Morphology of Polymer Materials and their Characterization

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# Polymer based materials

<table>
<thead>
<tr>
<th>Synthetic polymer materials</th>
<th>Biomaterials</th>
</tr>
</thead>
<tbody>
<tr>
<td>Polymer blends</td>
<td>Natural fibres (Silk, Cellulose)</td>
</tr>
<tr>
<td>Fibres</td>
<td>Wood, ivory</td>
</tr>
<tr>
<td>Fibre reinforced materials</td>
<td>Bone, Mussel shells</td>
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<tr>
<td>Particle filled polymers</td>
<td>Diatomes, cork</td>
</tr>
<tr>
<td>Foams</td>
<td>Agarose</td>
</tr>
<tr>
<td>Hydrogels</td>
<td></td>
</tr>
</tbody>
</table>
Microscopic techniques

- Foam cells
- LIMI (Lamellar Interface of Micro-Electronics)
- Blend Phases
- SEM (Scanning Electron Microscopy)
  - Glass fibres
  - Latex particles
- TEM (Transmission Electron Microscopy)
  - Blockcopolymers
  - Polymer crystals
- AFM (Atomic Force Microscopy)

Resolution:
- 100 µm
- 10 µm
- 1 µm
- 0.1 µm
- 0.01 µm
Optical microscopic techniques

<table>
<thead>
<tr>
<th>Light Microscopy</th>
<th>Electron Microscopy</th>
</tr>
</thead>
<tbody>
<tr>
<td>Confocal Laser Scanning Microscopy</td>
<td>Scanning Electron Microscopy</td>
</tr>
<tr>
<td>Conventional Light Microscopy</td>
<td>Transmission Electron Microscopy</td>
</tr>
</tbody>
</table>

Similar optical principles but different
b) Resolution
c) Contrast mechanisms
d) Technical Realisation
Optical beam path and basic optical elements

TEM

Light microscope

1. intermediate image plane phototube
2. eyepiece
3. intermediate image plane frontport
4. intermediate image plane baseport
5. beam path switching between baseport/ frontport/vis
6. sideport prisms
7. tube lens
8. analyzer
9. reflector module
10. luminous field diaphragm
11. aperture diaphragm
12. filter slider
13. HBO lamp
14. HAL lamp
15. luminous field diaphragm
16. polarizer
17. aperture diaphragm
18. condenser
19. objective

Winfried Wiegraebe, Stowers Institute for Medical Research
Polymer morphology

Homopolymer
- Amorphous
- Crystalline

Copolymer
- Block copolymer
- Statistical
Polymer morphology

Homopolymer
- crystalline

Copolymer
- blockcopolymer

Solid state

Aqueous
Microtome sectioning

- movement of microtome arm
- specimen embedded in wax or resin
- steel blade
- ribbon of sections
- ribbon of sections on glass slide, stained and mounted under a cover slip
Microscopic techniques

Electron microscopy

Low electron density difference

Contrast enhancement through selective staining

High electron density difference
Microscopic techniques

Microscopic technique

Light microscopy

Electron microscopy

Contrast mechanism

Refractive Index

Polarizibility

Electron density

Atomic number

Polymers: C, O, H, N

C = (G1 - G2) / G1 + G2
Microscopic techniques

Staining
Typical Blend morphology

Dispersed phase
TEM

Bicontinuous phase
Fracture surface SEM
Typical Blend morphology

Dispersed phase

Bicontinuous phase
Particle morphologies
High Impact Polystyrene – HIPS – TEM stained
Frozen hydrated specimen preparation

Cryo Electron Microscopy

Anja Rank
Diss. Uni. Freiburg
Herstellung und Charakterisierung von Vesikeln aus amphiphilen Blockcopolymeren
Cryo Electron Microscopy

Rapid freezing with a simple freeze punger

\[ V_c > 10^4 \text{ K/s (cooling rate)} \]
Cryo Electron Microscopy

Frozen hydrated specimen

Polymerosomes
EG$_{16}$ PS$_{50}$ EG$_{16}$

ALESSANDRO NAPOLI, MASSIMILIANO VALENTINI, NICOLA TIRELLI, MARTIN MÜLLER and JEFFREY A. HUBBELL
Particle morphologies
Latex particles

bimodal

unimodal
Transmission Electron Microscope

Diagram showing the components and process of transmission electron microscopy:
- Electron source
- Condenser lens
- TEM sample
- Objective lens
- Focal plane of objective
- Selector aperture
- 1st Intermediate Image
- Intermediate lens
- 2nd Intermediate Image
- Projector lens
- Image

Microscopy (a) vs. Diffraction (b)
Percolating structures
(Sand particles in cement)

Percolation

Percolation threshold

Fraction Connected

Sand Volume Fraction

SC-EMT
Percolating structures
Surface induced structures
Surface induced structures
Fractal structures
DLA Clusters
DLA Clusters
DLA Clusters
DLA Clusters in nature
Fractal characterisation
Fractal characterisation

Gleichung:
\[ F = \pi R^b \]

\[
\begin{align*}
a & = 3.1418 \pm 0 \\
b & = 1.84869 \pm 0.0026
\end{align*}
\]
Voronoi Polyhedra
Low Voltage Scanning Electron Microscope
Carbon fibres composites

Fracture surfaces
Foam structures

Light microscope

Scanning Electron Microscope
3 dimensional information by LSM
Fluorescence Microscopy

Phase characterisation in PS/PMMA blends
Fluorescence Microscopy

Fluorescence dyes
Fluorescence Microscopy

Fluorescence
Within femtoseconds after light excites the fluorophore at an appropriate wavelength, its electrons jump from the ground state to a higher vibrational state. Within picoseconds these electrons decay to the lowest excited state, and then decay more slowly (nsec) back to the ground state with the emission of a photon whose wavelength is longer than the exciting wavelength. Click to continue >>

Fluorescence dyes – the physical principle
Fluorescence Microscopy

Fluorescence absorption – emission – optical elements
Fluorescence Microscopy

Na Emission spectra
Fluorescence Microscopy

Hg emission spectra
Two-Photon Fluorescence Microscopy Principles

Helmchen, Denk, Nature Methods 2(12), (2005), 932
Two-Photon Fluorescence Microscopy Applications

Helmchen, Denk, Nature Methods 2(12), (2005), 932
Phase separation and diffusion limited growth in Phospholipid membranes
Bagatolli, Gratton, Biophysical Journal 78. (2000), 290
Two-Photon Fluorescence Microscopy

Applications

Phase separation and diffusion limited growth in Phospholipid membranes
Bagatolli, Gratton , Biophysical Journal 78. (2000), 290
Ein gesundes und erfolgreiches Jahr