Chemical and physical processes for nano particles synthesis

Process : Aerosol process - flame hydrolysis
Aerosil process Degussa 1942 - synthesis of silica

chemical reactions :

\[
\begin{align*}
\text{H}_2 + \text{O}_2 & \rightarrow 2 \text{H}_2\text{O} \\
\text{SiCl}_4 + 2 \text{H}_2\text{O} & \rightarrow \text{SiO}_2 + 4 \text{HCl} \\
\text{SiCl}_4 + 2 \text{H}_2 + \text{O}_2 & \rightarrow \text{SiO}_2 + 4 \text{HCl}
\end{align*}
\]

silicon tetra chloride

“fumed silica”

principle :

production in flame reactor

particle size range :

- primary particle size 7 – 40 nm, spherical, amorphous particle
- powder as agglomerated particles of high porosity
- specific surface area 50 – 400 m² / g

product : titanium dioxide, aluminium oxide, zirconium oxide, zinc oxide
Aerosol processes

Images (transmission electron microscopy) of different oxides, produced by direct oxidation in an arc
Particle formation in aerosol processes

Gas to particle conversion GPC

Solution of precursor:

Particle to particle conversion PPC
Particle morphology during flame hydrolysis

- particle concentration
- aggregation
- particle growth
- temperature
Chemical and physical processes for nano particle synthesis

process: aerosol process - flame hydrolysis

synthesis of titanium dioxide - chlorine process

chemical reaction:

\[
\begin{align*}
\text{TiCl}_4 + 2 \text{H}_2\text{O} & \quad \xrightarrow{1000 - 1300 \, ^\circ \text{C}} \quad \text{TiO}_2 + 4 \text{HCl} \\
4 \text{HCl} + \text{O}_2 & \quad \xrightarrow{1000 - 1300 \, ^\circ \text{C}} \quad 2 \text{H}_2\text{O} + 2 \text{Cl}_2 \\
\text{TiCl}_4 + \text{O}_2 & \quad \xrightarrow{} \quad \text{TiO}_2 + 2 \text{Cl}_2 
\end{align*}
\]

principle:

apparatus for titanium dioxide powder

particle size: 100 - 400 nm, amorphous particles, product of anatase / rutile, part of rutile increases with temperature

advantage: minimum aggregation and high dispersity of powder

products: titanium dioxide, zirconium dioxide
Chemical and physical processes for nano particle synthesis

process: aerosol process – powder synthesis using laser light

synthesis of silicon carbide and silicon nitride

chemical reaction:

\[
\begin{align*}
\text{SiH}_4 & + \text{CH}_4 \rightarrow \text{SiC} + 4 \text{H}_2 \\
3 \text{SiH}_4 & + 4 \text{NH}_3 \rightarrow \text{Si}_3\text{N}_4 + 12 \text{H}_2
\end{align*}
\]

silicon hydride \hspace{1cm} silicon carbide

3 silicon hydride \hspace{1cm} silicon nitride

principle:

advantage: particle of high purity, monodisperse particle size distribution, exact stoichiometry

disadvantage: precursor has to absorb laser light

only on a laboratory scale, mass produced 1 – 100 g

Reaction chamber for powder synthesis using a laser
## Methods for powder generation with spray processes

<table>
<thead>
<tr>
<th>methods</th>
<th>starting material</th>
<th>process steps</th>
</tr>
</thead>
<tbody>
<tr>
<td>spray drying</td>
<td>suspension</td>
<td>solvent evaporation</td>
</tr>
<tr>
<td>spray drying</td>
<td>solution</td>
<td>solvent evaporation, crystallization</td>
</tr>
<tr>
<td>spray calcination</td>
<td>suspension</td>
<td>solvent evaporation, calcination</td>
</tr>
<tr>
<td>spray decomposition</td>
<td>solution of inorganic salts</td>
<td>solvent evaporation, calcination</td>
</tr>
<tr>
<td>spray hydrolysis</td>
<td>non aqueous solution or liquid metal compound</td>
<td>hydrolysis by water vapour, for solutions</td>
</tr>
<tr>
<td>spray pyrolysis</td>
<td>solution or melt of metal organic salts</td>
<td>thermal decomposition, for solutions</td>
</tr>
<tr>
<td>plasma evaporation of solutions</td>
<td>solution</td>
<td>solvent evaporation and nucleation / crystallization in a gaseous phase</td>
</tr>
</tbody>
</table>
Spray hydrolysis

chemical reaction: hydrolysis

\[ \text{Ti}(	ext{OC}_2\text{H}_5)_4 + 2 \text{H}_2\text{O} \rightarrow \text{TiO}_2 + 4 \text{C}_2\text{H}_5\text{OH} \]

\[ \text{TiCl}_4 + 2 \text{H}_2\text{O} \rightarrow \text{TiO}_2 + 4 \text{HCl} \]

particle: mostly non-agglomerated, spherical particle with high purity

hollow and porous particle can be formed easily

controlling of powder porosity by concentrations in droplets and by temperature gradients

Formation of nonporous and porous particle by spray hydrolysis