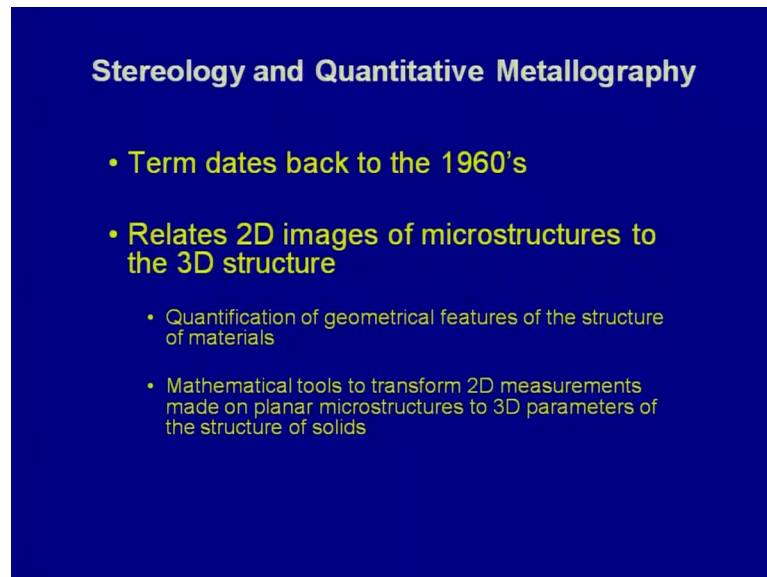


Heat Treatment and Surface Hardening - II
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Lecture – 34
Stereology and quantitative metallography – I

So, in this lecture we are going to look at the tools used for quantifying microstructure. This is an area called stereology and correspondingly in metallurgy it is often called as quantitative metallography.

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Stereology and Quantitative Metallography

- Term dates back to the 1960's
- Relates 2D images of microstructures to the 3D structure
 - Quantification of geometrical features of the structure of materials
 - Mathematical tools to transform 2D measurements made on planar microstructures to 3D parameters of the structure of solids

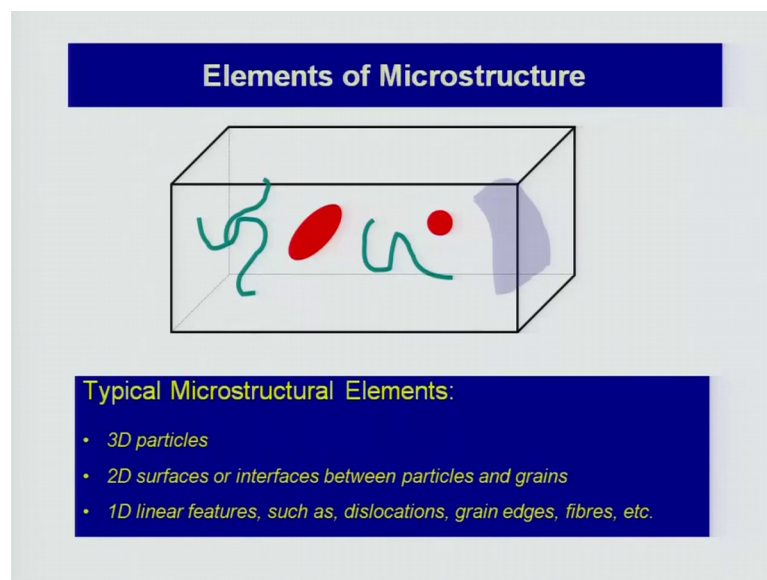
Now, what is stereology the term itself dates back to the 1960's when for the first time this term was invented by metallographic material scientists, where it was required to quantify microstructures. Now what is the problem that that was being addressed at that time? The problem is that our metallic structures are opaque and when we look at a microstructure. We look at a planar section of the structure while the structure of the material itself is a 3 dimensional structure, and what you are looking at is a microstructure is a 2 dimensional image.

Now, from this 2 dimensional image we have to reconstruct or estimate some other parameters of the 3 dimensional structure. For example, we have to find volume fraction for a certain phase. Then what kind of measurements we must make on over 2

dimensional image to be able to estimate the volume fraction, or if you are looking at size of particles then we need to make certain kind of measurements to be able to estimate the size of particles and so on. So, we will we looking at various measures on the 2 dimensional structure to be able to estimate the 3 dimensional parameters of the structure of the material.

So, essentially stereology relates 2 dimensional images of microstructures through the 3 dimensional structure of materials. Quantification of geometrical features of the structure of materials is a key of for stereology. And what does stereology provides it provides mathematical tools to perform this transformation of 2 dimensional measurements on the planar microstructure to the 3 dimensional parameter of the solvents.

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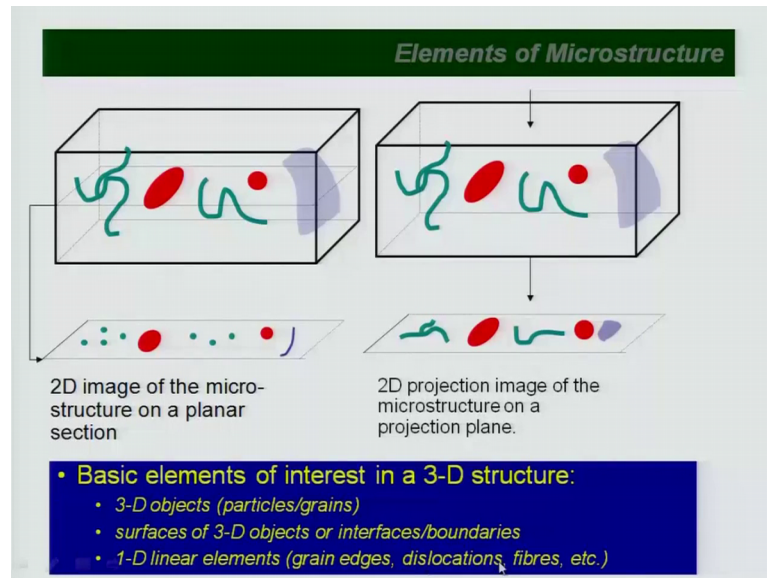


Now, before we go into this let us try to understand what are the elements 3 elements of a 3 dimensional micro structure. So, if you look at this image of a block of material. This block of material it is essentially an abstraction of what kind of geometrical elements that we normally want to estimate. The geometrical elements could be 3 dimensional particles present in the structure of the material, they could be surfaces which grain boundaries of vertical boundaries that would be presents. So, this is shown as a surface here. Or this could be line elements that are shown here there could also be points in the 3 dimensional structure, but here we are just going to focus on these 3 elements. So, the typical microstructure elements are 3 particles, 3 dimensional surface or interface

between particles and grains or one dimensional linear features such as this locations drainages fibers etcetera.

Now, let us see how we get an image out of this 3 dimensional structure.

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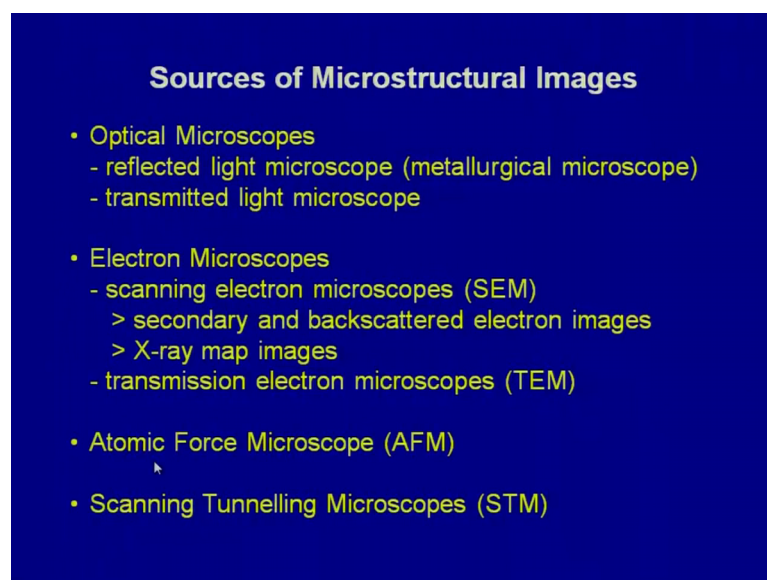
Well, this is one way we produce an image. We cut this a section through the material and then we what we have typically do? If we are going to an optical microscope or scanning electron microscope. We would polish the surface checked to reveal the various elements of a structure, like grade boundaries second phase and other a structural elements. So, if I take a section through my solid and it cuts the various geometrical elements that are presents. For examples 3 dimensional solids that are present are particles that are presents like ones that marked red here, they will get cut into a into a 2 dimensional profiles or area profiles. So now, they would be shown as 2 dimensional images.

A surface bit gets cut it would produces a line. If the line elements that are present like for example, this locations they will produce points. So, this these are the kind of elements that would be present in our 2 dimensional image of the microstructure. From such images we have to make measurable elements to be able to predict for example, what is the volume fraction of these particles, or what is the size of these particles. Or what is the density of these surfaces? Or what is the density of these line elements?

Now, this is one way we produce an image. This would typically be done in a optical microscope or a scanning electron microscope. Now coming to second way of forming an image, in our transmitter light microscope or in a transmission electron microscope. Here the beam whether it is a light beam or electron beam it is passing through a volume of the solid. And we obtain what is called as a projected image. Now if I look at this projected image. I will the particles the 3 dimensional particle will get projected as area on to my image. The surfaces would also get projected as an area, at the line elements instead of being points in the section image now get projected as linear elements or lines.

Hence the stereology that we develop the tools that we develop for making the measurements in the section image here is different from the stereology that is used here. The projection brings in some additional complications; however, for this particular set of lectures we will not be discussing the projected images we will be only concentrating on this section images which constitutes the large part of our quantitative metallography. The basic elements of interest in a 3 dimensional structure just to summaries a 3 d objects particles or grains surfaces of 3 dimensional objects of the interfaces and boundaries of one dimensional linear elements like drainages dislocations fibers optical.

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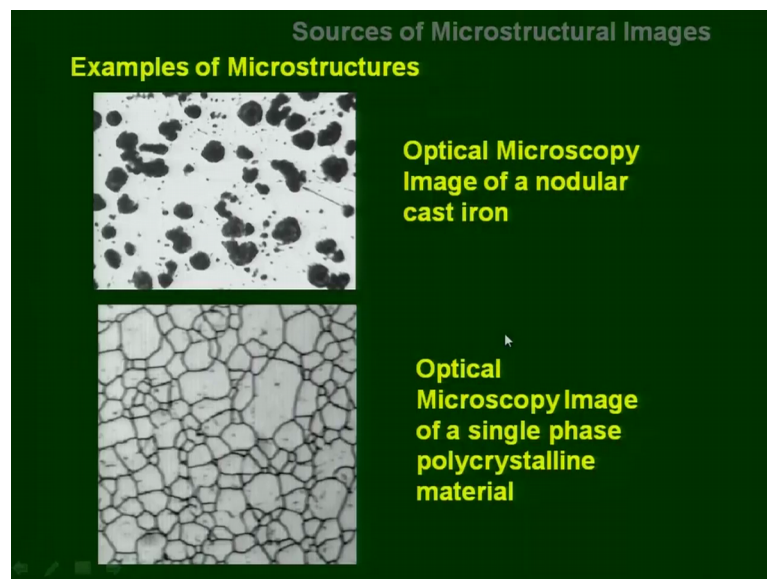
Now, before I go into the actual tools let us look at some microstructural images that we obtain from optical microscopes which could be a reflected light microscope, or

popularly called as a metallurgical microscope, or a transmitted light microscope, but for our metallurgy and material science applications normally it is a reflected light microscope that we are interested in because transmitted light microscope can only be used in those materials which are transparent light for light.

So, this is one way we would get our microstructural images. Electron microscopes like the scanning electron microscope will produce images from secondary electrons and backscattered electrons. Or even x ray map images. Now for most practical purposes secondary and backscattered electron images could be treated as single section of planar section images; however, this may not be true for x ray map images where the x ray is coming from a large volume of the material.

That transmission electron microscope would produce projected images. Then you also have any other microscopes like the atomic force microscope and the scanning tunneling microscope.

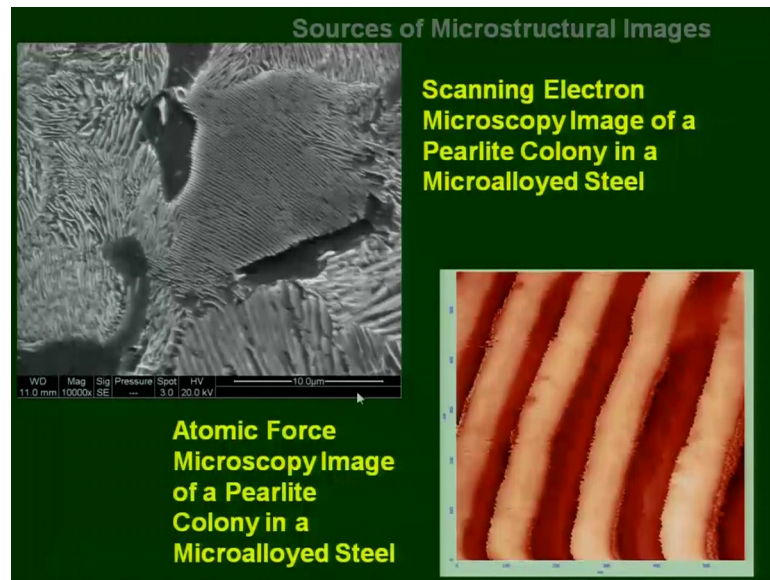
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Looking at some of the microstructures, For example here are couple of microstructures shown from a metallurgical optical microscope. This is a microstructure of nodular cast iron where the dark regions are the graphite nodules. And this is a single phase polycrystalline a material a showing us grain boundaries, and also you can see where 3 boundaries beat triple points, which are actually in the 3 dimensional image are actually lined. So, triple edges. So, using stereology we will see that we can estimate size of this

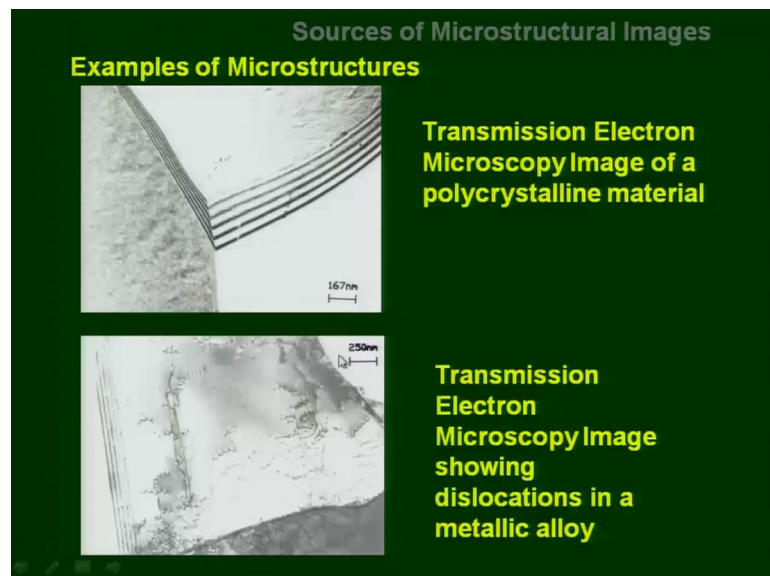
nodules the volume fraction of the nodules. Out here we can estimate grain size of these on an average grain size using stereological techniques. We can even measure what is the density of triple edges, that is the length of these triple edges per unit volume in the 3 dimensional structure.

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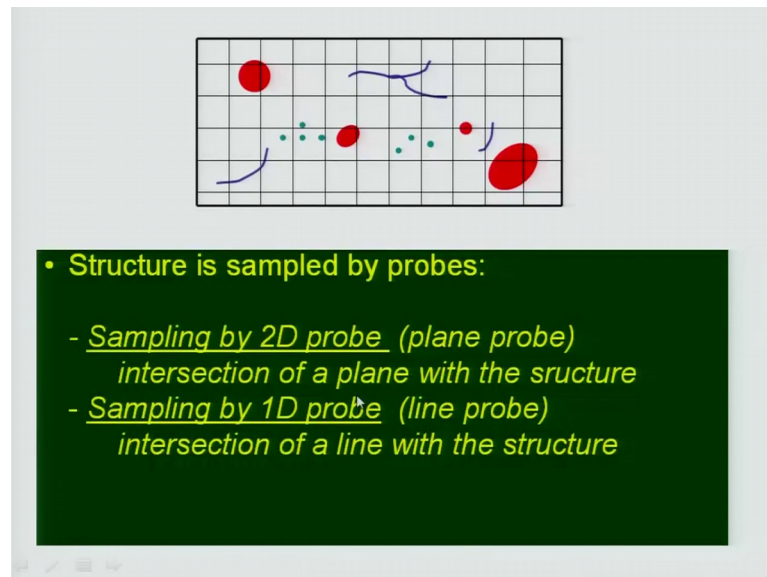
We can this is an image of a few pearlite colonies in a in a steel. Here we may be interested in measuring the inter leveling spacing again stereology can help us in doing that. This is just a magnified image of the same thing in a atomic force microscope.

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Now, these are projected images in a transmission electron microscope of polycrystalline material. This image shows 3 boundaries meeting at a triple edge, while the image at the bottom here shows this location and stereology can be used to determine for example, the density of dislocation which is line length per unit volume.

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Now, let us see how actually stereology is implemented. So, one other things that we need to understand of few terms. One of the terms in stereology are probes which are used to promote the microstructure and the 3 dimensional structure of the material. Or what is called here structure is sample by probes, and here the a the sample yeah the meaning of the sample is in it is statistical sense, that we sample a representative volume of the material in order to estimate. What is the a particular a parameter that we want to measure? Now how is sampling done one is; obviously, when you take a section we are actually sampling the material. So, sampling by a 2 dimensional probe or what we can call the plane section or a plane probe. So, may use section. So, the intersection of a plane with the structure is a process of sampling the 3 dimensional structure.

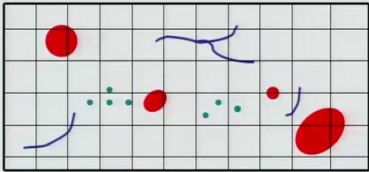
Second why with a 2 dimensional plane we can also section, by a line probe or a one dimensional probe? We can hypothetically thing that we can draw a line through the through the structure, and see the intersection make some measurements with the of the intersection the line with the structure. We will soon see what it all means. Similarly I can go down to the 0 dimensional probe, or the point probe which is the intersection of

point with the structure. If I look at this particular image here at the top we can try to understand what the these 3 probes are doing.

First is a section we have taken. So, this could be 3 dimensional particles for instance and they have section and now I have the area profiles. These are lines that you see which are nothing but the plane probe cutting a surfaces in the 3 dimensional structure. And the points are represented by the plane probe cutting a set of lines. Now what about the one dimensional probe? Well, you can see the lines here why horizontal lines or the vertical lines. Now they are sampling through the 3 dimensional structure for example, I could take these lines and look at what is the length of these lines a within the individual particles. So, intersection of a line with the 3 dimensional object or it could be the intersection of the lines with these line with these linear elements that I see on the on the on the plane probe, or these are lines which are actually cutting the 2 dimensional surfaces.

Similarly, if I look at the 0 dimensional probe we can look at the intersection of the horizontal lines and the vertical lines are the points. So, these are the point probe. So, I could see the intersection of the point with the 3 dimensional particles. And we will see what kind of measurements we can make with these with these intersections.

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Probes interact with microstructural features to produce "events" (e.g., interaction of plane probes with a volume feature produces 2D cross-section):

<u>3D Feature</u>	<u>Probe</u>	<u>Event</u>	<u>Measurement</u>
volume	plane	cross-section	area
surface	plane	line trace	length
line	plane	point-intersection	count
volume	line	intercept-length	length
surface	line	point-intersection	count
volume	point	point-intersection	count

Going to the next slide. What are these probes doing? Probes interact with the microstructural features to produce events now these events are in the sense of

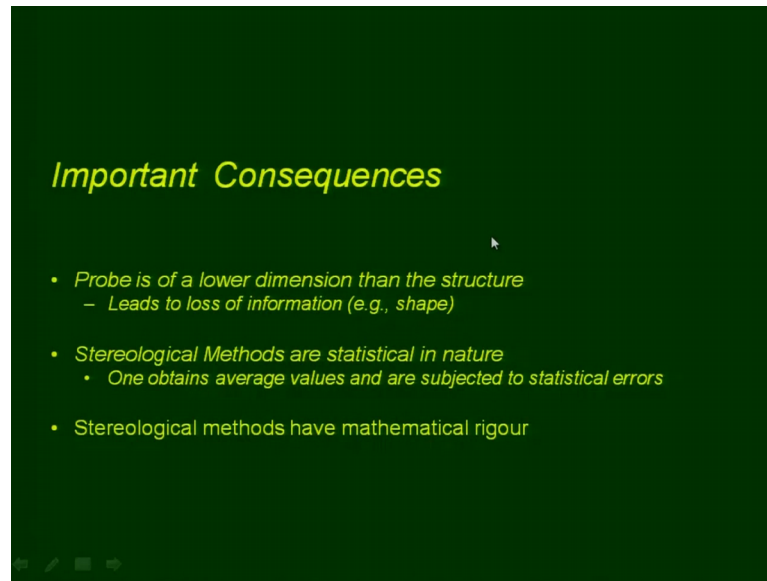
probabilistic events that is there are certain probability of some of these events taking place. For example, interaction of the plane probe with volume features or solid three dimensional particles produces uses 2 dimensional cross section. So, I have an table in which I have given some of the events and what are the kind of measurements I can make on these events.

For example if I the feature is volume which is like a 3 dimensional particle or a 3 dimensional grain a plane probe will intersect some of these volume features or this particles and produce cross sections that are shown here. One of the measurements I can make on this among many other is the area of these cross sections are surface feature, in the 3 dimensional structure intersected by a plane probe produces a line trace. Well, I can measure the length of these lines as a measurement, I can take a linear feature. In the 3 dimensional structure like triple edges or dislocations of plane probe will produce point inter section, and a measurement a simple measurement that I can make on these points is too simply count that how many points I have in a given area.

Similarly, a line probe with the volume will produce an intercept lengths. So, if I look at for example, this horizontal line it makes at intersection with the 3 dimensional particle. And I can measure this length of the intercept contained inside the particles. So, I can make a length measurement. A surface with the line will produce a point intersection, and I can count the number of point in points inter such points for example, if I look at this horizontal line I have only one intersection here. If I count with this I have another one if I look at this I have yet another one if I look at this horizontal line I have yet another one is similarly I can do these things with the vertical lines as well.

Finally I have a volume feature an point probe I have point intersection, and I can make a measurement of counting the number of such points. So, for example, I can count this as one point which is inside the particle I can count another point like this which is inside the particle I can count the third one which is also inside the particle. Now these are kind of measurements I can make and then we will see how stereology can be made to relate to the 3 dimensional structure.

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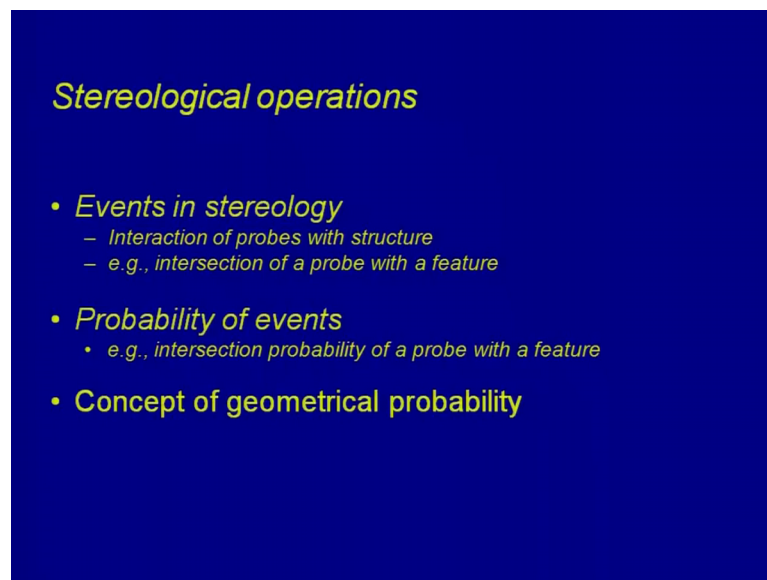


Now, what are the consequences of the intersection of the probe with the 3 dimensional structure? Well, as you have seen probe is a low dimension than the structure like plane probe is a 2 dimensional a 2 dimensional and my structure is 3 dimensional or a line probe is one dimensional or point probe is a 0 dimensional. So, whatever probes I am using have has a lower dimension. And as a consequence this leads to loss of information. For example, a plane probe intersecting a cylinder will produce various kinds of profiles a for example, it can produce circular profiles if the plane products the cylinder perpendicular to it is axis. It will produce an elliptical profile if it cuts at a angle to the axis of the cylindrical particles and I may not be able to tell from the various shapes that I get that these are actually cylindrical particles.

So, this is what it means that I am I one of the things that I lose is information or shape that makes sometimes analysis difficult unless we assume a certain shape. For example, the graphite nodule microstructure I had shown if we assume them to be spherical particles as an approximate shape, then I can go ahead and perform certain stereological analysis. Stereological methods are statistical in nature, and hence they will be subjected to a statistical errors and we have to obtain average estimates. That means, we have to cover large enough a area in the sample to get a good average estimate of the 3 dimensional parameter. And most importantly stereological methods have mathematical rigger, and that we will see soon.

Some other relationships that you may see in some other classical relationships or stereology look appear to be very simple, but they have been rigorously derived. So, we have already seen the stereological operations which are produce events stereology which is the interaction of probes with structure.

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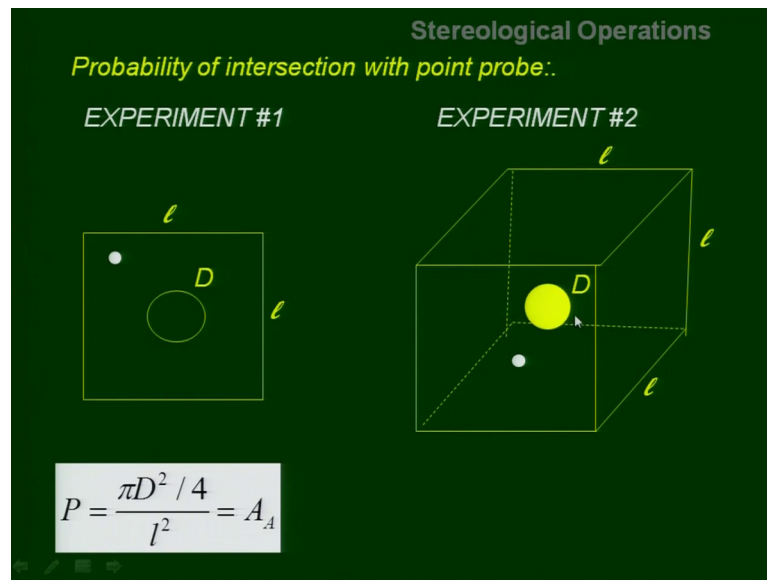


Stereological operations

- *Events in stereology*
 - *Interaction of probes with structure*
 - *e.g., intersection of a probe with a feature*
- *Probability of events*
 - *e.g., intersection probability of a probe with a feature*
- **Concept of geometrical probability**

And we will now see that these events have a certain probability, that what is the interest probability of a particle getting intersected by plane probe. What is the probability of a point falling inside a certain fails? In a inside a certain kind of particles in the microstructure, and hence we bring in the concept of geometrical probability, which is no different from our classical definition of probability as we shall see.

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So, I am going to consider some hypothetical experiments. Let us consider these are all thought experiments. So, this is considered experiment number 1, yeah it is done on a 2 dimensional plane. Let us consider a 2 dimensional box a square box of size l by l . Inside this box I have a shape a circular shape of diameter D . And I randomly put a point anywhere inside this box as represented by such a point. Now this point to I have just been exaggerated dimensions to clearly show it otherwise this point has to be considered as a 0 dimensional point which has no size.

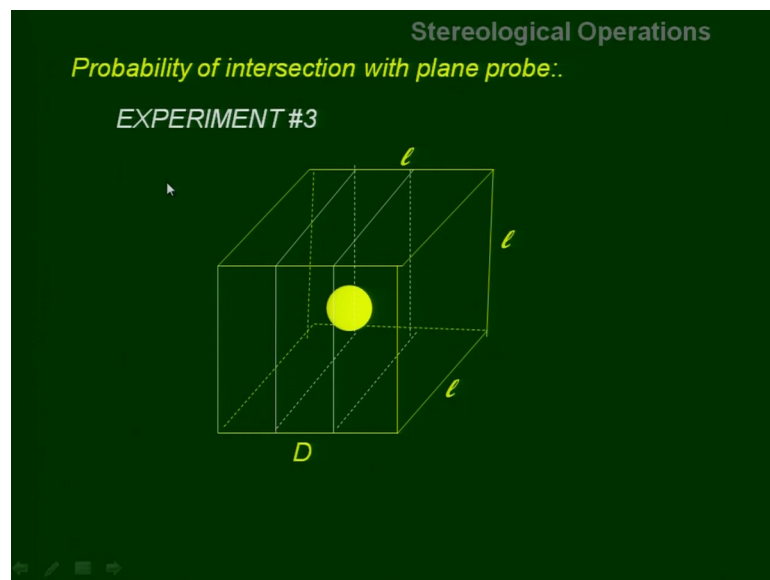
Now, the question that I ask is what is the probability of intersection of this point or what is the probability of this point falling inside this circular shape of diameter D . If you think about it a little bit. It should become evident that the probability of the point falling inside the circular shape is nothing but the area of the shape area of the circle divided by the total area of the box containing the shape. Hence the probability is nothing but πD^2 upon $4l^2$ that is area of the circle divided by l^2 which is the area of the box.

And if you look at what is this ratio this ratio is actually nothing but the fraction of the area occupied by the circular shape inside this box of area l^2 and this on the right hand side I am calling it as area fraction. Similarly I could go to a 3 dimensional space as experiment number 2 where now instead of a circular shape now I have a spherical shape of diameter D inside a box in the shape of a cube of dimensions l by l , and again I ask what is the probability of a randomly placed point inside this cubic box that the point

will fall inside this spherical shape. And this is an analogous problem to the 2 dimensional problem on the instead of areas now we are talking about volumes.

So, the probability of a point falling inside the spherical shape is nothing but the ratio of the volume of the sphere to the volume of the cubic box, and which very clearly then the probability would be $\pi D^3 / 6$ divided by l^3 . And if you look at it this ratio is nothing but the fraction of the volume occupied by the sphere inside the cubic box and this is what we can call it as the volume fraction. This can work both of these, these results can be proved rigorously, but I am going to skip in these set of lectures the rigorous proof behind them.

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A third experiment in geometrical probability could be that I know I have a sphere inside again a cubic box, and I ask the question what is the probability that if I place a random section I cut this randomly in such a way that the cut is made parallel to this particular phase, but that cut can be anywhere along this. So, I could make a cut here.

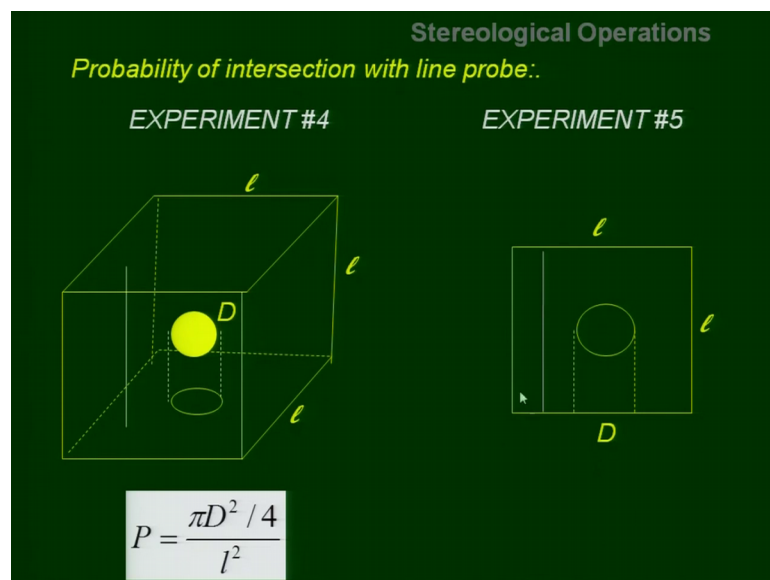
I could make a cut here or I could make a cut here, and it is a randomly placed cut. The question of probability here is what is the probability that a random cut will cut this spherical particle.

Now, in order to look at this consider these 2 planes, which are being drawn in such a way that they are tangent to the opposite ends of this spherical particles. So, this plane

makes a point contact with the spherical particle on this side and this plane makes us a points. Contact on this side of the particle what would be the distance between these plans the distance between these 2 planes would be D. So, the probability of a random cut parallel to these tangent planes would be intuitively we can guess, that it would be nothing but the ratio of this length versus the total length which is nothing but the ratio D upon l.

Similarly, let us have another experiment, where now I have a line probe, which can be randomly placed anywhere inside the cube, but keeping it parallel to this vertical edge.

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There is a spherical particle present and the question again is what is the probability that this line probe will intersect the spherical particle of diameter D inside the cubic box of size l by l by l. Well, this problem can be reduced to actually experiment number 1 of the point being dropped on a 2 dimensional plane and how well project the spherical particle on to the bottom plane. The projection will show up as a circle. Now this random location of this random line is nothing but the location of the end point of this line on the project on the surface on which we are projected the spherical particle.

So, therefore, this line will interested the sphere if this point the end point of the line falls inside the project itself. Hence the intersection of the line with this spherical particle is nothing but the area of the particle divided by the area of this plane, and hence the probability of the line intersecting thus spherical particle is nothing but pi D square upon

4 divided by 1 square. Consider yet another experiment in 2 dimensions and I leave this to you to work this out probability of this line intersecting this circular object in this 2 dimensional box of 1 by 1 is nothing but the ratio D upon 1.

We look now at a couple of more experiments with the one I am looking at is a now take a now on spherical object. Here what would be the probability of a random plane parallel to this face of the cube cutting this non spherical object. As you can see now the 2 tangent planes that I have drawn the distance between these 2 tangent planes would depend on what is the orientation of this non spherical particle. So, this orient the distance h between these 2 planes is now going to vary with the orientation of this particle.

Now, the question is what is the probability of a random plane cutting an object which also takes up a random orientation if it was a fixed orientation. Then the probability would simply be h the distance between these 2 planes which is also called as the tangent diameter of the particle divided by the total length 1. But since the particle also takes up a random orientation the probability then we have to consider what is the average tangent diameter of this particle the main tangent diameter of this particle would be we have to take average of all possible orientations of the particle, hence the probability would be equal to \bar{h} upon 1 where \bar{h} is the mean tangent diameter.