Elementary Stereology for Quantitative Microscopy
Prof. Sandeep Sangal
Department of Materials Science and Engineering
Indian Institute of Technology, Kanpur
Prof. S. Sankaran
Department of Metallurgical and Materials Engineering
Indian Institute of Technology, Madras

Lecture – 16
Counting of grains and particles

So, in the last lecture we were looking at Methodology for counting of particles.

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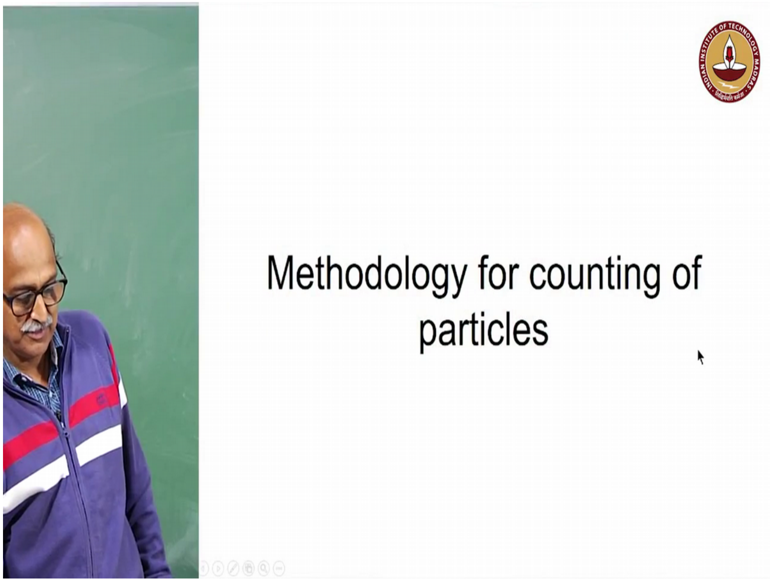

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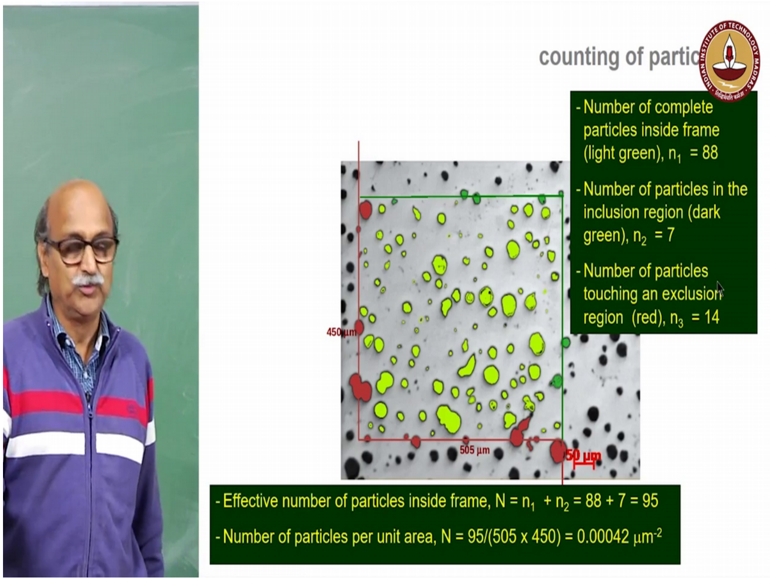

And very quickly if we just repeat this methodology that you put this kind of a counting frame which has a inclusion zone marked with green lines and an exclusion zone marked by the red line. So, any particle which touches the red line would be excluded from the count; particles touching green lines will be included and particles inside the frame would be included. Based on this we were able to separate out these 3 kinds of particles.

So, there were 88 particles which are completely inside 7 touching the green line and 14 of them touching the red line. Based on this 14 we will not include in the count, we will just include 88 and 7 giving us a count of 95 in this frame and then from that you can calculate number of particles per unit area. Now, this particular idea of a count on 2 dimensional structure which does not rely on what kind of shape of particles there are, particle can be of any shape and you can still count them.

This idea can be extended to the 3 dimensions and then you can do a count and get the actual numerical density n v in a unit volume without knowing their mean tangent diameter, without knowing the shape, that can be particles of different shapes you would be able to count those particles as well.

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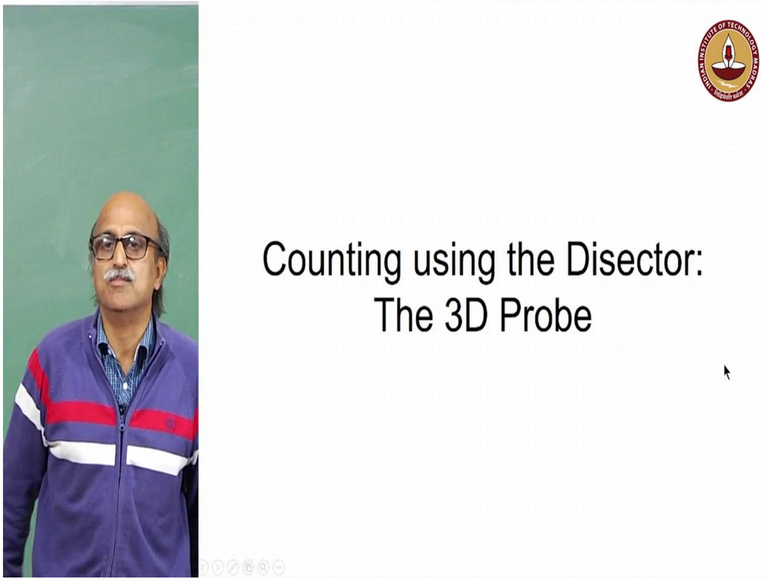

This requires an introduction of a new kind of probe; so far the kind of probe we have been looking at are the plane probe, the line probe and the point probe. Now, we introduce this so, called Disector the 3 dimensional probe which would enable us to do a count in 3 dimensions even though our material is typically are opaque and we are not able to see we are not able to pass light through them.

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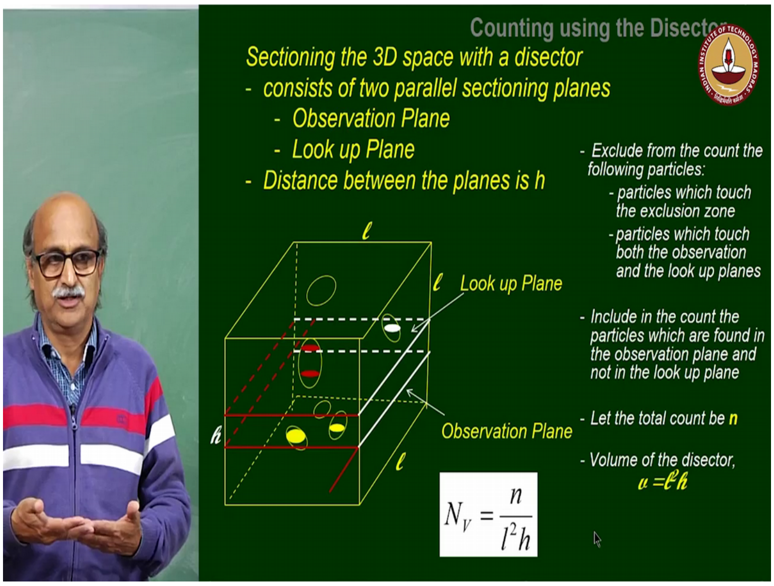

So, let us first look at how this, what is this disector the 3 dimensional probe, it consists of 2 parallel sections 2 parallel plane probe see.

One we will refer to it as the observation plane and the other we will refer to as a look up plane and these two, 2 dimensional sections are separated by a known distance h ok. So, I look at the microstructure on these two sectioning sectioned planes individually ok. Now, how can we do that, well if a materials are opaque which typically they are in your metallography sample preparation; you will first observe on one plane then you will remove some of the materials by polishing ok

By polishing some of the material is removed and there you basically you will get another plane on which you will again observe the microstructure. One of the planes we will call as the observation plane otherwise a look up plane we can reverse them it does not matter which one you call as the observation plane which one you call as the look up plane.

The other thing is we must also know the distance h between these 2 planes which means in a sense we must know how much material we have removed after polishing. Now, an auto material that we typically be removing will be very small because if you are dealing with particles of just a few microns we will remove only material of also couple of microns not more than that. So, we must have some technique by which we should be able to estimate accurately how much material is removed so, that we get a distance between the 2 planes.

We will look at the methodology later how to get h, first let us look at the method methodology of the count both the planes have this counting frame that we have already looked at in the 2 dimensional count. So, exclude from the count the following particles in each of the planes particles which touch the exclusion zone, exclude them particles which touch both the observation plane and the look up plane you have to exclude.

So, if I look at this figure here this particular particle has a section in both the plane the observation plane the section is here and the look up plane the section is here this particle will be excluded from the count. While this particular particle has only a section in the observation plane this particle is a has a section only in the observation plane which will be included in the count.

So, in the end which are the particles to be included in the count, include in the count the particles which are found in the observation plane and not in the look up plane which means that this particular particle which is section is in color is in white color this will not be counted because this is in the look up plane only and not in the observation plane.

Once we get this count of only those particles which does not touch the exclusive zone in the observation plane and those particles which are which are only in the observation plane and do not show up in the look up plane that particular count let that count be n, then this is the effective number of particles present in the disector volume ok.

So, the disector which consist of 2 parallel planes the volume in between these 2 disector plane is the volume of the disector which would be nothing, but area of one of the sectioning plane and that would be l square multiplied by h and that is why you need h.

So, volume of the disector is l square h and the numerical density number per unit volume of the particles is this total effective count n divided by l square h. In this particular methodology of counting we do not need to know the shape we do not need to know the mean tangent diameter we are directly doing this count and. In fact, this method will enable us to obtain the mean tangent diameter because now that I know N v.

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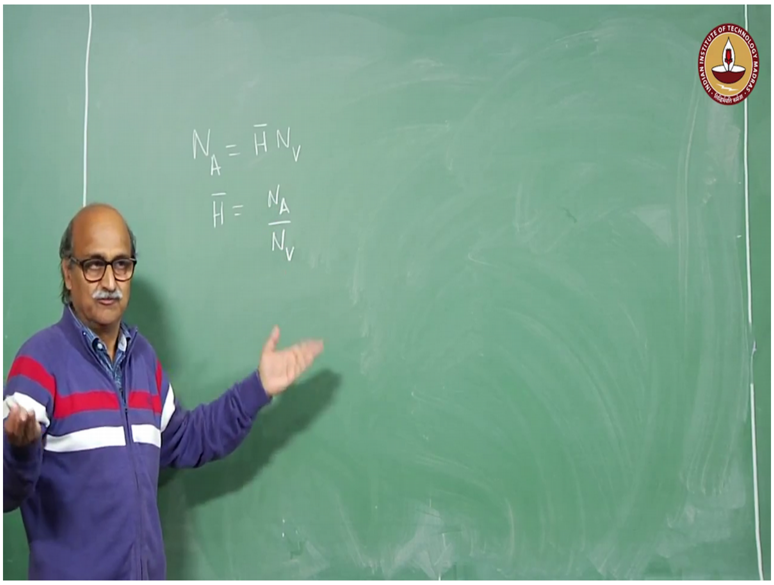

Then it is a simple matter that we already know that N A equals H bar N V and hence we would get the mean tangent diameter as N A upon N V.

So, N V we get from the disector N A is the effective number of particles on a section which also we can count. So, this method that is quite a powerful method somewhat tedious of course, because you will have to do this count carefully on the 2 planes you have to then exclude the particles as per the rules that I have already stated and you have to do a very careful experimentation. So, that you get a very accurate value of the distance h between the 2 planes.

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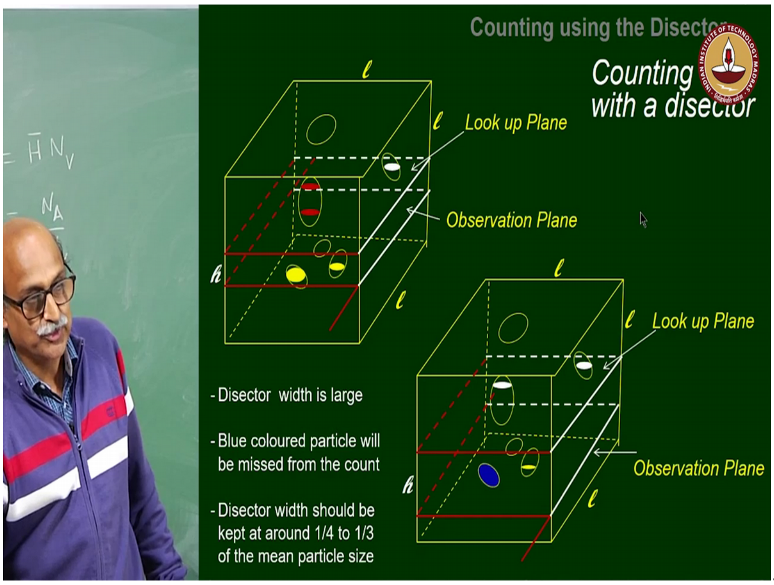

Now, something more on the distance h if we do not choose a proper spacing between the 2 disector planes then there can be counting error and hence you will get erroneous value of the count. Now let us consider 2 disectors here, one is a disector with some h value and here it is a disector with a larger h value.

Now in this particular case the error can occur as a result that this blue colored particle that you have seen is in between the 2 planes, but it does not intersect the it does not intersect either of the planes. So, it will not show up in either of the planes it will be missed from the count ok. So, this will be missed from the count and hence you will get and if you have larger number of such particles you can get large errors in your numerical density.

So, general thumb rule you can take it as that disector which should be kept around one fourth to one third of the average particle size that should give you a reasonably good count. Now, let us look at how we can obtain the value of h what could be a method of getting an accurate value of h any anything you could think of take a.

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FIB, oh no we do not want to go to very sophisticated techniques like FIB; yes you could you should be able to get it yes. But we are not looking at very we are not going into nanometers, we are going into nanometer then FIB could be a good idea, but otherwise something very simple.

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But that you do not know that is what you want to find out so, ok. So, here is a very simple way of finding h and very accurately as well, you are all aware of the micro hardness indenter right it is a pyramidal in shape. So, what you do is, on the first plane on the first section you put an indent.

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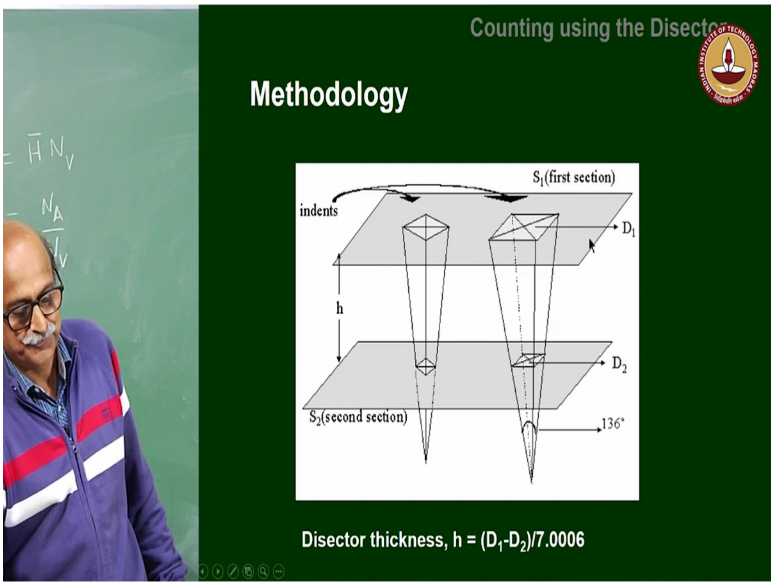

It will have a certain dimension the diagonal lengths D 1. When you remove the material this diagonal lengths will go down right this much material has gone. So, your indenter should be long enough of course. So, now, on the second plane you will see an indentation which is smaller and which can be measured as D 2.

Then simply the disector thickness because we know the geometry of this indenter with the Vickers indenter; if we take the Vickers indenter there from the geometry this is the relation we get for h. The difference in the 2 diagonal length divided by approximately 7 yeah that gives you h and so, this will give you a very accurate result.

One of the things that you should be careful about is when you get the first section do not put the indent immediately; first etch it because otherwise etching will ruin the geometry of the indent. So, with first etch then put the indent and you will put several indents not one and you will take an average value for h. Then after that when you want to reveal the second section you will polish it, polish some of the material of then measure the indent before etching on the second section that is what you have been careful about and that way you would be able to get h.

Other thing that these indents are going to help you is you want to observe the structure at the same in the same area. So, if you put your indents properly you can locate your area after removing the material. So, indents will give you the disector thickness and it will also enable you to locate your area because you have to go back to the same area and do the count.

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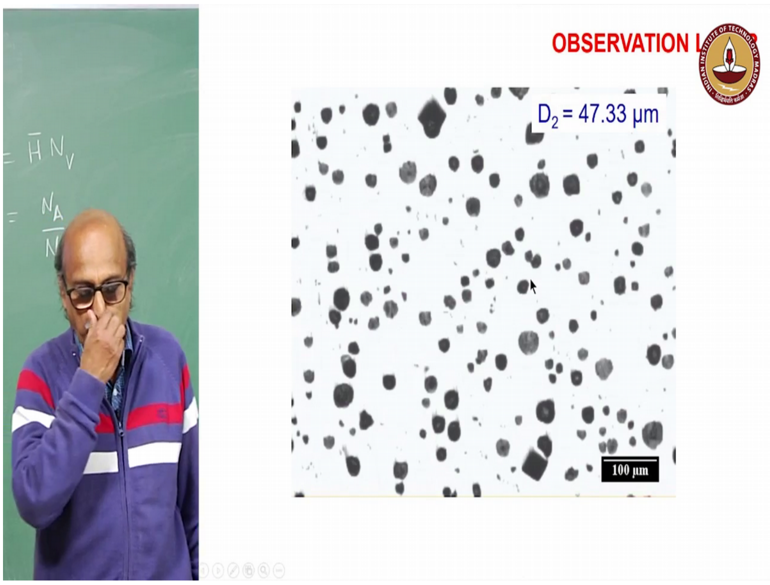

Now, here is one example that was taken so, this is let us say it is called observation layer this is the look up layer.

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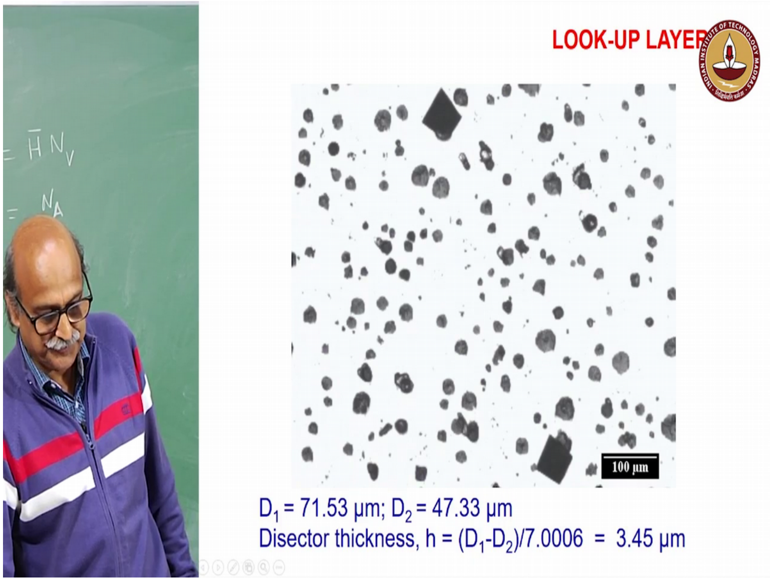

Now, as you can see this was the basically the first layer that we took. So, this indents are sharp this is an this is that micro hardness indent, the second layer which we have called it as observation layer; the indents have become somewhat diffused here because of etching.

So, before etching one would have measured it, so here this diagonal length is 47 microns for this the diagonal length is about 71 microns and using this relationship we get a spacing between the 2 sectioning planes as 3.45 micrometers and this is done by measuring at least 7 8 indents and an average value is obtained.

Now, this is my observation layer.

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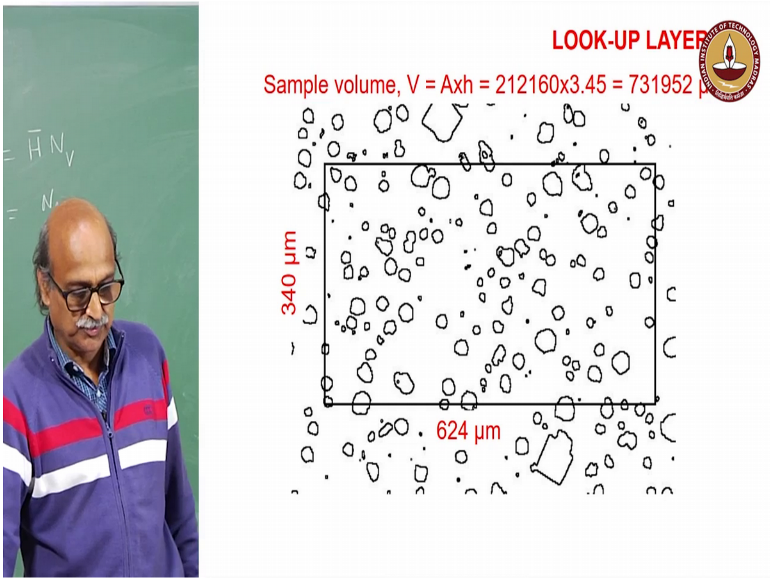

I have done some image processing to reveal only the contours of the grief nodules and removed some of the other so, that counting is easy put the counting frame ok. So, this counting frame is put as before and then do count of this.

So, the particles which are retained in the count are the yellow particles and that number comes out to 95 and if you divide by the area which is 3 624 into 340, you get a number per unit area as 4.4 7 into 10 to power minus 4 per micrometer square.

Now, go to the look up layer we again just reveal the contours put the counting frame.

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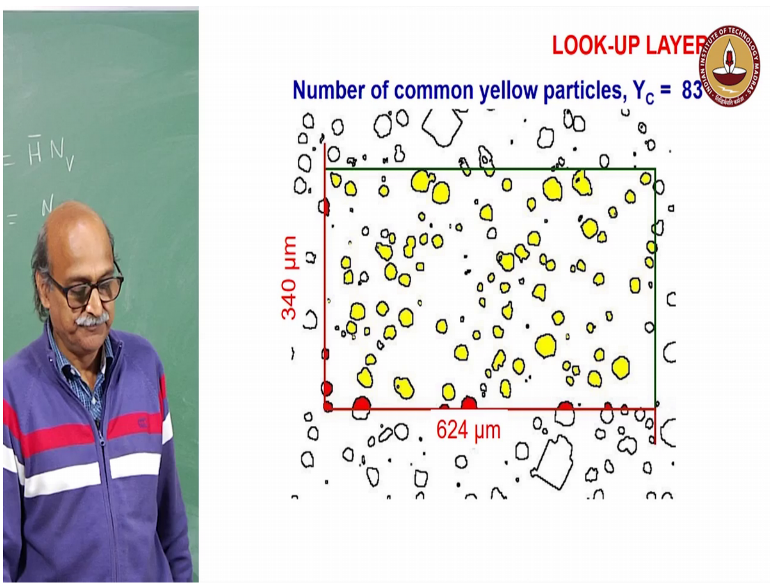

And again counted only the yellow particles, now it is a question that you have to now compare which are the common particles.

So, here are actually the here the yellow particles which are retained in the count, but they are also common with the observation layer and they are 83 in number and we had a total of in the observation layer 95; that means, the difference would be the particles which are only in the observation layer and not in the look up layer and they have to be retained in the count.

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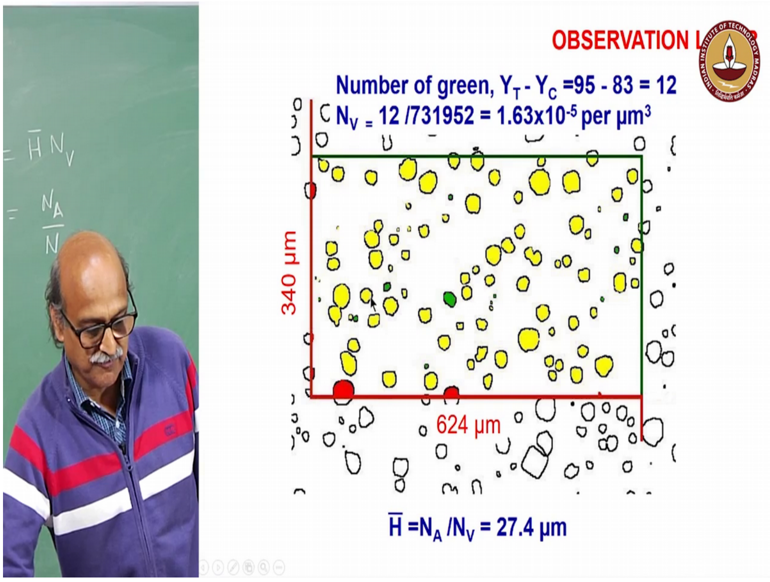

So, which I have now ref put them as green colored particles in the observation layer so, these are the particles which are retained in the count the yellow ones here are excluded from the count because they are appearing in both the observation layer and the look up layer.

So, there are only 12 particles 12 such particles which are only in the observation layer and not in the look up layer. This number 12 you divide by the volume of the dissector, so, the area of the disector plane you know multiplied by h which we had calculated as what 3 point which was 3.4 5 microns. So, one would multiply it by 3.4 5 microns to get this as the volume of the disector.

And this gives me a number density of 1.6 3 into 10 to power minus 5 per micrometer cube. So, I had really got the actual numerical density of the graphite nodules, I can get the main tangent diameter as N A upon N V and that turns out to be 27.4 microns. Now you can also just flip the layers what you called as the observation layer, you can call it as the look up layer and what you call it as the look up layer, you can call it as observation layer and do a count again. So, you will get from the same disector you will be able to get two counts and you can take an average of that.

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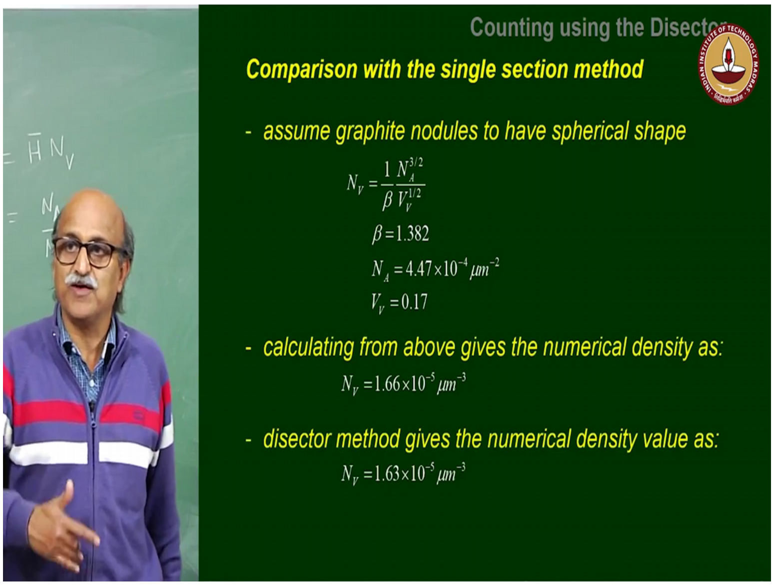

Now, just I just want to give you that if our shape assumptions are then what kind of a count I would, what kind of a numerical density I would have got for the same material based on only a single section count. Then remember from the last lecture that numerical density can be given by this relationship where beta is a shape measure N A is the number per unit area on a section and V s of V is the volume fraction.

Assuming spherical particles near about spherical particles so, then we can take the shape measure beta as 1.3 8 2 from the last lecture n a I just now mentioned on that, but one section was this much 4.4 7 10 to power minus 4 volume fraction of these particles were measured using point count that gave me 0.1 7 and calculating from this it gave me a numerical density of 1.6 6 into 10 to power minus 5 per micrometer cube and the disector method gave me a density which was very close to this density as you can see.

So, perhaps our assumption of spherical shape is if our assumption was wrong then these 2 values could be very different.

So, this kind of concludes some of the basic stereology related to single section plus an introduction to the disector. But so, far what we have assumed is that our materials are isotropic, which means that whatever features we are looking at they are randomly oriented they are randomly distributed.

Now, you can have microstructures which are not randomly oriented; what are they have some kind of an orientation and a good an extreme example could be if I had a fiber composite material and all fibers are in the same direction that that is the example of a highly oriented structure.

Somewhat less oriented structure could be I roll a material grains become elongated in the rolling direction then again not all directions of the grain boundaries are have the same uniform orientation there is a preferred orientation in the rolling direction. Then some of these methods will not work directly you have to modify their working you have to be careful with such microstructures and that is what we are going to see now.