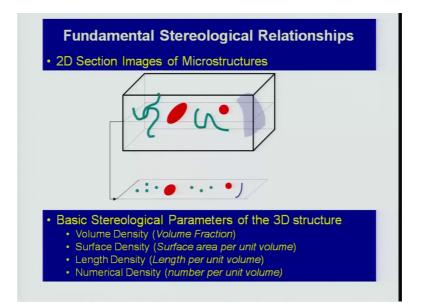
Heat Treatment and Surface Hardening - II Prof. Kallol Mondal Prof. Sandeep Sangal Department of Material Science & Engineering Indian Institute of Technology, Kanpur

## Lecture – 35 Stereology and quantitative metallography – II

In this lecture we will look at now some of the Fundamental Relationships in Stereology, what is called as classical Stereology on which we have all images from a 2 dimensional section.

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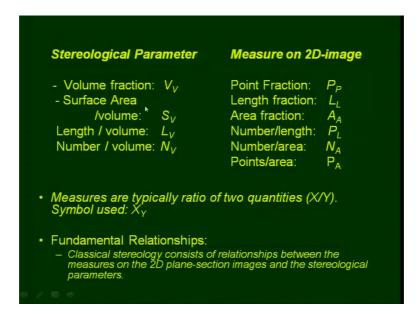


This is a slide I have already shown that how a section produces different geometrical features on a 2 dimensional structure, and what are the stereological parameters of the 3D structure that we are interested in generally Volume Density, Surface Density, Length Density and Numerical Density. Volume density means essentially volume fraction, Surface density is surface of for example, grain boundaries in a unit area it could be surface of interface boundaries per unit area length density.

For example of triple edges just total length of edges per unit volume it could be Length density of dislocations and Numerical density implies number of particles if you are looking at particles in a given area. For example, we will be one may doing a nucleation study in which one wants to measure number of nuclei per unit volume as a function of

time, then one has to go for some kind of a measurement from which one can get an estimate of this numerical density.

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So, here I have a number of 2 dimensional measurements or on our 2 dimensional image and on the right hand, on the left hand side here we are the stereological parameter that we want to estimate.

First look at the measures that we can make for example, if you are using a point probe one may get up point fraction out of that that is number of events from a total possibility of all events it will become clear by I will take an example, similarly there could be a Length fraction with the line probe, there could be an Area fraction with the with a plane probe, it could be Number per unit length which I call it as P sub L, Number per unit area which is N sub A points per unit area which is P sub A, all of these things will become clearer in a little bit in the next few slides, and from these measurements on the right we want to estimate these parameters here on the left, Volume fraction V sub V surface area per unit volume S sub V length per unit volume L sub V number per unit volume N sub V.

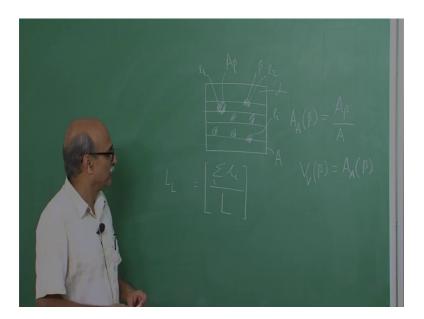
As you can see these are all the measurements as well as a quantities we want to estimate are ratio of 2 quantities X upon Y, in some cases it is dimensionless like point fraction and other cases for example, points per unit area has a dimension of length per, lengths square and so on. And the symbol that we have used is quite consistent as X or Y, what is in the numerator and what is in the denominator becomes clear. Now going to investigate part of classical stereology the relationship that link these measurements to these stereological parameters.

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Methods for Volume Fraction Estimation	
<ol> <li>Areal Measurement         <ul> <li>Direct measurement of area of the phase of interest</li> <li>Calculate the area fraction, A<sub>A</sub> as the volume fraction estimator</li> </ul> </li> </ol>	
<ul> <li>2. Lineal Measurement</li> <li>Use a line probe on a 2D section</li> <li>Measure the fraction of line intercepted, i.e., L<sub>L</sub> as the unbiased estimator of volume fraction</li> </ul>	
<ul> <li>3. Point Counting <ul> <li>Random Point Count</li> <li>Systematic Point Count</li> <li>Coarse Mesh</li> <li>Fine Mesh</li> </ul> </li> <li>Calculate Point fraction, P<sub>P</sub>, for estimating volume fraction</li> </ul>	

Let us first look at Volume Fraction Estimation, this is one way we can estimate the volume fraction and let me just go to the board and just show you what an aerial measurement technique implies. I have 2 dimensional image which is consisting of some second phase particles.

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This could be now alpha phase and this is a beat phase that is precipitating and I want to measure the volume fraction of the beta particles. So, what I do is measure it is area fraction.

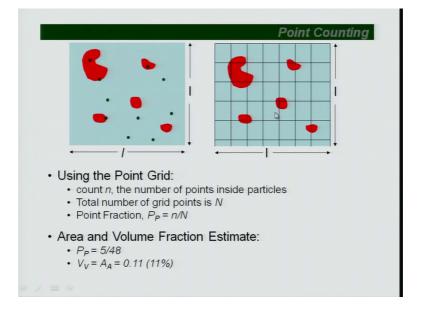
So, area fraction of beta and what is the area fraction of beta well I have to measure the area of these cross sections, if the individual cross sections in the given area of the microstructure. So, this could add up to total area of the beta phase let us says a beta divided by the total area or which of the image or which I am measuring this parameter a beta, this divided by the total area gives me the area of fraction of beta. This we can prove in the stereology though I am not going to prove it here that this area fraction is nothing, but the volume fraction of beta. So, area of fraction is an estimate of the volume fraction of beta similarly, this becomes one way of measuring volume fraction a, second method of measuring volume fraction is using a line probe on a 2 dimensional section this is not a very common technique of measuring volume fraction in metallurgy or material science.

However I would just show you what this means we use a line probe and measure what is called as a fraction of line intercepted L sub L as the estimator for volume fraction. Now let me just understand what this line fraction means again on the board on this image I impose a grid of parallel lines, actually they need not be parallel they need not even be straight, but we impose a certain set of lines and usual practice as well as it is simpler to implement is to put a set of parallel lines and measure the lengths intercepted inside the beta particles.

This is one such length, this is another such length, this is a third such length and so on, add up all of these lengths. Individual lengths L 1, L 2 and so on L I so summation overall all I s is a total length intercepted inside beta, divided by the total length of these parallel lines that I have superimposed on the microstructure. This quantity is what is called as the length fraction or the lineal friction L sub L, this turns out to be estimate of the area fraction and hence this also turns out to be an estimate of the volume fraction of the beta phase here.

Now, as I said this technique is not very popular, we will not dell deeper into this I will come to the most important technique of measuring volume fraction and that is using point counting. Now here I impose a certain set of points on the microstructure and then count all those events which are the points intersecting the beta phase in a 2 phase alpha beta structure. This number of points divided by the total number of points I have imposed on the structure gives me what is called as the point fraction P sub P and this turns out to be an estimate of the area fraction and hence also an estimate of the volume fraction. This is coming from the experiment number 1 and experiment number 2 we had seen in the last lecture the thought experiment in which a point is placed inside a square box or a point is placed inside a cube, in one case I have a circular object, in the second case I have a spherical object, and we had shown or rather we had simply given the result that an intuitive result then the probability of point falling inside the circular shape or inside the spherical object was nothing, but the area of fraction or the volume fraction.

Hence when I do this experiment of and obtain point fraction this is nothing, but actually estimating the probability of point falling inside the beta phase and hence point fraction becomes an estimate of area fraction or volume fraction. Now how do I superimposed the points on the on the microstructure well I can superimpose the point as a set of random points or I can superimpose the points systematically in a form of a grad both will give me the volume fraction. And thus the systematical or a grid of points could be forming a Coarse Mesh or a Fine Mesh this has some statistical consequences, but; however, you will not be discussing this here.



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Let us look at the next slide to make this point counting much more clear, here on the left I have a microstructural image of these red colored particles of arbitrary shape, on which I have superimpose the set of random points and one the right I have the same microstructural image on which I have imposed a grid of points and what is the grid it is a points of the intersections of the horizontal and the vertical lines.

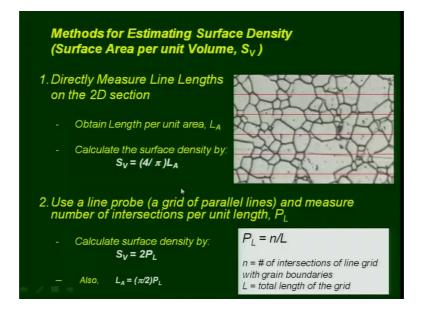
Let us just look at this grid of points both will give me an estimate of the area of fraction and volume fraction hence we will only look at the grid of points, why we will only look at the grid of points well it is far easier to count points in a grid because we have just out of a follow, a systematically or the follow the horizontal lines and count the points in random point count we are able to make many mistakes in accounting.

Let us see using the point grid count N the number of points inside particles. These are this is a point inside the particle this is a point inside particle and so on and we should also know what is the total number of points, if I look at this the total number of points here counting these points as well at the very edge is 48.

So, capital N is 48, number of points in side if I count this as one, this as second, third, fourth and perhaps this also is another point. I may have 5 points inside the second phase particles if we can call them as a beta particles in a 2 phase alpha beta alloy. Then the Area and the Volume Fraction Estimate is nothing, but point fraction which is 5 upon 48 and that gives me about 11 percent volume fraction. So, very simple to implement, but we have to get a good statistical estimate for that one area of the images not sufficient we must make, this measurement in large number of areas to get a statistical statistically a good estimate and we will see a little later that once we have this data we can also analyze what kind of errors are involved in the measurement.

This is what simple point counting does and it is very very easy to implement. So, you have a microstructure. In fact, you can take a printout of the microstructure and put a grid like this on a transparency and just superimposed transparency on the microstructure and do the point counting.

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Now, suppose I want to estimate surface area per unit volume of grain boundaries in this polycrystalline microstructure that is shown here. So, I these are lines grid boundaries are observed as lines on this to 2 dimensional image, but these are actually surfaces in the 3 dimensions therefore, they have a certain surface area and I want estimate the surface density which is nothing, but the total area of these boundaries in a unit volume called as the S sub V, well here if I look at it I could directly measure the total length of all of these lines in a given area obtain length per unit area call L sub A and the surface density then is given by the following stereological relationship S V is equal to 4 by pi is a multiplying factor to the measure of length per unit area.

So, on the right hand side you can see the unit of this equation matches in both sides say units are per length square. Now this is one way of doing it, but; however, the measurement of these lengths are not straight forward it could be tedious and time consuming and hence error prone as well and you would be difficult to cover a large area of the sample. So, there is actually a simple counting procedure by which I can measure the surface area per unit volume of boundaries and this is a procedure one can adopt put a line probe on this microstructure a line probe by as a grid of parallel lines and measure the number of intersections of those lines with the grain boundaries.

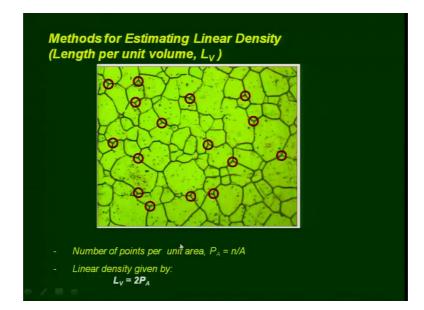
For example, this is the grid of lines I have put I can count this is an intersection with the boundary, this is an intersection with the boundary, this is an intersection with the boundary, this is an intersection with the boundary and keep counting for all of these lines. So, I will get some number n that number I divide by total length of the grid, which

means the total length of all of these lines added together and that is n upon l. This is what is meant by the measurement P sub L number of intersections per unit length and the simple counting procedure gives me an estimate of the surface area per unit volume as simply 2 times this a intersection count per unit length 2 times P L.

So, this is a far easier procedure than this and you can quickly implement this procedure over a large area of the sample to get a good statistical estimate, where do you get this relationship S V is equal to 2 P L, how does Stereology get it, well this relationship is can be obtained by considering one of the real thought experiments of geometrical probability we are done in the last lecture. Where we had looked at intersection of the lines with the with the particles in 3 dimensions and what is the probability of intersection using that we can derive this relationship of surface area per unit volume is equal to 2 P L.

But; however, the actual derivation would be beyond the scope of this course; however, there are many references available which can be looked at as to how one gets this particular relationship, and how to even get this particular relationship independently of this and a third result is also available to us if I just want to know what is the length per unit area which I was trying to measure actually measure, but from this count of P L, I can get the length per unit area as pi by 2 times P L this is also a simple stereological relationship that can come from another thought experiments of geometrical probability. The come to the third parameter estimating of Linear Density length per unit are now where are the linear elements in 3 dimensional here.

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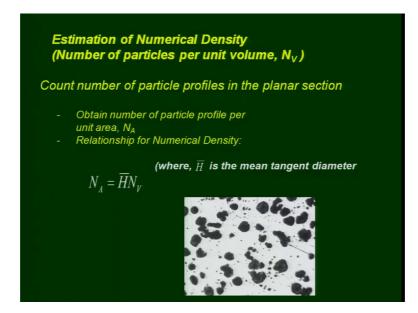
Well the linear elements in 3 dimensions are these triple points that one sees where 3 boundaries are meeting triple points are very important in polycrystalline materials, because these are points where Heterogeneous Nucleation could take place and hence it would be important to quantify the density of such triple points, but that would give us an estimate of the nucleation sites in the microstructure.

So, I fact on the actual nucleation can take place heterogeneously on those triple edges. Therefore, we would like to know what is the total triple edge length in a unit volume, larger this length 5 more such nucleation sites that are available, similarly the larger the grain boundary density again more heterogeneous nucleation sites are available all of that affects our nucleation kinetics and as well as the total phase transformation kinetics. In order to estimate the triple edge length per unit volume as I am calling it as L sub V lengths per unit volume, let us simply do a count all the triple edges all the triple points on this and on this 2 dimensional microstructure. I have not circled all the triple point for available here, but in a given area you have to count all triple points no triple point should be left.

If the area is too large to count 1 can divide this into smaller areas by putting a mask, smaller mask on the microstructure. Get that length get this number of points n divided by the area over which of the image over which this measurement has been made, that is equal to number of points per unit area with the symbol being used is P sub A. Now how is this number of points per unit area related to the triple edge density which is length per unit volume well, it is again a very simple stereological relationship as usual see here is

linear density or length of the triple edge per unit volume l sub V is given by 2 times this count a very very simple measure again this can be implemented very easily this count and a large area of the sample can be covered giving us a good estimate of the of the linear density of triple edges. Again you can see that the units on the left and the right match this is essential to make sure that our relationship is correct.

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This is last of the fundamental measure that I am going to talk about which is the estimation of numerical density number of particles per unit volume N sub V. So, this is the microstructure this is a microstructure of nodular cast iron and I want to count the number of nodules per unit volume.

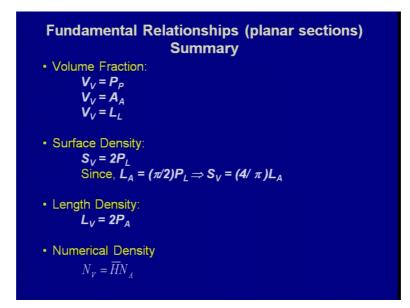
Now how to do that well on my 2 dimensional image I can count the number of nodules in a given area, hence I can obtain the number of particle profiles per unit area N sub A. I simply count the nodules and divide by the total area, now how to do an actual count I will be coming in this lecture just in a short while how to do a proper count unbiased count of these particles as we will see soon.

We obtain number of particle profiles per unit area and how to related to number of particles per unit volume well this relationship that was there was wrong I have corrected it. It is the number of particle profiles per for unit area that will make a measure on the 2 dimensional section is related to H bar which is the mean tangent diameter of these particles times the number of particles per unit volume. So, from here if I know what is

the mean tangent diameter of these particles, then from this measure of number of particles per unit area I can make an estimate of the number of particles per unit volume.

Though this relationship looks very simple we have a problem with this relationship the problem is that N A is a simple measure, however the mean tangent diameter of the 3 dimensional particle is still not known from this single section and unless we make a certain assumption about shape we would not be able to estimate the mean tangent diameter. If you assume that these graphite particles are spherical almost spherical then there are techniques for estimating the mean tangent diameter which will not being discussed here and then working relationship can be obtained to get the numerical density of the graphite nodules.

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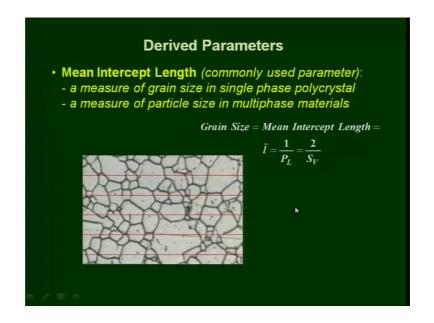


Just to summaries what are the fundamental relationship we have covered well Volume Fraction, can be estimated by point counting as point fraction, measure actually measuring the area of the phase of interest as area fraction or using a line probe and measuring a line fraction.

Similarly Surface Density is equal to 2 times the number of intersection count per unit length or the density as 2 times the number of points that are observed on the 2 dimensional structure in a unit area or just now we saw the Numerical Density as I and again you note that this relationship is reversed out N N V should be on the right hand side as in the previous slide and N A should be on the left hand side.

So, the otherwise the as you will see the units do not match. So, this relationship is incorrectly put you can correct it by putting changing N A to the left and N V to the right.

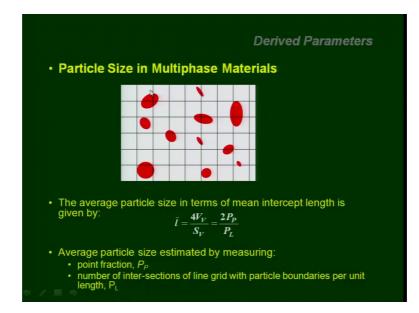
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Couple of Derived Parameters for example, we want to measure grain size and particle size here in materials what kind of a measure we can use a commonly used parameter is the mean intercept length, for grain size as well as particle size now how to let us in this is a polycrystalline microstructure and I want to get an average grain size for this. So, I superimpose the line probe on this microstructure do account of the intersection of the lines with the grain boundaries and obtain the intersection count per unit length as I had done before in order to get the surface density of boundaries.

The reciprocal of this measurement 1 upon P L is nothing, but the average length of these lines that are intersected within the grain and this is a very standard measure that is used as a measure of average grain size of the polycrystalline material and it is interesting to see that the average grain size is related actually to the surface density as 2 upon S V. If you look at what is the relationship between S V and P I than this 2 upon S V would become clear. So, as surface density increases very clearly the average grain size must reduce as expected, a second measure for particle size can also be used as a mean intercept length, but how to do that measure well here I have a set of arbitrarily shape particles and I want to get an average intercept length of these particles.

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Now this can also be given by a very simple relationship L bar is equal to 4 times the volume fraction of these particles divided by surface area per unit volume. We already know how to get the volume fraction by doing a point count so 2 times well simply not 2 times of the volume fraction I replace it with point fraction and S V is equal to 2 times P L. So, I substitute here 2 times P L to get a result of 2 times of point fraction of on P L. I have to just to count 2 methods of counting I put this grid get a point count get point fraction and then using the same grid used this horizontal lines as a line grid and do a front of P L getting this I will get the mean intercept length or the particle size.

The next lecture we will look at how to count grains and particles and we will also look at how to you know get average values from our measurements and the error associated with those measurement with this I close this lecture.