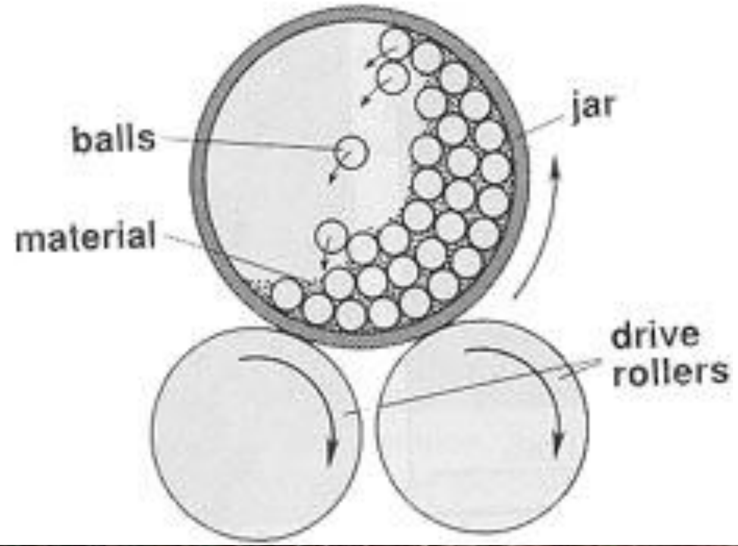
Powder Production

Powder production methods for ceramic powders can be classified as;

- Mechanical
- Chemical



Milling

It is a mechanical method for ceramic powder production. Reduction of particle size is beneficial for sintering, which depends on diffusion of atoms.

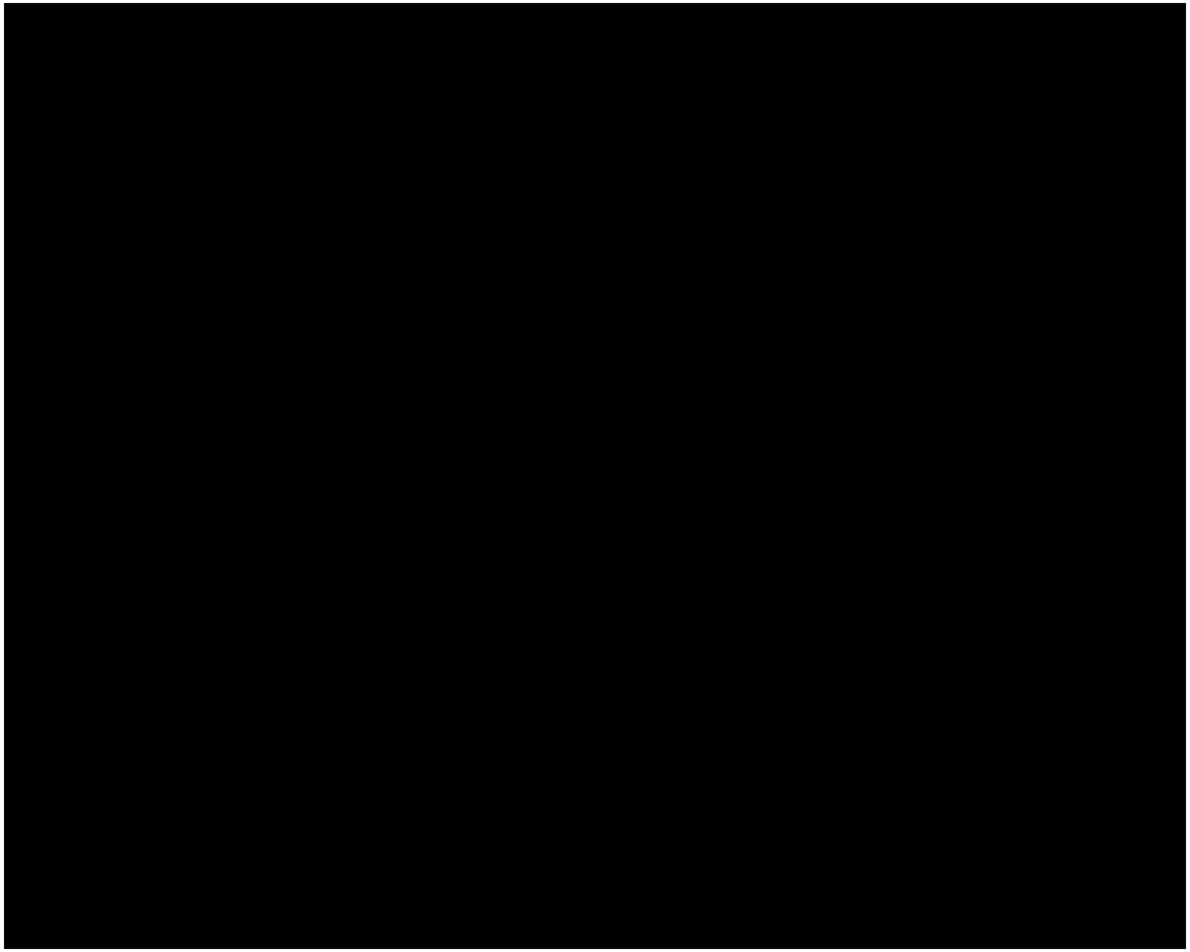
Most common milling method is ball milling

- Balls are used for milling. Hardness of the balls must be equal or greater than the hardness of the ceramic will be milled.
- Amount and size of the balls are critical for the final powder size.
- Wet or dry milling can be applied.
- From balls or barrel impurities may added to the ceramic powder

Powder Production

Apart from the ball mill, there are other milling types;

- Fluid energy mill (jet mill)



Powder Production

Apart from the ball mill, there are other milling types;

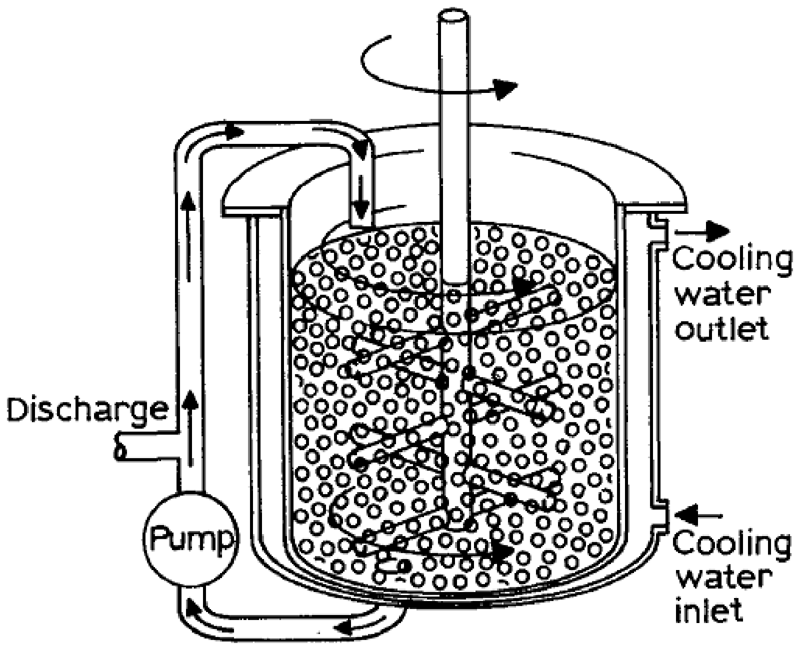
- Vibrational mill

Powder Production

Apart from the ball mill, there are other milling types;

- Attrition mill

Attritor Mill



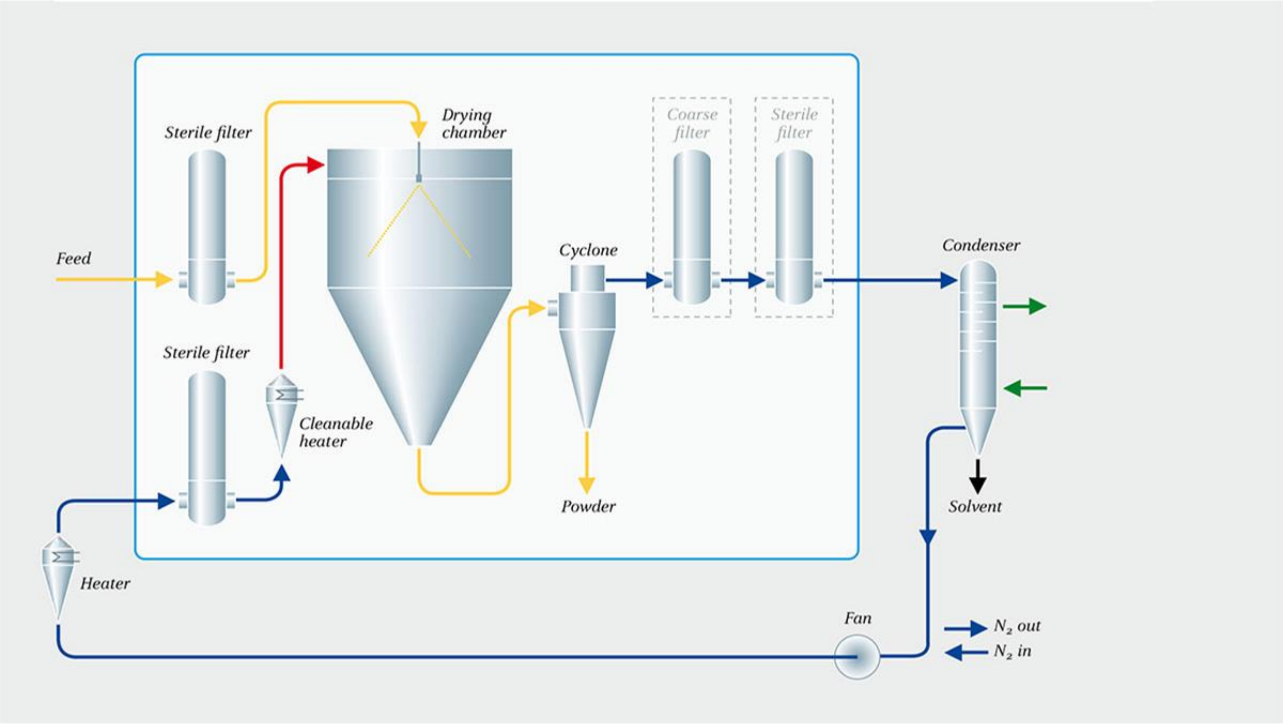
An attritor is a ball mill system in which the balls, together with the material to be milled are set in motion by a shaft with stirring arms, rotating 100-2000rpm.

Cylindrical vessel is usualy water cooled because of the considerable heat generated by the process.
Dry,wet (water, inorganic liquids) milling is possible inert gas supply possible for reactive materials.
More effective than conventional ball mill.

Powder Production

Spray Drying

It is commonly used in ceramic processing to achieve uniform, free-flowing powder. It has an atomizer (nozzle or centrifugal) which atomize fluid with a droplets around 30 to 250 μm through a pressure nozzle with pressures up to 10.000 psi.



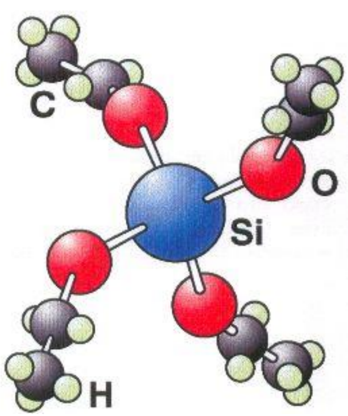
Powder Production

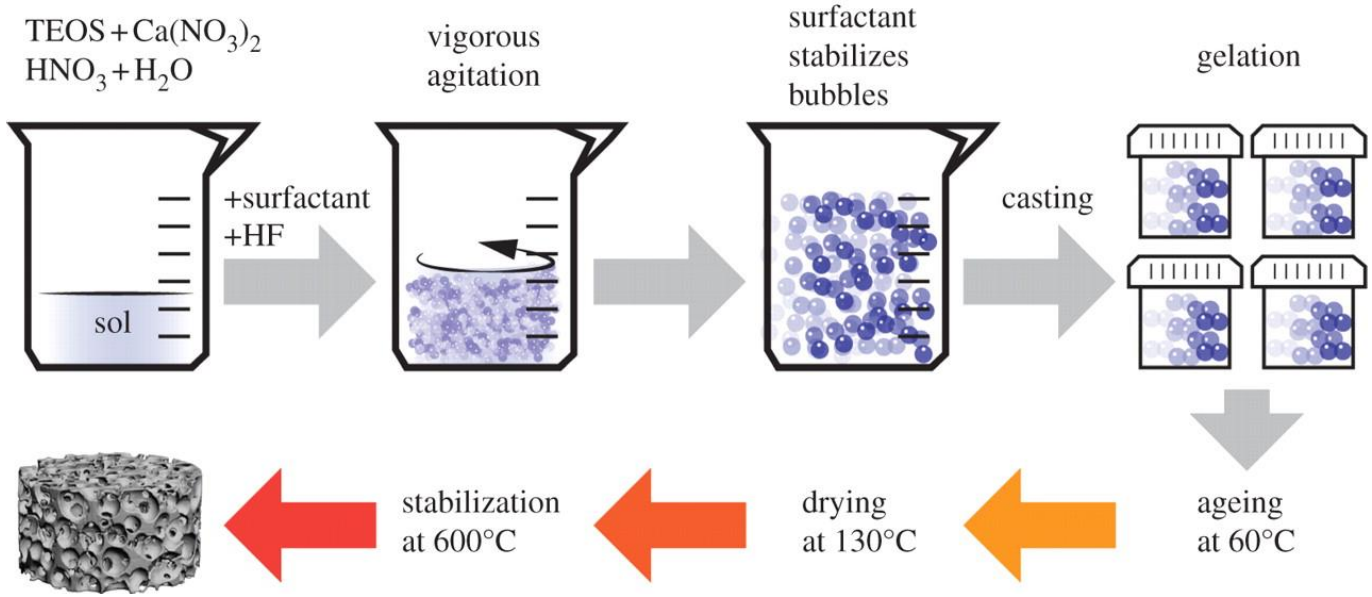
Sol-Gel Processing

Sol-gel is a generic term that includes a variety of techniques to achieve a high-purity composition with homogeneity at the molecular level.

Involves following steps:

- Form a stable dispersion (sol) of particles
- By changing concentration, aging, or addition of suitable electrolyte, induce polymer-like, 3D bonding to form a gel from the sol.
- Evaporate the remaining liquid from the gel
- Increase the T to convert dehydreated gel to the ceramic composition.



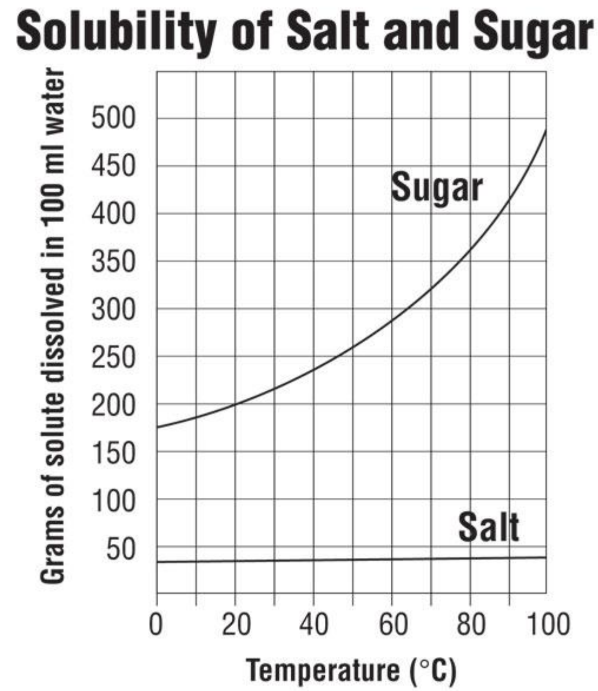


Powder Production

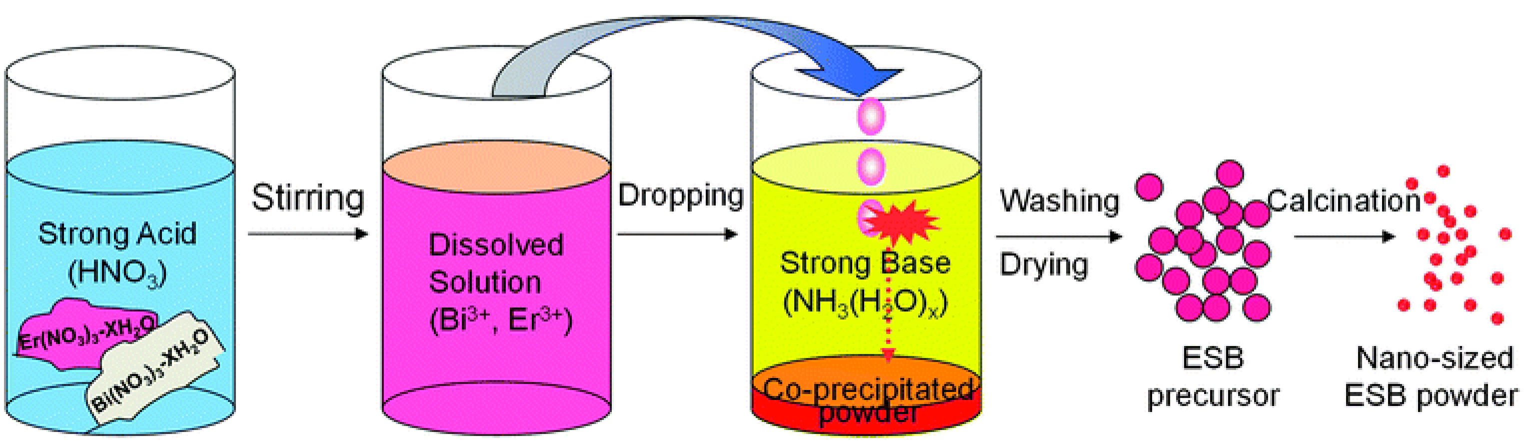
Precipitation

Precipitation depends on the solubility vs temperature (or pH, pressure)behaviour of the solutes in associated solvents.

T example



pH example

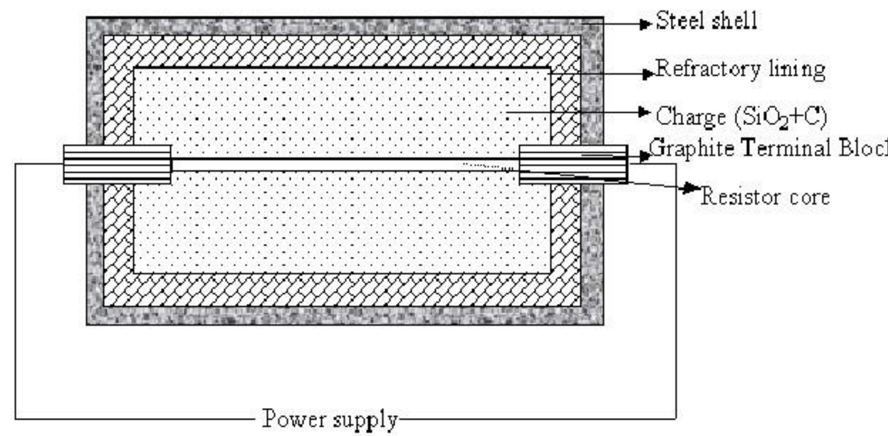


Powder Production

Production of Non-oxide Powders – SiC

SiC and Si_3N_4 are very rare in nature. Their powders are produced by chemical methods.
For example, SiC is produced generally by Acheson Process; lower grade SiC for abrasives, higher grade SiC for electrical applications.

Acheson process is a process which is used mainly for the manufacture of silicon carbide. Currently coke and quartz are used as major raw materials to produce SiC in bulk quantities. A schematic of a resistance furnace of the type used in the Acheson process, is shown in Figure. SiC has extreme hardness, sharpness and good thermal properties and hence it is employed as a abrasive and refractory material. In Acheson process, resistance-heating used up to around 2200^0C.



$$SiO_2 + 3C \xrightarrow{1600°C} SiC + 2CO$$

Powder Production

Production of Non-oxide Powders – SiC, Si_3N_4

SiC is used for high-temperature kiln furniture, electrical-resistance heating elements, grinding wheels and adrasives, wera-resistance applications, varistors, LEDs

Silicon nitride does not occur naturally. Several methods are present to synthesize it.

Silicon powder with nitrogen gas at 1250^0C-1400^0C range forms Si_3N_4.

High purity silicon nitride can be made by reduction of SiO_2 with carbon in the appropriate nitrogen environment.
Also, high purity silicon nitride can be produced by reaction of $SiCl_4$ or silane with ammonia. This reaction results in silicon diimide, and by decomposition of silicon diimide (around 1000^0C) gives rise to formation of silicon nitride powder.

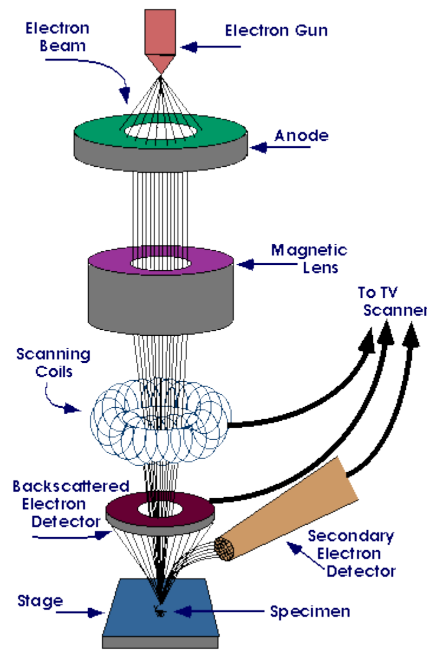
$$3Si(NH)_2 = Si_3N_4 + 2NH_3$$

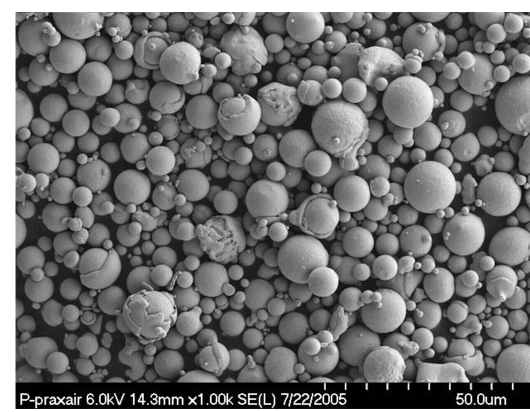
Powder Characterization

Powder/particle size and its distribution is essential. There are a couple methods to determine particle size range of the powders.

Microscopy

It is a direct method to measure particle size of the particles. Optical microscopy or electron microscopy can be used to determine particle size.





Powder Characterization

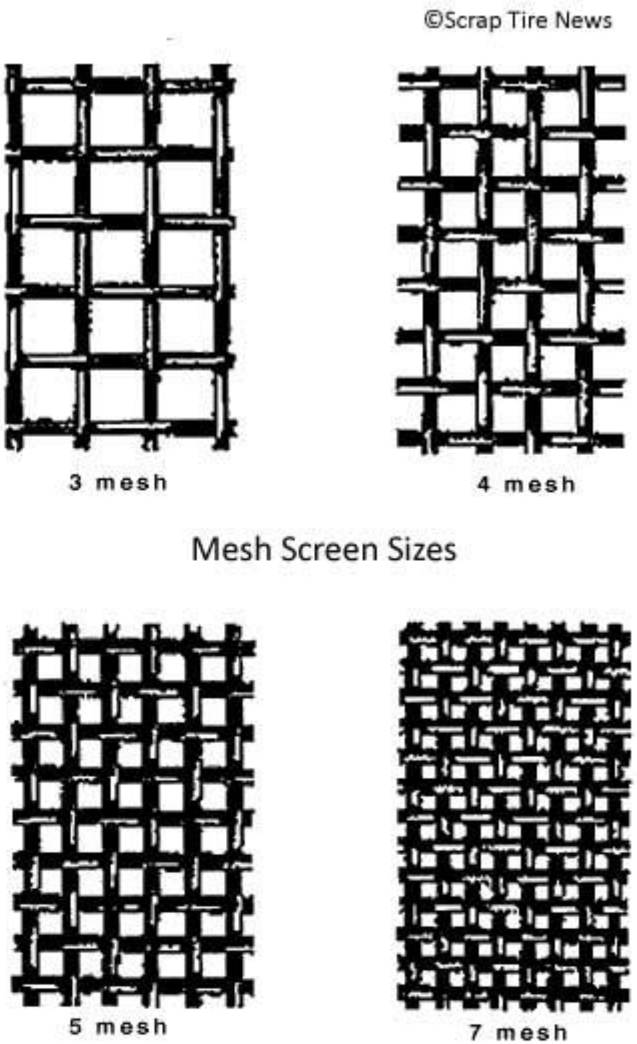
Sieving

Sieving is used to separate particles according to their particle sizes.

Powder Characterization

Sieving

Table shows sieve numbers and their corresponding openings in mm and inches.



(ASTM) U.S. Sieve Number	Particle Size	Tyler Screen Scale Equivalent	Sieve Opening Millimeters	Sieve Opening Inches	International Standard Organization Millimeters
3 ½		3 ½	5.660	0.223	5.6
4		4	4.760	0.187	
5		5	4.000	0.157	4
6		6	3.360	0.132	
7		7	2.830	0.111	2.8
8		8	2.380	**0.0937**	
10		9	2.000	0.0787	2
12		10	1.680	0.0661	
14		12	1.410	0.0555	1.4
16		14	1.190	0.0469	
18		16	1.000	0.0394	1
20		20	0.840	0.0331	
25		24	0.710	0.0280	0.71
30		28	0.590	0.0232	
35		32	0.500	0.0197	0.5
40		35	0.420	0.0165	
45		42	0.350	0.0138	0.355
50		48	0.297	0.0117	
60		60	0.250	0.0098	0.250
70		65	0.210	0.0083	
80		80	0.177	0.0070	
100		100	0.149	0.0059	
120		115	0.125	0.0049	
140		150	0.105	0.0041	
170		170	0.088	0.0035	
200		200	0.074	0.0029	
230		250	0.062	0.0024	
270		270	0.053	0.0021	
325		325	0.044	0.0017	
400		400	0.037	0.0015	

Powder Characterization

Sedimentation

If a particle with diameter d is dropped into a liquid, two forces act on it. Gravitational force and and the upward thrust of the liquid. (check the units)

Stokes Law

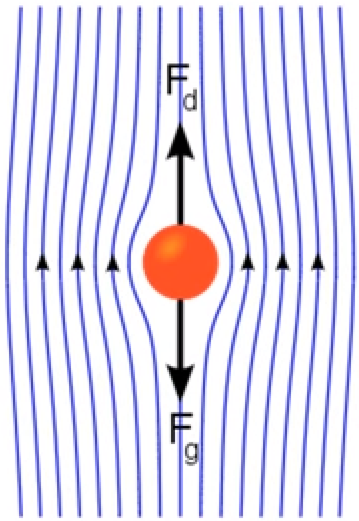
- Drag force $F_d = 6\pi\eta ru$

  η = viscosity
- Force of gravity

$$F_g = \frac{4}{3}\pi r^3(\rho_s - \rho_f)g$$

- At terminal velocity $F_g = F_d$
- Solve for velocity

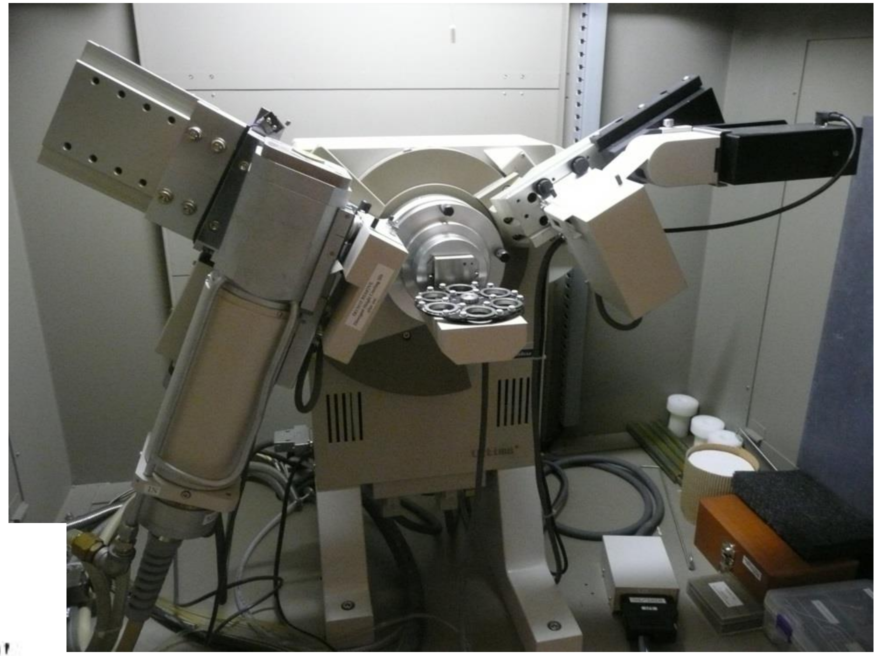
$$u = \frac{d^2 g(\rho_s - \rho_f)}{18\eta}$$



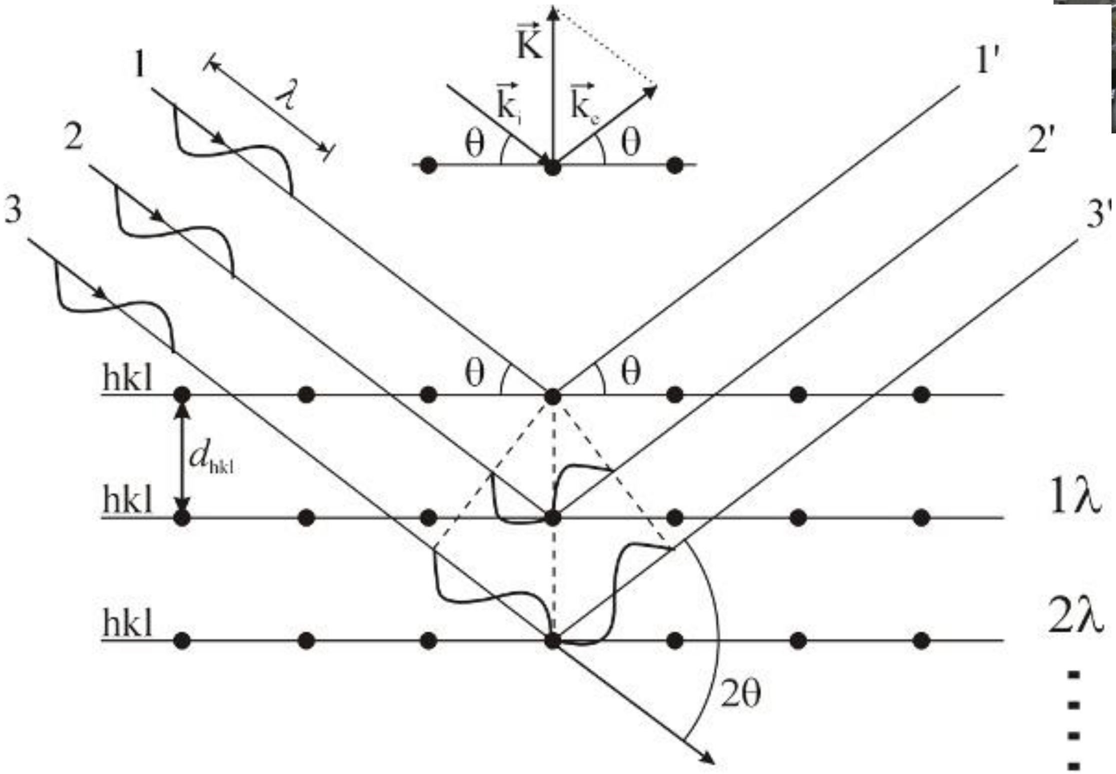
Gravity sedimentation is useful for particle size range 0.2 – 100 μm.

Powder Characterization

X-ray Diffraction



$n\lambda=2d\sin\theta$ - Bragg's Law

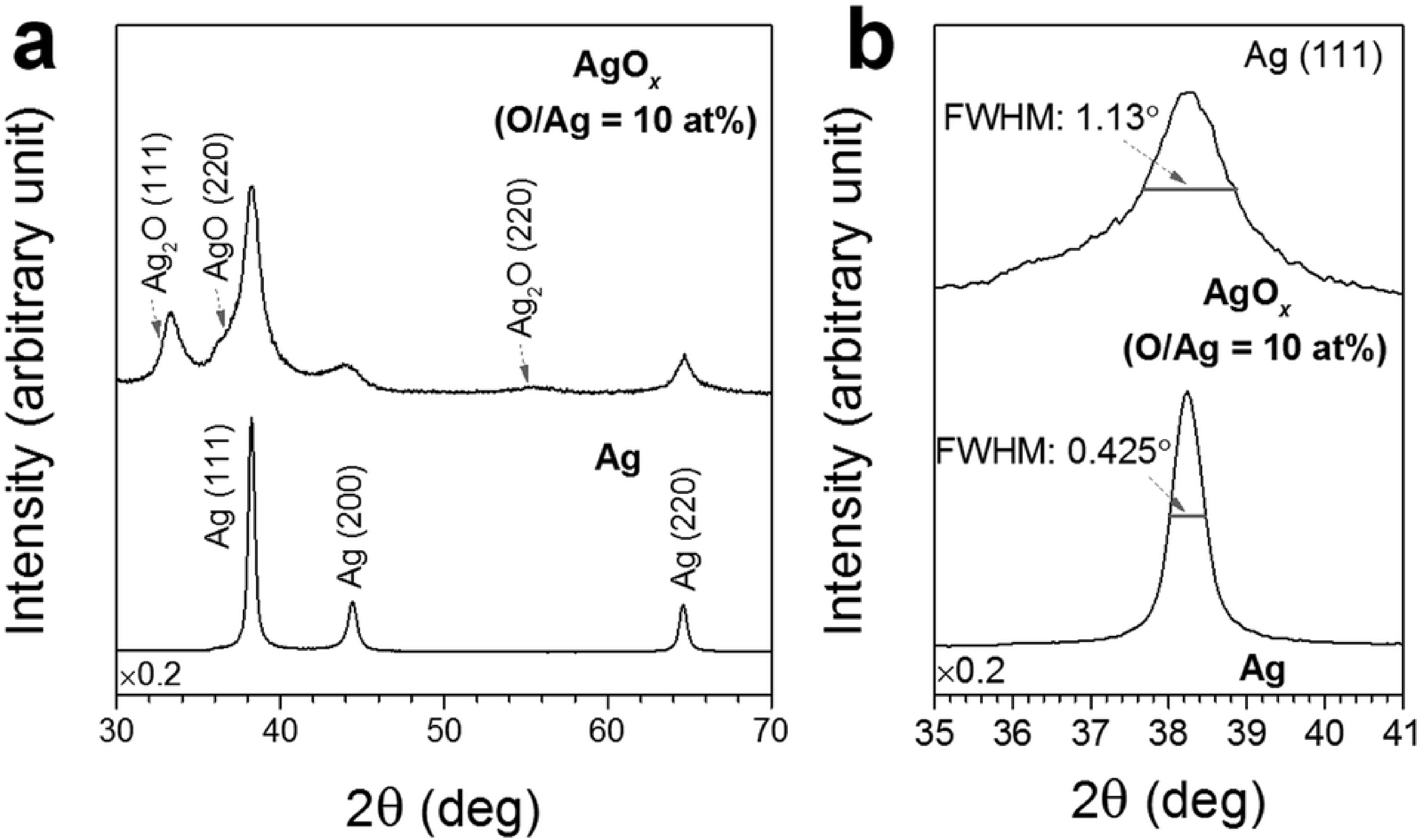


Powder Characterization

X-ray Diffraction

$$t = \frac{0.9\lambda}{B_{size}\cos\theta_B}$$ Scherrer Equation

Useful for particles less than 100nm



Powder Characterization

Surface Area Determination

Partice size can be calculated by measuring surface area of the particles. The surface area can be measured by adsorbing of surface a monomolecular layer of a gas. The pressure of gas before and after adsorbtion is measured. From the difference in pressure, mass of the gas adsorbed by the powder surface can be calculated. From this mass, total surface area can be calculated.

This method is called BET (Brunauer, Emmet, Teller) method. In this method, surface area is used to find the particle size.

$$Surface\ Area\ of\ a\ Sphere:\ A = 4\pi r^2$$

$$r = 3/\ \ S$$; S is total surface area, ρ is density