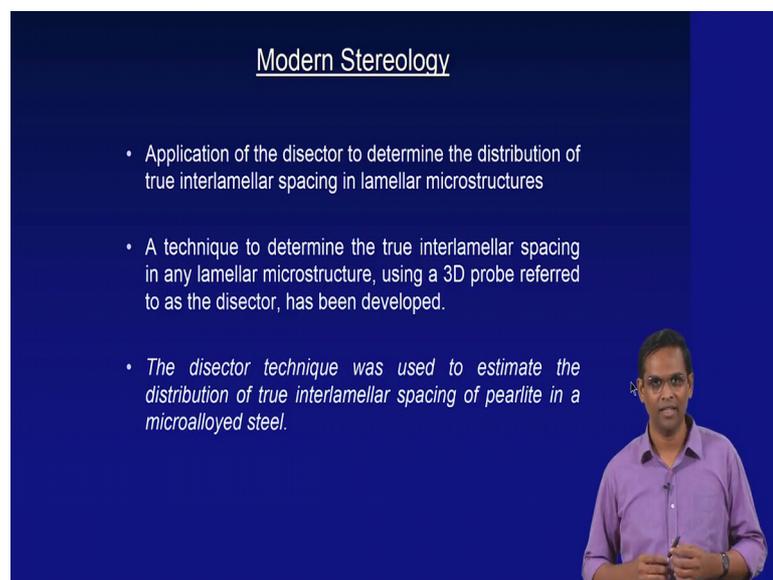


Elementary Stereology for Quantitative Microscopy
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Lecture – 18
Other Applications of the Disector

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Modern Stereology

- Application of the disector to determine the distribution of true interlamellar spacing in lamellar microstructures
- A technique to determine the true interlamellar spacing in any lamellar microstructure, using a 3D probe referred to as the disector, has been developed.
- *The disector technique was used to estimate the distribution of true interlamellar spacing of pearlite in a microalloyed steel.*



Hello everyone. Welcome to this tutorial session. In the last lec theory of the stereology course, you would have observed that process angle talked about disector right. It is again a point probe, blind probe. This is a 3 dimensional probe ok.

Sometimes it is referred as a using disector for microstructural analysis also considered as a modern stereology in a at times. So, what I am going to do today is, I am going to just demonstrate some of the case studies how this disector is being used in the quantification of microstructures.

So, if you look at this slide, what I have shown is the application of disector to determine the distribution of true interlamellar spacing in a lamellar microstructures. So, in general, in a any lamellar microstructures, when you look at the especially when you look at the microstructure under 2 D plane in an optical microscope, what you have to understand is, you are cutting a very random section. So, a plane which you are or you are polishing is

a random section of arbitrary orientation of the lamellar. It could be anything any lamellar eutectoid sorry, it could be lamellar microstructure or it could be pearlitic structure and so on ok.

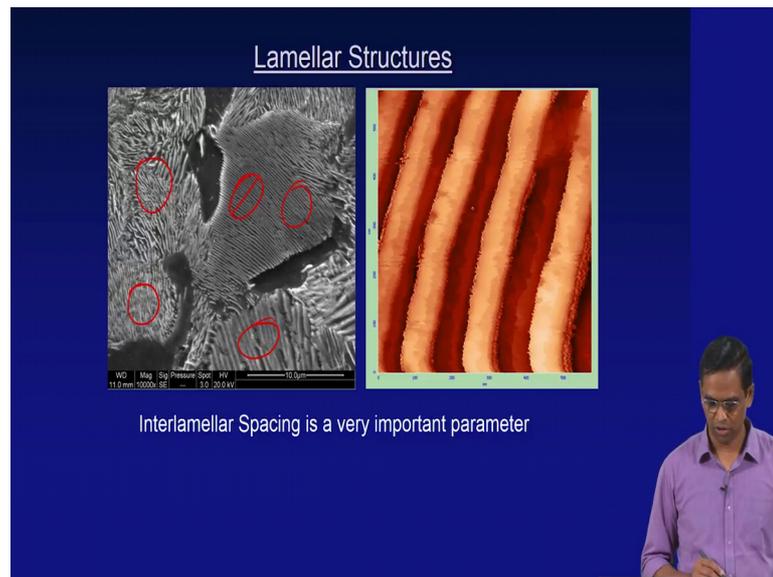
So, normally what we do to measure the interlamellar spacing, we take a polished section and take them micro recording the micrograph and then take a scale and put it on the 2 D section and then measure the interlamellar spacing. What we are now going to demonstrate is, what you actually measure on a 2 D section of a interlamellar spacing may not represent the actual true interlamellar spacing. So, we are going to demonstrate today, how a true interlamellar spacing can be found out using a disector technique.

The technique what we have developed or demonst going to demonstrate is for a generalized a technique. It can be applied to any lamellar microstructure but for this particular case study, we have taken the some a well known microstructure called a pearlite microstructure or pearlitic microstructure.

So, as I just said that this technique to determine the true interlamellar spacing in any lamellar microstructure using a 3 D probe, referred as a disector. And later, we will show how this can be used for some other application; for example, the dihedral angle of the grain boundaries which also I am going to present it after this.

So, the first one, which I am going to demonstrate is to estimate the distribution of two interlamellar spacing of a pearlite in a micro applied steel.

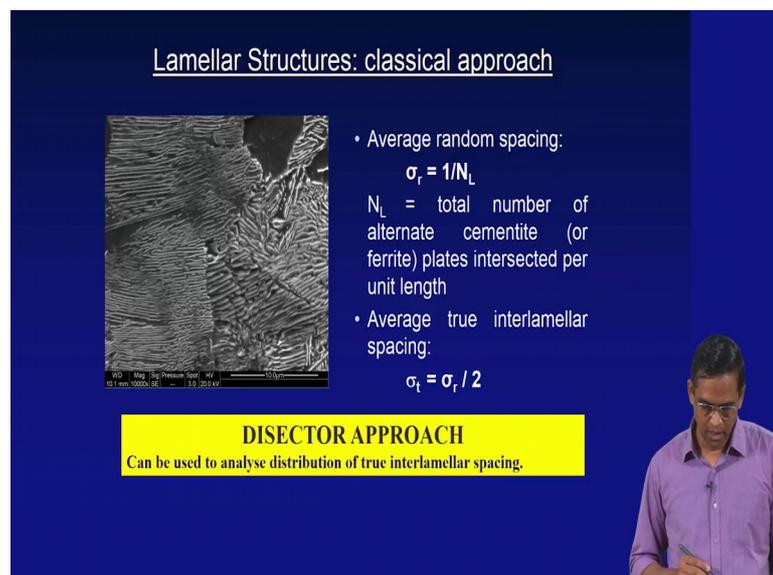
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So, this is what I was mentioning in the beginning. If you look at this micrograph, this is a typical ferrite pearlite microstructure where you see that the cemented plates, platelet us are just intersecting the plane of polish as a the relief.

You can see that the normally what we do is we just measure this length and then take the average. So, that interlamellar spacing also is going to vary for the different different colonies like this. And, and the right one is a very high magnified image of these cementite and ferrite region. It is done by atomic force microscopy.

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So, what is done in this approaches, to have the random spacing what we are actually measuring in a 2 D section is random spacing which is σ_r is a total number of alternate cementite or ferrite plates intersected per length that is $N L$, the average the true interlamellar spacing is σ_t is σ_r by 2.

So, to estimate this kind of a parameter, we can use this disector approach.

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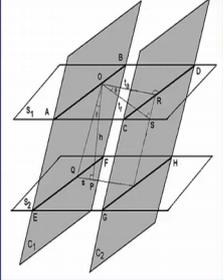
And if you look at this microstructure the, this is an again and a atomic force microscopy, image of a pearlitic colony where you can clearly see that the cementite plates platelet us all; just is a stack of plates in between the ferrite region. So, what we are interested is actually the spacing between these two or the set of cementite plates.

You can clearly see that this spacing is not a uniform, even across the colony here, you have a very different spacing and here it is very different spacing. Please understand this spacing, even though it is cutting the plane of polish it is not a true spacing.

Whatever you measure it is not a true spacing, it is going to we will just demonstrate how these are all this the 2 D measurement what you are going to make for a interlamellar spacing is going to be very different from the true spacing.

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New and Novel approach to characterization of lamellar structures: *DISECTOR*



Now from triangle OSR →
 $t_r = t_m \cos\theta$ (1)

and from triangle OPQ →
 $s = h \tan\theta$ (2)

Therefore,
 $T_r = (T_m \times h) / \sqrt{(s^2 + h^2)}$ (3)

Here
 T_r = True spacing between lamellae plates
 T_m = Measured distance between pearlite lamellae
 h = Depth of the material layer removed
 s = the shift distance of cementite plate with respect to lookup plane.



So, to do that, there is a geometrical model which has been developed. If you try to understand this you mean I mean this model, what you are seeing here is that C 1 and C 2 are the two parallel lamellar which is oriented in arbitrary direction. And then you, you know that how the disector is defined which process angle already explained. You have a look up plane S 1 and then observe observation plane that is in S 2.

You see that this the disector is intersecting the lamellar at again in arbitrary orientation at the point the intersections are noted as A, B and then C, D, E, F and G, H and if you try to draw a line here t a, that is in apparent spacing and which is again a perpendicular to this C D and you draw try to draw a perpendicular to this plane C 2 which is T r that is true distance, not true spacing or true thickness whatever it is.

And you can just, the distance between the two disector plane S 1 and S 2 is H that is how it is described. And now, you see that the if you look at this triangle O S R, O S R; t r can be written as t a cos theta ok. And from the triangle, O P Q, O P Q, you can write s is equal to h tan theta.

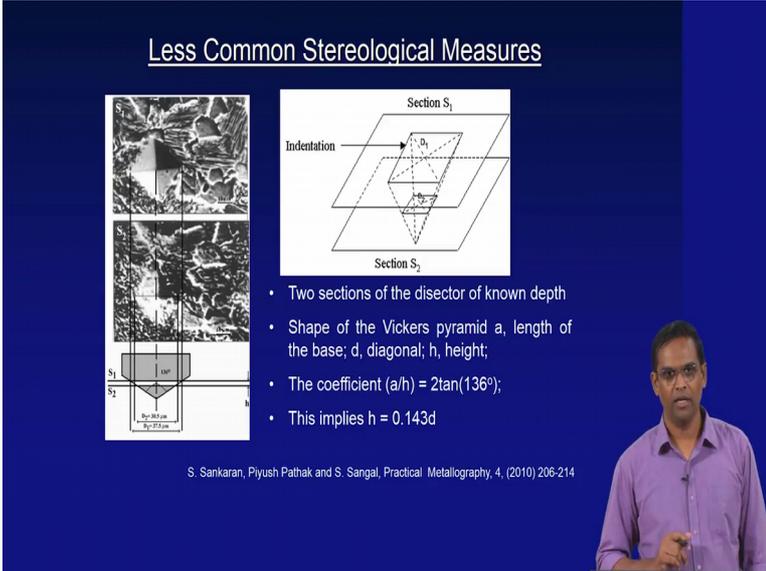
So, if you combine these, you can write T r is equal to T m into h divided by square root of s square plus h square where T r is a true spacing between the lamellar space. So, and T m is the measured between pearlite lamellar, h is a depth of material, material removed layer by layer and s is the shift distance of the cementite plate with respect to the look up plane.

So, what you measure here as an s is how that shift of this lamellar is oriented with respect to look up plane as well as the observation plane. So, this is what you have to understand. So, this is the about the complete description of the geometrical model about two lamellar oriented in a with an arbitrary θ and they were disector is again cutting these two lamellar at arbitrary sections.

So, how do we experimentally do this? This is a of course a model.

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Less Common Stereological Measures



- Two sections of the disector of known depth
- Shape of the Vickers pyramid a , length of the base; d , diagonal; h , height;
- The coefficient $(a/h) = 2 \tan(136^\circ)$;
- This implies $h = 0.143d$

S. Sankaran, Piyush Pathak and S. Sangal, Practical Metallography, 4, (2010) 206-214

So, how do we experimentally perform this and to estimate this? So, here you see the actual micrograph of this ferrite pearlite microstructure of a microalloy steel where you have, what you are seeing this is an S_1 that is a look up plane and the methodology is very simple. So, you make an indent like this and you just first polish the surface and then and then make an indent. If you make an indent and polish and then your indent indentation will have the itching effect.

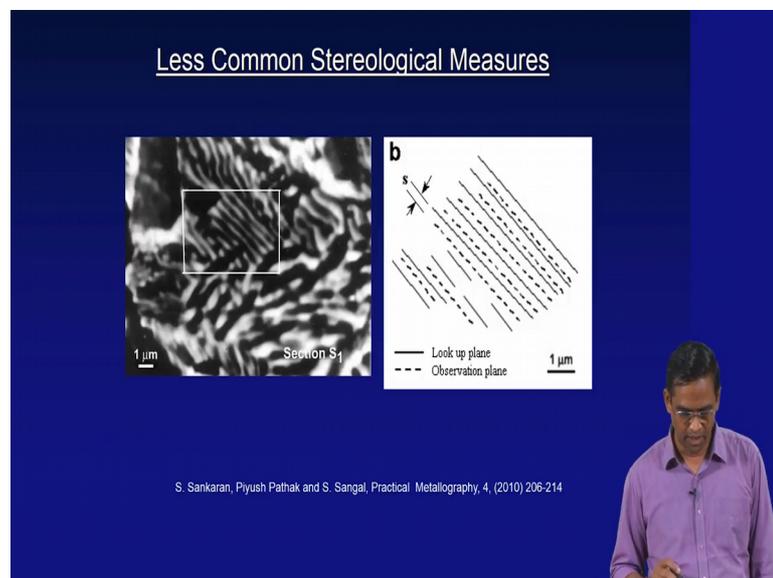
So, the you will not see this sharp boundary like this. So, you have to first polish the microstructure and then place this indent. Then and remove it, remove the material layer by layer and then after removing few microns from the layer again you make an indent, I mean look for this indents where you have already made.

This time you have to be very very careful because if you over itch, then again this indent boundary you will not be able to trace. And if you are able to trace, just the same indent just below that the previous indent.

So, you still see that the micrograph are the same region and we are now measuring the diagonals of this indent which is d_1 and d_2 . And you know that from this indent geometry, the shape of the Vickers pyramid, you can have this relation which is that a by h is equal to $2 \tan 136$ degree. So, that gives you h is equal to $0.143 d$. So, this d is an average d of these two. So, you can take the average of these two and then put just put it inside, then you will get the h that is the distance between the disector. So, that is how it is done.

So, it is bit involved. You have to be careful about the itching and you how to make several indents before you perform this measurements because you have to do this at a different places and what we have demonstrated here is they just doable and then still it is very powerful technique.

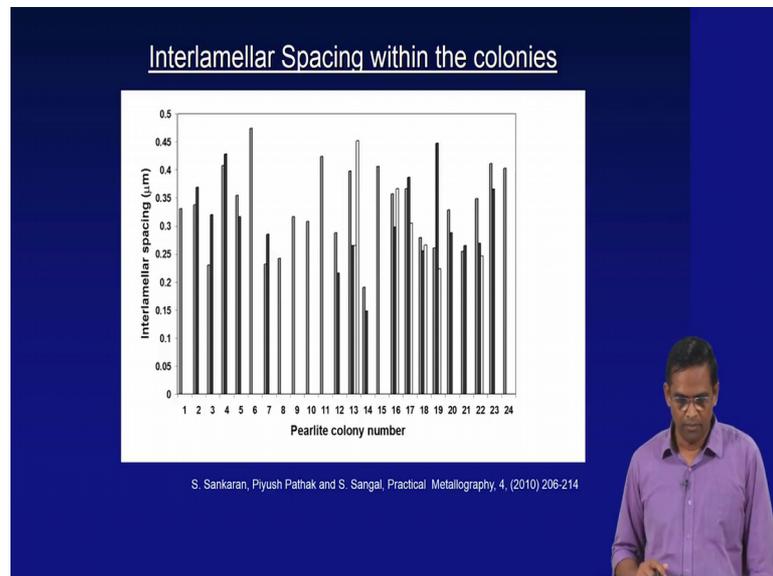
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So, and this is how the that colony which has been traced for the two planes using the disector is almost like this. And this is what we have traced the cementite plate in the look up plane as well as the observation plane and since it is all oriented in a arbitrary orientation, as per the model, you can see that there is a shift in the cementite plate plates from the observation plane versus the look up plane

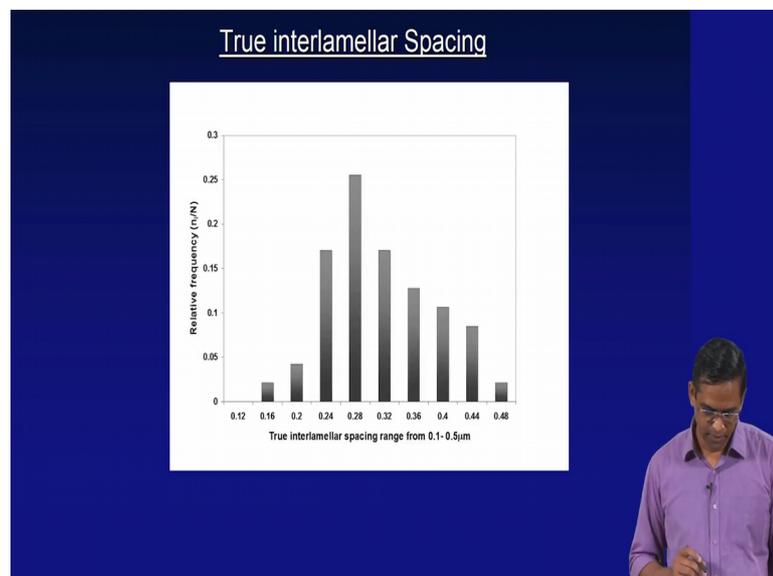
So, you have that (Refer Time: 15:01) which can be measured easily.

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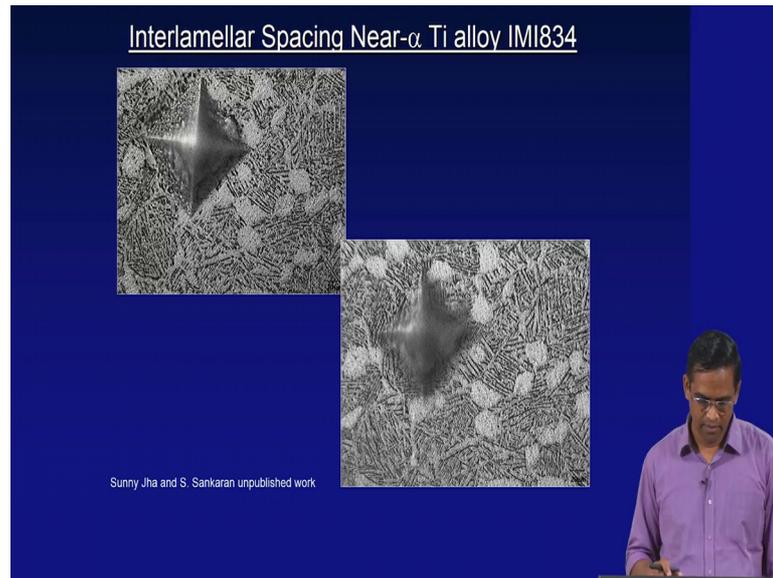
So, this is the plot of interlamellar spacing which is measured at a 2 D section in the conventional manner ok. So, what you have, you are seeing.

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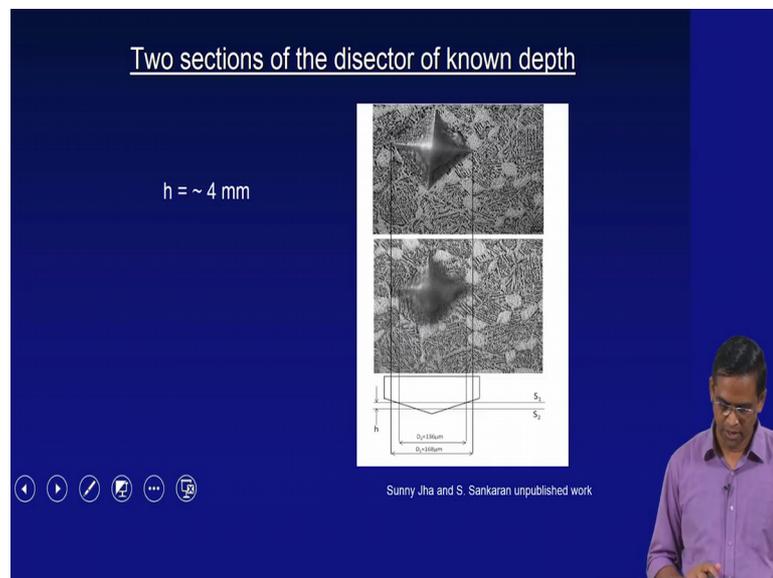
Now, is a true interlamellar spacing which is in the range of 0.01 to 0.05 micrometer. So, what you are seeing now, the true interlamellar spacing is looking very different as compared to the conventional measurement of interlamellar spacing on a 2 D section ok. So, you can see that the disector is. So, powerful and is able to give very good data.

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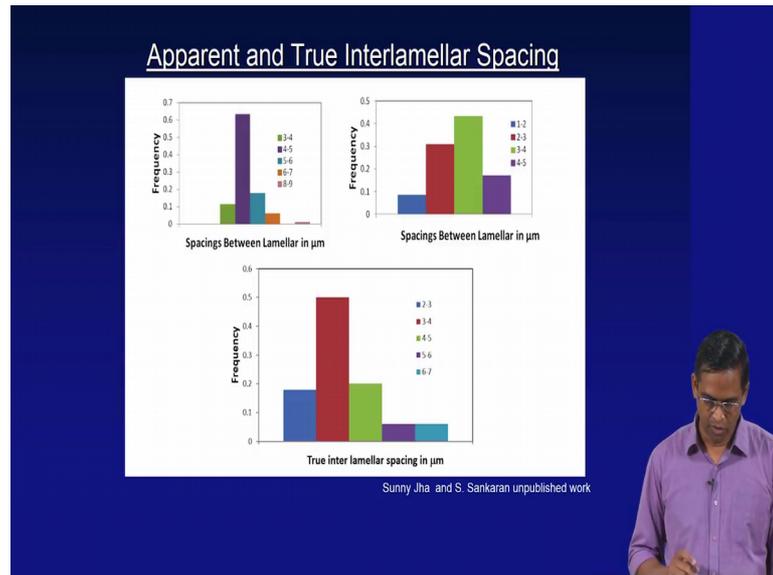
And similarly, we have done this exercise on a near alpha titanium alloy. We looked at this again this lamellar structure alpha beta lamellar colony. We have traced, this is the first plane, this is the second plane.

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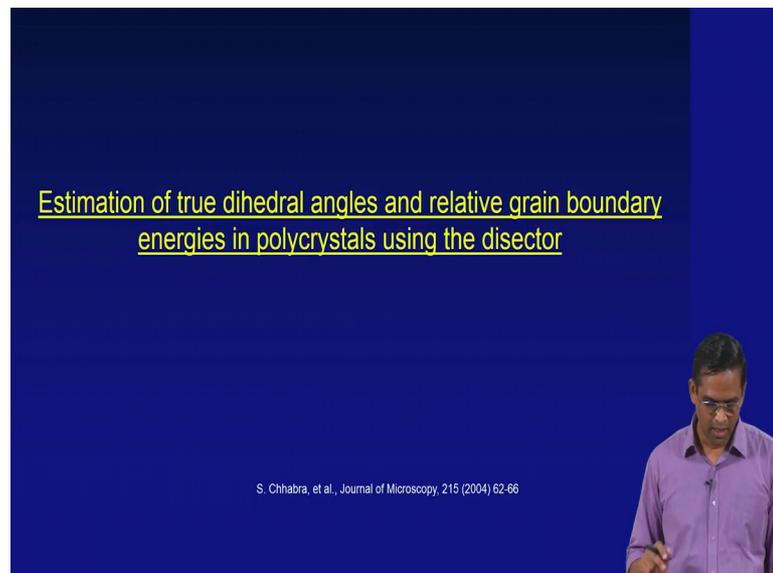
We looked at and you see that we have estimated the h is about 4 mm.

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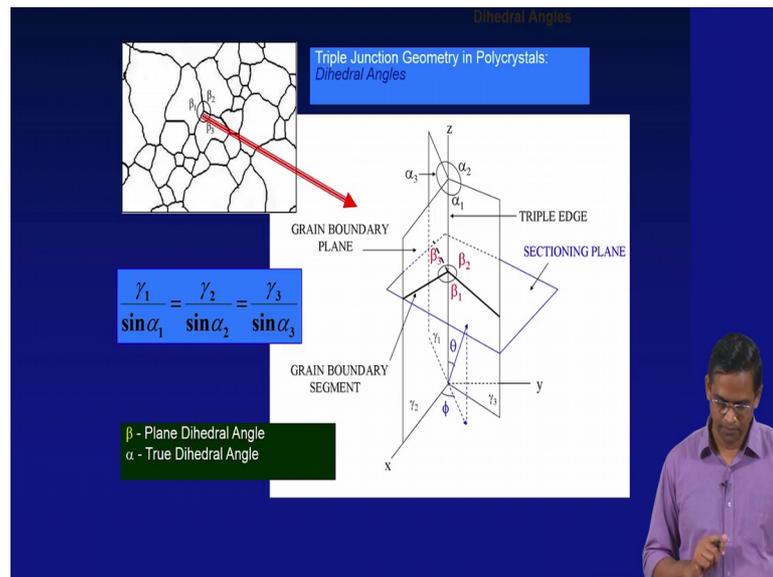
And we have also distributed this, I mean the spacing which is measured by the two techniques are very different. So, we have been demonstrated with the disector technique for a some other microstructure which is having a lamellar form of microstructural constituents.

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So, the another important aspect of the application of the disector is, the estimation of a true dihedral angles and the relative grain boundary energies in the polycrystalline or polycrystals using again a disector ok.

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So, we will look at this. So, what is this true dihedral angle. So, first, I will give an introduction. So, this is a typical microstructures where you have all triple junctions. So, like professor Sandeep said in the lecture, these boundaries are in reality, they are all surfaces. So, it is in a 2 D, it is all there are lines; that means, 3 D they are all surfaces. And these, triple junctions they are all edges; edges of a 3 grain where they meet. So, the edge points are all the triple junctions.

So, if you imagine that and you can just construct this a triple edge like along the z, and you can see that all this suppose, if you assume that this is what you are looking at the section plane, so the section plane is your nothing but your microstructure what we normally do. So, that section plane is this and then if you look at this, this is a triple edge which is here any one of them like this. There are so many triple edges. So, like this.

So, you have the plane, the you can see that the grain boundary plane, they are all shown like this and then you have alpha, alpha 1, alpha 2, alpha 3, they are all a true dihedral angle as compared to beta 1, beta 2, beta 3 which are all a apparent or plane dihedral angle, you can say that where we actually see in the microstructure 2 D section; the plane dihedral angle as well as a true dihedral angle.

So, this is a equation which relates the grain boundary energy. You can estimate the grain boundary energy by measuring the to true dihedral angle. So, that is our idea. So, this diagram clearly shows that why we measure the true dihedral angle as compared to the

plane dihedral angle. It is some something similar to what we have seen in the interlamellar spacing of the pearlite. What you see is a 2 D section is going to be very different in a 3 D. So, similar way, if you think about measuring this dihedral angle in the planar section, they are going to the angle distribution is going to be very different. So, which is again the plane boundary energy is depending upon this angle. So, that is going to vary. So, that is the idea.

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3D probe: the disector

$$\cos \alpha_1 = \hat{n}_1 \cdot \hat{n}_2 = \frac{\vec{E} \times \hat{r}_1}{E} \cdot \frac{\vec{E} \times \hat{r}_2}{E}$$

$$\cos \alpha_1 = \hat{n}_2 \cdot \hat{n}_3 = \frac{\vec{E} \times \hat{r}_2}{E} \cdot \frac{\vec{E} \times \hat{r}_3}{E}$$

$$\alpha_3 = 2\pi - (\alpha_1 + \alpha_2)$$

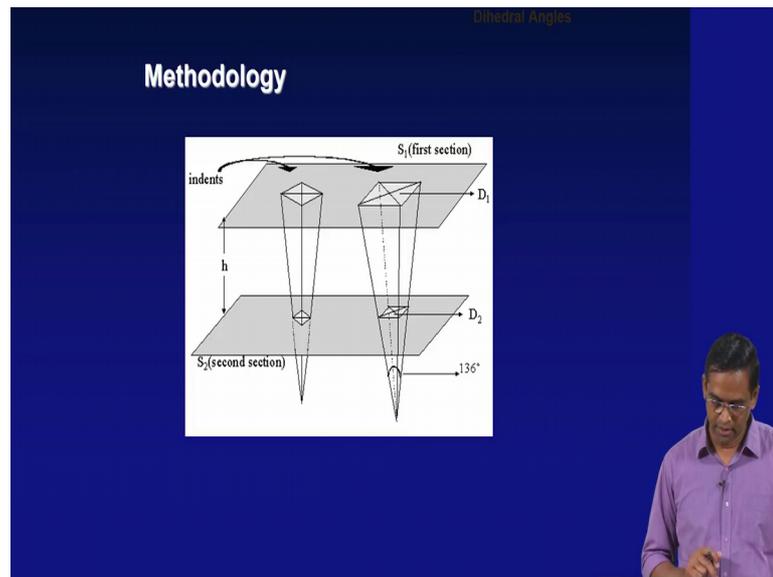
Random disector section of thickness h intersecting a triple edge.

S. Chhabra, et al., Journal of Microscopy, 215 (2004) 62-66

So, what is this ah? How the geometry of the disector described similar to this, the earlier one. So, you have 2 sections S 1 and S 2 and you see that the triple edge of the boundaries is represented by the vector E and if the grain boundary planes are represented by 1, 2 and 3 and n 1 and n 2 and n 3 are the plane normals, unit normals and you have alpha 1, alpha 2 and alpha 3 are the dihedral angle and which is again you know represented by the unit vectors r 1, r 2 and r 3, r alpha 1, alpha 2, alpha 3 are all the angle between this r 1 and r 2 and r 3 and so on.

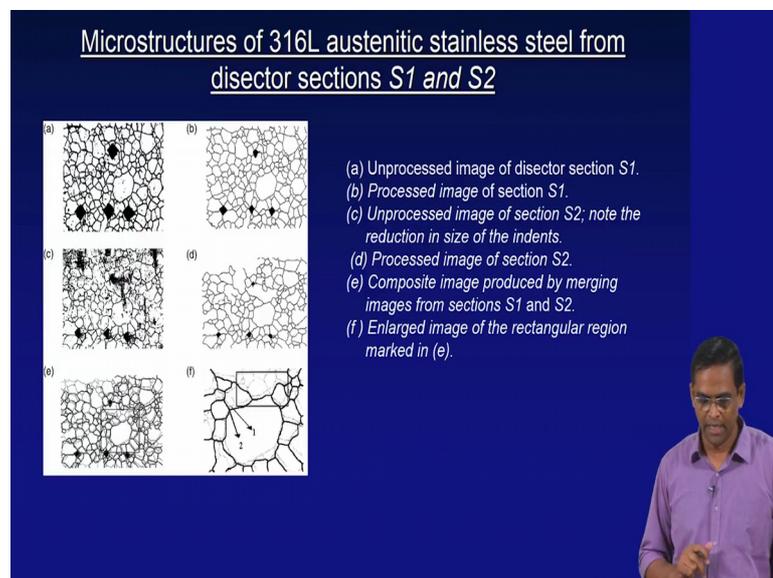
So, if you consider this geometry and the distance between the S 1 and S 2 is h, the which is the and it again and convention. So, then the angle can be found out simply by this relations and how this is going to be measured again. So, for all the mathematical derivation and details, you can refer through this publication as well as the thesis of these author and how this is implemented it.

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This is again by the indents. So, you have the Vickers hardness indent. So, you make a section 1 and section 2 and so on.

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So, what you are seeing here is like their actual microstructures of 316 austenitic stainless steel from the disector section S_1 and S_2 . So, you have a set of a micrographs where a, a is corresponding to unprocessed image of a disector section one; that means, it is actual polished section and b is processed image of a section 1, so that means, there is some image processing is involved in order to avoid this, all this noise or some

unnecessary features of the microstructure in order to because this particular estimation was done by computation. So, there was a computer program written into calculate identify certain things. I will just come to come back to that.

But let me describe this and the c is unprocessed image after S 2. This is after polishing you see that how a clumsy that micrograph becomes. So, it becomes little tedious to look at and this indents. So, after image processing, this indent shifts are better visible.

So, after it is done that, this two sections of a processed image, they are superimposed to one another; that is a composite image produced by wedging image from section 1 and section 2. So, you have this and you can see that enlarged view of this two micrographs, how they look like.

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Relative grain boundary energies in polycrystals using the disector

Table-1. Annealing treatment, grain size and disector thickness in 316L austenitic stainless steel samples.

Annealing temperature (° C)	Annealing time (min)	Grain size* (mm)	Disector thickness, h (mm)
900	60	24.9	3.8
950	60	35.9	3.2
1000	60	53.4	6.2
1050	60	62.3	6.3

* Mean intercept length has been taken as a measure of grain size



And then this is kind of exercises done on a material which is subjected to annealing treatment at four different temperatures, at for 1 hour and then this was a grain size and this is the disector thickness.

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Relative grain boundary energies in polycrystals using the disector

$$\frac{\gamma_1}{\sin \alpha_1} = \frac{\gamma_2}{\sin \alpha_2} = \frac{\gamma_3}{\sin \alpha_3}$$

- where γ_1, γ_2 and γ_3 the energies of the grain boundary planes meeting at a triple edge.
- Using above well known Young's equation the ratio of the minimum energy to maximum energy ($\gamma_{\min}/\gamma_{\max}$) was evaluated at each triple junction.

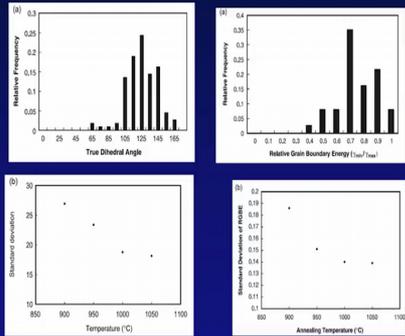
S. Chhabra, et al., Journal of Microscopy, 215 (2004) 62-66



So, this is a again a grain boundary energy relations and this is called an Young's equation. So, those whatever the true dihedral angle measured from this techniques, we have used that angles to estimate the relative grain boundary energies using this equations.

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Typical Histograms of true dihedral angle and relative grain boundary energy



The figure consists of four subplots arranged in a 2x2 grid:

- (a) Histogram of True Dihedral Angle: The x-axis is 'True Dihedral Angle' (0 to 165) and the y-axis is 'Relative Frequency' (0 to 0.3). The distribution is unimodal and centered around 120 degrees.
- (b) Histogram of Relative Grain Boundary Energy: The x-axis is 'Relative Grain Boundary Energy ($\gamma_{\min}/\gamma_{\max}$)' (0 to 1) and the y-axis is 'Relative Frequency' (0 to 0.4). The distribution is unimodal and centered around 0.7.
- (c) Scatter plot of Standard Deviation vs Temperature: The x-axis is 'Temperature (°C)' (850 to 1100) and the y-axis is 'Standard Deviation' (10 to 30). The data points show a decreasing trend.
- (d) Scatter plot of Standard Deviation of Relative Grain Boundary Energy vs Annealing Temperature: The x-axis is 'Annealing Temperature (°C)' (850 to 1100) and the y-axis is 'Standard Deviation of Relative Grain Boundary Energy' (0.1 to 0.2). The data points show a decreasing trend.

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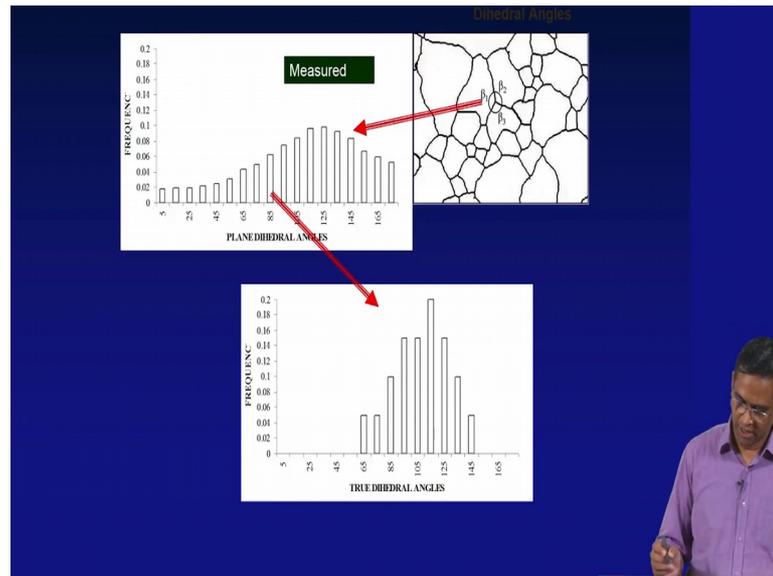


And you see that the typical histogram of true dihedral angle distribution and this is the standard deviation for a different material subjected to different annealing temperature

and also you can see that relative grain boundary energy distribution. And this is for the standard deviation of this both.

So, now we will see how this compares with the data which is measured with the a simple 2 D sections.

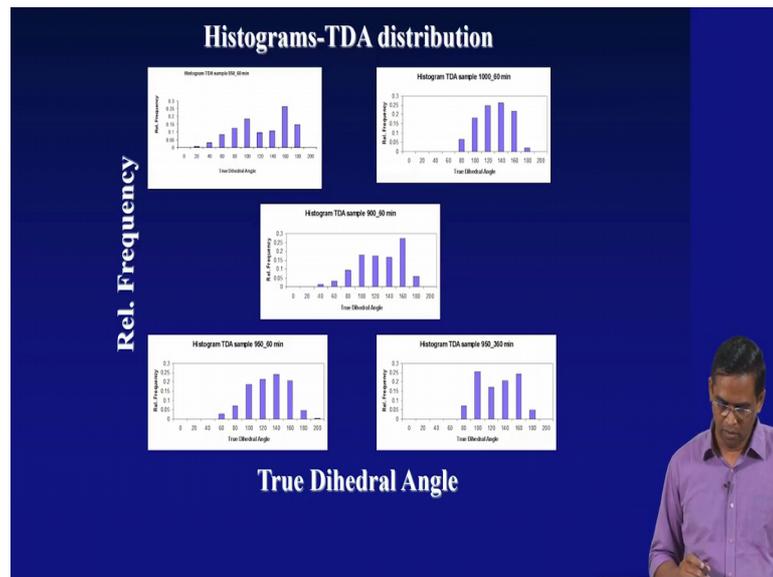
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So, this is measured a plane dihedral angle, this distribution that is and what you had seen here is a true dihedral angle distribution. So, these two looks very different, these two looks very very different.

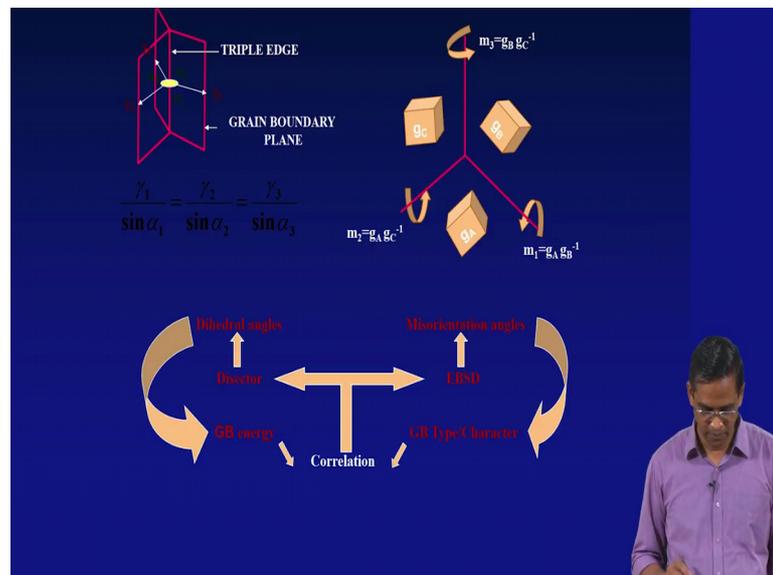
So, you can see that the effect of the 3 D probe disector which brings out a very different level of accuracy in terms of a bringing this true value of any parameter, whether it is a dihedral angle or a interlamellar spacing. It is so effective and powerful.

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And this is how the 2 dihedral angle distribution for different different annealing temperatures for this materials and this is a grain, relative grain boundary energy distribution plot for a different, I mean material subjected to different annealing temperatures.

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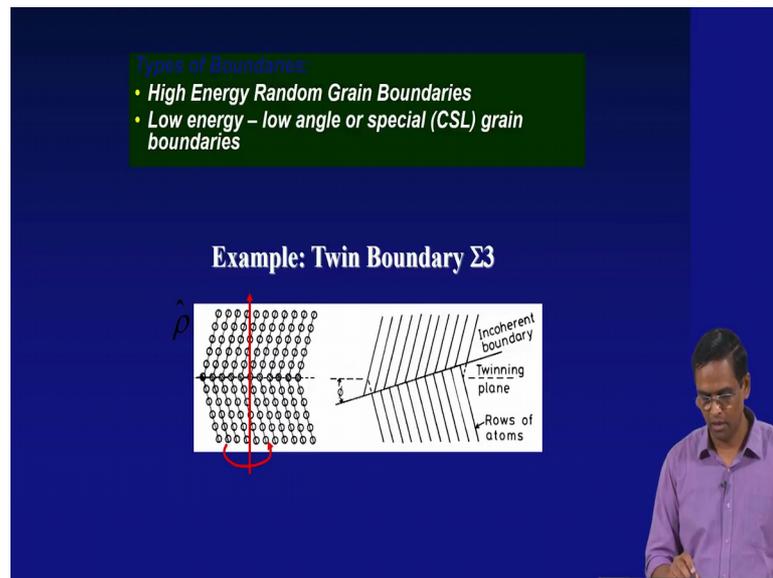


So, what is I am trying to say is like suppose if you want to correlate this with the, I mean all the properties with the microstructure. See if you look at, you can see that you know grain boundary characters and then you have EBSD which also gives

misorientation angles. And disector gives the dihedral angles and from the EBSD again, you can describe the grain boundary type and the character and again, but from the disector output you can talk about grain boundary energy again, this two will merge. So, that is the idea.

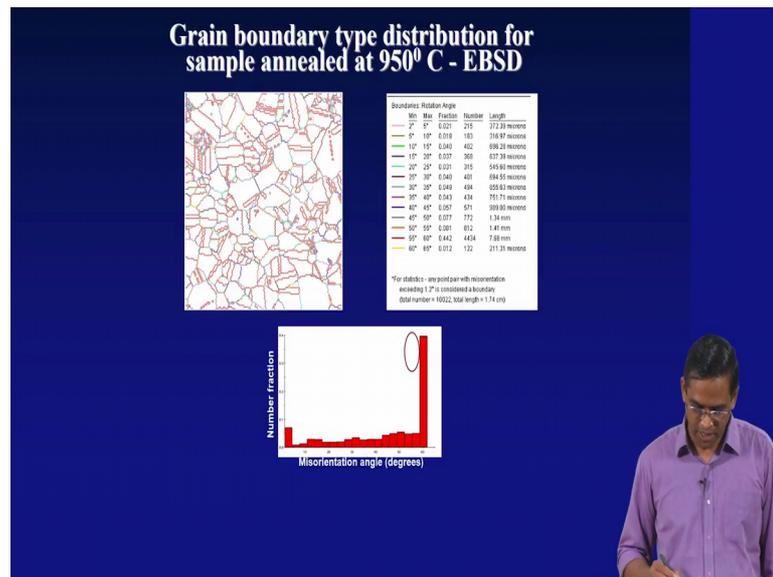
So, you have a powerful technique today called EBSD, then everything is possible even this you can come with the same conclusion.

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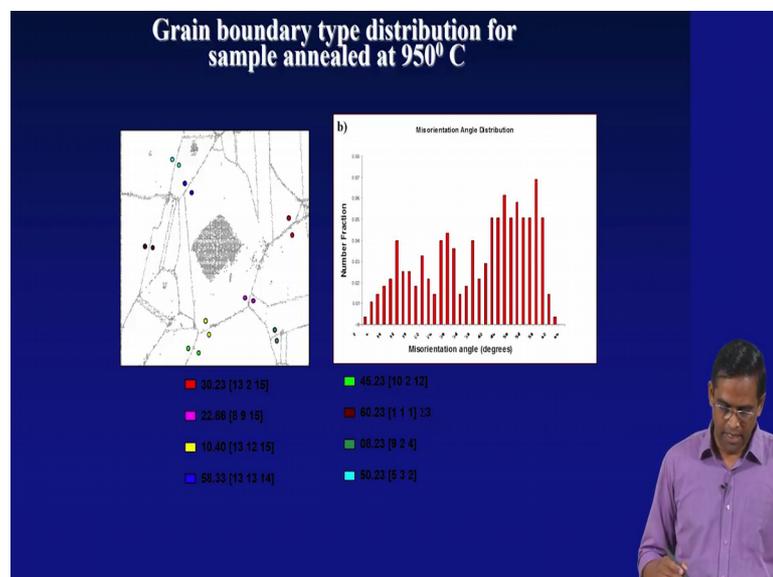
So, just give you an idea what is that type of boundaries, you have a twin boundary and as well as the which is the sigma 3 boundary. You can have a boundary with high energy random grain boundaries or low energy low angle or it could be a special CSL boundaries.

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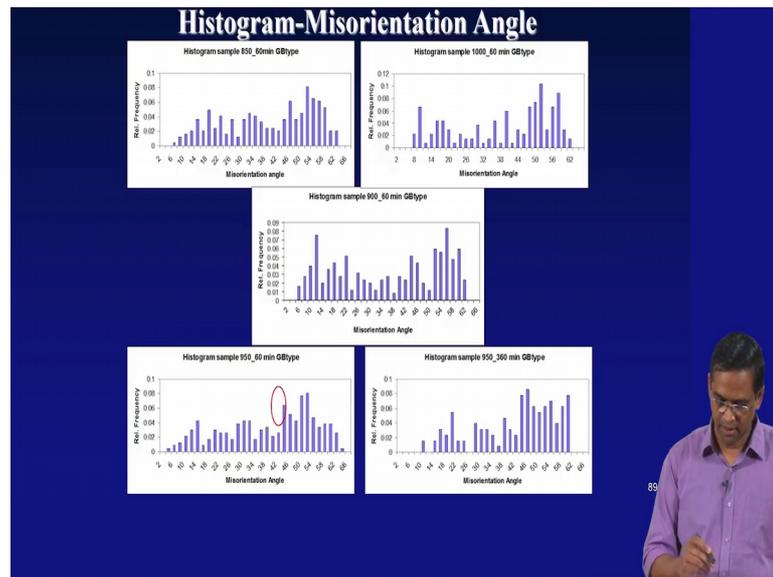
So, from the typical EBSD data, you have this grain boundary type distribution for the sample ah; how this is how the you get the disorientation angle a typically most of you have done this. This is how the map will look like.

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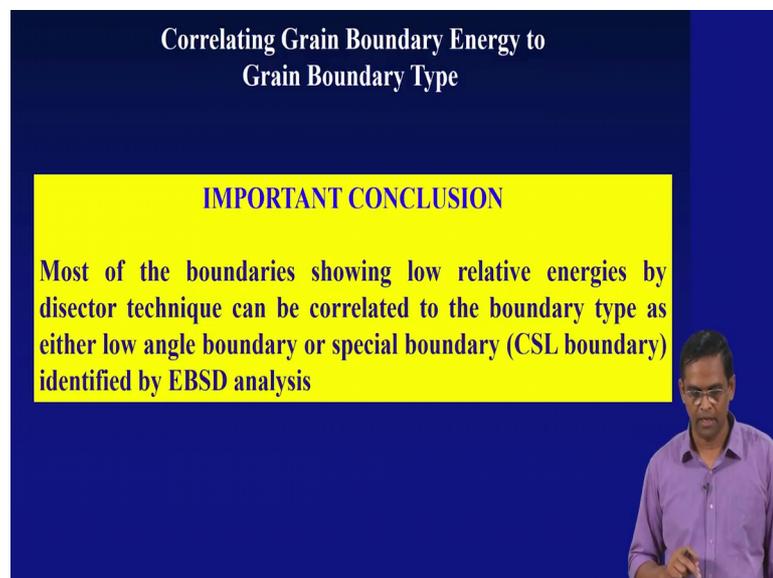
And again the grain boundary distribution disorientation angle for the similar typical mat materials will also appear like this.

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And this is a this is how the EBSD produces the data for the misorientation angle for a different, I mean material subjected to different heat treatment conditions and so on.

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So, similar data what we have generated using disector again, you can get it by the EBSD. So, the important point to note here is, most of the boundaries showing the low relative energies by the disector technique can be correlated to the boundary type as either low angle boundary or as special boundary that is the coincidence site lattice boundary identify by EBSD analysis.

So, these two techniques can be combined or correlated to arrive at much more useful data for the microstructural analysis. So, this is exactly, I just want to say it for this demonstration. So, with this demonstration, I close this tutorial and then I hope you had a much more you know, I would say a, I mean good experience in understanding the powerful technique of the disector.

Thank you.