

Powder Metallurgy
Prof. Ranjit Bauri
Department of Metallurgical and Materials Engineering
Indian Institute of Technology, Madras

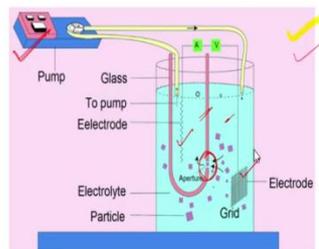
Lecture - 26
Particle size measurement – Other methods

So, in the past few classes, we have been talking about the Particle size measurement and in last couple of classes, we have discussed about the dynamic light scattering technique which is a well-known method for particle size analysis. Now, there are few more techniques which can be used to measure the particle size and that is what we are going to discuss in this particular class.

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Electrical zone sensing

- This technique is based on change in electrical conductivity of a fluid in a small aperture due to presence of particles.
- The change in conductivity is proportional to particle volume in the aperture.
- The apparatus consists of a nonconductive tube with a small aperture dipped in an electrolyte containing the suspended particles.
- The fluid is made to flow in the aperture by a small pressure difference achieved through a pump.
- An electrical current is maintained through two electrodes, one inside and the other outside the tube and the change in current flow is recorded.
- The dynamic ratio is around 30 and the lower limit of particle size is $0.5 \mu\text{m}$.

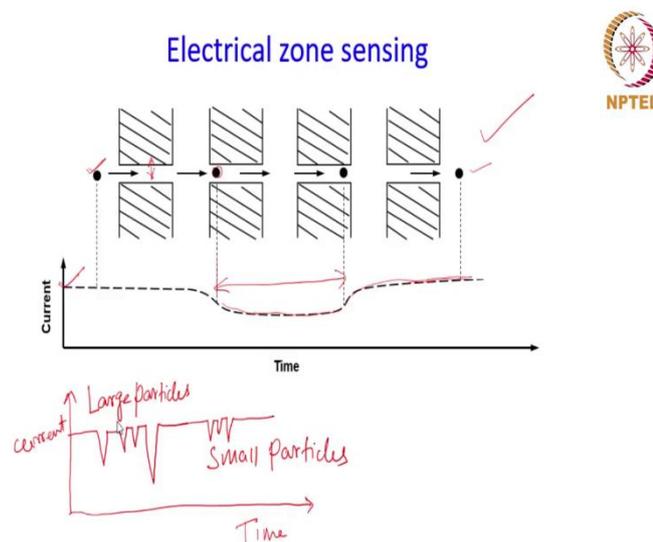


Let us first talk about electrical zone sensing method and it is based on the change in electrical conductivity of a fluid in a small aperture due to presence of particles. And this change in the conductivity is proportional to particle volume in the aperture and from there we can calculate the size of the particles. The apparatus which is used for this technique basically consists of a non-conductive tube with a small aperture dipped in an electrolyte containing the suspended particles.

So, the fluid is made to flow in the aperture by a small pressure difference which is created through a pump and in order to maintain the electrical current through the aperture, two electrodes are used; one is inside the tube and the other is outside the tube.

And through this, the change in the current flow is recorded and from there the change in the conductivity can be obtained and that is finally used to calculate the size of the particles. And the dynamic ratio that can be obtained from this technique is around 30 and the lower limit of particle size that can be determined by this technique is 0.5 micrometer.

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So, the change in the electrical conductivity and the way it varies depending on whether the particle is inside or outside the aperture that can be seen from this particular diagram from here (slide above). So, this is the aperture, the gap that you see over here and this is the particle that we are talking about.

When the particle is outside the aperture into the electrolyte, there is a particular current which is flowing, which is the initial current. Now, as you can see, as the particle enters the aperture, the current starts to drop and you can see that drop, as long as the particle is inside the aperture. The current which is flowing is lower compared to the initial current and the moment the particle goes out of the aperture, the current again comes back to the initial value.

So, this drop in the conductivity that you have here when the particle is moving into the aperture, is actually recorded in this technique and used to calculate the particle size because, here, the change in the current or the conductivity is proportional to the volume of the particles flowing into the aperture.

So, if you see the change in the current with respect to the size of the particles, the variation could be something like this (current vs time plot in above slide). The first section is for large particles and the second one is for small particles. You can clearly see that the change in the current or the conductivity is proportional to the size or the volume of particles and that is how the size can be calculated based upon the change in the conductivity. But of course, for that the instrument has to be properly calibrated before the measurements are done and once that is done and you measure the current which is flowing and from there measure the change in the conductivity and the particle size can be obtained.

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Electrical zone sensing



- Valid only for $D(\rho_m - \rho_f)^{3/2} < 500$.
- Particle settling is an issue – more for high density materials (Fe, W).
- Need to maintain low particle concentration to avoid coincidence (multiple particles at the same time) in the aperture.
- Aperture plugging and electrical noise.

This technique is valid only when the following relationship holds:

$$D(\rho_m - \rho_f) < 500$$

wherein D is the particle size, ρ_m is the density of the metal or the particles and ρ_f is the density of the fluid in which the particles are dispersed. And in this technique, particle settling is an issue and especially for high density materials like iron and tungsten which

can settle down easily because of their higher density and you also need to maintain a low concentration of particles to avoid coincidence in the aperture. Coincidence means multiple particles entering at the same time into the aperture.

And the other problem is aperture plugging which could happen due to the particles settling down into the aperture and not moving out and also at times there are electrical noises. So, these are some of the issues that you can encounter in this technique and as I said this technique is only valid for a particular relationship when it holds.

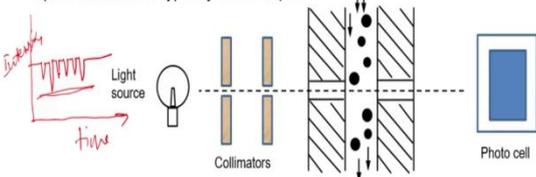
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Light blocking technique

- Similar to electrical zone sensing but instead of conductivity, the decrease in light intensity caused by particles is recorded and correlated with the particle size.
- The basic apparatus consists of a transparent tube in which particles are dispersed.
- The particles are exposed to the light beam by collimators and as a particle comes in front of the beam it partially blocks light and the intensity captured in the photocell decreases.
- Assuming spherical shapes, the amount of light blockage is equated to an equivalent circular cross-sectional area.
- The dynamic ratio is 45 and the lower limit of particle size determined by the optical resolution is typically around $1\ \mu\text{m}$.



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The next technique that we are going to discuss is known as light blocking technique. This is similar to electrical zone sensing, but instead of conductivity, the decrease in light intensity caused by the particles is recorded and correlated with the particle size.

The basic apparatus that you can see over here in this picture (slide above) basically consists of a transparent tube in which particles are dispersed and then, the particles are exposed to light beam. So, a light source is channeled towards the particles with the help of the collimators. Collimators are essentially guide tubes which can channel the light in a particular direction.

So, as the particles come in front of the beam, they partially block the light and the intensity captured at the photocell detector decreases. The photocell detector captures the intensity which is coming out after passing through the transparent tube.

So, how much of the intensity will be decreased will depend on the size of the particles. So, assuming spherical shape for the particles, the amount of light blockage is equated to an equivalent circular cross sectional area and from that the size of the particles can be calculated.

Now, if you see the output from this photocell, it may appear something like this (intensity vs time plot in slide above). So, the intensity can vary something like this depending on the size of the particles and if you assume a spherical particle shape from that the extent of this intensity variation can be correlated with the size and from that the particle size can be obtained.

The dynamic ratio for this particular technique is about 45 and the lower limit of the particle size that can be determined depends on the optical resolution of the system and it is typically around 1 micrometer.

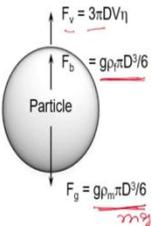
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Sedimentation

- Particles settling in a fluid reach a terminal velocity that depends on particle size and fluid viscosity. Particle size can be estimated from the settling velocity.
- The settling velocity is obtained from Stokes Law as $V = gD^2(\rho_m - \rho_f)/18\eta$ (1)
- This technique basically involves measuring the time (t) for a particle to move a distance H in a tube filled with a liquid of viscosity η to determine particle velocity
 $V = H/t$
- V is substituted in equation (1) to get the particle size, $D = [18H\eta / (g t(\rho_m - \rho_f))]^{1/2}$
- The settling time can be measured by measuring turbidity using a light source, a slit and a photodetector.
- Dilute suspension to avoid interparticle interactions.
- Limited to narrow particle size distribution

Stokes Law: $F_v = 3\pi DV\eta$

$$\underline{F_v} + \underline{F_b} = \underline{F_g}$$

The next method to determine the particle size is based on sedimentation, which is nothing, but settling down of particles in a fluid. You might know that particles settling in a fluid reach a terminal velocity that depends on the particle size and the fluid viscosity and therefore, the particle size can be estimated from the settling velocity. And here, the Stokes law is used to derive the settling velocity and from that the particle size is obtained.

So, the apparatus basically consists of a tube which contains a fluid in which the particles are dispersed and when a particle moves through a fluid, these are the forces which will act on it. The frictional drag force F_v which is given as

$$F_v = 3\pi DV\eta$$

wherein D is the diameter of the particle, V is the velocity and η is the viscosity of the fluid. So, this is the Stokes law which gives this frictional drag force acting on the particles.

And the other force which will provide a resistance to the movement of particles is the buoyancy which is given by

$$F_b = g\rho_f\pi\frac{D^3}{6}$$

wherein ρ_f is the density of the fluid and the gravitational force (mg) which is essentially, $F_g = m \times g = g\rho_m\pi\frac{D^3}{6}$, will act downward.

So, when the terminal velocity is reached, the forces acting on the particle will be balanced; that means, the forces acting upwards will balance the forces acting downward which means that F_v and F_b , the frictional drag force and the buoyancy will be balanced by the gravitational force F_g and from this, we can derive an expression for V .

$$F_v + F_b = F_g$$

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$$3\pi DV\eta + \frac{\pi D^3 \rho_f g}{6} = \frac{\pi D^3 g \rho_m}{6}$$
$$\underline{V} = \frac{g D^2 (\rho_m - \rho_f)}{18\eta}$$

Settling height H ,
Settling time t is measured

$$V = \frac{H}{t}$$
$$D = \sqrt{\frac{18H\eta}{(\rho_m - \rho_f)gt}}$$



So, let us write them in terms of their corresponding expressions.

$$3\pi DV\eta + g\rho_f\pi\frac{D^3}{6} = g\rho_m\pi\frac{D^3}{6}$$

F_v is this plus the buoyancy will equal the gravitational force and from here, you can derive V as

$$V = \frac{gD^2(\rho_m - \rho_f)}{18\eta}$$

So, now, if we can measure V , D can be easily obtained from this equation.

So, in order to measure V , there is an apparatus which is used that apparatus basically consists of a tube in which the particles are dispersed in a fluid and a known settling distance is used, a settling height let us say that is H and in the measurement process, the time taken for settling t is measured and from here, V can be easily calculated

$$V = \frac{H}{t}$$

So, once this is obtained, it is replaced in the equation below and D is obtained. So, if you substitute V over here, D will be obtained as

$$D = \sqrt{\frac{18H\eta}{(\rho_m - \rho_f)gt}}$$

So, this is how you will get the particle size D from this sedimentation technique.

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Sedimentation



- Limited to narrow size range.
- Particle below 1 μm – slow settling and turbulence.
- Internal porosity reduces the mass and leads to slow settling
- Irregular particles – trajectory is not straight and hence velocity is not constant.
- Prior knowledge (density, size) about particles is necessary. Hence, difficult to apply for unknown powders.

But it has its own limitations. For example, it is limited to a narrow size range and for particles below 1 micrometer, there is slow settling and turbulence and if there are internal porosity in the particle that will reduce the mass and that in turn will lead to slow settling.

And apart from that, the other difficulties that you can encounter in this technique are related to the type of particles. For example, if you have irregular particles, the trajectory of the movement of particles through the fluid is not straight and hence, a constant velocity cannot be expected and hence, the accuracy of the measurement will not be good when the particles are irregular in shape.

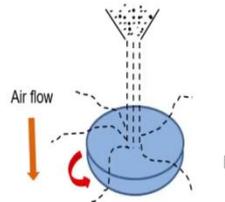
And also some prior knowledge about the particles in terms of their density and an approximate size is also needed beforehand to be able to carry out the measurement by this technique and therefore, it is difficult to apply for unknown powders where such properties are not known beforehand.

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Air classification



- A variation of the sedimentation technique. The powder is separated into selected size fractions using a cyclone or a spinning disc (up to 12000 rpm) and cross current air flow.
- The centrifugal force provides a constant particle velocity and the air flow results in separation by Stokes Law effect.
- The deceleration of the particles out of the disc depends on particle dia and mass and hence, the lighter or smaller particles are deflected by the air flow and are separated from the larger ones.
- Applicable to size range from 1 to 150 μm .



Then, we have the technique called air classification. This is nothing, but a variation of the sedimentation technique. So, here, the powder is separated into selected size fractions using a cyclone or a spinning disc which rotates at high velocity up to 12000 rpm and cross current airflow is used to separate out the particles as they fall on the rotating disc. So, the centrifugal force due to the rotation of the disc will provide a constant particle velocity and the air flow will result in separation by Stokes law effect.

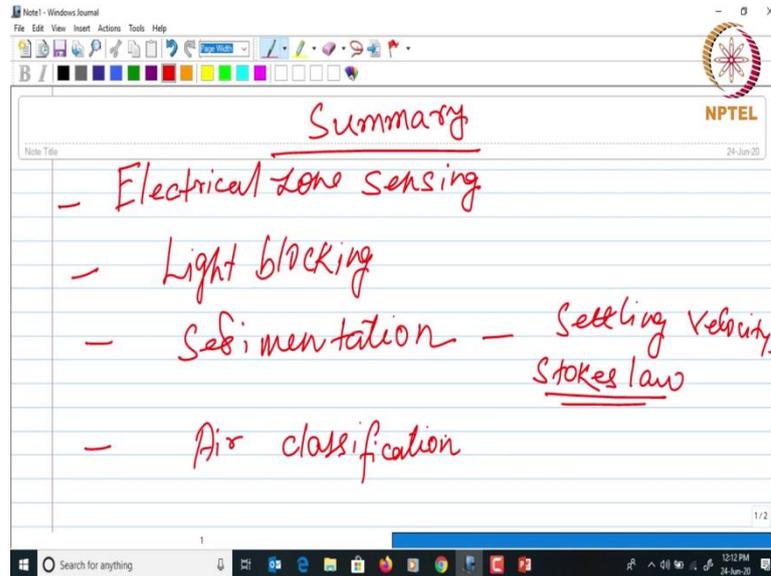
Now, the way, the particle size is distinguished here or the way smaller particles are distinguished from the bigger ones is because of the fact that the deceleration of the particles out of the disc depends on the size of the particle or the diameter of the particle and their mass and therefore, the lighter and smaller particles are deflected by the air flow and are separated from the larger ones. So, that is how the size-based separation happens in this case.

The schematic of the apparatus is shown over here (slide above). It basically consists of a feeding hopper which feeds the powder particles onto a rotating disc and as the powder particles fall on this rotating disc, they are given a velocity due to the centrifugal force and the cross current air flow from here will result in the separation of the particles based on their size.

The rotational speed of the disc and the air flow velocity can be controlled to change the particle size separation and that is how you can handle powders having a different

particle size range and the applicable size range for this particular technique varies from 1 to 150 micrometer.

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So, before we finish this lecture, let us take a moment to summarize it. In this lecture, we learned about the four techniques of particle size measurement. These were:

1. Electrical zone sensing which is based upon the conductivity change of a fluid as the particles move through a aperture in a tube which is dipped in that particular fluid in which the particles are dispersed.
2. Light blocking which is similar to the electrical zone sensing technique, but in this case, instead of the conductivity, the change in light intensity is captured and from that the particle size is correlated to that intensity change and the size of the particles is obtained.
3. Sedimentation which is based upon the settling velocity of particles in a fluid and the Stokes law is applied in order to get the settling velocity and from that, the particle size is obtained.
4. Air classification, a variation of the sedimentation method, uses a rotating disc and a cross current airflow to separate the particles based upon their size and from that the particle size can also be obtained.

So, with that we come to the end of this class. Thank you for watching.