to Thin Film Deposition
- 1.4 Applications of Thin Films
- 1.5 Fabrication of microstructures/microdevices
1.1 Fields of Expertise, Suggested Literature
What Expertise is Necessary?

Material processing requires knowledge of processes:
- **gas kinetics** (for processes from vapor/gas phase)
- **film growth** (general views like adsorption, desorption, utilization etc.)
- **interaction of ions with solid** (for ion beam and plasma techniques)
- **chemistry** (for chemical and plasmachemical methods)
- **plasma-related phenomena**, i.e. plasma physics, principles of electrical discharges, elementary processes in plasma, plasma-surface interaction

The processes often takes places at decreased pressure. Therefore, a knowledge of **vacuum technology** is also required.

This information are then applied to master the **material processing techniques**:
- **etching** (physical sputtering, chemical etching, plasma etching)
- **vacuum evaporation** for thin film deposition
- **magnetron sputtering** for thin film deposition
- **chemical vapor deposition** (CVD)
- **plasma enhanced chemical vapor deposition** (PECVD)
- etc.
Handbooks of Technologies

▶ Handbook of Thin-Film Deposition Processes and Techniques, ed. K. K. Schuegraf, Noyes Publications 1988
▶ Handbook of Plasma Immersion Ion Implantation and Deposition, Wiley 2000
▶ Handbook of Thin Film Deposition Techniques (Materials and Processing Technology), by Krishna Seshan, (Noyes Publications 2002)
▶ Handbook of Nanotechnology (Springer 2010), B. Bushan
Books Focused on Specific Processes and Technologies

- Thin-Film Deposition, Principles and Practice by Donald L. Smith, McGraw-Hill, 1995
- Chemical reactor, analysis and design, G. F. Froment and K. B. Bischoff, John Wiley 1990
- Principles of plasma discharges and materials processing, M. A. Lieberman and A. J. Lichtenberg, John Wiley 1994
- Lecture notes on principles of plasma processing, F. F. Chen and J. P. Chang, Kluwer Academic 2003
Books focused on Specific Materials

- The Science and Technology of Carbon Nanotubes, K. Tanaka, T. Yamabe, F. Fukui eds., Elsevier 1999
- Nanostructures & Nanomaterials: Synthesis, Properties & Applications by Guozhong Cao, Imperial College Press, 2004
There are several electronic information resources http://knihovna.sci.muni.cz/:

- Databases of scientific publications that collect information independently on the publisher and often contain links to full texts
  - Web of Science
  - Scopus
  - INSPEC

- Databases of scientific publications from given publisher - always connected with full texts but the download must not be for free (depends on the institutional domain, e.g., sci.muni.cz), some journals are “open access” (authors pay for the publication)
  - Science Direct
  - IOPscience
  - PROLA
Related courses

- F4160 Vakuová fyzika 1 (jarní semestr)
- F6450 Vakuová fyzika 2 (podzimní semestr)
- F5170 Úvod do fyziky plazmatu (podzimní semestr)
- F7241 Fyzika plazmatu 1 (podzimní semestr)
- F7360 Charakterizace povrchů a tenkých vrstev (jarní semestr 2018)
- FB100 Plazmochemické procesy
- F3390 Výroba mikro a nanostruktur (jarní semestr)
- FC250 Nano- a mikrotechnologie (jarní semestr 2017)
- atd.
1.2 Overview of Material Processing
Surface Treatment

What can happen after surface treatment?

- change of surface roughness
- change of surface chemistry

What can be these changes used for?

- change of surface free energy, i.e. wettability
- improved adhesion of further coatings
- immobilization of biomolecules

Preparation of Films

Difference between thin-film and thick-film technology:

- **thin-film technology**: deposition of individual molecules, film thickness 10 nm–10 µm
- **厚-film technology**: involves deposition of particles (e.g. painting, silk screening, spin-on-glass coating, plasma spraying)

Plasmachemical methods compete with several other approaches on the field of thin film deposition and synthesis of nanostructures

Several aspects have to be taken into account:

- functional properties of the deposition
- uniformity of the processes
- step coverage
- conformality
- reproducibility
- simplicity
- price
- etc.
Etching/Sputtering Processes

ion sputtering
★ purely physical approach, removal by energy transfer
★ slow process, no selectivity
★ ions are directed by electric field, i.e. anisotropic process

chemical etching
★ purely chemical processes that requires aggressive chemicals and/or elevated temperature for reaction activation
★ can be very fast, selective
★ chemical reactions with surface are not directed, i.e. isotropic process

plasma etching
★ combination of physical and chemical approaches
★ directional process
Unique Features of Plasma Technologies

- dry process, i.e. with low consumption of chemicals,
- offering replacement of toxic and explosive reactants
- environmentally friendly
- preparation of new materials

Why? **Plasma** of laboratory electrical discharge provides **environment of**

- **hot electrons** \( T \approx 10000 \text{ K} \) \( \Rightarrow \) dissociation of molecules into reactive species

- **positive ions** that can be accelerated by \( \approx 100 \text{ eV} \) near solid surface \( \Rightarrow \) sputtering of targets, implantation, modification of surfaces and growing films

- **cold neutral gas** \( \Rightarrow \) highly energetic process can be kept in a vessel, heat sensitive materials can be treated (e.g. polymers, even polymer nanofibers)
Plasma Processing Methods

**Plasma etching**
anisotropic dry etching: combination of chemistry and effect of ions (reactive ion etching)

**Plasma treatment** in O2, NH3, CF4 . . . creation of surface chemical group

**Plasma deposition of thin films**
plasma enhanced chemical vapor deposition (PECVD)

**physical vapor deposition** (PVD) - dc diode sputtering, magnetron sputtering
Plasma reactors can also look very differently, like plastic boxes :-)

Oxford Instruments, PlasmaPro 100
- **reactive ion etching**

Oxford Instruments, NanoFab
- **high T (plasma enhanced) chemical vapor deposition** for deposition of carbon nanomaterials and other 2D materials
1.3 Introduction to Thin Film Deposition
Thin-Film Deposition Process Steps

All thin-film processes contain the four (or five) sequential steps.

1. A source of film material is provided.
   Solid, liquid, vapor or gas source. Solid materials need to be vaporized (by heat or energetic beam of electrons, photons, i.e. laser ablation, or positive ions, i.e. sputtering) - physical vapor deposition (PVD). The methods using gases, evaporating liquids or chemically gasified solids are chemical vapor deposition (CVD) methods.

2. The material is transported to the substrate.
   The major issue is uniformity of arrival rate over the substrate area. Transport in a high vacuum = straight travelling lines → importance of geometry. Transport in a (gaseous) fluid = many collisions → gas flow patterns, diffusion of source molecules through other gases present.

3. The film is deposited onto the substrate surface.
   It is influenced by source and transport factors and the conditions at the deposition surface. Three principal surface factors: (i) surface condition (roughness, contamination, degree of chemical bonding with the arriving materials and crystallographic parameters in the case of epitaxy), (ii) reactivity of arriving material (sticking coefficient $S_c$ from 1 to less than $10^{-3}$) and (iii) energy input (substrate heating, photons, ions, chemical energy).
4. (Optionally, **annealing** takes place)

5. The final step is **analysis** of the film.

   One level of analysis is the determination of functional properties important for given application and optimization of the deposition process for these processes (empirical approach). A deeper level of analysis involves probing the film structure and composition (better understanding of the overall processes).

Analysis of the films after deposition - kind of final process monitoring. However, **monitoring** is important in all steps!
## Overview of Deposition Methods I

<table>
<thead>
<tr>
<th>Method/Processes</th>
<th>Specification</th>
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<tbody>
<tr>
<td><strong>Evaporative Techniques:</strong></td>
<td></td>
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<tr>
<td>Thermal (vacuum) evaporation</td>
<td>Resistive heating, flash evaporation, arc evaporation, exploding-wire technique, laser evaporation, rf heating, electron-beam evaporation</td>
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<tr>
<td><strong>Molecular Beam Epitaxy (MBE)</strong></td>
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<td><strong>Liquid-Phase Chemical Techniques:</strong></td>
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<td>Electro processes</td>
<td>Electroplating, electrolytic anodization</td>
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<td>Mechanical techniques</td>
<td>Spray pyrolysis</td>
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<tr>
<td><strong>Gas-Phase Chemical Techniques:</strong></td>
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<tr>
<td>Chemical vapor deposition (CVD)</td>
<td>CVD epitaxy, metalorganic CVD (MOCVD), low-pressure CVD (LPCVD), atmospheric-pressure CVD (APCVD), atomic layer deposition (ALD)</td>
</tr>
<tr>
<td><strong>Gas-Phase Physical-Chemical Techniques (except plasma and ion beam):</strong></td>
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<tr>
<td>Modifications of CVD</td>
<td>Hot filament CVD (HFCVD), laser-induced CVD (PCVD), photo-enhanced CVD (PHCVD), electron enhanced CVD</td>
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# Overview of Deposition Methods II

<table>
<thead>
<tr>
<th>method/processes</th>
<th>specification</th>
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<tr>
<td><strong>plasma techniques:</strong></td>
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<tr>
<td>sputter deposition</td>
<td>dc sputtering</td>
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<td>rf diode sputtering</td>
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<td>magnetron sputtering</td>
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<td>PECVD in low temperature discharges</td>
<td>dc discharge</td>
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<tr>
<td>rf capacitively coupled plasma (CCP)</td>
<td></td>
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<tr>
<td>rf inductively coupled plasma (ICP)</td>
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<tr>
<td>microwave ECR deposition</td>
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<td>microwave resonantor reactor</td>
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<tr>
<td>atmospheric pressure dielectric barrier discharge (DBD)</td>
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<td>atmospheric pressure glow discharge (APGD)</td>
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<td>atmospheric pressure surface barrier discharge</td>
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<td>etc.</td>
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<tr>
<td>plasma processing in high temperature discharges</td>
<td>vacuum arc</td>
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<td>dc torch</td>
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<td>microwave torch</td>
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<td>etc.</td>
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<td><strong>ion beam techniques:</strong></td>
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<td>sputter deposition</td>
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<td>reactive ion beam sputtering</td>
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<td>ion deposition</td>
<td>ion beam deposition</td>
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<td>ionized cluster beam deposition (ICB)</td>
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<tr>
<td>dual processes</td>
<td>ion beam assisted deposition (IBAD)</td>
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<tr>
<td>dual ion beam sputtering</td>
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</tbody>
</table>
1.4 Applications of Thin Films
Overview of Applications Related to Particular Properties

- **Optical properties**
  - Antireflection coating
  - Filters (interference coatings)
  - Decoration (color, color effects)

- **Thermomechanical properties**
  - Scratch resistant coatings (hardness)
  - Thermal protection/heat barriers
  - Tribology (friction control, wear resistant films)

- **(Bio)chemical properties**
  - Corrosion resistant coatings
  - Permeation barriers
  - Biocompatible surfaces, not-fouling surfaces

- **(Photo)Electrical properties**
  - Conductors
  - Insulators
  - Semiconductor devices (microelectronics)
  - Photovoltaic materials (solar cells)

- **Magnetic properties**
  - Magnetic storage devices
Thin Films for Optical Applications

Antireflection coatings:

\[ r = \frac{r_{12} + r_{23}e^{-i2\Delta}}{1 + r_{12}r_{23}e^{-i2\Delta}} \]

with \( n_2d = \frac{\lambda}{4} \) and \( \alpha = 0 \):

\[ R = \left( \frac{n_1n_3 - n_2^2}{n_1n_3 + n_2^2} \right)^2 \]

with \( n_1 = 1 \) (air) and \( n_2 = \sqrt{n_3} \):

\[ \Rightarrow R = 0! \]
Interference filters and mirrors: multilayer structure

Important – control of film thickness, roughness (interface) and refractive index
Interference filters and mirrors: multilayer structure

Important – control of film thickness, roughness (interface) and refractive index

Fig. 2. Refractive index (at $\lambda=550$ nm) of different PECVD optical film materials; comparison with selected substrate and PVD materials.

Cutting tools:

*Which properties can be improved?*

*What do we achieve with it?*

*What are the challenges?*

- Hardness - wear resistance, range of materials
- Friction - wear resistance, cutting speed
- Thermal stability - cutting speed
- Heat conductivity - cutting speed
- Chemical stability - cutting speed and range of materials
- Color - more attractive for customer

*Challenges:*

- adhesion, cohesion, thermal expansion, chemical stability
- Complex shape of the object
Thin Films for Mechanical Protection

Function: corrosion protection and tribology enhancement

Important – control of film thickness, roughness and uniformity

Challenge – measurement of film properties at thickness < 3 nm
## Thin Films for Mechanical Protection

### Table 12-1. Mechanical and Thermal Properties of Coating Materials

<table>
<thead>
<tr>
<th>Material</th>
<th>Melting or Decomposition Temperature (°C)</th>
<th>Hardness (kg-mm(^{-2}))</th>
<th>(H_0) (kg-mm(^{-2}))</th>
<th>(a) (10(^{-4}) °C(^{-1}))</th>
<th>Density (g-cm(^{-3}))</th>
<th>Young's Modulus (kN-mm(^{2}))</th>
<th>Thermal Expansion Coefficient (10(^{-6}) K(^{-1}))</th>
<th>Thermal Conductivity (Wm(^{-1}) K(^{-1}))</th>
<th>Fracture Toughness (MPa-m(^{1/2}))</th>
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<tbody>
<tr>
<td>Ionic</td>
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<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>Al(_2)O(_3)</td>
<td>2047</td>
<td>2100</td>
<td>2300</td>
<td>7.85</td>
<td>3.98</td>
<td>400</td>
<td>6.5</td>
<td>~ 25</td>
<td>3.5</td>
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<tr>
<td>TiO(_2)</td>
<td>1867</td>
<td>1100</td>
<td>1250</td>
<td>5.99</td>
<td>4.25</td>
<td>200</td>
<td>9.0</td>
<td>9</td>
<td></td>
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<tr>
<td>ZrO(_2)</td>
<td>2710</td>
<td>1200</td>
<td></td>
<td></td>
<td>5.76</td>
<td>200</td>
<td>8.0</td>
<td>1.5</td>
<td></td>
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<tr>
<td>SiO(_2)</td>
<td>1700</td>
<td>1100</td>
<td></td>
<td></td>
<td>2.27</td>
<td>151</td>
<td>0.55</td>
<td>2</td>
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<tr>
<td>Covalent</td>
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<tr>
<td>C (Diamond)</td>
<td>3800</td>
<td>~ 8000</td>
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<td></td>
<td>3.52</td>
<td>1050</td>
<td>1</td>
<td>1100</td>
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<tr>
<td>B(_4)N</td>
<td>2450</td>
<td>~ 4000</td>
<td></td>
<td></td>
<td>2.52</td>
<td>660</td>
<td>5</td>
<td></td>
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<tr>
<td>BN</td>
<td>2730</td>
<td>~ 5000</td>
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<td>3.48</td>
<td>440</td>
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<td>SiC</td>
<td>2760</td>
<td>2600</td>
<td>2800</td>
<td>0.90</td>
<td>3.22</td>
<td>480</td>
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<td>Si(_3)N(_4)</td>
<td>1900</td>
<td>1700</td>
<td>1900</td>
<td>2.79</td>
<td>3.19</td>
<td>310</td>
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<td>AlN</td>
<td>2250</td>
<td>1200</td>
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<td></td>
<td>3.26</td>
<td>350</td>
<td>5.7</td>
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<td>Metal Compounds</td>
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<tr>
<td>TiB(_2)</td>
<td>3225</td>
<td>3000</td>
<td>3500</td>
<td>18.9</td>
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<td>560</td>
<td>7.8</td>
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<td>TiC</td>
<td>3067</td>
<td>2800</td>
<td>3300</td>
<td>18.3</td>
<td>4.9</td>
<td>460</td>
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<td>TiN</td>
<td>2950</td>
<td>2100</td>
<td>2100</td>
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<td>5.4</td>
<td>590</td>
<td>9.3</td>
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<td>HfN</td>
<td>2870</td>
<td>2000</td>
<td>2000</td>
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<td>12.3</td>
<td>460</td>
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<td>HfC</td>
<td>3928</td>
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<td>3000</td>
<td>14.7</td>
<td>14.5</td>
<td>560</td>
<td>7.1</td>
<td>23</td>
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<td>TaC</td>
<td>3985</td>
<td>1600</td>
<td>1800</td>
<td>6.75</td>
<td>15.7</td>
<td>720</td>
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<tr>
<td>WC</td>
<td>2776</td>
<td>2300</td>
<td>2350</td>
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<td>250</td>
<td>14</td>
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<td>50–170</td>
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<td>Substrate Materials</td>
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<td>High-Speed Steel</td>
<td>1400</td>
<td>900</td>
<td></td>
<td></td>
<td>7.8</td>
<td>250</td>
<td>14</td>
<td>30</td>
<td>50–170</td>
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<tr>
<td>WC-6%Co</td>
<td>1500</td>
<td></td>
<td></td>
<td></td>
<td>640</td>
<td>5.4</td>
<td>5.4</td>
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<tr>
<td>Ti</td>
<td>1667</td>
<td>250</td>
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<td></td>
<td>120</td>
<td>11</td>
<td>13</td>
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<tr>
<td>Ni Superalloys</td>
<td>1280</td>
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<td></td>
<td></td>
<td>4.5</td>
<td>12</td>
<td>13</td>
<td></td>
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</tbody>
</table>

M. Ohring, The Materials Science of Thin Films
Alloys can have properties superior to each component

Multilayer structures can combine properties of different compounds

![Microhardness of mixed carbides](image1.png)

**Figure 12-5.** Microhardness of mixed carbides due to solid solution and precipitation hardening (From Ref. 3).

![SEM images of CVD multilayer coatings](image2.png)

**Figure 12-6.** SEM images of CVD multilayer coatings for cutting tool inserts. (a) Carbide substrate/TiC/TiCN/TiN (5500 ×). (b) Carbide substrate–TiC–Al2O3–TiN (3500 ×). (Courtesy of S. Wertheimer, ISCAR Ltd.)
Barrier coatings – permeation barriers

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**Figure 5.** Permeation rates of 60 nm thick SiO$_x$ films for various pulse powers $600 \text{ W} \leq P \leq 3000 \text{ W}$ at plasma conditions $\Phi_{\text{HMDSO}}=4 \text{ sccm}$, $\Phi_{\text{O}_2}=400 \text{ sccm}$, $P=30 \text{ Pa}$, $t_{\text{on}}=4 \text{ ms}$ and $t_{\text{off}}=40 \text{ ms}$.
Barrier coatings – permeation barriers

Practical problem: Bottles are filled at pressure of ~ 6 bar!

Figure 7. SEM micrographs of SiOx films on PET after 5 h etching in CCP oxygen plasma.

Important: adhesion, microstructure (defects), elasticity, biocompatibility
1.5 Fabrication of microstructures/microdevices
Increase of integration:

- **Small-Scale Integration (SSI)**
  few transistors on chip,

- **Medium-Scale Integr. (MSI)**
  hundreds of transistors on chip (end of 60ties),

- **Large-Scale Integration (LSI)**
  10,000 transistors on chip (70ties),

- **Very Large-Scale Integr. (VLSI)**
  100,000 transistors on chip (begining of 80ties),
  1,000,000,000 in 2007
Microelectronics - Fabrication of Integrated Circuits

- **Front-end-of-line (FEOL) structure**: complementary metal-oxide-semiconductor (CMOS) technology is the dominant semiconductor technology for microprocessors, microcontrollers, static RAM and other ICs. CMOS uses complementary and symmetrical pairs of p-type and n-type metal oxide semiconductor field effect transistors (MOSFETs) for logic functions.

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- **Back-end-of-line (BEOL) structure**: interconnect metallization, Cu instead of Al and low-k materials are used to decrease the R and C, i.e. BEOL delay.

SEM view of three levels of copper interconnect metallization in IBM's CMOS integrated circuits (Photograph courtesy of IBM Corp., 1997.)
What are MEMS/NEMS?

The acronym MEMS/NEMS (micro / nanoelectromechanical systems) originated in the USA. The term commonly used in Europe is microsystem technology (MST), and in Japan it is micro/nanomachines. Another term generally used is micro/nanodevices.

- MEMS - microscopic devices with characteristic length < 1 mm and > 100 nm
- NEMS - nanoscopic devices with characteristic length < 100 nm

MEMS/NEMS terms are also now used in a broad sense and include electrical, mechanical, fluidic, optical, and/or biological functions. They are referred to as intelligent miniaturized systems comprising e.g. sensing, processing and/or actuating functions.

MEMS/NEMS for

- optical applications - micro/nanooptoelectromechanical systems (MOEMS/NOEMS),
- electronic applications - radio-frequency-MEMS/NEMS or RF-MEMS/RF-NEMS.
- biological applications - BioMEMS/BioNEMS.
MEMS/NEMS examples shown are of a vertical single-walled carbon nanotube (SWCNT) transistor (5 nm wide and 15 nm high), of molecular dynamic simulations of a carbon-nanotube-based gear, quantum-dot transistor, and digital micromirror device (DMD http://www.dlp.com)
Examples of MEMS - gears/motors

- MEMS motor was developed in late 1980s using polycrystalline silicon (polysilicon) technology.
- Left-bottom SEM photo - microengine output gear and two additional driven gears. Gear extreme diameter is approximately 50 micrometers and gear thickness is 2.5 micrometers (J. J. Sniegowski et al.)
Two principle approaches can be used for micro/nanofabrication:

**top-down approach:**
- deposition of thin films
- doping
- etching/sputtering (lithography, i.e. through a mask, and nonlitographic fabrication)
  - anisotropic etching of Si
- preparation of surfaces (cleaning, polishing, functionalization)

**bottom-up**
- building using nanoobjects (atoms, molecules),
- self-assembly of structures
Lithography - process flow

Microlithography is a technique that creates microstructures after given geometrical template:

- Lithography is usually applied to shape a thin film ⇒ deposition of thin film
- Photosensitive material (resist) is coated on the material that should be shaped
- Resist is irradiated through a mask, by projection of UV image or by directed electrons (photolithography or electron lithography)
- Resist development:
  - positive resist: soluble in developer at the irradiated places
  - negative resists: unsoluble in developer at the irradiated places
- Etching of the film through photoresist pattern
- Rest of the resist is removed

lithography patterning with positive resist