Indian Institute of Technology
Kanpur

NP-TEL
National Programme
On
Technology Enhance Learning

Course Title
Advanced Characterization Techniques

Lecture-11

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So we will now see how the example of EBSD as I have discussed and shown you in the lab is extensively used now for different micro structural analysis , so I am going to show you one example which we have carried out to analyze these solidified micro structure in which EBSD was used to show how the gain refinement happens during under cooling experiments.

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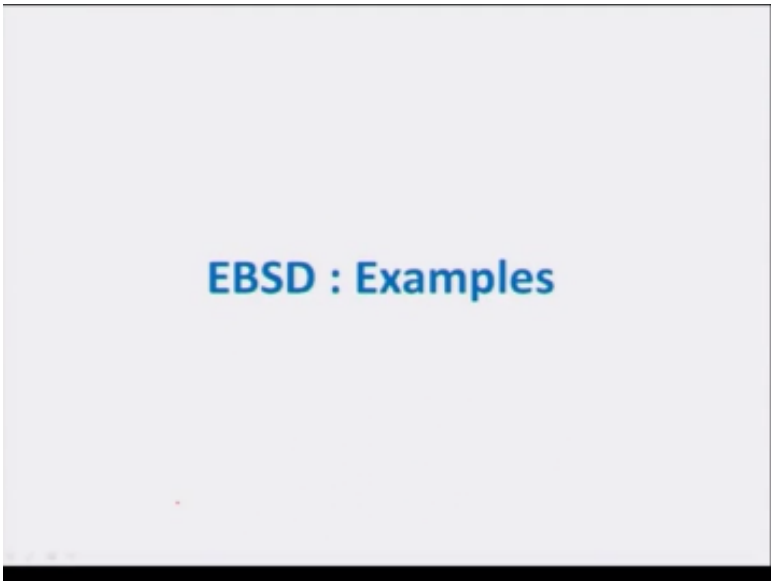

Well as you know that refinement of any material can be either intrinsic or extrinsic in case of extrinsic in the point meant we add certain defilement agents like titanium diorite in case of

aluminum alloys but in case of instinct confinement we know add ironing elements iron again definer gain refinement happens because of the fragmentation of the dendrites.

Which formed is our definition of the alloys or metals and this can happen because of many reasons so one of the reason is the distillation and the recovery and recrystallization which takes place during the certification are just after the certification event as you know the certification leads to volume change of the material from the liquid to solid and this can lead to stress generation and stress can cause at high temperature recrystallization and crystallization can lead to the can department.

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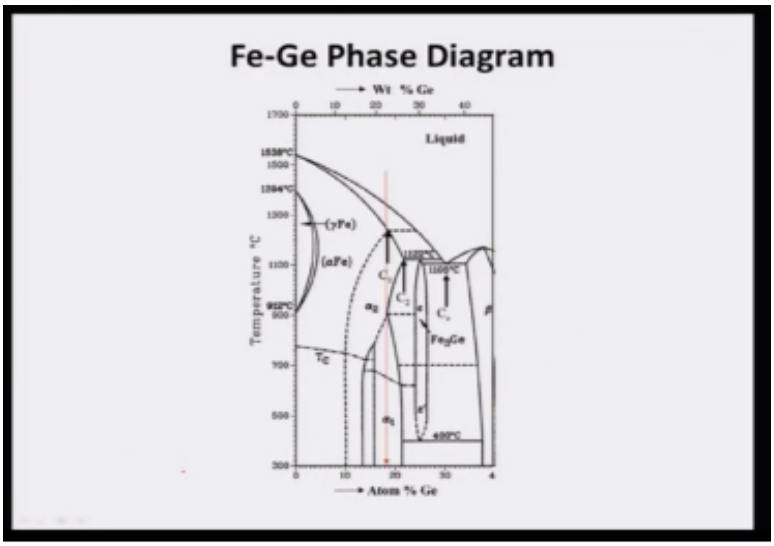

So let us see one example like that so the one which we have done in our research is are from Fe-Ge phase diagram so if you look at iron and a phase diagram it is basically a crowded with so many phases but I am going to consider all the alloy which 18% germanium the one which are marked by rate and as you see here if we start certifying from the liquid state it will form α Fe that is BCC α RN with certain amount of germanium and then just below the solidus temperature it will undergo ordering so that BCC α iron will become v2 and then further cooling leads to do second order ordering so therefore this ordering in a solid state can also lead to stress three or in a generalization and that can also cause recrystallization to happen.

So by knowing that we are going to see how this review HD technique can be used to analyze the microstructures I will show you some micro structures which are basically obtained by conventional optical microscope and then I will show you some analysis by BST.

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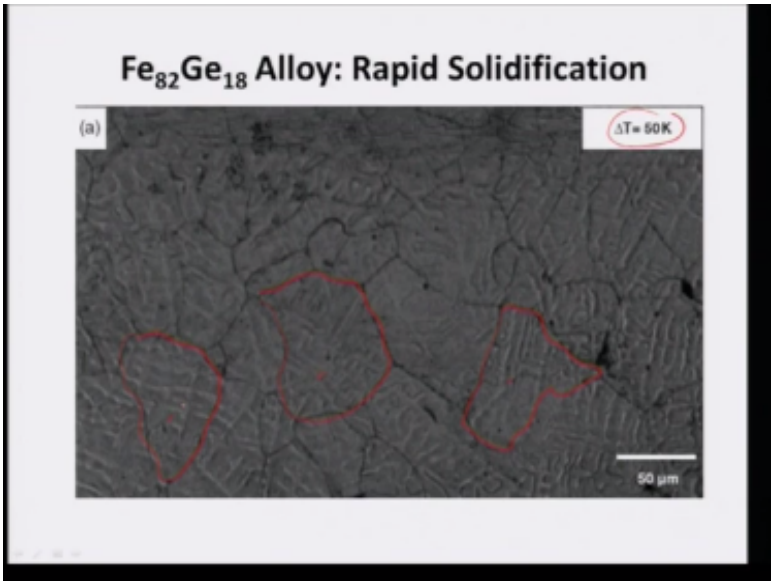

So as you see if we under cool the alloy by only 50 degrees that is very small amount we could see the micro structure consisting of grains like this and which is gain we can see segregation patterns that basically signature of the certification at low under cooling and not only that we can see when the dendrites present within the grains and, I am marking some of the grains here as you can clearly see here this is another grain and within the gains you can see the dendrite structure built in so therefore at low under cultivation happens normally by dendrite manner.

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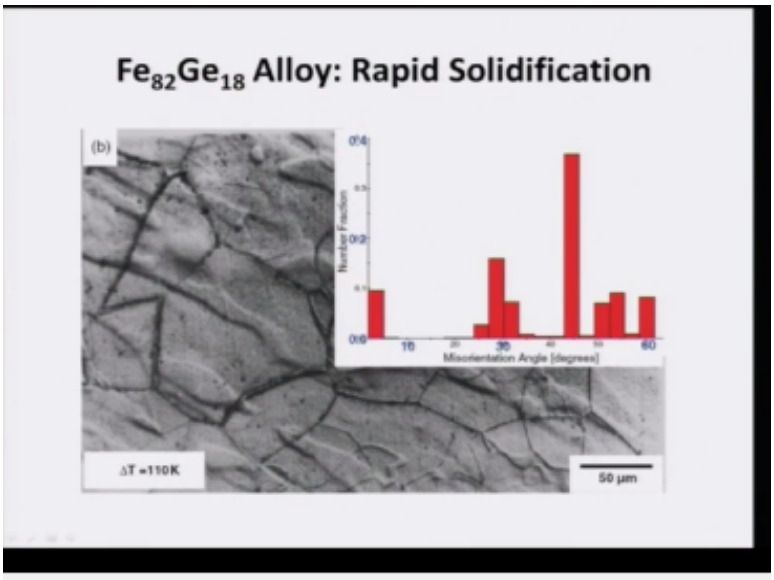

And as you increase the under cooling level by more than 100 degree 110 you can see the grain structure much little bit finer than the earlier one and we can also see the segregation pattern inside the grains inside it is again with a the genetic morphology remaining in the grain now once you do the VC analysis we can find out the nature of this gain boundaries okay ,which are there and the inner in these microstructure and if we look at it most of the grain boundaries the hair are shown the as a function of mis-orientation angle okay.

Number of grains as a function of missile test angles miss orientation basically means a one grain I with respect to the other gain is mis-oriented by certain angle and that is basically the reason for the gain boundary we know that within one grain the orientation of the crystal is same as we cross over the boundary we go to the next screen orientation changes and this orientation change is represented by missile into an angle in the ABS T so this data is I will obtained from VST after just collecting the EVSD informations and plotted as a number fraction of grains versus missile denotation angle as you see most of the grains.

Are actually having mis-initial angle more than 25 degrees very small amount is basically having less than 5 degrees so move so again boundaries her hair high angle been boundaries that is very clear so therefore one can obtain the nature of the gain boundaries of only based analysis directly .

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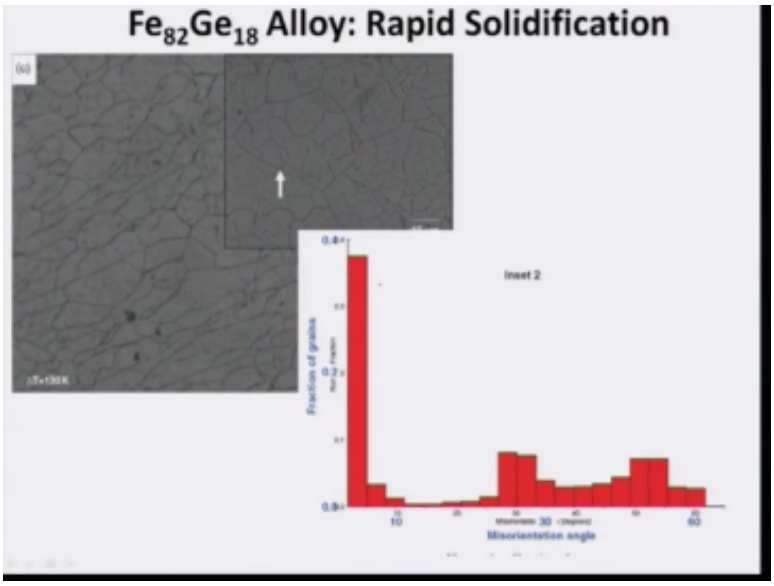

Now this is the one which I am showing if one work will be 130 degrees k-30 k and you can see you in the microstructure there are big grains like this and within the big grain there are small grains this got a small gains peasants so by looking at this optical micrograph any ask all avid reader of this what is called microstructure understand we have, I having good understand the micros will tell that these greens are actually act form by some means from the big grains so therefore the boundaries of this games will be small angle grain boundaries.

And this big grain boundaries will be large Heineken boundaries this is what is we find in main case of doing VST if we do a BST and then find out the missile interesting angle law passes fraction of grains for some games as a functional mission at this angle you can see that there are large number of grains having missile triangle less than 15 degrees when in fact if you look at it less than 10 degrees there are large number of grains present but these grains actually are small angle gay boundary.

They have the boundaries on the other hand there are grains which are having large angle grain boundaries which are present here like the one which has shown you and there is another one, I can show you here so that means there is a distinct change of the gain boundary nature of the game boundaries as the under cooling increases as you know under cooling increases me Society which increases so that will there be no stress in a more stage donation and chances of recrystallization is more so it looks like that these gains which are formed here may be due to recrystallization which will be clearer more as I go on.

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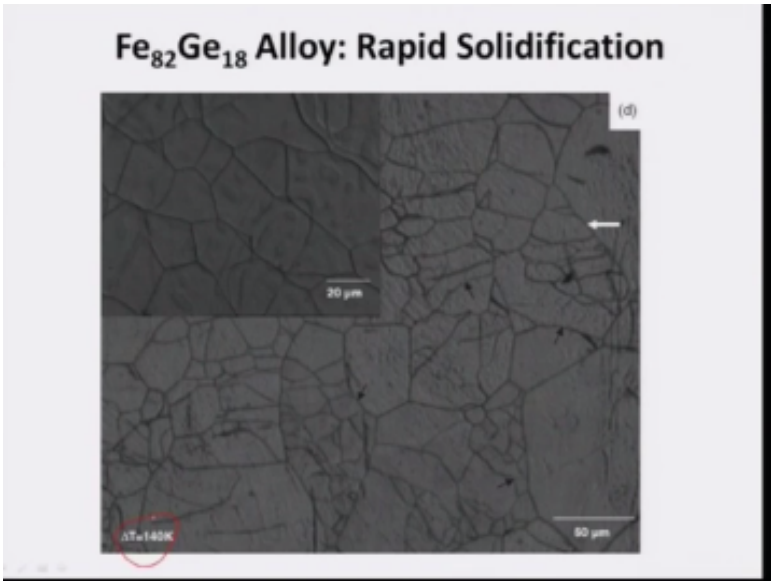

If we under cumulative n little deeply 140 k I am just showing the optical micrograph can see the big grains here again and within the big gain there are so many small gains patients similar nature which have shown just one slide before and if we zoom out the picture you can see the segregation of genetic pattern inside the grains actually if you are under cool it even much higher level by 190 k.

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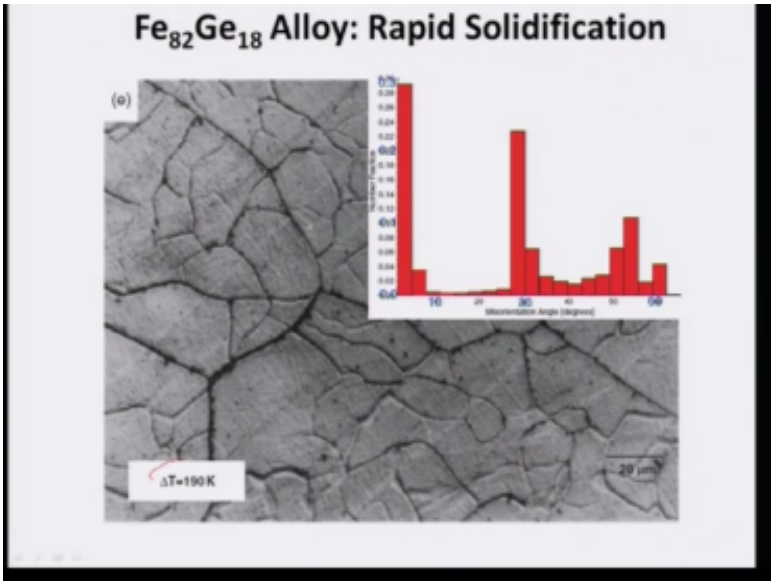

Which is very extensively high under cooling you can see that the big grains are present within the big graves there are so many small dense presents and abuse the analysis taken down on this sample it can be representative as submit the number of fraction of grains a boxes here again you see the large number of grains having less than a certain angle less than 10 degrees signifying that they are small angle grain boundaries the alarm of molecule came boundaries which can be seen here also on our end there are also large number of grains.

Which high merchants angle so they are this big grains which are actually this so this two things are very clear from the one is there is a subsequent gain refinement grains are you getting smaller and smaller as we under cooling increases and at the same time these finer gains are basically having small like a boundaries so therefore just by doing this alloys analysis one can tell all that these grains most likely has formed by recrystallization and if they are formally crystallized obviously distinct ting disting texture of the material also will be visible.

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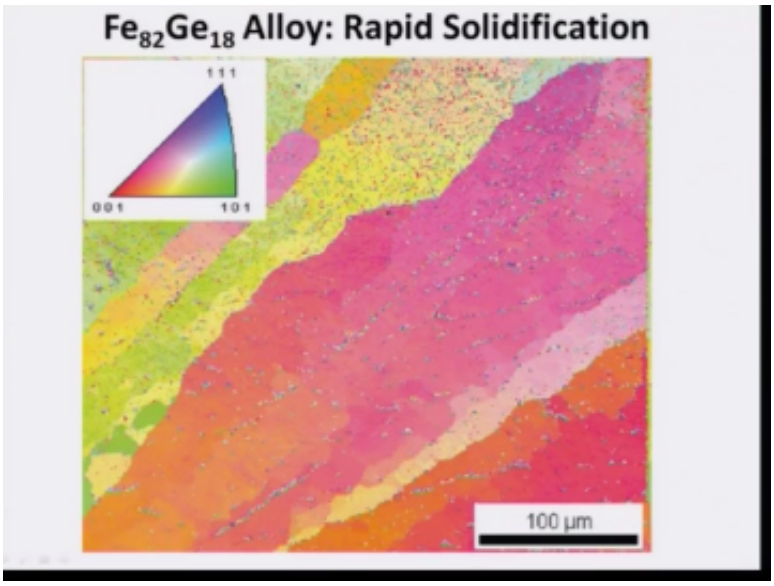

So if I just do a Oscar and gain map and plot it in different color format we can see there are very large gains you can see this one has a very large gain with pink color and within this large grain there are so many small gain space and evens are small gain presence here and there also and if we get this ask all in the small figure you can see this lead this pink are very close to just one orientation the Green way actually 0101 orientation and the blue or that little bit bluish grains which are present very small the small ones are actually 1,1,1 and 2 orientations very clearly.

So you can see the depth of information which one can obtain from one sample by an alloy TBST.

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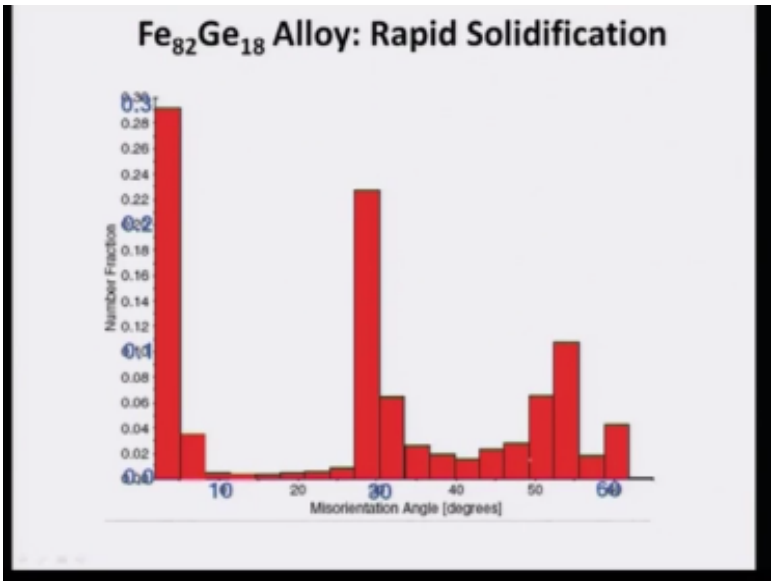

And if we do this again this from the same grain if we if I plot the grain boundary number of fractional gain boundary submission triangle again we see large number of gains of the base small mis-orientation less than about 7.5 degrees and they are actually having small angle boundaries what are there are grains which having large angle gain motives so gain bound the information be easily obtained.

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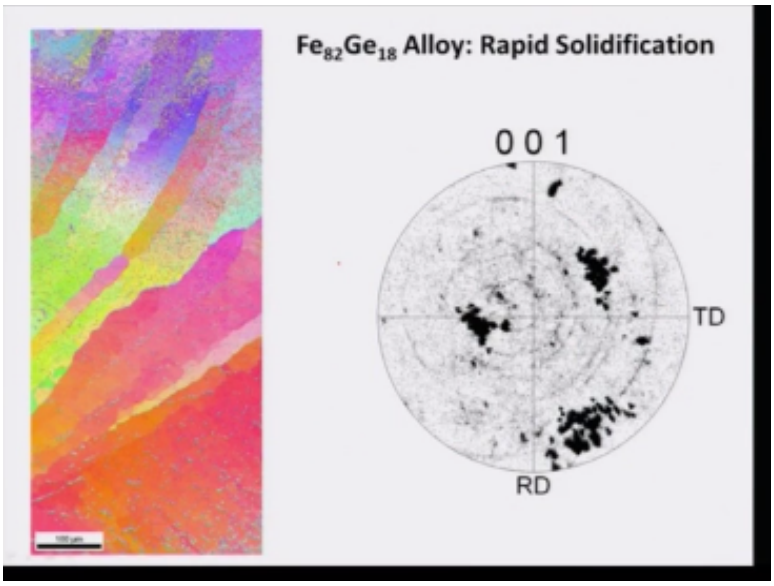

Now as for this texture is cancer which are just now I mentioned if I just use this end put a plot inverse pole figure from the gain map you can see there is fibrous texture paste and you can see this kind of nature of this texture clearly present on 11 orientations in a typical ID wealth and TD plot and there are obviously orientations of the grains which are predominantly patient in along these directions ,so one can actually obtain exactly the nature of texture from this analysis.

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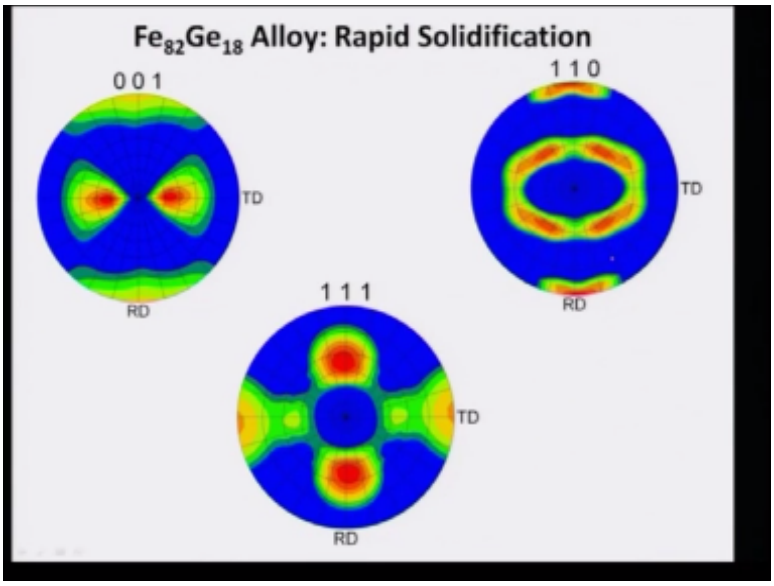

And if we do careful analysis that means if we take inverse bowl figure of 0 0 1 110 and 111 of this grains or this line number of grains we can actually get an idea of the kind of texture presence you can clearly see that along 110 directions the picture is much more clear showing the distribution of the orientation of different grains and signifying that there is a distinct texture so this itself tells us that the grains has formally crystallization and there is a texture of the grains present in the sample.

So by this way as I we can actually understand the whole mechanism again refinement in a in an alloy where there is no game department presents but because of under cooling gain defilement happens and this was not possible without EVSD obviously one cannot do with the other any other analyses BSG because in TM you cannot see so many grains as you seen the gains are very big so it is not possible to do analysis by electron microscope only possible way dialysis is CBSD well.

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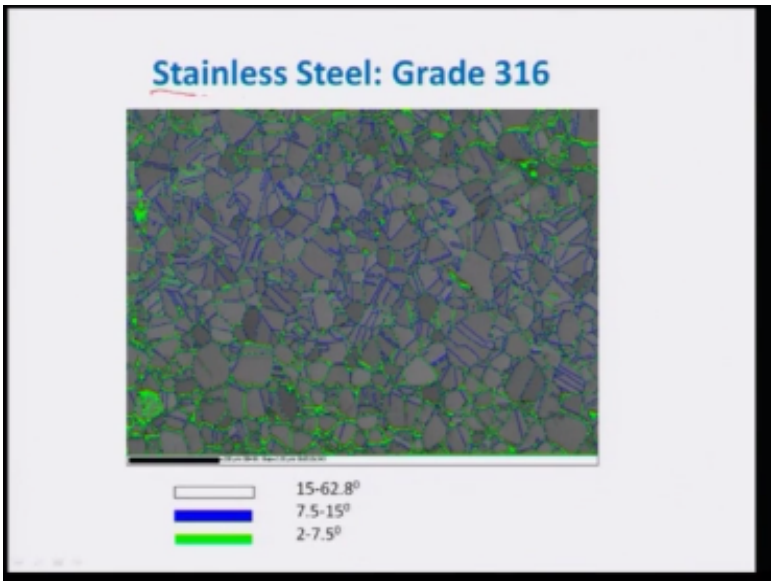

So now I am going to basically show you different pictures which tripping alloys which you have done while giving you the demo in front of them SEM in the different metals as engineering at IIT Kanpur and here we are looking at another sample of stainless steel off steady stainless steel 316 and as you know it is a basically yes FCC crystal structure containing about 18% chromium and 8% nickel and one can take the sample analyzed it again the grains can boundaries mis-orientation and many other features.

This sample are basically prepared by simple casting route and followed by rolling and so therefore we expect some petal kind of what crystallization happen in the during the processing what you see here is basically again map okay ,of the stainless steel sample and after taking the VST data and here I am showing you grain boundaries by plotting different colors okay, so if you look at if the grain boundary is having white and then it is a high angle grain boundary the most mysterious beasts orientation will be 15 to 15 to 60 2.8 degrees that is a basically definition.

And if the gain body is shown by blue color which are they have so many large number of denounced represents have a gain mount facilitation angles between the grains to be 7.5 to 15 degrees and if it is in the green color it is seven less than seven point five degrees so as you can see there are a large number of grains of having less than seven point five percent orientation and 15 degree orientation differences so that clearly tells us there is some kind of what is called

texture first of all the certain kind of grain refinement happen because of the distillation or whatever happened is dual process.

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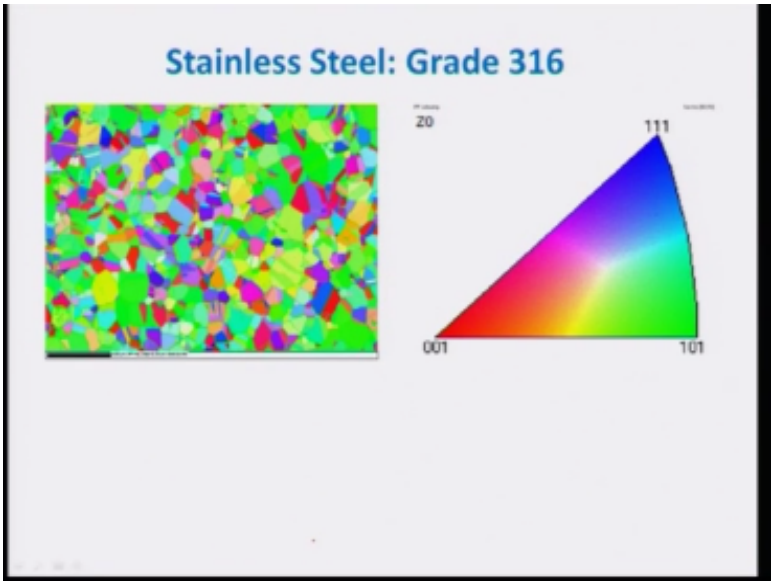

No one can actually do this gain map from the from the VHD data and those are shown here you can see even the very clearly the gain map one can even see the twins in the stainless stacking fault in stainless steel this is the one that is another one there is another one there is another one lot up and from the gain map which is very clear gain map one can actually see this poll where 0 0 1 1 1 1 and 1 10 FCC is plotted they here you can see that this red color gains are again having orientation close to 0 1 and they are there but not very large on.

The large number of gives largely green color which orientation of 101 type so therefore predominantly the grains are having 101 orientations in this crystal and presence of the grains of was called 1110 attrition is very less and when the greens of on orientation is little higher than the one on orientation but still it is predominantly 101 type of orientation present in the grains.

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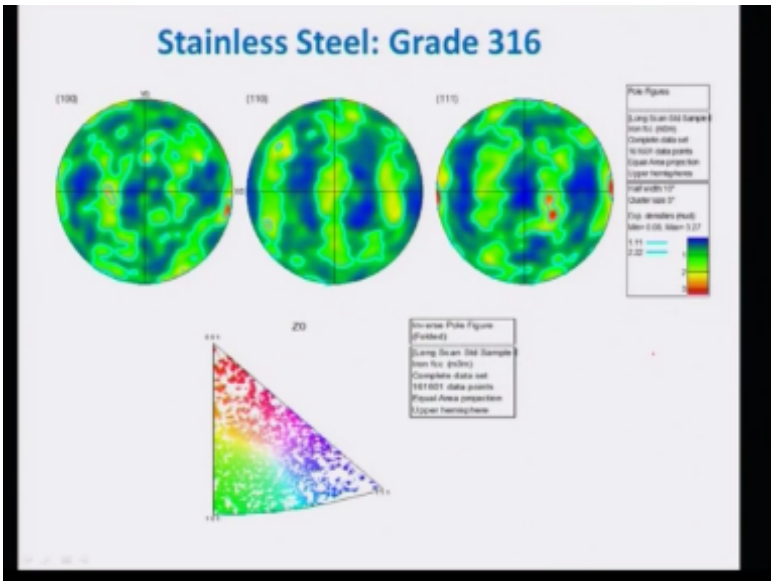

No one can actually take the inverse pole figure and analyze they basically pole figure analyze this different texture this is along 100 110 111 as you can see here 110 is not clearly visible what kind of texture is present but along 110 it is very clear that a long 110 there are distinct regions where the two orientations are very fixed so that is it with certain amount of texture presents another 11 on also you can see clearly this was predominantly featuring orientation present in the sample in the one can do better.

Nowadays with the help of the better software's so what you can see here that the number of greens having different rotation is plotted here this is 0 1 this is 1 0 1 this one on one and you can see that predominantly the green line number of green areas present here so therefore grains are predominately 101 which are just now, I showed you.

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